2019 Edition

Standard Test Methods for Metal Powders and Powder Metallurgy Products

Metal Powder Industries Federation
Standard Test Methods for Metal Powders and Powder Metallurgy Products

Introduction & Scope

The Metal Powder Industries Federation (MPIF) is a voluntary-membership, not-for-profit trade association formed by the members of the PM and particulate materials industry to promote the advancement of the metal powder producing and consuming industries and the practice of powder metallurgy and particulate materials technologies. MPIF is a federation of six trade associations, each of which is concerned with some aspect of powder metallurgy (PM), metal injection molding (MIM), metal additive manufacturing (AM), metal powders or particulate materials: the Powder Metallurgy Parts Association (PMPA), Metal Powder Producers Association (MPPA), Refractory Metals Association (RMA), Powder Metallurgy Equipment Association (PMEA), Metal Injection Molding Association (MIMA), and the Association for Metal Additive Manufacturing (AMAM).

MPIF standards cover five categories:
1. PM Nomenclature
2. Powder (Material) Test Method Standards (Testing Procedures)
3. Materials Standards/Specifications for PM Structural Parts, PM Self-Lubricating Bearings, PF Steel and MIM Parts
4. Product (Parts) Test Method Standards (Testing Procedures)
5. PM Press Safety Standards (ANSI/MPIF)

Certain trade associations within MPIF have established standards committees composed of technical people who are responsible for developing standards within their area of expertise and proposing them to the MPIF membership. Before a standard can be issued as an official MPIF standard, it must be approved by the MPIF corporate-voting membership as a whole. The standards contained in this book have all been adopted under this procedure.

MPIF standards are intended to present and clarify PM technology so as to aid in the conduct of business. PM materials specifications and testing procedure standards relate to those activities that concern designers and users of PM parts as well as the producer of the material. The use of any MPIF standard is entirely voluntary. Existence of an MPIF standard does not in any respect preclude any member or non-member of MPIF from manufacturing or selling products that use materials or testing procedures not included in MPIF standards. Other materials, products or testing procedures other than those identified in any MPIF standard may exist.

Neither MPIF nor any of its members assumes or accepts any liability resulting from use or non-use of any MPIF standard. MPIF does not accept any liability or responsibility for the compliance of any product with any standard, the achievement of any minimum or typical values by any supplier, or for the results of any testing or other procedure undertaken in accordance with any standard. By publication of these standards, no position is taken with respect to the validity of any patent rights nor does MPIF undertake to insure anyone utilizing the Standards against liability for infringement of any Letters Patent or accept any such liability.

MPIF standards are subject to periodic review and may be revised at any time by the group responsible for their creation. Users are cautioned to refer to the latest edition. Comments from any source concerning the standards or MPIF’s standards programs are welcome.
OF SPECIAL INTEREST TO USERS OF PM...

MPIF
Standard 35—
Materials
Standards

The latest editions of MPIF materials standards are available for purchase in printed and electronic formats. Visit the Publications area at mpif.org

The MPIF Standard 35 Family of Materials Standards

- MPIF Standard 35-SP, Materials Standards for PM Structural Parts—for structural parts made by the powder metallurgy process
- MPIF Standard 35-SLB, Materials Standards for PM Self- Lubricating Bearings—for bearings and bushings made by the PM process
- MPIF Standard 35-PF, Materials Standards for PM Steel Parts—for steel components made by the powder forging (PF) process
- MPIF Standard 35-MIM, Materials Standards for Metal Injection Molded Parts—for components made by the metal injection molding (MIM) process

MPIF GUIDE TO PM MICROSTRUCTURES

The goal of this Guide is to assist PM parts manufacturers and the end user community to interpret powder metallurgy (PM) microstructures. It will help build appreciation of what a powerful tool metallography can be when: engineering new components; designing new materials; solving various types of quality problems; and as a most effective means for quality control.

The user will be able to reference materials processed by conventional sintering; elevated temperature sintering; accelerated cooling rates; etc. to determine phase and structures of the materials. Specimen preparation and proper selection of etchants is also covered. Galleries available for viewing include: Iron and Carbon Steel; Iron- Copper and Copper Steel; Iron-Nickel and Nickel Steel; Prealloyed Steel; Hybrid Low-Alloy Steel; Sinter-Hardened Steel; Diffusion-Alloyed Steel; Copper Infiltrated Iron and Steel; Soft Magnetic Alloys along with other examples.

Free access at mpif.org.
# General Information 1

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Sources* of Specialized Equipment Noted in
Standard Test Methods for Metal Powders and Powder Metallurgy Products

Std. 01 - Sampling
Keystone Sampling Thief and Sample Splitter
Gilson Co., Inc.
P.O. Box 200
Lewis Center, OH 43035-0200
www.globalgilson.com

Seedburo Equipment Co.
2293 S. Mt Prospect Road
Des Plaines, IL 60018
www.seedburo.com

Sepor, Inc.
718 N. Fires Ave
Wilmington, CA 90744
www.sepor.com

Std. 01 - Spinning Rifflers
Gilson Co., Inc.
P.O. Box 200
Lewis Center, OH 43035-0200
www.globalgilson.com

Microscal Ltd.
79 Southern Row
London
United Kingdom W 10 SAL
info@microscal.com

Quantachrome Instruments
1900 Corporate Drive
Boytont Beach, FL 33426
www.quantachrome.com

Std. 03 - Flow Rate
Std. 04 and 28 - Apparent Density
Hall Flowmeter, Carney Funnel and Standard Powder

Kymera International
901 Lehigh Avenue
Union, NJ 07083-7632
www.kymerainternational.com

Std. 05 - Sieve Analysis
Sieve Shaker
Fisher Scientific Co.
(major U.S. cities)
www.fishersci.com
Gilson Co., Inc.
P.O. Box 200
Lewis Center, OH 43035
www.globalgilson.com
W.S. Tyler, Inc.
8570 Tyler Blvd.
Mentor, OH 44060
www.wstyler.com

Std. 06 - Tension Test Strength
15 - Green Strength
41 - Transverse Rupture Strength
44 - Dimensional Change
45 - Compressibility
60 - PM Test Specimen Preparation
Compacting Dies, Transverse Rupture Strength Fixture and Green Strength Tester
Gasbarre Products, Inc.
Precision Tooling
159 McKee Road
Olanta, PA 16863
www.gasbarre.com

Green strength tester engineering drawings available from:
Kymera International
901 Lehigh Avenue
Union, NJ 07083-7632
www.kymerainternational.com

Std. 32 - Average Particle Size
Sub-sieve AutoSizer
Micromeritics Instrument Corporation
4356 Communications Drive
Norcross, GA 30093-2901
www.micromeritics.com

Std. 46 - Tap Density
Tap Density Tester
Quantachrome Instruments
1900 Corporate Drive
Boynton Beach, FL 33426
www.quantachrome.com

Std. 58 - Surface Finish
Chisel Stylus
Precision Devices, Inc.
P.O. Box 220
Milan, MI 48160
www.predev.com

Std. 63 - MIM Density
Gas Pycnometer
Micromeritics Instrument Corporation
4356 Communications Drive
Norcross, GA 30093-2901
www.micromeritics.com
Quantachrome Instruments
1900 Corporate Drive Boynton Beach, FL 33426
www.quantachrome.com

Other - Apparent Density
Paint Pigment Volumeter, Metal Powder Volumeter, Scott Volumeter
Steiner Enterprises, Inc.
3532 Coleman Ct., Suite B
Lafayette, IN 47905
www.steineronline.com
VWR International
(major U.S. cities)
www.vwr.com

*Note: Other sources may exist for equipment listed.
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#### Quantities/Terms Used in MPIF Standards

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<th>Designation</th>
<th>Inch-Pound Units</th>
<th>Preferred Working Unit</th>
<th>Symbol</th>
<th>Approx. Conversion to SI Units*</th>
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<tr>
<td>Apparent Density</td>
<td>( \rho_a )</td>
<td>g/cm(^3)</td>
<td>gram per cubic centimetre</td>
<td>g/cm(^3)</td>
<td>-</td>
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<tr>
<td>Applied Magnetic Field</td>
<td>H</td>
<td>oersteds (Oe)</td>
<td>amperes-turns/metre</td>
<td>A/m</td>
<td>X 79.6</td>
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<td>Atmosphere Flow</td>
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<td></td>
<td></td>
<td>CFH</td>
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<td>cm(^3)/s</td>
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<td>Belt Speed</td>
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<td></td>
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<td>MPa</td>
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<td>Crush Strength</td>
<td>K</td>
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<td>MPa</td>
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<td>second per 50 grams</td>
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<td>Fracture Toughness</td>
<td>( K_{IC} )</td>
<td>10(^3) psi-inch(^{1/2})</td>
<td>megapascals root metre</td>
<td>MPa ( \cdot m^{1/2} )</td>
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<td>Green Density</td>
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<td>joule</td>
<td>J</td>
<td>X 1.356</td>
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<td>Kinematic Viscosity</td>
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<td>metres squared per second</td>
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<td>B</td>
<td>kilogauss (kG)</td>
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<td>T</td>
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<td>micrometre</td>
<td>( \mu ) m</td>
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<td>megagram</td>
<td>Mg</td>
<td>X 0.907</td>
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<tr>
<td></td>
<td></td>
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<td>metric ton</td>
<td>t</td>
<td>X 0.907</td>
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<td>g/cm(^3)</td>
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<td>Surface Finish</td>
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<td>micrometre</td>
<td>( \mu ) m</td>
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<td>g/cm(^3)</td>
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<td>degree Celsius</td>
<td>°C</td>
<td>5/9 (°F -32)</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td></td>
<td>10(^3) psi</td>
<td>megapascals</td>
<td>MPa</td>
<td>X 6.895</td>
</tr>
<tr>
<td>Thermal Conductivity</td>
<td></td>
<td>Btu-ft/(h-ft(^{2})-°F)</td>
<td>watts per metre kelvin</td>
<td>W/(m·K)</td>
<td>X 1.731</td>
</tr>
<tr>
<td>Torque</td>
<td></td>
<td>lbf-ft</td>
<td>newton metres</td>
<td>N·m</td>
<td>X 1.356</td>
</tr>
<tr>
<td>Transverse Rupture Strength</td>
<td></td>
<td>10(^3) psi</td>
<td>megapascals</td>
<td>MPa</td>
<td>X 6.895</td>
</tr>
<tr>
<td>Yield Strength</td>
<td></td>
<td>10(^3) psi</td>
<td>megapascals</td>
<td>MPa</td>
<td>X 6.895</td>
</tr>
<tr>
<td>Young's Modulus</td>
<td></td>
<td>10(^6) psi</td>
<td>gigapascals</td>
<td>GPa</td>
<td>X 6.895</td>
</tr>
</tbody>
</table>

* Example: 1 psi = 0.0069 MPa
If 100,000 psi, then MPa = 0.0069 \times 100,000 = 690 MPa
General Information VI

Video Examples of the Standard Test Methods for Metal Powders and Powder Metallurgy Products

Scan a QR code below

By scanning the QR Codes or visiting the appropriate link via the internet, the user will be able to view video clip demonstrations of the working mechanics of the cited test method. Examples are intended for educational purposes only. Any reference to text, tables or figures may not be 100% accurate, as standards are reviewed and revised on a periodic basis. Users should always refer to and use the latest edition of the standard.

No position should be taken by the user with respect to the validity of the contents of the videos. Neither MPIF nor any of its members assumes or accepts liability resulting from their use.

---

**STANDARD 01**
Method for Sampling Metal Powders
https://youtu.be/XEEsEuNBD-U

---

**STANDARD 02**
Method for Determination of Loss of Mass in a Reducing Atmosphere for Metal Powders (Hydrogen Loss)
https://youtu.be/PX8oiAa_ZAs

---

**STANDARD 03**
Determination of Flow Rate of Free-Flowing Metal Powders Using the Hall Apparatus
https://youtu.be/Dpw8MZ1qYJM

---

**STANDARD 04**
Determination of Apparent Density of Free-Flowing Metal Powders Using the Hall Apparatus
https://youtu.be/zOxHkiV4570

---

**STANDARD 05**
Determination of Sieve Analysis of Metal Powders
https://youtu.be/DX7c_Bi5Vmo

---

**STANDARD 08**
Determination of Apparent Density of Free-Flowing Metal Powders Using the Carney Apparatus
https://youtu.be/PdcoUT7yhE

---

**STANDARD 10**
Determination of the Tensile Properties of Powder Metallurgy (PM) Materials
https://youtu.be/Ep97CV0SRck

---

**STANDARD 15**
Determination of Green Strength of Compacted Powder Metallurgy Materials
https://youtu.be/UdHAb9Eqi7M
<table>
<thead>
<tr>
<th>STANDARD</th>
<th>Method</th>
<th>Video Link</th>
</tr>
</thead>
<tbody>
<tr>
<td>46</td>
<td>Determination of Tap Density of Metal Powders</td>
<td><a href="https://youtu.be/mXBthlopJqw">https://youtu.be/mXBthlopJqw</a></td>
</tr>
<tr>
<td>41</td>
<td>Determination of Transverse Rupture Strength of Powder Metallurgy (PM) Materials</td>
<td><a href="https://youtu.be/_JqpUKurnSI">https://youtu.be/_JqpUKurnSI</a></td>
</tr>
<tr>
<td>48</td>
<td>Determination of Apparent Density of Metal Powders Using the Arnold Meter</td>
<td><a href="https://youtu.be/D5QQXoO7hsM">https://youtu.be/D5QQXoO7hsM</a></td>
</tr>
<tr>
<td>42</td>
<td>Determination of Density of Compacted or Sintered Powder Metallurgy (PM) Products</td>
<td><a href="https://youtu.be/iDHw3r9Ww8I">https://youtu.be/iDHw3r9Ww8I</a></td>
</tr>
<tr>
<td>53</td>
<td>Measuring the Volume of the Apparent Density Cup Used with the Hall and Carney Apparatus (Standards 04 and 28)</td>
<td><a href="https://youtu.be/Bc954mfy6FQ">https://youtu.be/Bc954mfy6FQ</a></td>
</tr>
<tr>
<td>55</td>
<td>Determination of Radial Crush Strength (K) of Powder Metallurgy (PM) Materials</td>
<td><a href="https://youtu.be/M5-EaAGuF74">https://youtu.be/M5-EaAGuF74</a></td>
</tr>
<tr>
<td>45</td>
<td>Determination of Compressibility of Metal Powders</td>
<td><a href="https://youtu.be/qU7GOFtiS54">https://youtu.be/qU7GOFtiS54</a></td>
</tr>
</tbody>
</table>
1. SCOPE

1.1 This standard describes methods used to sample metal powders and the procedures that are recommended for reducing this sample into smaller test portions for subsequent testing.

1.2 A number of sampling practices are described. Their applicability depends on how the powder is stored or being transported through the process.

1.2.1 Practice 1A is applicable to sampling moving streams of powder such as those being transferred from one container to another, or those in a process stream or falling from a conveyor. This is the preferred method for obtaining the several increments that are combined to form the gross sample.

1.2.2 Practice 1B is applicable to sampling powders that have already been packaged for transport, as in a flexbag, bulkpack, or drum.

1.2.3 Practice 2 is applicable to obtaining test portions from the composite sample.

1.3 These practices apply to metal powders, and mixes or premixes of metal powders that may contain lubricants and other non-metallic additives.

1.4 This standard does not cover the sampling of flake powders or pastes. For procedures on the sampling of flake metal powders and pastes, refer to ASTM D480.

1.5 The values stated in inch-pound units are to be regarded as standard. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.6 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. DEFINITIONS

2.1 The following terms are defined (see Fig. 1):

2.1.1 Increment—a quantity of powder obtained by a sampling device at any one time from a larger quantity of powder.

2.1.2 Gross sample—the total quantity of powder adequate for the intended purpose(s), consisting of all the increments combined.

2.1.3 Composite sample—the entire gross sample after blending.

2.1.4 Test portion—a quantity of powder (generally taken from the composite sample) on which the test is performed, or from which a test specimen is produced.

3. APPARATUS

3.1 A rectangular receptacle, having a length and width greater than the stream of powder and capable of being moved completely across a stream of flowing powder. It must be large enough that no overflow of powder occurs when collecting the sample.

3.2 A small blender of sufficient capacity to blend the entire gross sample.

3.3 A commercial sampling device for collecting powder samples from a flowing stream. Powder transfer systems are often sealed for dust control. Commercial devices are available for insertion into a section of a pipe to collect powder increments while maintaining the flowing stream.

3.4 A powder sampler such as the Keystone sampler or other appropriate device with an auger point that can be screwed to the bottom of a filled container and is designed to collect powder at one (single level) or more (multi-level) depths—see Figs. 2 and 3.

3.5 A non-magnetic stainless steel scoop with a sharp edge and high sides, of sufficient size to obtain the desired powder increment—see Fig. 4.

3.6 A chute splitter of sufficient size and capacity to split the required amount of powder into two approximately equal portions—see Fig. 5. Several different sizes of chute splitter may be needed throughout the sampling process, depending on the sample size at each step. The chute openings should be at least three times the size of the powder particle size that is being split and the chute angle should be at least 45°. The smallest chute splitters are often referred to as “micro-splitters”.

3.7 A charging pan that is the same width as the chute splitter and large enough to contain the entire sample that is to be split.

3.8 A spinning riffler of sufficient size and capacity to split the required amount of powder into at least eight approximately equal portions—see Fig. 6.
4. PROCEDURE

PART 1—OBTAINING THE COMPOSITE SAMPLE

4.1 Practice 1A—Sampling a Moving Powder

4.1.1 The preferred method for sampling metal powder is when the powder is in motion; for example, being transferred from a blender, falling from a conveyor, or in a moving process stream.

4.1.2 Equal powder increments should be taken at random intervals over the time of the flow.

4.1.3 The number of increments that make up the gross sample shall be agreed upon between the involved parties.

4.1.4 Pass the rectangular container at a constant speed through the steady state stream of flowing powder starting near the beginning of the initial flow of powder. Repeat at random intervals until the agreed-upon number of increments have been obtained with the last increment being taken near the end of the final flow of the powder.

4.1.5 Alternatively, collect the agreed-upon number of increments using a commercial sampling device starting near the beginning of the flow of powder with the final increment being taken near the end of the powder flow.

4.1.6 The total amount of powder in all the increments shall be adequate for the tests or evaluations that are to be performed.

4.1.7 Combine all the increments to form the gross sample.

NOTE 1—Individual increments may be tested to investigate within-lot variability.

4.1.8 Blend the gross sample in a small blender to form the composite sample.

4.2 Practice 1B—Sampling a Stationary Powder

4.2.1 It is not always possible to take samples from a moving powder so samples often need to be taken from metal powders that are, for example, packed in flexbags, bulkpacks, or drums. Such stationary powder masses must be sampled using either a tube (slot) sampler or a simple scoop as specified in the following sections.

4.2.2 Sampling a Stationary Powder Using a Slot or Tube Sampler

4.2.2.1 Use a hollow tubular multi-slot sampler (Fig. 2) to take powder samples from several depths within the container. This is the recommended way to sample powder that has been packaged. Alternatively, a single-level tube sampler (Fig. 3) may be used multiple times to take samples from various depths within the container. In this case, equal increments of powder should be taken at each depth.

4.2.2.2 The number of increments to be taken from each container and the number of containers to be sampled should be agreed upon by the concerned parties. The minimum number of containers to be selected at random for sampling is listed in Table 1.

<table>
<thead>
<tr>
<th>No. of Containers in the Lot</th>
<th>No. of Containers to be Sampled (randomly selected)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-5</td>
<td>All</td>
</tr>
<tr>
<td>6-11</td>
<td>5</td>
</tr>
<tr>
<td>12-20</td>
<td>6</td>
</tr>
<tr>
<td>21-35</td>
<td>7</td>
</tr>
<tr>
<td>36-60</td>
<td>8</td>
</tr>
<tr>
<td>61-99</td>
<td>9</td>
</tr>
<tr>
<td>100-149</td>
<td>10</td>
</tr>
<tr>
<td>150-199</td>
<td>11</td>
</tr>
<tr>
<td>200-299</td>
<td>12</td>
</tr>
<tr>
<td>300-399</td>
<td>13</td>
</tr>
<tr>
<td>More than 400</td>
<td>13 + 1 per 100 additional containers</td>
</tr>
</tbody>
</table>

Adapted from, “Powder Sampling and Particle Size Analysis”, by Terence Allen, published by Elsevier 2003, p. 10

4.2.2.3 Obtain powder increments from each container using either the multi-level slot sampler or the single-level tube sampler.

4.2.2.4 Insert the sampler in the closed position into the packed powder at a point about 70% of the distance from the periphery of the container. Insert the multi-level slot sampler straight down to the bottom of the container and the single-level tube sampler straight down to an appropriate depth for the increment that is required (repeat, in this case, at various depths for the number of increments needed).

4.2.2.5 Open the sampler, allowing powder to flow into the sampler tube. Close the tube when filled and remove the sampler from the container.

4.2.2.6 Empty the contents of the sampler onto a sheet of waxed or glazed paper or into a container.

4.2.2.7 Repeat 4.2.2.4 through 4.2.2.6 until the required number of containers have been sampled and the required number of increments have been obtained.

4.2.2.8 The total amount of powder in all the increments shall be adequate for the tests or evaluations that are to be performed. Additional increments may be taken from other randomly selected containers if necessary.

4.2.2.9 Combine all the increments to form the gross sample.

NOTE 2—Individual increments may be tested to investigate within-lot variability.

NOTE 3—The single-level tube sampler can be used to collect discrete samples from several levels that may then be individually evaluated to investigate within-container variability.

4.2.2.10 Blend the gross sample in a small blender to form the composite sample.

4.2.3 Sampling a Stationary Powder Using a Scoop Sampler

4.2.3.1 Scoop sampling is a common practice that has been used successfully in the metal powder...
industry. When applied using a standardized procedure, it can provide samples that are reliable examples of the powder being tested.

4.2.3.2 The scoop design is very important to minimize sampling error. It shall have a sharp edge and high sides that will contain the sample without overflowing during use, as partial loss of powder may compromise the sample. The scoop shall be made from a non-magnetic stainless steel. Plastic or cast aluminum food scoops or flat spoon-like implements shall not be used.

4.2.3.3 Consult the sampling schedule in Table 1 to determine the recommended minimum number of randomly selected containers from the lot or batch of powder that are to be sampled.

4.2.3.4 Hold the scoop sideways to scrape away the top 2 inches (approx. 50 mm) of the surface powder from the central region, exposing about a 10 by 10 inch (approx. 250 by 250 mm) area of fresh powder.

4.2.3.5 Skim through a short distance of the sub-surface powder with an arcing motion of the scoop to collect a powder sample from the container. The sample taken shall be confined completely within the scoop without any powder overflowing. This is one increment.

4.2.3.6 Empty the contents of the scoop into a container or onto a sheet of glazed or waxed paper.

4.2.3.7 Repeat 4.2.3.4 through 4.2.3.6 until the required number of containers have been sampled and the required number of increments have been obtained. Be sure to duplicate the scooping motion in order to collect about the same quantity of powder in each increment.

4.2.3.8 The total amount of powder in all the increments shall be adequate for the tests or evaluations that are to be performed. Additional increments may be taken from other randomly selected containers if necessary.

4.2.3.9 Combine all the increments to form the gross sample.

NOTE 4—Individual increments may be tested to investigate within-lot and sampling variability.

4.2.3.10 Blend the gross sample in a small blender to form the composite sample.

PART 2—OBTAINING THE TEST PORTIONS FROM THE COMPOSITE SAMPLE

4.3 Practice 2—Obtaining the Test Portions Using a Chute Splitter or Spinning Riffler

4.3.1 Divide the composite sample into smaller portions using a chute splitter, until each division either becomes small enough to use a spinning riffler or reaches the test portion size. If the composite sample is small enough, or when the divisions from a chute splitter are small enough, the use of a spinning riffler is the preferred method for obtaining one or a number of nearly identical test portions of powder. Chute splitters and spinning rifflers of various sizes are commercially available.

4.3.2 Chute Splitter

4.3.2.1 Place the entire composite sample into the charging pan of the splitter so that it is spread evenly in the pan.

4.3.2.2 Place the edge of the charging pan over the center line of the chute bank and pour the composite powder sample gently through the splitter at a rate where it flows freely through all of the chutes and into the collecting pan.

4.3.2.3 Determine the mass of the sample in one of the collection pans after the initial split has been made.

4.3.2.4 Adjustments to this mass can be made by repeatedly splitting the contents of one of the collection pans to increase the quantity in the previously filled pan, or repeatedly splitting the contents of one pan to produce two smaller size samples until the desired sample size or test portion size is reached.

4.3.3 Spinning Riffler

4.3.3.1 Pour the entire composite sample or one of the divisions from the prior chute splitter into the powder hopper of the spinning riffler and pass it through the riffler, maintaining a constant feed rate and rotational speed such that the entire sample passes through in no fewer than 100 rotations.

4.3.3.2 Measure the mass of powder in one of the riffler containers after the initial distribution.

4.3.3.3 Make adjustments by either using one of the previously riffled samples, combining two or more of the previously riffled samples, or using one or more of the previously riffled samples to increase the quantity in the remaining previously filled containers: then repeatedly riffling to produce smaller size samples until the desired test portion size is reached. If each riffled sample becomes too small to be used as a test portion, two or more riffled samples may be combined to make up the test portion quantity.

APPENDIX

A1. COMPARABLE STANDARDS

ASTM B215
ISO 3954
**FIGURE 1:** Sampling Scheme

- Entire Starting Powder
- Sampling
- Increments
- Combining
- Gross Sample
- Blending
- Composite Sample
- Splitting or Riffing
- Test Portions
- Compacting
- Green Test Specimens
- Sintering
- Sintered Test Specimens

**FIGURE 2:** Keystone Sampler

**FIGURE 3:** Alternative Sampler

**FIGURE 4:** Stainless Steel Scoop
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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This standard describes a method for determining the loss of mass of metal powders after heating for a specified time in a reducing atmosphere. This value is commonly referred to as hydrogen loss and approximates the oxygen content of the powder.

1.2 Only oxygen from oxides that are reduced by hydrogen gas under the test conditions, is measured. The test does not measure oxygen from oxides such as SiO₂, Al₂O₃, MgO, CaO, BeO, TiO₂.

1.3 The loss of mass may also include carbon, sulfur and other impurities that may react with the reducing gas or reaction products. Compensations should be made for this loss.

1.4 The loss of mass may include oxygen from oxides reduced by carbon, if present.

1.5 The overall change in mass may include a mass gain resulting from oxidation of hydrides formation of components present in the mixture.

1.6 For materials susceptible to reaction with nitrogen, such as stainless steels, the furnace should be purged with an inert gas, such as argon, and reduction carried out in a hydrogen atmosphere.

1.7 The loss of mass may include moisture and gases desorbed during the heat cycle.

1.8 With the exception of density, for which g/cm³ is the industry standard, SI units are used in this standard. Values in inch-pound units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.9 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Furnace capable of operating at the prescribed temperature.

2.2 Temperature control capable of maintaining temperature ± 15 °C (± 25 °F).

2.3 Gas-tight ceramic or metallic combustion tube.

2.4 Flow meter to measure flow of reducing gas.

2.5 Quartz, nickel, or alundum combustion boats.

2.6 Balance: A balance readable to 0.0001 g, having a capacity of at least 100 g.

2.7 Reagents

2.7.1 Nitrogen having an oxygen content less than 20 ppm and a dew point lower than -40 °C (-40 °F).

2.7.2 Reducing gas, either hydrogen having an oxygen content less than 20 ppm and a dew point lower than -40 °C (-40 °F), or dissociated ammonia having a dew point lower than -40 °C (-40 °F).

3. TEST PORTION

3.1 The test portion obtained in accordance with MPIF Standard 01, Sampling Metal Powders, shall be approximately 5 g (4 to 6 g).

4. PROCEDURE

Caution – Hydrogen and dissociated ammonia form explosive mixtures with air. Proper purging with nitrogen can reduce the risks of explosions.

4.1 Ensure that the furnace, with combustion tube inserted, has a temperature inside the combustion tube within the temperature range prescribed for the metal being tested. See Table 1.

4.2 Spread the test portion to a uniform depth in a combustion boat that has been preconditioned under test conditions to a constant mass. Ensure that the depth of the powder in the boat is approximately 3 mm (1/8 inch). Measure the mass of boat and specimen and record it to the nearest 0.0001 g.

4.3 Pass nitrogen through the combustion tube for a period of at least one (1) minute prior to insertion of the boat. Then place the boat at the center of the zone of uniform temperature in the furnace.

4.4 Ensure that the pilot flames at both ends of the tube furnace are lit and then start the flow of reducing gas (typically 10–30 mL/min); then stop the nitrogen gas flow. Record the time when the flow of reducing gas was started.

4.5 Ensure that a positive flow of reducing gas is maintained through the system for the time of reduction. Also ensure that the temperature inside the combustion tube is maintained within ± 15 °C (± 25 °F) of the temperature recommended for the metal being evaluated. See Table 1.
At the end of the prescribed time listed in Table 1, stop the flow of reducing gas and immediately start the flow of nitrogen into the tube.

After nitrogen has been flowing through the combustion tube for at least one minute, open the tube and withdraw the boat under nitrogen atmosphere to the cooler part of the combustion tube.

Allow the boat to cool in the nitrogen atmosphere until there is no possibility for oxidation of the sample material (normally 15 to 30 minutes). Then remove it from the tube and cool it to room temperature in a desiccator.

After the boat with the test portion has cooled to room temperature, measure the mass, and record it to the nearest 0.0001 g.

### Table 1.

<table>
<thead>
<tr>
<th>Metal Powder</th>
<th>Time (minute)</th>
<th>Temperature °C</th>
<th>Boat</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cobalt</td>
<td>30</td>
<td>875</td>
<td>Alundum, Nickel</td>
</tr>
<tr>
<td>Copper</td>
<td>30</td>
<td>875</td>
<td>Quartz</td>
</tr>
<tr>
<td>Copper-tin</td>
<td>30</td>
<td>775</td>
<td>Quartz</td>
</tr>
<tr>
<td>Iron &amp; Steel</td>
<td>60</td>
<td>1120</td>
<td>Alundum, Nickel</td>
</tr>
<tr>
<td>Nickel</td>
<td>60</td>
<td>1120</td>
<td>Alundum, Nickel</td>
</tr>
<tr>
<td>Tin</td>
<td>30</td>
<td>550</td>
<td>Alundum, Nickel</td>
</tr>
<tr>
<td>Tungsten</td>
<td>60</td>
<td>1120</td>
<td>Quartz</td>
</tr>
</tbody>
</table>

### 5. CALCULATION

5.1 Calculate the loss in mass as follows:

\[
\text{Loss in mass, } \% = \frac{(\text{A} - \text{B})}{\text{C}} \times 100
\]

where:

A = original mass of boat and test portion, grams
B = mass of boat and test portion after reduction, grams
C = original mass of test portion, grams

### 6. REPORT

6.1 Percentage loss in mass as percent to the nearest 0.01 percent.

### 7. PRECISION AND BIAS

7.1 **Precision**—The precision of this test method has not been determined by a statistically valid inter-laboratory test because of the limited number of participating laboratories (less than six).

7.2 The following precision data were developed by ASTM Subcommittee B09.02 using the procedures contained in ASTM E159 from an interlaboratory test conducted by four laboratories in 1994. The percent loss in mass in hydrogen was determined for four samples, a cobalt powder, a copper powder, an iron powder, and a tungsten powder. Except for the use of only four laboratories, ASTM Practice E691 was followed for the design and analysis of the data. The details are given in an ASTM research report.

7.3 The precision information given below is for the results. The results were obtained from the running of three replicates by each lab on each sample.

<table>
<thead>
<tr>
<th></th>
<th>Cobalt</th>
<th>Copper</th>
<th>Iron</th>
<th>Tungsten</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average, %</td>
<td>0.45</td>
<td>0.15</td>
<td>0.22</td>
<td>0.47</td>
</tr>
<tr>
<td>r, %</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.09</td>
</tr>
<tr>
<td>R, %</td>
<td>0.12</td>
<td>0.03</td>
<td>0.07</td>
<td>0.11</td>
</tr>
</tbody>
</table>

### APPENDIX

#### A1. COMPARABLE STANDARDS

- ASTM E159
- ISO 4491-2

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Method for
Determination of Flow Rate of Free-Flowing Metal Powders Using the Hall Apparatus

MPIF Standard 03

1. SCOPE

1.1 This standard describes two methods for determining the rate of flow of metal powders. The methods are only suitable for those powders that will flow unaided through the specified Hall flowmeter funnel.

1.2 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Hall Flowmeter Funnel: A standard flowmeter funnel (Fig. 1) having a calibrated orifice of 0.10 inch (2.5 mm) in diameter.

2.2 The dimensions shown for the flowmeter funnel, including the orifice, are not to be considered controlling factors. Calibration with emery, as specified in Appendix A, determines the working flow rate of the funnel.

NOTE 1—Name of the manufacturer of this apparatus is listed in General Information IV.

2.3 Stand: A support (Fig. 2) to hold the flowmeter funnel.

2.4 Timing Device: A device indicating elapsed time to the nearest 0.1 second.

2.5 Workbench: A vibration-free table to support the flowmeter assembly.

2.6 Balance: A balance readable to 0.01 g with a capacity of at least 100 g.

2.7 Chinese Emery: An emery powder used to calibrate the flowmeter funnel.

NOTE 2—This emery powder shall be dry in accordance with the procedure outlined in Appendix A.

3. TEST PORTION

3.1 The test portion shall consist of a mass of 50.0 g of metal powder obtained in accordance with MPIF Standard 01, Sampling Metal Powders.

4. PROCEDURE

4.1 Clean the funnel with clean dry paper towel.

4.2 Clean the funnel orifice with a clean, dry pipe cleaner.

4.3 Method 1—Static Method

4.3.1 Weigh out a 50.0 g test portion into a clean weighing dish.

4.3.2 Block the discharge orifice at the bottom of the funnel with a dry finger.

4.3.3 Carefully pour the 50.0 g test portion into the center of the flowmeter funnel without any tapping, vibration or movement of the funnel.

4.3.4 Place the emptied weighing dish on the flowmeter stand directly under the funnel orifice.

4.3.5 Simultaneously start the timing device and remove finger from the discharge orifice.

4.3.6 If the powder fails to start flowing, one light tap on the funnel rim is permitted. Further tapping of the funnel, however, or poking or stirring of the powder in the funnel with a wire or any other implement is not permitted.

4.3.7 Stop the timing device the instant the last of the powder exits the orifice.

4.3.8 Record the elapsed time to the nearest 0.1 s.

4.3.9 More than one flow may be run if desired. Use a new 50.0 g quantity of powder for each flow test. Average the flow times.

4.4 Method 2—Dynamic Method

4.4.1 Place an empty receptacle directly under the discharge orifice.

4.4.2 Weigh out a 50.0 g test portion into a clean weighing dish.

4.4.3 Pour the 50.0 g test portion into the center of the funnel and start the timing device the instant the powder exits the orifice.

4.4.4 Stop the timing device the instant the last of the powder exits the orifice.

4.4.5 Record the elapsed time to the nearest 0.1 s.

4.4.6 More than one flow may be run if desired. Use a new 50.0 g quantity of powder for each flow test. Average the flow times.
5. **CALCULATION**

5.1 Calculate the flow rate by multiplying the elapsed time by the correction factor, which has been determined as indicated in Appendix A.

6. **REPORT**

6.1 Flow rate (s/50 g) to the nearest second.

7. **PRECISION AND BIAS**

7.1 **Precision**—Precision has been determined from an interlaboratory study performed in 1991 by seven laboratories of ASTM Subcommittee B09.02 using Method 1.

7.1.1 Repeatability limits, r, are listed in Table 1. In 95% of flow rate determinations, on the basis of test error alone, duplicate tests in the same laboratory by the same operator on one homogeneous lot of powder will differ by no more than the stated amount in seconds.

7.1.2 Reproducibility limits, R, are listed in Table 1. For 95% of comparative trials done in two different laboratories, and on the basis of test error alone, single tests on the same homogeneous lot of powder will differ by no more than the stated amount in seconds.

7.1.3 The repeatability of the flow times using both the static and the dynamic measurement methods has been determined for a sponge iron powder, a water atomized iron powder, and a spherical bronze powder. In addition, the methods have been compared for a mixture of water atomized iron powder with 2% Ni, 0.8% graphite, and 0.5% Acrawax C lubricant. The results of ten repetitions for each material are summarized in Table 2.

7.2 **Bias**—No statement can be made about bias, because there is no standard reference material for flow rate measurement.

---

### Table 1. Precision of Flow Rate Measurements of Metal Powders

<table>
<thead>
<tr>
<th>Powder</th>
<th>Apparent Density (g/cm³)</th>
<th>Flow Rate (s/50 g)</th>
<th>Repeatability r</th>
<th>Reproducibility R</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spherical Bronze</td>
<td>5.04</td>
<td>12</td>
<td>0.1</td>
<td>0.8</td>
</tr>
<tr>
<td>Iron #1</td>
<td>2.46</td>
<td>31</td>
<td>0.7</td>
<td>2.5</td>
</tr>
<tr>
<td>Iron #2 (lubricated)</td>
<td>3.03</td>
<td>26</td>
<td>1.0</td>
<td>2.1</td>
</tr>
<tr>
<td>Iron (lubricated)</td>
<td>3.18</td>
<td>26</td>
<td>1.3</td>
<td>2.2</td>
</tr>
<tr>
<td>Bronze Premix</td>
<td>3.31</td>
<td>31</td>
<td>1.7</td>
<td>2.6</td>
</tr>
<tr>
<td>Brass (lubricated)</td>
<td>3.61</td>
<td>42</td>
<td>4.1</td>
<td>8.7</td>
</tr>
</tbody>
</table>

7.2 The repeatability of the flow times using both the static and the dynamic measurement methods has been determined for a sponge iron powder, a water atomized iron powder, and a spherical bronze powder. In addition, the methods have been compared for a mixture of water atomized iron powder with 2% Ni, 0.8% graphite, and 0.5% Acrawax C lubricant. The results of ten repetitions for each material are summarized in Table 2.

7.2.2 **Bias**—No statement can be made about bias, because there is no standard reference material for flow rate measurement.

---

### Table 2. Repeatability of Static and Dynamic Measurements Methods

<table>
<thead>
<tr>
<th>Material</th>
<th>Static Average</th>
<th>Std Dev</th>
<th>Dynamic Average</th>
<th>Std Dev</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sponge Iron</td>
<td>31.3</td>
<td>0.18</td>
<td>31.1</td>
<td>0.13</td>
</tr>
<tr>
<td>Water Atomized Iron</td>
<td>23.5</td>
<td>0.17</td>
<td>23.5</td>
<td>0.18</td>
</tr>
<tr>
<td>Mixture (FN-0208)</td>
<td>34.2**</td>
<td>0.85</td>
<td>33.8**</td>
<td>0.93</td>
</tr>
<tr>
<td>Spherical Bronze</td>
<td>12.3</td>
<td>0.06</td>
<td>12.4</td>
<td>0.11</td>
</tr>
</tbody>
</table>

*Only three of the ten flow tests resulted in measurable flow times; the others showed a no flow condition.

**One of the ten tests resulted in a no flow condition.

---

### APPENDIX A

**CALIBRATION OF HALL FLOWMETER FUNNEL**

A1. The producer supplies the powder flowmeter funnel calibrated as follows:

A1.1 Heat an open glass jar of Chinese emery in a drying oven at a temperature of 215 °C to 225 °F (102 °C to 107 °C) for 1 hour.

A1.2 Cool the emery to room temperature in a desiccator.

A1.3 Follow the procedure outlined in steps 4.1 through 4.3.8.

A1.4 Repeat steps 4.3.2 through 4.3.8 using the identical 50.0 g mass of emery for all the tests until 5 flow times, the extremes of which shall not differ by more than 0.4 s, have been recorded.

A1.5 The average of these five flow times, reported to the nearest 0.1 s, is stamped on the bottom of the funnel. The correction factor for the unused flowmeter funnel is 40.0 divided by this number.

A2. The flow rate of Chinese emery powder was established by an interlaboratory study conducted by ASTM Subcommittee B09.02 in 1995. It represents the flow rate through the master flowmeter funnel that had been used in a previous interlaboratory study with the former Turkish emery calibration powder, which is no longer available. The value was 40.0 s.

A3. It is recommended that the flow rate be checked periodically, at least every six months, using the procedure outlined in steps A1.1 through A1.4. If the flow rate has changed from that stamped on the instrument, the new correction factor will be 40.0 divided by this new flow rate. Before adopting the new correction factor, however, it is recommended that the cause of the change be investigated. If the flow rate has increased (faster flow), it is probable that repeated use has burnished the orifice and the new correction factor may be used. A decrease in flow rate (slower flow) may indicate a plating of soft powder upon the orifice. This should be removed carefully with the aid of a pipe cleaner and the calibration test rerun, the new correction factor being calculated if required. It is recommended that the use of a funnel be discontinued after the flow rate of the emery has increased such that the flow time is less than 37.0 s.
APPENDIX B

B1. COMPARABLE STANDARDS
    ASTM B213
    ISO 4490

Disclaimer

By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability.

MPIF standards are adopted in the public interest and are designed to eliminate misunderstandings between the producer and the purchaser and to assist the purchaser in selecting and obtaining the proper material for his particular product. Existence of an MPIF standard does not in any respect preclude any MPIF member or non-member from manufacturing or selling products not included in this standard or from utilizing procedures or equipment other than those included in this standard.

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
METAL POWDER INDUSTRIES FEDERATION

Method for

Determination of Apparent Density of Free-Flowing Metal Powders Using the Hall Apparatus

MPIF Standard 04
Issued 1945

1. SCOPE

1.1 This standard describes a procedure for determining the apparent density of free-flowing metal powders and is suitable for only those powders that will flow unaided through the specified Hall flowmeter funnel.

1.2 To determine the apparent density of non-free-flowing metal powders see MPIF Standard 28.

1.3 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Hall Flowmeter Funnel: A standard flowmeter funnel (Fig. 1) having a calibrated orifice of 0.10 inch (2.5 mm) in diameter.

2.2 The dimensions shown for the flowmeter funnel, including the orifice, are not to be considered controlling factors. Calibration with emery, as specified in MPIF Standard 03, Appendix A, determines the working flow rate of the funnel.

2.3 Density Cup: A cylindrical cup (Fig. 2) having a capacity of 25 ± 0.03 cm³, as measured by MPIF Standard 53, with an inside diameter of 28 ± 0.5 mm.

2.4 Stand: A support (Fig. 3) to hold the flowmeter funnel concentric with the density cup so that the bottom of the orifice is approx. 1.0 inch (25 mm) above the top of the density cup when assembled (Fig. 4).

2.5 Workbench: A level and vibration-free surface to support the powder flowmeter assembly.

2.6 Balance: A balance readable to 0.001 g with a capacity of at least 200 g.

NOTE 1—Names of manufacturers of this apparatus are listed in General Information IV.

3. TEST PORTION

3.1 The test portion shall consist of a volume of approximately 30 to 40 cm³ of metal powder, obtained in accordance with MPIF Standard 01, Sampling Metal Powders.

4. PROCEDURE

4.1 Weigh the empty density cup and record the mass to 0.001 g. Alternatively, place the empty density cup on the balance and tare the balance to zero.

4.2 Carefully load the test portion into the flowmeter funnel and permit it to run into the density cup through the discharge orifice. Take care not to move the density cup.

4.3 When the powder completely fills and overflows the periphery of the density cup, rotate the funnel approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

4.4 Using a non-magnetic spatula with the blade held perpendicular to the top of the cup, level off the powder flush with the top of the density cup. Avoid jarring the apparatus at any time.

4.5 After the leveling operation, tap the density cup lightly on the side to settle the powder to avoid spilling during transfer. Wipe off any powder sticking to the outside wall of the cup.

4.6 Transfer the filled density cup to the balance and weigh it. Record the mass to 0.001 g.

4.7 If necessary, deduct the mass of the empty density cup to obtain the mass of powder in the cup, M.

4.8 More than one apparent density test may be run if desired. Use a fresh test portion of powder for each test. Average the apparent density values.

5. CALCULATION

5.1 Calculate the apparent density as follows:

\[
\text{Apparent Density - Hall} \ (\rho_{\text{Hall}}), \ g/cm^3 = \frac{M}{V}
\]

where:

\[M = \text{mass of powder from the density cup in grams}\]
\[V = \text{volume of the density cup, cm}^3\]

NOTE 2—Use MPIF Standard 53 to determine the volume of the density cup.
6. REPORT

6.1 Apparent Density - Hall to the nearest 0.01 g/cm³.

7. PRECISION

7.1 The repeatability limit, r, and reproducibility limit, R, values were determined (1991) from an interlaboratory study analyzed statistically according to ASTM E691 as follows:

7.1.1 Repeatability limit \( r = 1.5\% \). Duplicate analyses of unlubricated or lubricated metal powders by the same operator and same apparatus should not differ by more than 1.5\% at the 95\% confidence level.

7.1.2 Reproducibility limit \( R = 3\% \) for unlubricated and lubricated iron powder and \( R = 5\% \) for lubricated brass and bronze powders. The difference between two single and independent results obtained by different operators working in different laboratories on lubricated and unlubricated iron powders should not differ by more than 3\% at the 95\% confidence level. Analyses of lubricated brass and bronze metal powders should not differ by more than 5\% at the 95\% confidence level.

APPENDIX

A1. COMPARABLE STANDARDS
ASTM B212
ISO 3923/1

Disclaimer

By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability. MPIF standards are adopted in the public interest and are designed to eliminate misunderstandings between the producer and the purchaser and to assist the purchaser in selecting and obtaining the proper material for his particular product. Existence of an MPIF standard does not in any respect preclude any MPIF member or non-member from manufacturing or selling products not included in this standard or from utilizing procedures or equipment other than those included in this standard.

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FIGURE 1: Hall Flowmeter Funnel

FIGURE 2: Density Cup (25 ± 0.03 cm³)

FIGURE 3: Stand

FIGURE 4: Assembly

This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. **SCOPE**

1.1 This standard describes a method for determining the dry sieve analysis of granular metal powders.

1.2 The values stated in SI units are to be regarded as the standard. The inch-pound units in parentheses were converted in accordance with IEEE/ASTM SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. **APPARATUS**

2.1 Sieves: A set of standard sieves should be selected based upon the mesh size of powders being evaluated. The sieves shall be 203 mm (8 inch) in diameter and either 25 or 50 mm (1 or 2 inch) in depth and fitted with brass, bronze, stainless steel, or other suitable wire. The U.S. Series standard sieves are the preferred series to use.

**NOTE 1**—For more detailed information, refer to ASTM Standard E11.

**NOTE 2**—The 203-mm diameter sieve is most commonly referenced as an 8-inch diameter sieve. 8-inch diameter sieves do not nest with 200-mm diameter sieves.

**Table 1. Testing Sieves According to U.S. Standard Series**

<table>
<thead>
<tr>
<th>Mesh Designation</th>
<th>Sieve Opening (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>850</td>
</tr>
<tr>
<td>40</td>
<td>425</td>
</tr>
<tr>
<td>60</td>
<td>250</td>
</tr>
<tr>
<td>80</td>
<td>180</td>
</tr>
<tr>
<td>100</td>
<td>150</td>
</tr>
<tr>
<td>140</td>
<td>106</td>
</tr>
<tr>
<td>200</td>
<td>75</td>
</tr>
<tr>
<td>230</td>
<td>63</td>
</tr>
<tr>
<td>325</td>
<td>45</td>
</tr>
</tbody>
</table>

2.2 Sieve Shaker: A mechanically operated, single eccentric sieve shaker that imparts to the nest of sieves a rotary motion of 285 ± 15 rpm and a tapping action of 150 ± 10 taps per minute. The hold down arm of the sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be mounted rigidly, and preferably shall be provided with an accurate time switch.

**NOTE 3**—Other types of sieve shakers are also available but their precision and reproducibility have not been determined.

**NOTE 4**—Names of manufacturers of this apparatus are listed in General Information IV.

2.3 Balance: A balance readable to 0.01 g, having a capacity of at least 150 g.

**NOTE 5**—Use of a sound-proof enclosure is recommended.

3. **TEST SPECIMEN**

3.1 The test portion, obtained in accordance with MPIF Standard 01 and having an apparent density greater than 1.50 g/cm² as determined by MPIF Standards 04 or 28, shall be 90-110 g. If the apparent density of the powder is less than 1.50 g/cm², a 40-60 g sample shall be used.

4. **PROCEDURE**

4.1 Assemble the group of sieves selected in consecutive order by the size of their openings, with the coarsest sieve at the top, the assembly being completed by a solid collecting pan below the bottom sieve.

**NOTE 6**—Examine the sieves to make sure that they are not damaged and that they are clean.

4.2 Place the test portion on the top sieve and close this sieve with a solid cover; then fasten the sieve assembly securely in the sieve shaker and operate the machine for a period of 15 minutes.

4.3 Remove the screened fractions from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them onto a glazed paper. Dislodge and transfer any material adhering to the bottom surface and the lower frame of the sieve with a brush into the next finer sieve.

4.4 Turn the sieve just removed upside down, and tap it on the paper containing the portion that had been
retained on it. Weigh this fraction and remove it from the balance. Record the mass to the nearest 0.01 g.
4.5 Repeat this process for each sieve in the nest.
4.6 Remove the fraction collected in the pan and weigh it. Record the mass to the nearest 0.01 g.
4.7 The sum of the masses of all the fractions shall be not less than 99% of the mass of the test specimen.
4.8 Add the difference between this sum and the mass of the test specimen to the mass of the fraction collected in the pan.

NOTE 7—If the sum is less than 99%, check the condition of the screens and the pan. Also check for possible weighing errors. If necessary, replace any sieves or the pan and repeat the test.

5. REPORT
5.1 The masses of the fractions retained on each sieve and the mass of the fraction collected in the pan shall be expressed as percentages of the test portion mass to the nearest 0.1%. Report any fraction that is less than 0.1% of the mass of the test portion as “trace”. If a fraction is absent it shall be reported as “0.0”.
5.2 Report the actual time of sieving, if it is different than 15 minutes.
5.3 Form for reporting test data of a typical 100 mesh powder

<table>
<thead>
<tr>
<th>Particle Size (µm)</th>
<th>Mesh Designation</th>
<th>Percentage By Mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt; 180</td>
<td>+ 80</td>
<td></td>
</tr>
<tr>
<td>≤ 180</td>
<td>&gt; 150</td>
<td>+ 100</td>
</tr>
<tr>
<td>≤ 150</td>
<td>&gt; 106</td>
<td>+ 140</td>
</tr>
<tr>
<td>≤ 106</td>
<td>&gt; 75</td>
<td>+ 200</td>
</tr>
<tr>
<td>≤ 75</td>
<td>&gt; 45</td>
<td>+ 325</td>
</tr>
<tr>
<td>≤ 45</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

5.4 Interpretation of this report should be made with reference to the dimensional tolerance of the standard sieves as specified in ASTM E11.

6. PRECISION
6.1 An interlaboratory study of the sieve analysis of metal powders was run in 1993 and 1994 using the procedures contained in MPIF Standard 05 (1992). Each of twelve laboratories made three tests on four powder samples using each of two sets of sieves. One set of sieves was a standard set that was circulated to each laboratory in turn. A second set of sieves was chosen by each laboratory from its in-house sieve stock. ASTM Practice E691 was followed for the design and analysis of the data. The details are given in MPPA Research Report MPPA R-05-95.
6.2 There were five U.S. Series standard sieves in each sieve nest: 80 mesh, 100 mesh, 140 mesh, 200 mesh, and 325 mesh plus a cover and a pan.
6.3 The precision information given below covers the percent retained between any pair of sieves, the percent retained on the coarsest sieve, the percent passing the finest sieve, and the cumulative percentages calculated from all sieves of greater openings above any sieve in the set.
6.4 The 95% repeatability limit, r, (within a laboratory) is represented by the equation:

\[ r = 0.4 + 0.03 \times [SF] \]

where:

[SF] is the % retained on the sieve of interest.

6.5 The 95% reproducibility limit, R, (between laboratories) is smaller for the circulated sieves than for the in-house sieves. For in-house sieves R can be calculated from the following equations:

\[ R = 1.2 + 0.15 \times [SF] \text{ for } [SF] \text{ from } 0 \text{ to } 22, \text{ or} \]

\[ R = 4.5 \text{ for } [SF] \text{ from } 22.1 \text{ to } 50 \]

where:

[SF] is the % retained on the sieve of interest. For circulated sieves (or by analogy matched sieves obtained by two laboratories) R can be calculated from the following equations:

\[ R = 0.3 + 0.064 \times [SF] \text{ for } [SF] \text{ from } 0 \text{ to } 30, \text{ or} \]

\[ R = 2.2 \text{ for } [SF] \text{ from } 30.1 \text{ to } 50 \]

where:

[SF] is the % retained on the sieve of interest.

6.6 Duplicate results from the same laboratory should be considered acceptable at the 95% confidence level unless they differ by more than r, the repeatability limit.
6.7 Duplicate results from two different laboratories should be considered acceptable at the 95% confidence level unless they differ by more than R, the reproducibility limit.

APPENDIX

A1. Compliance, Inspection, and Sieve Calibration
Compliance, inspection, and calibration sieves conforming to Specification ASTM E11 can be obtained from the sieve manufacturers. If used continually, the sieves will, after a period of time, become less accurate and might no longer meet the requirements set in Specification E11. A common acceptable practice would be to use either certified-, matched-, or calibration-grade sieves as a master set for quality control of compliance-grade working sieves.
A2.

**Table 3. Suggested Sieve Series for Metal Powders**

<table>
<thead>
<tr>
<th>Nominal Powder Mesh Size</th>
<th>20</th>
<th>40</th>
<th>60</th>
<th>100</th>
<th>140</th>
<th>200</th>
<th>325</th>
</tr>
</thead>
<tbody>
<tr>
<td>.</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Sieve</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Series</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>(Mesh Sizes)</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>Pan</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>

A3. Report on Precision and Accuracy of MPIF Standard 05. The complete report on this work, MPPA R-05-95, is available in the offices of the Metal Powder Industries Federation.

A4. COMPARABLE STANDARDS

ASTM B214

ISO 4497

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**Disclaimer**

By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability.

MPIF standards are adopted in the public interest and are designed to eliminate misunderstandings between the producer and the purchaser and to assist the purchaser in selecting and obtaining the proper material for his particular product. Existence of an MPIF standard does not in any respect preclude any MPIF member or non-member from manufacturing or selling products not included in this standard or from utilizing procedures or equipment other than those included in this standard.

The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.
1. SCOPE
1.1 This standard describes an analytical method for determination of the acid insolubles content of elemental iron and copper metal powders.
1.2 The acid insolubles referred to are compounds that are not completely soluble in ordinary mineral acids. These are generally considered to be silica and silicates, carbides, alumina, clays, other refractory oxides, or difficult-to-dissolve oxides that may be present in the raw material from which the powders are made or were introduced in their manufacturing process. This method excludes insoluble material that is volatile or combustible at the specified ignition temperature.
1.3 The values stated in SI units are to be regarded as the standard. The inch-pound units in parentheses were converted in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.
1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS AND REAGENTS
2.1 Apparatus
2.1.1 Hot Plate.
2.1.2 Muffle furnace capable of operating at 980 °C (1800 °F).
2.1.3 250 and 750 mL non-metallic casserole or beaker.
2.1.4 Glass funnel.
2.1.5 Quartz or porcelain crucible.
2.1.6 Desiccator.
2.1.7 Balance: A balance readable to 0.0001 g, having a capacity of at least 100 g.
2.1.8 Fine, ash-less filter paper (Whatman 541 or one of equivalent pore size and ash content).
2.1.9 Vapor collection system suitable to provide adequate operator protection from chemical vapors resulting from the acid digestion steps and muffle furnace ignition steps.
2.2 Reagents to use in the iron powder procedure.
2.2.1 Hydrochloric acid solution (1:1) - Mix 1 volume of concentrated HCl with 1 volume of water.
2.2.2 Hydrochloric acid solution (1:25) - Mix 1 volume of concentrated HCl with 25 volumes of water.
2.2.3 Nitric acid (concentrated), HNO₃.
2.2.4 Potassium thiocyanate, KSCN (5%).
2.3 Reagents to use in the copper powder procedure.
2.3.1 Nitric acid solution (1:1) - Mix 1 volume of concentrated HNO₃ with 1 volume of water.
2.3.2 Ammonium iodide, NH₄I

3. TEST PORTION
3.1 The test portion shall be 5 g of metal powder, obtained in accordance with MPIF Standard 01 “Sampling Finished Lots of Metal Powders,” weighed to the nearest 0.0001 g.

NOTE 1—Some operators report better reproducibility of results when increasing the metal powder sample size to 10 g. However, the statistical analysis that supports the present method was based on 5 g samples.

4. PROCEDURE
4.1 Iron Powder
4.1.1 Weigh the test portion, record the mass to the nearest 0.0001 g, and place it in a 750 mL casserole. Add 100 mL of HCl (1:1) with caution and cover with a watch glass. Allow the solution to stand at room temperature until the reaction is complete. Place the casserole on the hot plate. Heat to boiling point and hold until reaction ceases. Add 150 mL of distilled water, reheat to boiling point and maintain for about 1 minute. Filter the hot solution and wash the residue on the filter paper alternately with hot distilled water, and hot HCl (1:25) six times with each to ensure that all iron salts are dissolved. The absence of iron salts in the filtrate may be checked by the addition of a 5% solution of potassium thiocyanate. If iron salts are present, the filtrate will turn blood-red.

NOTE 2—If it is desired to exclude carbides as part of the insoluble matter, add 20 mL of concentrated HNO₃ to the HCl (1:1) and proceed as indicated above. This will prevent the inclusion of combined carbon with the insoluble matter being determined.
4.1.2 Prepare a crucible by preheating in a muffle furnace for 40 minutes in air at 980 °C (1800 °F) and then cool it in a desiccator. Weigh the crucible, record the mass to the nearest 0.0001 g, and place the filter paper and residue in it. Dry and place in a muffle furnace at 980 °C (1800 °F). Burn off the paper for 1 hour. Cool in the desiccator. Re-weigh the crucible and the ash, and record the mass to the nearest 0.0001 g.
4.2 Copper Powder

4.2.1 Weigh the test portion, record it to the nearest 0.0001 g, and place it in a 250 mL casserole. Add 100 mL of HNO₃ (1:1) and cover with a watch glass. Allow the solution to stand at room temperature until the reaction is complete. Place the casserole on the hot plate, and heat to boiling point. Boil to half volume. Add distilled water to bring the volume to approximately 100 mL. Heat the solution to boiling point and maintain boiling for about 1 minute. Filter the hot solution and wash the filter with hot distilled water until all traces of blue color (copper salts) disappear.

4.2.2 Prepare a crucible by preheating for 40 minutes in air at 820 °C (1500 °F) and then cool it in a desiccator. Weigh the crucible and place the filter paper and residue in it, record the mass to the nearest 0.0001 g. Dry and place in a muffle furnace at 820 °C (1500 °F) for 1 hour. Cool in the desiccator. Re-weigh the crucible and the ash, and record the mass to the nearest 0.0001 g.

NOTE 3—If the ignited residue contains tin oxide, add 5 g of NH₄F to the prepared crucible and heat in a furnace, in air, at 600 °C (1100 °F) for 15 minutes. After the fumes have disappeared, remove the crucible and cool. Add 2 to 3 mL of concentrated nitric acid; evaporate to dryness, ignite and weigh. Repeat this procedure with NH₄F and HNO₃ until constant mass is obtained. The loss in mass represents tin oxide. Subtract this loss in mass from the mass of insoluble matter determined in section 4.2.2 to calculate an insoluble fraction that is free of tin oxide.

5. CALCULATION

5.1 Calculate the percentage of total insoluble matter as follows:

\[
\text{Total insoluble matter, } \% = \left( \frac{A-B}{C} \right) \times 100
\]

where:

A = mass of crucible and ash after ignition, in grams,
B = mass of crucible, in grams, and
C = grams of sample used

6. REPORT

6.1 Total insoluble matter as a percentage to the nearest 0.01%.

7. PRECISION

7.1 The following precision data were developed using the procedures contained in MPIF Standard 06 from an interlaboratory study that performed six sets of tests. The % insoluble was determined for four samples, a -325 mesh iron, a -60 mesh iron, a -325 mesh copper, and a -60 mesh copper. The different particle sizes were used to determine if there were any effects on the precision of testing based on differences in particle size distribution. ASTM Practice E691 was followed for the design and analysis of the data; the details are given in MPPA Research Report No. MPPA-R-06-98.

7.2 The precision information given below is for the comparison of two test results. The results were obtained from the running of three replicates in each test on each sample.

<table>
<thead>
<tr>
<th>Insoluble Content of Iron and Copper Powders</th>
<th>Precision Statistics</th>
</tr>
</thead>
<tbody>
<tr>
<td>-325 Iron</td>
<td>-60 Iron</td>
</tr>
<tr>
<td>Average (%)</td>
<td>0.29</td>
</tr>
<tr>
<td>(S_e) (%)</td>
<td>0.022</td>
</tr>
<tr>
<td>(S_R) (%)</td>
<td>0.051</td>
</tr>
<tr>
<td>(r) (%)</td>
<td>0.06</td>
</tr>
<tr>
<td>(R) (%)</td>
<td>0.14</td>
</tr>
</tbody>
</table>

7.3 Duplicate results from the same laboratory (individual determinations prior to averaging) should be considered acceptable at the 95% confidence level unless they differ by more than \(r\), the repeatability limit.

7.4 Duplicate results from two different laboratories (individual determinations prior to averaging) should be considered acceptable at the 95% confidence level unless they differ by more than \(R\), the reproducibility limit.

7.5 Data Source – Data used in developing this statement were contributed by the members of ASTM Committee B09 on Metal Powders and Metal Powder Products and were used for the determination of Precision with the permission of ASTM.

APPENDIX

A1. COMPARABLE STANDARDS

ASTM E194
ISO 4496

Disclaimer

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This terminology standard includes definitions that are helpful in the interpretation and application of powder metallurgy terms.

1.2 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. REFERENCED DOCUMENTS

2.1 ASTM Standards:
   B331 Test Method for Compressibility of Metal Powders in Uniaxial Compaction

3. TERMINOLOGY

3.1 Definitions:

acicular powder, \( n \)—needle-shaped particles.

activated sintering, \( n \)—a sintering process during which the rate of sintering is increased, for example, by addition of a substance to the powder or by changing sintering conditions.

agglomerate, \( n \)—several particles adhering together.

air classification, \( n \)—the separation of powder into particle size fractions by means of an air stream of controlled velocity.

angle of repose, \( n \)—the basal angle of a pile formed by powder when freely poured under specified conditions onto a horizontal surface.

apparent density, \( n \)—the mass of a unit volume of powder, usually expressed as grams per cubic centimetre, determined by a specified method.

apparent hardness, \( n \)—the hardness of a PM material (including the effects of porosity), measured using macroindentation hardness equipment.

Discussion—See general description of production, properties, and uses of sintered metal powder bearings and structural parts, paragraph on density and mechanical properties, information on hardness measurement, Volume 02.05.

apparent porosity, \( n \)—specific to cemented carbides, microstructural features that appear to be pores in a properly prepared, unetched surface; these features may result from uncombined carbon or nonmetallic inclusions as well as actual porosity.

atomization, \( n \)—the dispersion of a molten metal into particles by a rapidly moving gas or liquid stream or by mechanical means.

atomized metal powder, \( n \)—metal powder produced by the dispersion of a molten metal by a rapidly moving gas, or liquid stream, or by mechanical dispersion.

binder, \( n \)—a cementing medium; either a material added to the powder to increase the green strength of the compact, and which is expelled during sintering; or a material (usually of relatively lower melting point) added to a powder mixture for the specific purpose of cementing together powder particles which alone would not sinter into a strong body.

binder (MIM), \( n \)—a mixture of polymers or other materials, or both, that when combined with metal powders enables the mixture to flow during the injection molding process. The binder imparts green strength to the MIM part and allows it to retain its molded shape.

binder removal, \( n \)—the chemical or thermal extraction of binder from a compact. Synonymous with debinding.

blank, \( n \)—a pressed, presintered, or fully sintered compact, usually in the unfinished condition, requiring cutting, machining, or some other operation to give it its final shape.

blending, \( n \)—the thorough intermingling of powders of the same nominal composition (not to be confused with mixing).
blist er crack, $n$—typically small defects (star burst) over or around a bump or blister.

Discussion—These may occur during sintering as a result of rapid outgassing of the lubricant. The rapid outgassing may be caused by the specified amount of lubricant being subjected to an excessive heating rate. The defects may also be caused by “concentrated balls” of lubricant, or moisture. During the sintering of the copper base PM parts, hydrogen gas from the furnace atmosphere can diffuse into the compact and react with residual oxygen, producing steam that can form blisters and cracks. In that industry, this is also called embrittlement, and is not to be confused with the hydrogen embrittlement of high strength steel.

blistered compact, $n$—a blistered object characterized by having blisters or eruptions on the surface.

Discussion—In ferrous materials, this effect is often caused by in situ gas decomposition and soot formation that forces particles apart and causes the compact to blister.

bridging, $n$—the formation of arched cavities in a powder mass.

briquet, $n$—see compact.

brown part, $n$—an MIM part that has had a majority of the binder (first stage) removed. Synonymous with debound part.

bulk density, $n$—the mass per unit volume of a powder under nonstandard conditions, for example, in a shipping container (not to be confused with apparent density).

burn-off, $n$—removal of organic additives (binder or lubricant) from a compact by heating.

cake, $n$—a bonded mass of unpressed metal powder.

Discussion—Often refers to the form of powder as it exits a furnace.

carbonyl powder, $n$—a metal powder prepared by the thermal decomposition of a metal carbonyl

cavity (MIM), $n$—the hollow space of a mold that forms one part.

cemented carbide, $n$—sintered material characterized by high strength and wear resistance and comprising one or more carbides of refractory metals as the main component bonded by metallic binder phase.

cermet, $n$—sintered material containing at least one metallic phase and at least one nonmetallic phase that is generally of a ceramic nature.

c hemical deposition, $n$—the precipitation of one metal from a solution of its salts by the addition of another metal or reagent to the solution.

c hemically precipitated metal powder, $n$—powder produced by the reduction of a metal from a solution of its salts either by the addition of another metal higher in the electromotive series or by other reducing agent.

classification, $n$—separation of a powder into fractions according to particle size.

closed pore, $n$—a pore not communicating or connected with an exterior surface.

c coin, $v$—to repress a sintered compact to obtain a definite surface configuration. Synonymous with emboss (not to be confused with restrike or size).

cold isostatic pressing (CIP), $n$—isostatic pressing at ambient temperature.

cold pressing, $n$—the forming of a compact at room temperature.

cold welding, $n$—cohesion between two surfaces of metal, generally under the influence of externally applied pressure, at room temperature.

Discussion—Often used to describe the mechanism by which powder particles develop initial bonds and a pressed compact develops green strength.

co mmunited powder, $n$—a powder produced by mechanical attrition of solid metal or powder

communicating pores, $n$—see interconnected porosity.

compact, $n$—an object produced by the compression of metal powder, generally while confined in a die, with or without the inclusion of nonmetallic constituents. Synonymous with briquet.

compactibility, $n$—a conceptual term, encompassing the powder characteristics of compressibility, green strength, edge retention, and lamination tendency, that relates to the ability of a powder to be consolidated into a usable green compact.

compacting, $n$—a process in which a powder held in a die or other container is subjected to an external force in order to densify the powder and produce a compact of prescribed shape and dimensions.

compacting pressure (uniaxial), $n$—applied force divided by the projected area of contact with the punch(es).

compacting tool set, $n$—an assembly of tooling items in which powder is pressed.

Discussion—May include a die, punches, and core rods.

c ompletely alloyed powder, $n$—see pre-alloyed powder.

composite compact, $n$—a metal powder compact consisting of two or more adhering layers, rings, or other shapes of different metals or alloys with each material retaining its original identity.

composite powder, $n$—a powder in which each particle consists of two or more distinct constituents.

compound compact, $n$—a metal powder compact consisting of mixed metals, the particles of which are joined by pressing or sintering, or both, with each metal particle retaining substantially its original composition.

compounding (MIM), $n$—process whereby the metal powders and molten binder constituents are combined to produce a feedstock.
compressibility, n—the capacity of a metal powder to be densified under a uniaxially applied pressure in a closed die.

Discussion—Compressibility is measured in accordance with Test Method B331 and may be expressed numerically as the pressure to reach a specified density, or alternatively the density at a given pressure.3

compression ratio, n—the ratio of the volume of the loose powder to the volume of the compact made from it. Synonymous with fill ratio.

continuous sintering, n—presintering, or sintering, in such manner that the objects are advanced through the furnace at a fixed rate by manual or mechanical means. Synonymous with stoking.

cooling rate, n—the average temperature change per second between prescribed temperatures during the cooling phase of a thermal process.

core rod, n—a member of the compacting tool set that forms internal features such as splines, diameters, keyways, or other profiles in a PM compact.

cored bar, n—a compact of bar shape heated by its own electrical resistance to a temperature high enough to melt its interior.

crack (RD), n—generally a planar defect.

cracked ammonia, n—see dissociated ammonia.

cracks (rigid die system (RD)), n—the following names and definitions apply only to items produced in a rigid die system (RD) as opposed to those cracks produced by other systems, that is, metal injection molding, vacuum hot pressing, and so forth.4

cross-product contamination, n—the unintentional mixing of powders with distinct differences in either physical characteristics or chemical composition or both.

cut, n—see fraction. 

debinding, n—see binder removal.

debound part, n—see brown part.

dendritic powder, n—particles, usually of electrolytic origin, having the typical pine tree structure.

densification crack, n—a defect caused by differential stresses in a region of a part that has experienced large differences in shrinkage during sintering.

density (dry), n—see sintered density.

density (wet), n—see impregnated density.

density ratio, n—the ratio, often expressed as a percentage, of the density of a porous material to the density of the same material completely free of porosity. Synonymous with relative density.

die, n—a member of the compacting tool set forming the cavity in which the powder is compacted or a PM compact is repressed.

die body, n—the stationary or fixed part of a die.

die insert, n—a removable liner or part of a die body.

die set, n—the parts of a press that hold and locate the die in proper relation to the punches.

diffusion-alloyed powder, n—a partially alloyed powder produced by means of a diffusion anneal.

dimensional change of a compact, n—the difference, at room temperature, between the size of the sintered specimen and the die size.

Discussion—The difference in dimensions is usually reported as a percentage of the die size. It should include a (+) when the sintered part is larger than the die size and a (−) when the sintered part is smaller than the die size.

disintegration, n—the reduction of massive material to powder.

dispersion-strengthened material, n—a material consisting of a metal and finely dispersed, substantially insoluble, metallic or nonmetallic phase.

dissociated ammonia, n—a reducing gas produced by the thermal decomposition of anhydrous ammonia over a catalyst, resulting in a gas of 75% hydrogen and 25% nitrogen. Synonymous with cracked ammonia.

double-action pressing, n—a method by which a powder is pressed in a die between opposing moving punches.

double press-double sinter, n—to repress and sinter a previously presintered or sintered compact.

Discussion—Used to describe a four-step manufacturing process.

ejection crack, n—a defect that occurs during the removal of the compact from the tooling (usually occurs in multilevel parts that are not supported uniformly on all lower surfaces).

electrolytic powder, n—powder produced by electrolytic deposition or by the pulverization of an electrodeposit.

endermic gas, n—a reducing gas atmosphere used in sintering, produced by the reaction of a hydrocarbon vapor and air over a catalyst with the use of an external heat source. It is low in carbon dioxide and water vapor while containing combustibles of about 60 atomic percent hydrogen and carbon monoxide combined.

equalizing, n—see blending.

exothermic atmosphere (gas), n—a reducing gas atmosphere used in sintering, produced by partial or complete combustion of hydrocarbon fuel gas and air with the associated generation of heat. The maximum combustible content is approximately 25 atomic percent.

explosive compaction, n—high-energy consolidation of powders by means of a detonation shock wave.

exudation, n—the action by which all or a portion of the low melting constituent of a compact is forced to the surface during sintering. Sometimes referred to as “bleed out.” Synonymous with sweating.

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3 See ASTM Test Method B331.

4 There is detailed information on numerous cracks, their location, cause, and prevention in a handbook published by Metal Powder Industries Federation, Princeton, New Jersey, “The Common Cracks in PM Compacts” by D. Zenger and H. Cai.
feedstock, $n$—a part of the compacting press that delivers powder to the die cavity, usually by sliding an open-bottomed powder container over the open top of the die.

feedstock, $n$—in metal injection molding (MIM), a moldable mixture of metal powder and binder.

fill ratio, $n$—see compression ratio.

fines, $n$—the portion of a powder composed of particles which are smaller than a specified size, currently less than 44 μm. See also superfines.

flake powder, $n$—flat or scale-like particles whose thickness is small compared with the other dimensions.

flow rate, $n$—the time required for a powder sample of standard weight to flow through an orifice in a standard instrument according to a specified procedure.

fluid permeability, $n$—see permeability.

fraction, $n$—the portion of a powder sample that lies between two stated particle sizes. Synonymous with cut.

fully dense material, $n$—a material completely free of porosity and voids.

Discussion—This is a conceptual term. In practice, complete densification is difficult to achieve and some microporosity will generally be present. The measured density of a material depends on its specific chemistry, thermomechanical condition, and microstructure.

gas classification, $n$—the separation of powder into particle size fractions by means of a gas stream of controlled velocity.

granular powder, $n$—particles having approximately equidimensional nonspherical shapes.

granulation, $n$—the production of coarse metal particles by pouring the molten metal through a screen into water (shotting) or by violent agitation of the molten metal while solidifying.

green, $n$—unsintered (not sintered); for example, green compact, green density, green strength.

green crack, $n$—a defect that occurs prior to sintering.

green density, $n$—the mass per unit volume of an unsintered compact.

green expansion, $n$—the increase in dimensions of an ejected compact relative to the die dimensions, measured at right angles to the direction of pressing. Synonymous with springback.

green strength, $n$—stress required to break an unsintered compact.

growth, $n$—an increase in dimensions of a compact which may occur during sintering. (Converse of shrinkage.)

hardmetal, $n$—see cemented carbide.

heating rate, $n$—the average temperature change per unit time between prescribed temperatures during the heating phase of a thermal process.

hot densification, $n$—the consolidation, at an elevated pressure and at a temperature that often results in recrystallization, of an unsintered, presintered, or sintered powder preform, or of encapsulated or loose powder, to reduce porosity.

hot isostatic pressing (HIP), $n$—isostatic pressing at an elevated temperature.

hot pressing, $n$—pressure-assisted, low strain rate uniaxial densification of a powder preform, compact, or encapsulated or loose powder at a temperature sufficient to induce diffusion or creep.

hot repress powder forging, $n$—hot densification of a PM preform by forging where the material flow is mainly in the direction of forging.

hot upset powder forging, $n$—hot densification of a PM preform by forging where there is a significant amount of lateral material flow.

hybrid-alloy powder, $n$—a pre-alloyed or diffusion-alloyed powder to which either elemental or master-alloy metal powders have been admixed.

hydrogen loss, $n$—the loss in weight of metal powder or of a compact caused by heating a representative sample for a specified time and temperature in a purified hydrogen atmosphere—broadly, a measure of the oxygen content of the sample when applied to materials containing only such oxides as are reducible with hydrogen and no hydride-forming element.

hydrogen-reduced powder, $n$—powder produced by the reduction of a metal oxide in an atmosphere containing hydrogen.

impregnated density, $n$—the mass per unit volume of a sintered PM part or test specimen, impregnated with oil or other lubricants. Synonymous with density (wet).

impregnation, $n$—a process of filling the pores of a sintered compact, with a nonmetallic material such as oil, wax, or resin.

infiltrant efficiency, $n$—the ratio of the mass of infiltrant absorbed by the part to the mass of infiltrant originally used, expressed as a percentage.

infiltration, $n$—a process of filling the pores of a sintered, or unsintered, compact with a metal or alloy of lower melting point.

infiltration erosion, $n$—the pitting, channeling, and coarsening of the surface porosity that results from the dissolution of the base metal by the liquid infiltrant, as the infiltrant flows into the matrix.

infiltration loading density, $n$—infiltrant weight per unit area of contact between infiltrant and part.

infiltration residue, $n$—material that remains on the surface of the part after infiltration.

interconnected porosity, $n$—a network of mutually connected pores that may or may not extend to an exterior surface. Synonymous with communicating pores.

irregular powder, $n$—particles lacking symmetry.
isostatic pressing (IP), \( n \) — densification of a powder or compact, at an elevated pressure, nominally equal from every direction.

Discussion — Powders and compacts require a barrier envelope to prevent the pressurizing medium (gas or liquid) from infiltrating the item, unless the density of the compact is sufficient (for example, HIP of AM or MIM parts).

lamination crack, \( n \) — a defect(s) roughly parallel to the punch faces of the part (these defects usually occur when powder is compressed to high density and the relaxation forces during pressure release exceed the binding force between the particles).

liquid phase sintering, \( n \) — sintering of a compact, or loose powder aggregate, under conditions in which a liquid phase is present during part of the sintering cycle.

lot, \( n \) — a specified quantity of product manufactured under traceable, controlled conditions as agreed between producer and user.

lubricant, \( n \) — material used to reduce inter-particle friction and the friction between the powder mass and the tooling.

lubricant (admixed), \( n \) — a lubricant incorporated into a powder mixture.

lubricant (die-wall), \( n \) — a lubricant applied to the tooling surfaces to facilitate ease of movement of the tooling and the removal of the compact or part from the tooling.

master-alloy powder, \( n \) — a pre-alloyed powder of high concentration of alloy content designed to be diluted when mixed with a base powder to produce the desired composition.

matrix metal, \( n \) — the continuous phase of a polyphase alloy or mechanical mixture; the physically continuous metallic constituent in which separate particles of another constituent are embedded.

mechanically alloyed powder, \( n \) — a composite powder produced by mechanically incorporating other constituents which are generally insoluble within the deformable particles of the matrix metal.

metal filter, \( n \) — a metal structure having controlled interconnected porosity produced to meet filtration or permeability requirements.

metal injection molding (MIM), \( n \) — a process in which a mixture of metal powders and a binder system is forced under pressure into a mold. See also powder injection molding.

metal powder, \( n \) — particles of elemental metals or alloys, normally less than 1000 \( \mu \)m (1 mm) in size.

milling, \( n \) — the mechanical treatment of metal powder, or metal powder mixtures, as in a ball mill, to alter the size or shape of the individual particles or to coat one component of the mixture with another.

MIM, \( n \) — see metal injection molding.

minus sieve, \( n \) — the portion of a powder sample which passes through a standard sieve of specified number. (See plus sieve.)

mixed powder, \( n \) — see powder mixture.

mixing, \( n \) — the thorough intermingling of powders of two or more materials.

mold, \( n \) — in metal or powder injection molding, the member of the tooling into which the powder and binder mixture is forced, and the configuration of which forms the surfaces of the green part. In isostatic compacting, a mold is also the confining form in which powder is isostatically compacted.

molding, \( n \) — see compacting.

multiple pressing, \( n \) — a method of pressing whereby two or more compacts are produced simultaneously in separate die cavities.

nanopowder, \( n \) — a powder consisting of particles typically less than 100 nm in size.

neck formation, \( n \) — during sintering, the development of a neck-like bond between particles.

needles, \( n \) — elongated rod-like particles.

nitrogen alloying, \( n \) — the transfer of nitrogen from a furnace atmosphere to powder or a PM part, in such a way as to increase the nitrogen content of the material within controlled limits.

nodular powder, \( n \) — irregular particles having knotted, rounded, or similar shapes.

oil content, \( n \) — the measured amount of oil contained in an oil-impregnated object, for example, a self-lubricating bearing.

open pore, \( n \) — a pore communicating with an exterior surface.

oversize powder, \( n \) — particles coarser than the maximum permitted by a given particle size specification.

oxide network, \( n \) — continuous or discontinuous oxides that follow prior particle boundaries.

packing material, \( n \) — any material in which compacts are embedded during the presintering or sintering operation.

partially alloyed powder, \( n \) — a powder in which the alloy addition or additions are metallurgically bonded to an elemental or pre-alloyed powder.

particle size, \( n \) — the controlling linear dimension of an individual particle as determined by analysis with sieves or other suitable means.

particle size distribution, \( n \) — the percentage by weight, or by number, of each fraction into which a powder sample has been classified with respect to sieve number or microns. (Preferred usage: “particle size distribution by frequency.”)

particulate matter, \( n \) — see powder.

permeability, \( n \) — the rate of passage of a liquid or a gas through a porous material; determined under specified conditions.

PF, \( n \) — the acronym for powder forging. See powder forging.
PIM, *n*—see powder injection molding.
platelet powder, *n*—a powder composed of flat particles having considerable thickness (as compared with flake powder).
plus sieve, *n*—the portion of a powder sample retained on a standard sieve of specified number. (See minus sieve.)
PM, *n*—the acronym for powder metallurgy.
PM forging, *n*—see powder forging.
PM part, *n*—see powder metallurgy part.
pore, *n*—an inherent or induced cavity within a particle or within an object.
pore-free density (compacted powder mixture), *n*—density that a compacted powder mixture (green compact) would reach in the absence of any porosity.

**Discussion** —It may be calculated by dividing the mass of the green compact by the sum of the volumes occupied by all the constituents of the mixture.
pore-forming material, *n*—a substance included in a powder mixture that volatilizes during sintering and thereby produces a desired kind and degree of porosity in the finished compact.
porosity, *n*—the amount of pores (voids) expressed as a percentage of the total volume of the powder metallurgy part.
powder, *n*—particles that are usually less than 1000 μm (1 mm) in size.
powder flow meter, *n*—an instrument for measuring the rate of flow of a powder according to a specified procedure.
powder forging, *n*—densification (generally hot) of a PM preform by forging.

**Discussion** —In the case in which the preform has been sintered, the process is often referred to as “sinter forging.”
powder injection molding (PIM), *n*—a process in which a mixture of powders and a binder system is forced under pressure into a mold. See also metal injection molding.
powder metallurgy, *n*—the production and utilization of metal powders.
powder metallurgy part, *n*—a shaped object that has been formed from metal powders and bonded by heating below the melting point of the major constituent. A structural or mechanical component, bearing, or bushing made by the powder metallurgy process. Synonymous with PM part.
powder mixture, *n*—a powder made by mixing two or more powders of differing chemical composition, particle size distribution, particle shape, or a combination of these characteristics.
powder rolling, *n*—see roll compacting.
pre-alloyed powder, *n*—powder composed of two or more elements that are alloyed in the powder manufacturing process in which the particles are of the same nominal composition throughout. Synonymous with completely alloyed powder.
preform, *n*—a PM compact intended to be changed in shape through deformation and densification.
preforging, *n*—the initial pressing of a metal powder to form a compact that is subjected to a subsequent pressing operation other than coining or sizing. Also, the preliminary shaping of a refractory metal compact after presintering and before the final sintering.
premix, *n*—a uniform mixture of ingredients to a prescribed analysis, prepared by the powder producer, for direct use in compacting powder metallurgy products.
presintering, *n*—the heating of a compact at a temperature below the normal final sintering temperature, usually to increase the ease of handling or shaping the compact, or to remove a lubricant or binder before sintering.
presi forming, *n*—to apply force to a mass of powder, generally while confined in a die or container, to form a compact.
pressed bar, *n*—a compact in the form of a bar; a green compact.
presi density, *n*—synonymous with green density.
presi crack, *n*—a defect occurring as a result of the forming operation.
pulverization, *n*—the reduction in particle size of metal powder by mechanical means, a specific type of disintegration.
punch, *n*—a member of a compacting tool set used to close the die cavity and transmit the applied pressure to the powder or PM compact.

**Discussion** —Multiple upper or lower punches may be needed to compact multilevel parts.
push-off crack, *n*—a defect or crushed surface caused by the action of the feed shoe or other mechanism removing the compact from the area above the lower punch.
radial crushing strength, *n*—the relative capacity of a plain sleeve specimen of sintered metal to resist fracture induced by a load applied between flat parallel plates in a direction perpendicular to the axis of the specimen.
rate-of-oil flow, *n*—the rate at which a specified oil will pass through a sintered porous compact under specified test conditions.
reduced metal powder, *n*—metal powder produced, without melting, by the chemical reduction of metal oxides or other compounds.
relative density, *n*—see density ratio.
repress, *v*—to apply pressure to a previously pressed and either sintered or presintered compact. It includes restrike, coin, and size.
restripe, *v*—to repress for the purpose of increasing the density of a sintered compact (not to be confused with coin or size).
roll compacting, n—the progressive compacting of metal powders by the use of a rolling mill. Synonymous with powder rolling.

rolled compact, n—a compact made by passing metal powder continuously through a rolling mill so as to form relatively long sheets of pressed material.

rotary press, n—a machine fitted with a rotating table carrying multiple dies in which a material is pressed.

RSM—Rapidly Solidified Materials.

RSP—Rapid Solidification Processing or Rapidly Solidified Powders.

RST—Rapid Solidification Technology.

runner (MIM), n—(1) the secondary feed channel in an injection mold that runs from the inner end of the sprue to the cavity gate.(2) the piece formed in a secondary feed channel or runner.

screen analysis, n—see sieve analysis.

segment die, n—a die fabricated by the assembly of several die sections within a retaining bolster or shrinkage ring.

segregation, n—the separation of one or more constituents of a powder, for example, by particle size or chemical composition.

shot volume (MIM), n—the total hollow space of a mold including cavity or cavities, runner(s), and sprue.

shrinkage, n—a decrease in dimensions of a compact which may occur during sintering. (Converse of growth.)

sieve analysis, n—particle size distribution; usually expressed as the weight percentage retained upon each of a series of standard sieves of decreasing size and the percentage passed by the sieve of finest size. Synonymous with screen analysis.

sieve classification, n—the separation of powder into particle size ranges by the use of a series of graded sieves.

sieve fraction, n—that portion of a powder sample that passes through a standard sieve of specified number and is retained by some finer sieve of specified number.

single-action pressing, n—a method by which a powder is pressed in a stationary die between one moving and one fixed punch.

Discussion—Only during ejection does either the stationary die or punch move.

sinter, v—to increase the bonding in a mass of powder or a compact by heating below the melting point of the main constituent.

sinter forging, n—see powder forging.

sinter hardening, n—a thermal process in which a ferrous product (material) is sintered and then cooled at a rate sufficient to produce a predominantly martensitic microstructure.

sintered density, n—the mass per unit volume of a sintered PM part or test specimen not impregnated with oil or other lubricant. Synonymous with density (dry).

sintering crack, n—a defect that occurs during the sintering operation.

sintering time, n—the total elapsed time during which the PM part/specimen is within (±) a specified percentage of the stated sintering temperature.

size, v—to repress a sintered compact to decrease the dimensional variation (not to be confused with coin or restrike).

slip casting, n—a method of forming metal or ceramic shapes by pouring a stabilized suspension of a powder in a fluid, usually water, into the shaped cavity of a fluid-absorbing mold, followed by debinding and sintering.

slip (rupture) crack, n—a defect that occurs typically at the junction between levels of a multilevel part (occurs during the pressing cycle while powder is transferring from one level (area) to another).

slumping, n—the lack of shape retention of a molded part, during subsequent processing, because of the effect of gravity.

soft magnetic composite, n—a compacted PM product in which individual ferrous powder particles are separated by a dielectric material.

solid-state sintering, n—sintering of a powder or compact without formation of a liquid phase.

solids loading, n—the relative volume of metal powder in a feedstock designed for metal injection molding, expressed as a volume percent, e.g. 65 % solids loading.

specific surface, n—the surface area of one gram of powder, usually expressed in square centimetres.

spherical powder, n—globular-shaped particles.

split die, n—a die made of parts that can be separated for ready removal of the compact.

sponge iron, n—a coherent, porous mass of substantially pure iron produced by solid-state reduction of iron oxide (for example, iron ore or mill scale).

sponge iron powder, n—ground and sized sponge iron, which may have been purified or annealed or both.

sponge metal, n—any porous metal produced by the reduction or decomposition of a compound at temperature below the melting point of the metal.

sponge metal powder, n—a powder produced from a sponge metal by mechanical methods of size reduction.

springback, n—see green expansion.

sprue (MIM), n—(1) the primary feed channel that runs from the outer face of a n injection mold to the runner.(2) the piece formed in a primary feed channel or sprue.

steam blackening, n—the superheated steam treatment of a ferrous PM component to form a thin, dark, oxide layer, primarily FeO, on the outside surfaces of the component, and extending into the surfaces of the interconnecting porosity.

steam treatment, n—see steam blackening.
stoking, n—see continuous sintering.

stripper punch, n—a punch that, in addition to forming the top or bottom of the die cavity, later moves further into the die to eject the compact.

subsieve fraction, n—the portion of powder passing through a 45-μm (no. 325) sieve.

superfines, n—the portion of a powder composed of particles that are smaller than a specified size, currently less than 10 μm.

surface-connected porosity, n—a network of interconnected pores or isolated pores that are connected to an exterior surface.

surface finger oxide, n—the oxide that follows prior particle boundaries into a part from the surface and cannot be removed by physical means, such as rotary tumbling.

sweating, n—see exudation.

tap density, n—the apparent density of the powder in a container that has been tapped under specified conditions.

transverse rupture strength, n—the stress calculated from the flexure formula, required to break a specimen supported near the ends as a simple beam; the load is applied midway between the center lines of the supports.

warm compaction, n—the consolidation of a pre-heated powder in a pre-heated die.

warm-die compaction, n—the consolidation of an unheated powder in a pre-heated die.

warpage, n—distortion that may occur in a compact during sintering.

withdrawal pressing, n—a powder consolidation method in which the die moves downward in relation to the lower punch(es) during compaction. It further descends over the fixed lower punch(es) for ejection, so that the compact may then be pushed off the tooling at this point.

APPENDIXES
(Nonmandatory Information)
X1. ACRONYMS USED IN THE PM INDUSTRY, LITERATURE, AND STANDARDS
X1.1 Subcommittee B09.01 has collected the following acronyms, typically used within the Powder Metallurgy Industry, and has chosen to include this table as an aid to those using this Terminology who may be unfamiliar with some of the PM acronyms currently in use.
## TABLE X1.1 PM Acronyms

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Meaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>A2LA</td>
<td>American Association of Laboratory Accreditation</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>BET</td>
<td>Brunauer, Emmett and Teller Theory</td>
</tr>
<tr>
<td>CAD</td>
<td>computer aided design</td>
</tr>
<tr>
<td>CAE</td>
<td>computer aided engineering</td>
</tr>
<tr>
<td>CAM</td>
<td>computer aided manufacturing</td>
</tr>
<tr>
<td>CIP</td>
<td>cold isostatic pressing</td>
</tr>
<tr>
<td>CNC</td>
<td>computer numerical control</td>
</tr>
<tr>
<td>CTE</td>
<td>coefficient of thermal expansion</td>
</tr>
<tr>
<td>CYS</td>
<td>compressive yield strength</td>
</tr>
<tr>
<td>EDM</td>
<td>electro-discharge machining</td>
</tr>
<tr>
<td>FEA</td>
<td>finite element analysis</td>
</tr>
<tr>
<td>FGM</td>
<td>functionally graded materials</td>
</tr>
<tr>
<td>HIP</td>
<td>hot isostatic pressing</td>
</tr>
<tr>
<td>HSS</td>
<td>high-speed steel</td>
</tr>
<tr>
<td>HT</td>
<td>heat treated</td>
</tr>
<tr>
<td>HTS</td>
<td>high temperature sintering</td>
</tr>
<tr>
<td>ICP</td>
<td>inductively coupled plasma (spectrometer)</td>
</tr>
<tr>
<td>IGA</td>
<td>inert gas analysis</td>
</tr>
<tr>
<td>IP</td>
<td>isostatic pressing</td>
</tr>
<tr>
<td>K</td>
<td>radial crushing strength</td>
</tr>
<tr>
<td>K(I)</td>
<td>fracture toughness (stress state defined by subscript)</td>
</tr>
<tr>
<td>LPS</td>
<td>liquid-phase sintering</td>
</tr>
<tr>
<td>MIM</td>
<td>metal injection molding</td>
</tr>
<tr>
<td>MMC</td>
<td>metal matrix composite</td>
</tr>
<tr>
<td>MPIF</td>
<td>Metal Powder Industries Federation</td>
</tr>
<tr>
<td>NDT</td>
<td>non-destructive testing, non-destructive techniques</td>
</tr>
<tr>
<td>PF</td>
<td>powder forging</td>
</tr>
<tr>
<td>PIM</td>
<td>powder injection molding</td>
</tr>
<tr>
<td>PM</td>
<td>powder metallurgy</td>
</tr>
<tr>
<td>PV</td>
<td>pressure/velocity factor</td>
</tr>
<tr>
<td>RBF</td>
<td>rotating beam fatigue</td>
</tr>
<tr>
<td>RBO</td>
<td>rapid burn off (sintering)</td>
</tr>
<tr>
<td>RCF</td>
<td>rolling contact fatigue</td>
</tr>
<tr>
<td>TRS</td>
<td>transverse rupture strength</td>
</tr>
<tr>
<td>tsi</td>
<td>tons/square inch (compacting)</td>
</tr>
<tr>
<td>UTS</td>
<td>ultimate tensile strength</td>
</tr>
<tr>
<td>YS</td>
<td>yield strength</td>
</tr>
</tbody>
</table>

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1. SCOPE

1.1 This standard describes a method for determining the tensile properties of powder metallurgy materials.

1.2 With the exception of density, for which g/cm$^3$ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Universal testing machine of suitable capacity to break the tensile specimens. The machine must meet the requirements of ASTM E8.

2.2 Gauge suitable for measuring the extension of the test specimen gauge length, typically a caliper accurate to 0.001 inch (0.025 mm).

2.3 Extensometer suitable for the test method selected. If left on through fracture, some extensometers may be damaged.

3. TEST SPECIMEN

3.1 Different test specimens are used depending on whether the material is in the as-sintered or a heat-treated condition; either quench-hardened and tempered or sinter-hardened and tempered.

As-Sintered Ferrous and Non-Ferrous Materials

3.2 The test specimens shall be pressed in dies with the dimensions of Fig. 1 (conventional flat test specimen) or Fig. 2 (modified flat test specimen) in accordance with MPIF Standard 60.

3.2.1 The modified flat test specimen (Fig. 2) is gripped on the 20° wedge surfaces.

NOTE 1—The gauge length and fillets of the conventional flat test specimen shall be as shown. The ends, as shown in Fig. 1, are designed to provide a total pressing area of 1.00 in$^2$ (645 mm$^2$). Other end designs are acceptable and may be necessary in order to provide adequate gripping load for high strength sintered materials. Some suggested alternative end designs include:

1. Longer ends of the same general shape and configuration as the conventional flat test specimen, but which provide more surface area for gripping, and which increase the total pressing area of the test specimen.

2. Shallow transverse grooves, or ridges, may be pressed in the ends to be gripped by the jaws. The gripping jaws should be machined to fit the contour providing some positive loading.

NOTE 2—Test specimens for as-sintered materials may also be machined from suitably sized, sintered compacts or PM parts. The dimensions of the machined specimen shall conform to those in Fig. 3. Stress relieve the test specimens at 350 °F (180 °C), or some other agreed upon temperature, for 1 h after machining, unless agreed otherwise.

Quench-Hardened and Tempered Ferrous Materials

3.3 The test specimens shall be machined from pressed and sintered compacts of sufficient size to give a bar that is 0.40–0.43 inch by 0.40–0.43 inch (10–11 mm by 10–11 mm) in cross-section and at least 3 inches (75 mm) long. The compacts shall be prepared in accordance with MPIF Standard 60.

NOTE 3—Test specimens cut from a heat-treated PM part should be stress relieved at 350 °F (180 °C), or some other agreed upon temperature, for 1 h unless agreed otherwise.

NOTE 4—Use of either of the flat bars in the heat-treated condition may result in lower values than can be obtained with the machined round test bar (Fig. 3).

3.3.1 Turn the bars to a 0.390 inch (9.90 mm) diameter on centers.

3.3.2 Austenitize at the appropriate temperature for the alloy and the carbon content of the material, and quench into an agreed, appropriate, agitated quenchant.

NOTE 5—Test specimens should be suspended vertically during quenching to minimize distortion.

3.3.3 Temper for 1 h at the temperature recommended for the material.

3.3.4 Machine to the final dimensions shown in Fig. 3, thread the ends for gripping, and polish the gauge section to a surface finish of 16 microinch (0.4 μm) Ra or better.
NOTE 6—Remove material at a rate of about 0.015 inch (0.375 mm) per pass until the test specimen is about 0.002 inch (0.05 mm) oversize. Then remove material at no more than 0.0002 inch (0.005 mm) per pass.

NOTE 7—The surface finish may be achieved by mechanically polishing in a longitudinal direction on all sides using successively # 240, # 400, # 500, and # 600 grades of emery paper.

3.3.5 Stress relieve the test bars at 350 °F (180 °C), or some other agreed upon temperature for 1 h, unless agreed otherwise.

Sinter-Hardened and Tempered Ferrous Materials

3.4 The test specimens shall be machined from pressed and pre-sintered compacts of sufficient size to give a bar that is 0.40–0.43 inch by 0.40–0.43 inch (10–11 mm by 10–11 mm) in cross-section and at least 3 inches (75 mm) long. The compacts shall be prepared in accordance with MPIF Standard 60.

3.4.1 Pre-sinter the compacts, at 1500–1600 °F (815–870 °C) in an inert or reducing gas atmosphere for 15–20 minutes.

3.4.2 Turn the bars to a 0.390 inch (9.90 mm) diameter on centers.

3.4.3 Sinter harden the machined, pre-sintered bars using the appropriate conditions for the material.

3.4.4 Temper for 1 h at the temperature recommended for the material.

3.4.5 Machine to the final dimensions shown in Fig. 3, thread the ends for gripping, and polish the gauge section to a surface finish of 16 microinch (0.4 μm) Ra or better.

NOTE 8—Remove material at a rate of about 0.015 inch (0.375 mm) per pass until the test specimen is about 0.002 inch (0.05 mm) oversize. Then remove material at no more than 0.0002 inch (0.005 mm) per pass.

NOTE 9—The surface finish may be achieved by mechanically polishing in a longitudinal direction on all sides using successively # 240, # 400, # 500 and # 600 grades of emery paper.

3.4.6 Stress relieve the test specimens at 350 °F (180 °C), or some other agreed upon temperature for 1 h, unless agreed otherwise.

4. PROCEDURE

4.1 The specimens shall be prepared in accordance with the appropriate procedure in Section 3.

4.2 In tensile testing, recognized standards of gripping, speed, and methods of determining tensile strength, elongation, and yield point such as described in ASTM E8 shall be used (See Note 4).

4.2.1 Elongation may be determined graphically as plastic strain at fracture. For this measurement, the extensometer must remain on the specimen until fracture. Plastic strain at failure is calculated by subtracting elastic strain from the total strain at failure as shown in Fig. 4.

NOTE 10—The result of graphical determination of elongation as plastic strain at failure is generally lower than that of the conventional gauge mark method. However, the latter is only accurate to ± 1% elongation and is not appropriate for low elongation materials. Choice of the gauge mark or graphical method should be made by agreement between the manufacturer and purchaser.

4.3 Test at least three (3) and preferably five (5) specimens that break within the gauge length.

5. REPORT

5.1 The following shall be reported:

5.1.1 Type of test specimen used (conventional flat test specimen or modified flat test specimen, or machined round test specimen).

5.1.2 Compaction method and processing sequence.

5.1.3 Ultimate tensile strength and yield strength at 0.2% offset as the average of at least three (3) but preferably five (5) tests to the nearest 1,000 psi (10 MPa).

5.1.4 Percent elongation in 1 inch (25 mm) to the nearest 1%, unless reporting plastic strain at fracture to the nearest 0.1%.

5.2 The following supplemental information may also be reported:

5.2.1 Chemical composition of the powder mix and alloying method used.

5.2.2 Type, brand, and percent of lubricant.

5.2.3 Density.

5.2.4 Apparent hardness.

5.2.5 Sintering temperature.

5.2.6 Sintering time.

5.2.7 Furnace atmosphere.

5.2.8 Final total carbon content, where applicable.

5.2.9 Other processing information, as required.

5.2.10 Any deviation from this test method.

6. PRECISION

6.1 As-Sintered Materials

6.1.1 The following precision data were developed by an interlaboratory study using the procedures contained in MPIF Standard 10. The results of the study are summarized in MPIF Research Report No. MPIF R-10/1-2014.

6.1.2 Flat, unmachined test specimens were compacted from the following materials:

   FC-0208 at a 6.9 g/cm³ density
   FY-4500 at a 7.0 g/cm³ density

Twelve different laboratories each received five (5) test specimens. They were asked to report the 0.2% offset yield strength and the ultimate tensile strength of the materials. The resulting data were evaluated using ASTM E691 to determine the repeatability limit, r, and reproducibility limit, R, of the data. The results of the analysis are shown in Table 1.
Table 1. Precision of Tensile Properties for As-sintered Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Property</th>
<th>Average Value (psi)</th>
<th>Repeatability r</th>
<th>Reproducibility R</th>
</tr>
</thead>
<tbody>
<tr>
<td>FC-0208</td>
<td>UTS</td>
<td>72,000</td>
<td>8,023</td>
<td>8,023</td>
</tr>
<tr>
<td></td>
<td>Yield</td>
<td>57,000</td>
<td>6,975</td>
<td>7,528</td>
</tr>
<tr>
<td>FY-4500</td>
<td>UTS</td>
<td>45,000</td>
<td>4,557</td>
<td>4,960</td>
</tr>
<tr>
<td></td>
<td>Yield</td>
<td>34,000</td>
<td>2,503</td>
<td>4,310</td>
</tr>
</tbody>
</table>

6.1.3 Duplicate results (individual determinations prior to averaging) from the same laboratory should be considered acceptable at the 95% confidence level unless they differ by more than r, the repeatability limit.

6.1.4 Duplicate results (individual determinations prior to averaging) from two different laboratories should be considered acceptable at the 95% confidence level unless they differ by more than R, the reproducibility limit.

6.2 Heat-Treated Materials

6.2.1 The following precision data were developed by an interlaboratory study using the procedures contained in MPIF Standard 10. The results of the study are summarized in MPIF Research Report No. MPIF R-10/2-2015.

6.2.2 Machined, round test specimens were prepared for the following materials:

- FC-0208 HT at a 6.9 g/cm³ green density
- FL-4405 HT at a 7.0 g/cm³ green density

Ten (10) different laboratories each received five (5) test specimens of each material/condition. They were asked to report the ultimate tensile strength of the materials. Some samples failed outside the gauge length so three (3) acceptable values were used for statistical evaluation. The resulting data were evaluated using ASTM E691 to determine the repeatability limit, r, and reproducibility limit, R, of the data. The results of the analysis are shown in Table 2.

Table 2. Precision of Tensile Properties for Heat-Treated Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Property</th>
<th>Average Value (psi)</th>
<th>Repeatability r</th>
<th>Reproducibility R</th>
</tr>
</thead>
<tbody>
<tr>
<td>FC-0208HT</td>
<td>UTS</td>
<td>140,326</td>
<td>13,522</td>
<td>18,367</td>
</tr>
<tr>
<td>FL-4405HT</td>
<td>UTS</td>
<td>156,278</td>
<td>9,829</td>
<td>11,109</td>
</tr>
</tbody>
</table>

6.2.3 Duplicate results (individual determinations prior to averaging) from the same laboratory should be considered acceptable at the 95% confidence level unless they differ by more than r, the repeatability limit.

6.2.4 Duplicate results (individual determinations prior to averaging) from two different laboratories should be considered acceptable at the 95% confidence level unless they differ by more than R, the reproducibility limit.

APPENDIX

A1. COMPARABLE STANDARDS

ASTM E8
ISO 2740

Disclaimer

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The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.
FIGURE 1: Conventional Flat Test Specimen
Dimensions shown in inches

Tolerances unless otherwise stated to be ± 0.001 inch (inch-pound)

Dimensions shown in millimetres

Tolerances unless otherwise stated to be ± 0.03 mm (SI)

FIGURE 1: Conventional Flat Test Specimen
Dimensions specified are those of the die cavity. (See NOTES 1 and 2) The tensile specimen design shown in Fig. 1 was developed by ASTM Committee B09 on Metal Powders and Metal Powder Products and is used herein with their permission.
FIGURE 2: Modified Flat Test Specimen
Dimensions specified are those of the die cavity. (See NOTE 2) The tensile specimen design shown in Fig. 2 was developed by Byron B. Beldon and is used herein with his permission.

Tolerances unless otherwise stated to be ± 0.001 inch (inch-pound)

Tolerances unless otherwise stated to be ± 0.03 mm (SI)
**FIGURE 3**: Machined Round Test Specimen  
(See NOTES 3 and 4)
NOTE: Elongation (EL) = EL_{plastic} = EL_{total} - \varepsilon_{elastic}

FIGURE 4: Example of Elongation Determined by the Extensometer Method
1. SCOPE

1.1 This method covers the apparatus, test specimens and procedure for determining the green strength of compacted powder metallurgy specimens by subjecting them to a uniformly increasing transverse loading under controlled conditions. The green strength, as used in this standard, is the stress, calculated from the flexure formula, required to break the powder metallurgy compact as a simple beam. The specimen is supported near the ends, and broken by applying the force midway between the fixed center of the supports.

1.2 With the exception of density, for which g/cm$^3$ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 The following apparatus is required to perform this operation:

2.1.1 Punches and die for producing a test specimen 0.50 by 1.25 inch (12.7 by 31.8 mm), nominal dimensions. An example of tooling is shown in Fig. 1.

2.1.2 Compression testing machine, capable of applying the required load with an accuracy of at least ± 1%.

2.1.3 Either of the following testing apparatus:

2.1.3.1 Transverse rupture test fixture, as shown in Fig. 2, for locating the test bar in a compression testing machine so that the breaking load can be measured to the nearest 0.1 lbf (0.4 N). The cross-head velocity of the compression testing machine should be capable of being set at approximately 0.1 inch/minute (2.5 mm/minute).

2.1.3.2 A balance, readable to 0.001 g with a capacity of at least 100 g.

2.1.3.3 A constant loading beam device, an example of which is shown in Fig. 3, capable of metering shot at a constant rate.

NOTE 1—Names of manufacturers of this apparatus are listed in General Information IV.

2.1.4 Micrometer or other suitable apparatus capable of measuring the green compacts to the nearest 0.001 inch (0.02 mm).

3. TEST SPECIMEN

3.1 The test portion used to compact the test specimens shall be obtained in accordance with MPIF Standard 01, Sampling Metal Powders.

3.2 The recommended green strength test specimen has the following nominal dimensions: 1.25 inch (31.8 mm) long by 0.50 inch (12.7 mm) wide by either 0.25 inch (6.4 mm) or 0.50 inch (12.7 mm) thick. The specimen may be compacted from either a non-lubricated or lubricated metal powder following the procedure in MPIF Standard 60.

3.3 The test specimens may be compacted to a predetermined green density or at a specified compacting pressure.

NOTE 2—A test specimen 30 mm long by 12 mm wide by 5 to 7 mm thick may be used. In this case, however, the Precision statement in this standard is not appropriate.

4. PROCEDURE

4.1 Compression testing machine.

4.1.1 Position the specimen in the transverse rupture test fixture (Fig. 2) so that it is centrally located and perpendicular to the supporting rods, with the top surface facing upward.

4.1.2 Place the loaded fixture between the platens of the compression testing machine and apply a load at a uniform rate such that the test specimen fractures within a time not less than 10 seconds. (Note: approximately 20 lbf/minute [89 N/minute] is suggested.)

4.1.3 Record the breaking load to the nearest 0.1 lbf (0.4 N). NOTE 3—Use of a scale calibrated to the correct load range and capable of being read to the nearest 0.1 lbf (0.4 N) is critical.
4.1.4 If the upper portion of the test fixture is not attached to the upper press platen, then the force in lbf (N), exerted by the mass of the upper portion of the test fixture, shall be determined and added to the read-out to determine the true breaking force.

4.2 Constant loading beam device

4.2.1 Using the level incorporated in the loading beam as a guide, adjust the balance of the loading beam so that it is horizontal. Position the specimen in the loading beam device (Fig. 3) so that it is centrally located and perpendicular to the supporting rods, with the top surface facing upward.

4.2.2 Allow the shot to fall into the collecting container at a uniform rate such that the test specimen fractures within a time not less than 10 seconds. (Note: a rate of approximately 2 lb/minute [1 kg/minute] is suggested for a typical loading beam device shown in Fig. 3 having A = 10 B.)

4.2.3 Weigh the collected shot. Record the mass to the nearest 0.01 lb (0.005 kg).

5. CALCULATIONS

5.1 Calculate the density of the test bar from its mass and volume. Calculate the arithmetical mean of the densities of the three different test pieces to the nearest 0.01 g/cm³.

5.2 Calculate the green strength as follows:

5.2.1 For compression testing machines, the green strength, S, is given in pounds per square inch (MPa) by the following formula:

\[ S = \frac{3PL}{2t^2w} \]

where:
- \( P \) = force is lbf (N) required to rupture
- \( L \) = length of specimen span of fixture in inches (mm)
- \( t \) = thickness of specimen in inches (mm), and
- \( w \) = width of specimen in inches (mm)

5.2.2 For constant loading beam device, use the same formula as in 5.2.1 except for the breaking load, \( P \), which is calculated as follows when using inch-pound units:

\[ P = \frac{AX}{B} \]

where:
- \( P \) = force on test specimen in lbf
- \( A \) = length A in inches; see Fig. 3
- \( B \) = length B in inches; see Fig. 3, and
- \( X \) = mass of shot to the nearest 0.01 lb required to rupture

5.2.2.1 When using metric units, calculate the breaking load \( P \), as follows:

\[ P = 9.8AX \]

\[ B \]

where:
- \( P \) = load on test specimen in N
- 9.8 = conversion factor 9.8 N = 1 kg
- \( A \) = length A in mm; see Fig. 3
- \( B \) = length B in mm; see Fig. 3, and
- \( X \) = mass of shot to the nearest 0.005 kg required to rupture

6. REPORT

6.1 Green strength in psi (MPa) as the arithmetical mean of the three determinations to the nearest 100 psi (0.5 MPa).

6.2 Nominal thickness of the test specimen.

6.3 Density of the test specimen.

6.4 Lubrication method used—die wall or admixed.

6.5 Lubricant type and percentage, if admixed.

6.6 Chemical composition of the powder mix if other than elemental powders are being tested.

6.7 Identification of the powder by brand, grade, and lot number.

6.8 The following supplementary information for clarification:

6.8.1 Brand of lubricant and lot number, if admixed.

7. PRECISION

7.1 The precision of this test has been determined from an interlaboratory study performed in 2005 in which 32 MPIF laboratories participated. ASTM E691 was used to perform a statistical analysis of the data at the 95% confidence level. The results are summarized in MPIF Research Report: MPIF R-15-05.

7.2 Repeatability limit values, \( r \), are listed in Table 1. On the basis of test error alone, duplicate results from the same laboratory (individual determinations prior to averaging) should be considered acceptable at the 95% confidence level unless they differ by more than \( r \), the repeatability limit.

<table>
<thead>
<tr>
<th>Table 1. Precision of Green Strength Measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Material</strong></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Water atomized prealloyed powder + 0.5% wax lubricant (green density = 6.8 g/cm³)</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Sponge iron + 0.5% wax lubricant (green density = 7.2 g/cm³)*</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

*Not a typical green density for this material.
7.3 Reproducibility limit values R, are listed in Table 1. On the basis of test error alone, test results from two laboratories (individual determinations prior to averaging) should be considered acceptable at the 95% confidence level unless they differ by more than R, the reproducibility limit.

Some of the participants in the interlaboratory study used a constant loading beam device (see Fig. 3). For the lower green strength material, the precision appears to be better when using the constant loading beam device – see Table 2.

#### Table 2. Precision of Green Strength Measurements on Lower Green Strength Material When Using a Constant Loading Beam Device

<table>
<thead>
<tr>
<th>Material</th>
<th>Thickness inch (mm)</th>
<th>Number of Labs</th>
<th>Mean r (psi)</th>
<th>R (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water atomized prealloyed powder + 0.5% wax / lubricant (green density = 6.8 g/cm³)</td>
<td>0.250 (6.35)</td>
<td>9</td>
<td>1274</td>
<td>113 201</td>
</tr>
<tr>
<td></td>
<td>0.500 (12.7)</td>
<td>9</td>
<td>1703</td>
<td>136 518</td>
</tr>
</tbody>
</table>

APPENDIX

A1. COMPARABLE STANDARDS

ASTM B312

ISO 3995

FIGURE 2: Transverse Rupture Test Fixture

Tolerance on all dimensions to be ± 0.001 inch (0.025 mm). Hardness on 0.125 inch ⌀ pins to be 60 HRC minimum

FIGURE 3: An Example of a Constant Loading Beam Device
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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
METAL POWDER INDUSTRIES FEDERATION

Method for
Determination of Apparent Density of Non-Free-Flowing Metal Powders Using the Carney Apparatus

MPIF Standard 28
Issued 1959

1. SCOPE

1.1 This standard describes a procedure for determining the apparent density of non-free-flowing metal powders. It is designed for those powders that do not flow freely through the Hall flowmeter funnel.

1.2 To determine the apparent density of free-flowing metal powder see MPIF Standard 04.

1.3 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Carney Funnel: A Carney funnel (Fig. 1) having an orifice of 0.20 inch (5 mm) in diameter.

2.2 Density Cup: A cylindrical cup (Fig. 2) having a capacity of 25 ± 0.03 cm³, as measured by MPIF Standard 53, with an inside diameter of 28 ± 0.5 mm.

2.3 Stand: A support (Fig. 3) to hold the flowmeter funnel concentric with the density cup so that the bottom of the orifice is approx. 1.0 inch (25 mm) above the top of the density cup when assembled (Fig. 4).

2.4 Workbench: A level and vibration-free surface to support the powder flowmeter assembly.

2.5 Balance: A balance readable to 0.001 g with a capacity of at least 200 g.

2.6 Wire: A wire not exceeding 0.10 inch (2.5 mm) in diameter by 6 inches (150 mm) long.

NOTE 1—Names of manufacturers of this apparatus are listed in General Information IV.

3. TEST PORTION

3.1 The test portion shall consist of a volume of approximately 30 to 40 cm³ of metal powder, obtained in accordance with MPIF Standard 01, Sampling Metal Powders.

4. PROCEDURE

4.1 Weigh the empty density cup and record the mass to 0.001 g. Alternatively, place the empty density cup on the balance and tare the balance to zero.

4.2 Load the test portion carefully into the flowmeter funnel and permit it to run into the density cup through the discharge orifice. If necessary, agitate or push the powder using the wire, but take care to prevent the wire from entering the density cup. Take care not to move the density cup during the filling operation.

4.3 When the powder completely fills and overflows the periphery of the density cup, rotate the funnel approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

4.4 Using a non-magnetic spatula with the blade held perpendicular to the top of the cup, level off the powder flush with the top of the density cup. Avoid jarring the apparatus at any time.

4.5 After the leveling operation, tap the density cup lightly on the side to settle the powder to avoid spilling during transfer. Wipe off any powder sticking to the outside wall of the cup.

4.6 Transfer the filled density cup to the balance and weigh it. Record the mass to 0.001 g.

4.7 If necessary, deduct the mass of the empty density cup to obtain the mass of powder in the cup, M.

4.8 More than one apparent density test may be run if desired. Use a fresh test portion of powder for each test. Average the apparent density values.

5. CALCULATION

5.1 Calculate the apparent density as follows:

\[ \rho_a = \frac{M}{V} \]

where:

- \( M \) = mass of powder in the density cup, g
- \( V \) = volume of the cup, cm³

NOTE 2—Use MPIF Standard 53 to determine the volume of the density cup.

6. REPORT

6.1 Apparent Density - Carney to the nearest 0.01 g/cm³.
7. PRECISION

7.1 The precision of this test method has not been determined by a statistically valid interlaboratory test as defined by ASTM E691. Results obtained (1991) by eight laboratories testing a sample of tin powder suggest the following:

7.1.1 Repeatability limit \( r = 1\% \) for tin powder. Duplicate analyses of a tin powder by the same operator and same apparatus should not differ by more than 1\% at the 95\% confidence level.

7.1.2 Reproducibility limit \( R = 4\% \) for tin powder. The difference between two single and independent results obtained by different laboratories on tin powder should not differ by more than 4\% at the 95\% confidence level.

APPENDIX

A1. COMPARABLE STANDARDS

<table>
<thead>
<tr>
<th>ASTM</th>
<th>ISO</th>
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<tbody>
<tr>
<td>B417</td>
<td>3923/1</td>
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</tbody>
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1. SCOPE

1.1 This standard defines and illustrates schematically (Figs. 1a and 1b) those segments of a metal powder compacting press and the tooling used therein that are directly involved in forming a powder metallurgy compact. Its purpose is to provide the producer and purchaser of such equipment with a uniform and universally acceptable glossary of terms.

1.2 Multiple upper and lower punches are common today. A scheme is presented for identifying tooling members in multiple upper and lower punch presses—see Figs. 2a, 2b and 2c.

Compacting Tool Set—the part or parts making up the confining form in which a powder is pressed. Parts of the tool set may be some or all of the following: die, punches, and core rods.

Core Rod—member of the tool set that forms a through hole in the compact.

Core Rod Adapter—the press member used to adapt the core rod to the core rod support.

Core Rod Support—the press member supporting the core rod. It may be movable or stationary.

Die—member of the tool set that forms the periphery of the part being produced.

Die Adapter—a sleeve or component that may be required to adapt the die to the die table bore.

Die Case—a member that retains or supports the die insert.

Die Insert—a member sometimes used inside of a die case, to prolong die life.

Die Set—that part of the press (sometimes removable) that holds and aligns the tool set in proper relation to the press.

Die Table—the member of the press or die set that holds the die.

Die Table Bore—the bore in the die table to fit the outside diameter of the die or its adapter.

Die Table Clearance Hole—the hole in the bore of the die table necessary to permit clearance for the lower punch when two counterbores are used.

Die Table Counterbore—the bore in the die table with tapped holes used for clamping the die in place.

Lower Punch—a member of the tool set that determines the powder fill and forms the bottom of the part being produced. Secondary lower punches may be necessary to facilitate filling, forming and ejecting of multiple level parts.

Lower Punch Adapter—a sleeve or component that may be required to adapt the lower punch to the lower punch holder.

Lower Punch Holder—that part of the press which is fastened to the lower ram.

Upper Punch—member of the tool set that closes the die and forms the top of the part being produced. Multiple punches can be used for multi-level parts.

Upper Punch Adapters—a sleeve or a member that may be required to adapt the upper punch to the upper punch holder.

Upper Punch Holder—that part of the die set which is fastened to the upper ram.

Zone and Tooling Position Designations—(starting from the inside working toward the outside—see Figs. 2a, 2b and 2c.).

Zone U is the designation for upper press levels.

Zone L is the designation for lower press levels.

Zone C is the designation for core rods.

Zone D is the designation for the die platens. (The D designation can be used to identify the use of “split dies”; D1 for half of the split die and D2 for the other half.)

1 is the inner most tooling member/core rod.

2 is the next outer tooling member/core rod.

3 is the next outer tooling member/core rod.

4 is the next outer tooling member/core rod.

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FIGURE 1a: Example Schematic of Compacting Press Tooling
FIGURE 1b: Example Schematic of Compacting Press Tooling for a Multi-level Part
(This figure was developed by Al Dornisch and is used herein with his permission.)
**FIGURE 2a:** Tooling and Position Designations

(Example: a set of tooling with a single upper punch, single lower punch and a core rod)
FIGURE 2b: Tooling and Position Designations
(Example: a set of tooling with two upper punches, three lower punches and a core rod)
Top view of possible core rod arrangement

C1 – Center Core Rod
C2 – Outer Core Rods within the same pitch circle diameter (PCD) closest to C1
C3 – Outer Core Rods within the same pitch circle diameter but with a larger PCD than C2 core rods

Lower case letters represent Core Rod positions:

a – located in quadrant 1, from 12 o’clock position up to but not including the 3 o’clock position
b – located in quadrant 2, from 3 o’clock position up to but not including the 6 o’clock position
c – located in quadrant 3, from 6 o’clock position up to but not including the 9 o’clock position
d – located in quadrant 4, from 9 o’clock position up to but not including the 12 o’clock position

Note: Similar terminology should be used for satellite punches if applicable. Example: L2a, L2b

FIGURE 2c: Top View of Possible Core Rod Arrangement
1. SCOPE

1.1 This standard describes methods for determining the average particle size of a sample of fine metal powders using air permeability of a packed powder bed. This method is not generally used for mixed powders. The values obtained may not be absolute, but are reproducible, and are highly useful on a relative basis for control purposes.

1.2 With the exception of the units for density and mass, for which g/cm³ and g are the industry standards; and the units for pressure, cm H₂O—long standing industry practice—values in SI units are to be regarded as standard.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. PRINCIPLE

2.1 These methods employ the air permeability method of measuring the average particle size of powders. It is based upon the principle that a current of air flows more readily through a bed of coarse powder than through an otherwise equal bed of fine powder. The pressure drop is related to the total surface area of the powder mass. A measurement is actually made of the envelope specific surface area of the powder, but the average particle size is calculated by the instrument software (Method 1) or read directly from the scale of the instrument (Method 2) in micrometres (µm).

3. APPARATUS

3.1 Micromeritics Sub-sieve AutoSizer (MIC SAS II)—Method 1—consisting of an air pump, a calibrated gas mass flow controller, a precision-bore sample tube, a sample tube retaining collar, a spacer tool, a gas flow metering valve, two precision pressure transducers (inlet and outlet), a stepper motor controlled ball screw-mounted piston, and computer hardware and software for instrument control and calculation and reporting of results. Included is accessory equipment consisting of a plug manipulator (extraction rod), two porous plugs, and a supply of paper disks.

NOTE 1—When homing the piston, adjust the sample packing assembly (1) as described in the manufacturer’s directions, with the plugs and paper disks stacked together and placed on the fixed anvil spigot, or (2) using a specially designed baseline (homing) gauge instead of the plugs and paper disks. This baseline gauge shall have a height of 20.30 ± 0.10 mm. Check all plug heights when new plugs are purchased and periodically thereafter to make sure all are equal in height.

3.1.1 Powder funnel—stainless steel, with spout outside diameter slightly smaller than the sample tube inside diameter.

3.1.2 The manufacturer provides instructions which should be followed, using the “Inorganics Test” procedure when testing metal powders. Particular attention should be given to proper maintenance of the instrument with special reference to the instructions on (1) “homing” the piston when turning on from an unpowered state, (2) setting the pressure and periodic checking of the pressure, (3) condition of O-rings on the piston and sample spigot, and (4) the sample packing assembly (plugs and paper disks).

3.2 Sub-sieve Sizer (Method 2): A Fisher Sub-sieve Sizer (FSSS) complete with air pump, air pressure regulating device, standardized double range air flowmeter, calculator chart and accessory equipment consisting of sample tube and porous brass plugs, plug manipulator, powder funnel, rubber tube support stand and filter paper disks.

3.2.1 Sub-sieve sizer calibrator: A jewel with a precision orifice mounted in a tube similar to the sample tube.

3.2.2 Torque Screwdriver: A calibrated screwdriver or torque wrench capable of applying a force of 222 N through the pinion knob in the instrument.

3.3 Operating Instructions: Manufacturer’s directions for the installation, operation, and maintenance of the instrument.

3.4 Balance: An analytical balance, readable to 0.01 g with a minimum capacity of 100 g.

NOTE 2—Name of the manufacturer is listed in General Information IV.
NOTE 3—The Fisher Sub-sieve Sizer (FSSS) is no longer commercially available, nor is it supported with parts and service. It is included here as apparatus for Method 2 because of several instruments still operating in the field. In-house repair or parts replacement is discouraged, as these are likely to affect results and precision detrimentally.

4. TEST PORTION

4.1 The test portion shall be a weighed amount of powder equal in grams to the published density of the material within ± 0.1 g.

4.2 The Micromeritics Sub-sieve AutoSizer (MIC SAS II) may be used for materials up to 75 μm in particle size.

4.3 The Fisher sub-sieve sizer (FSSS) is primarily useful for materials less than 50 μm in particle size. The determination should be made on the minus 325 mesh fraction from the screen analysis of the material. If a minimum of 95% of the material is minus 325 mesh, the total sample may be used, but this shall be reported.

5. PROCEDURE

5.1 METHOD 1—Micromeritics Sub-sieve AutoSizer (MIC SAS II)—0.2 to 75 μm

5.1.1 Standardize and check the instrument as per Appendix A1.1 and Special Precautions therein before measuring the average particle size of a material.

5.1.2 Press the “Inorganics” button.

5.1.3 Determine the mass of the sample to the nearest 0.1 g.

5.1.4 Select the test parameters: 3 compressions; slow decompression; slow termination.

5.1.5 Press the “Run Test” button and enter the Sample Details, including the true density of the material and the actual mass of the sample used.

5.1.6 Lay a paper disk over one end of the sample tube using one of the porous plugs with the perforated surface of the plug against the surface of the paper disk. This crimps the paper around the edges and the paper precedes the plug into the sample tube. Push the plug into the tube until it is even with the end of the sample tube. Place the sample tube in a vertical position in a support with the paper side of the plug up.

5.1.7 With the aid of the powder funnel, completely transfer the sample into the sample tube by tapping the side of the tube and funnel. Lay a second paper disk over the top of the sample tube. Place the perforated surface of a porous brass plug on top of the paper disk and force the plug and paper disk down into the sample tube until the plug is just inside the sample tube.

5.1.8 Push the sample tube retaining collar onto the sample tube.

5.1.9 Push the sample tube onto the fixed anvil spigot with the retaining collar below the sample tube holder, centered in the sample tube holder and leaving enough of a gap at the bottom of the sample tube to fit the SAS spacer tool below the sample tube.

NOTE 4—The sample tube may eventually wear and cause faulty values. When this condition is suspected, replace the tube. Sample tubes with obvious wear or scratches, or both, should be discarded.

5.1.10 Insert the SAS spacer tool into the gap below the sample tube.

5.1.11 Using an Allen key or cam lock device, lock the sample tube retaining collar into position just below the sample tube holder arms.

5.1.12 Press the “Next” button and the test will automatically run.

5.1.13 Monitor the test and remove the spacer (washer) after the first compression.

Warning—The piston moves slowly but with considerable force. Keep all body parts clear of the mechanism while in motion. Do not operate with any guards removed.

NOTE 5—The sample tube must be held off the spigot to ensure that the full force is applied to the sample and not dissipated through the spigot.

5.1.14 When the test is finished, the results will be displayed on the instrument’s screen. Record the Porosity, (Average) Particle Size, and Specific Surface Area (SSA). The data will automatically be saved with the file name indicated during entry of the sample details.

5.1.15 Test each specimen in duplicate using a freshly prepared bed of the material for each determination.

5.1.16 For later data extraction, refer to the manufacturer’s instructions.

5.2 METHOD 2—Fisher Sub-sieve Sizer (FSSS)—0.2 to 50 μm

5.2.1 Standardize and check the instrument as per Appendix A1.2 and Special Precautions therein before measuring the average particle size of a material.

5.2.2 Set the range control indicator to the proper range of the instrument. The low range is used if it is suspected that the average particle size is between 0.2 and 20.0 μm. The high range is used for materials from 20.0 to 50.0 μm.

5.2.3 The liquid level of the liquid meniscus in the manometer tube located over the calculator chart should be made to coincide exactly with the upper edge of the curved portion of the metal cross bar attached to the rack when the tip of the pointer coincides exactly with the base line on the calculator chart. This adjustment is made by turning the manometer control knob, and only when no sample tube is in the instrument. After using the instrument, this adjustment should not be attempted until the water has had sufficient time to drain down the walls of the tube.

5.2.4 Screw one of the porous plugs into the plug manipulator. Lay a paper disc over one end of the sample tube and push the plug into the sample tube, forcing the paper to crimp around the edges of the porous plug. Push the plug into the sample tube approximately 10 mm and then unscrew the plug.
manipulator. Place the sample tube in a vertical position in the rubber stand with the paper side of the plug up.

5.2.5 Using the glass powder funnel, completely transfer the weighed sample into the sample tube, tapping the side of the tube to settle the powder. Lay a second paper disk over the top of the sample tube and force the other plug and paper disk into the sample tube. Place the filled sample tube on the brass post beneath the rack and pinion with the lower plug in contact with the upper end of the brass post.

5.2.6 Lower the rack, guiding it until the flat bottom end comes in contact with the upper plug. Pack the sample by turning down the pinion knob with a torque screwdriver. Use a compressive force limited to a maximum of 222 N or that force necessary to achieve a porosity value of 0.400, whichever is smaller. The sample tube must not be permitted to contact the block holding the brass post. When using a force of 222 N apply and release this force three times. Make certain the rack does not move upward after final release of pressure.

NOTE 6—Alternative methods of packing the sample may be mutually agreed upon by the parties involved.

5.2.7 Shift the calculator chart laterally until the extreme tip of the pointer just coincides with the sample height curve on the chart. The chart must not be moved after this setting until the determination is finished. Record the porosity.

5.2.8 Without disturbing the sample in any way, mount the sample tube between the rubber cushioned supports just to the right of the brass post. Clamp the upper cap down onto the sample tube until an airtight seal is obtained at both ends.

5.2.9 Turn the electrical switch at the lower right hand corner of the front panel to “on.” If necessary adjust the pressure control knob, located near the bubpler observation window at the lower left of the panel, until the bubbles rise in the standpipe at the rate of 2 to 3 bubbles per second. This will cause the water lines to rise above the calibration mark on the upper end of the standpipe. This is normal and does not mean the calibration is in error.

5.2.10 The liquid level in the manometer tube will rise slowly and reach a maximum within 30 seconds to several minutes. At the end of this period, using care not to disturb the chart, turn the rack up until the upper edge of the cross bar coincides with the liquid meniscus in the manometer. The particle size is indicated by the location of the tip of the pointer with relation to the curves on the calculator chart.

5.2.11 Test each specimen in duplicate using a freshly prepared bed of the material for each determination.

### 6. REPORT

6.1 Report whether Method 1 (Micrometrics Sub-sieve AutoSizer) or Method 2 (Fisher Sub-sieve Sizer) was used.

6.2 If the two values agree within 3 percent, report the particle size as the average of the two determinations. Report the average porosity of the packed sample along with the average particle size. If the values do not agree within 3 percent, review the equipment and procedure and repeat the test.

6.3 The recommended reporting practice is listed in Table 1.

<table>
<thead>
<tr>
<th>Table 1. Recommended Reporting Practice</th>
</tr>
</thead>
<tbody>
<tr>
<td>Range</td>
</tr>
<tr>
<td>-------------------</td>
</tr>
<tr>
<td>0.20 to &lt; 4.0 μm</td>
</tr>
<tr>
<td>4.0 to &lt; 8.0 μm</td>
</tr>
<tr>
<td>8.0 to &lt; 15.0 μm</td>
</tr>
<tr>
<td>15.0 to &lt; 20.0 μm</td>
</tr>
<tr>
<td>20.0 to &lt; 50.0 μm</td>
</tr>
</tbody>
</table>

### 7. PRECISION AND BIAS

7.1 Precision—The precision of this test method has not been determined by a statistically valid interlaboratory test because of the limited number of participating laboratories (less than six).

7.2 Method 1 (Micrometrics Sub-sieve AutoSizer):

7.2.1 The within-laboratory repeatability standard deviation, based on repetitive testing of a single sample in the same laboratory, has been determined to be: 0.013 μm at an average particle size of 1.08 μm; 0.021 μm at an average particle size of 2.75 μm; and 0.042 μm at an average particle size of 4.02 μm.

7.2.2 The between-laboratory reproducibility of Method 1 is being determined.

7.3 Method 2 (Fisher Sub-sieve Sizer):

7.3.1 The following precision data were developed by ASTM Committee B09 on Metal Powders and Metal Powder Products using the procedures contained in ASTM Practice E691 and while not in conformance with E691 (six laboratories are required), the user of this test method may infer its precision from the interlaboratory study.

7.3.2 The results of the interlaboratory study to determine the precision of this test method are available from ASTM in Research Report No. B09-1010—a report done in five laboratories on tungsten carbide powders in both the as-supplied and laboratory-milled conditions.

7.3.3 The within-laboratory repeatability limit, r, was estimated to be 2 to 6% of the measured Fisher Number. Duplicate results from the same laboratory should not be considered suspect unless they differ by more than r.
7.3.4 The between-laboratory reproducibility limit, \( R \), was found to be estimated by the following equation:

\[
R = 0.173F - 0.042
\]

where:

- \( R \) = the reproducibility limit and
- \( F \) = the measured Fisher Number

Results from two different laboratories should be not considered suspect unless they differ by more than \( R \).

7.2 Bias—The average particle size is a calculated estimate of average particle diameter in a powder. No absolute method of determining powder particle size exists, nor are there any universally recognized standard or reference powders for this measurement; therefore it is not possible to discuss the bias of results by this test method.

NOTE: Precision data were developed by ASTM Committee B09 on Metal Powders and Metal Powder Products and are reprinted, with permission, from ASTM B330-12, Standard Test Method for Estimating Average Particle Size of Metal Powders and Related Compounds Using Air Permeability, copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428

APPENDIX

A1. STANDARDIZATION OF APPARATUS

A1.1 METHOD 1—Micromeritics Sub-sieve AutoSizer (MIC SAS II)

A1.1.1 Before proceeding with standardization of the Micromeritics Sub-sieve AutoSizer (MIC SAS II), the following items shall be checked:

A1.1.1.1 The sample tube and plugs shall not be worn to the point where results are affected.

A1.1.1.2 Inspect the O-ring seals for tears and abrasion marks. The O-ring seals shall not be worn to the point where the sample tube moves easily by hand or the pressure reading varies as the sample tube is moved.

A1.1.1.3 The drying agent shall be in proper condition.

A1.1.1.4 Whenever the instrument is turned on from an unpowered state, the piston shall be “homed” according to the manufacturer’s instructions. See NOTE 1 above.

A1.1.1.5 Before running the initial sample, the pressure shall be set to 50.0 (+0.1, -0.5) cm H\(_2\)O, using the metering valve; then checked and reset if necessary every few hours, or if the ambient temperature changes more than ± 2 °C.

NOTE 7—The metering valve position should not be adjusted for repeat runs of the same sample as this will likely lead to a loss of precision even if the inlet pressure reading has drifted a little outside the 50.0 (+0.1, -0.5) cm H\(_2\)O range. Further adjustment is not necessary as the pressure is controlled precisely during the particle size measurement.

A1.1.1.6 Standardization is recommended before and after any series of determinations or at least every 4 hours of continued operation. Warm-up of the instrument is required if it has been off for more than 30 minutes.

A1.1.17 Calibration of the pressure transducers is recommended every 3-6 months, using a traceable external pressure gauge per the manufacturer’s instructions.

A1.2 METHOD 2—Fisher Sub-Sieve Sizer (FSSS)

A1.2.1 The standardization of the Fisher Sub-sieve Sizer shall be made using the Fisher jewel orifice as the primary standard. Standardization should be made at both ranges of the machine.

NOTE 8—Because only one flowmeter is used for the low (0.2 to 20.0 μm) range while both flowmeters are used for the high (20.0 to 50.0 μm) range, the low range should be standardized first. After the low range is standardized, the high range is then standardized, making adjustments only to the one Flowmeter opened up by the range-control knob.

NOTE 9—Care should be taken to keep the calibrator tube clean as any slight obstruction can cause a significant shift in values. Periodic microscopic examination of the actual orifice is necessary.

A1.2.2 With the sub-sieve sizer properly adjusted and set to the proper range, proceed as follows:

A1.2.2.1 Mount the Fisher jewel orifice tube between the rubber cushioned supports just to the right of the brass post. Clamp the upper cap down onto the tube so that an airtight seal is obtained at both ends.

A1.2.2.2 Adjust the calculator chart so that the porosity reading corresponds to the value indicated on the tube.

A1.2.2.3 Proceed with the standardization as described in sections 5.9 and 5.10 of the test procedure.

A1.2.2.4 The value obtained in this manner shall correspond to the particle size indicated on the jewel orifice tube within ± 3 percent.

A1.2.2.5 If the average particle size value as indicated on the chart does not correspond to ± 3 percent of the particle size value indicated on the jewel orifice tube, calibrate the sub-sieve sizer as follows: Remove the rear panel and loosen the set screw securing the needle valve or tungsten wire (depending on the model of sub-sieve sizer used) in the flowmeter and adjust as required to bring the average particle size indicated on the chart to the value indicated on the jewel orifice tube. Check adjustment by repeating test.

A1.2.2.6 Standardization with the Fisher jewel orifice is required before and after any series of determinations and at least every four hours of continued operation.

A2. SPECIAL PRECAUTIONS

A2.1 The following items shall be checked at regular intervals per the Fisher operating instructions:

A2.1.1 The water level in the pressure regulator standpipe shall be maintained.

A2.1.2 The sample packing assembly shall be adjusted for wear. This is best done as described in the manufacturer’s directions with the exception that the plugs and paper disks are not inserted in the sample tube but are merely stacked together and placed between the brass support and the “flat” of the bottom of the rack.
A2.1.3 The chart shall be properly aligned horizontally with the indicator pointer.
A2.1.4 The rack and pinion shall be properly aligned vertically with the chart.
A2.1.5 The sample tube or plugs shall not be worn to the point where they affect results. (The sample tube and plugs will eventually wear and be the cause of faulty values.) When this condition is suspected, a spare unused tube and plugs shall be substituted and a determination repeated on a sample of known particle size.
A2.1.6 The manometer and air resistors shall be free of dirt.
A2.1.7 The rubber sample tube cushions shall not be worn to the point where leakage occurs.
A2.1.8 The drying agent shall be in proper condition.
A2.1.9 All particle size determinations shall be made within ± 3 °C of the temperature, measured within the cabinet, at which the standardizations were made.
A2.1.10 The torque screwdriver should be standardized at least once every month.

A3. REFERENCE MATERIAL
Where a vendor and customer wish to use the result of this test as a specified property it is helpful to split and share a properly mixed sample of a stable metal powder, with an average particle size near the specified value, to serve as a reference material.

A4. COMPARABLE STANDARDS
ASTM B330
ASTM E2980
MIL-STD-1233

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By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability.

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The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.

This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-8692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This standard describes recommended test methods for evaluating the sintered properties of bronze filter powders. It includes methods for the determination of dimensional change, air permeability, maximum pore diameter and strength of standard flat sintered porous metallic bronze filter specimens.

1.2 The supplier of filter powder and the parts fabricator shall agree to the test methods used and should report the supplementary information listed in Section 5.6.

1.3 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Stainless steel molds that have been oxidized, or graphite molds, are used to contain the loose filled bronze filter powder during sintering. Dimensions are shown in Fig. 1.

2.2 Sintering furnace, preferably of muffle design, capable of maintaining temperatures as high as 1700 °F (925 °C). It must have a non-oxidizing, controlled atmosphere in both the heating and cooling chambers.

2.3 Micrometer capable of measuring the specimen to the nearest 0.001 inch (0.02 mm).

2.4 Test fixture to hold the sintered filter for the air permeability and maximum pore diameter measurements. Dimensions are shown in Fig. 2. The exposed area of test filter is 1 square inch (645 mm²).

2.5 Isopropyl alcohol to wet the test filter for the determination of maximum pore size.

2.6 Flowmeter capable of measuring air flow up to 1 ft³/min (500 cm³/s or 30 L/min).

2.7 Manometer, standard open end type, capable of measuring pressure up to 50 inches (1.5 m) of water.

NOTE 1—Other manometers may be used as necessary depending on the anticipated pressure drop.

2.8 Test panel including a flowmeter, manometers, air filter, pressure regulator, pressure gauge, valves, piping and the test fixture of Fig. 2 connected in a manner shown schematically in Fig. 3.

2.9 Test fixture to hold the sintered filter during the break strength test. Dimensions are shown in Fig. 4.

2.10 Compressive testing machine with a capacity of 1,000 lbf (4,500 N) capable of measuring the strength of the test filter to the nearest 2 lbf (9 N).

3. PROCEDURE

3.1 Determination of radial shrinkage. Use this procedure to determine the percent change in radial dimension (shrinkage) from mold size upon sintering loose bronze filter powder in comparison with that of a control reference.

3.1.1 Pour the bronze filter powder to be tested into at least two reference molds (Fig. 1). Tap the molds lightly and uniformly on their sides to settle the loose powder. Carefully remove excess powder with a straight edge drawn across the top of the molds.

3.1.2 Follow the procedure in 3.1.1 to determine the radial shrinkage of a mutually accepted control reference bronze filter powder.

3.1.3 Place the four or more filled molds on a graphite plate, equally spaced in a symmetrical configuration with the test filter molds and the reference powder molds alternating in such a manner that they will receive the same amount of heat from the furnace.

3.1.4 Allow the sintering furnace to soak at the appropriate temperature for the grade of material being tested for a minimum of one hour.

3.1.5 Push the filled molds on the graphite plate directly into the center of the hot zone for a time sufficient to achieve proper sintering for the grade being tested. (Exact time and temperature will vary from furnace to furnace. Both concerned parties shall agree on the conditions that are to be used.) After the agreed upon time at temperature, push the molds on the graphite plate into the cooling zone, where they should remain under protective atmosphere until they reach room temperature.
FIGURE 1: Material stainless steel or graphite. Stamp I.D. dimension on bottom of mold. Grind and polish upper surface, oxidize stainless steel before using.

3.1.6 Remove all the molds from the furnace, strip the sintered filters from the molds, and label them. Measure the diameters of the filters made from the reference powder at circumferential points 45° apart (four measurements per filter) and average the measured values.

3.1.7 Calculate the percent shrinkage from the size of the mold for the control reference filters.

3.1.8 If the control reference filters have shrinkages in a range acceptable to both concerned parties, repeat step 3.1.7 for the test filters and calculate the percent shrinkage.

3.1.9 If the control reference filters have shrinkages outside the agreed upon allowable range, reset the furnace temperature to adjust the shrinkage (usually higher temperature to achieve greater shrinkage) then repeat steps 3.1.1 through 3.1.8 or 3.1.9 until the control reference filters have the appropriate shrinkage.

3.2 Determination of air permeability. Use this procedure to measure the air permeability of the test filters prepared using procedure 3.1. The permeability of a sintered filter is defined as the resistance or backpressure measured when air at a specified flow rate passes through the filter. The air permeability test fixture is illustrated in Fig. 2, and Fig. 3 is a schematic of the air permeability test assembly.

3.2.1 Use the test fixture illustrated in Fig. 2 to clamp each test filter tightly between the two rubber gaskets by screwing down the upper part of the fixture snugly by hand. This exposes 1 in² (645 mm²) of filter surface to the airflow.

3.2.2 Gradually increase the airflow through the test filter to determine if the backpressure will exceed the capability of the water manometer. If it does not, hold the flow rate at 1 ft³/minute (30 L/minute) and read the backpressure off the water manometer to the nearest 0.1 inch (2 mm) of water.

3.2.3 For finer grades of filter powder that have low permeability and create back pressures so high that the water would be blown out of the manometer an alternative manometer should be chosen.

3.2.4 Repeat steps 3.2.1 and 3.2.2 or 3.2.3 for each test.
filter and calculate the average backpressure measured to the nearest 0.1 inch (2 mm).

3.3 **Determination of maximum pore diameter.** Use this procedure to measure the maximum pore diameter of the test filter prepared using the procedure in section 3.1. A schematic of the assembly for maximum pore diameter measurement is provided in Fig. 3. The maximum pore diameter of sintered bronze filters is defined as the maximum pore opening measured as a backpressure in the bubble point test.

3.3.1 Immerse all the test filters in isopropyl alcohol for a minimum of two minutes.

3.3.2 Clamp an alcohol soaked filter tightly, with the smoother side up, between the two rubber gaskets of the test fixture illustrated in Fig. 2, by screwing down the upper part of the fixture snugly by hand. (The side of the filter that contacted the mold is smoother.)

3.3.3 Apply a small amount of air pressure to the test filter through needle valve B (needle valve A closed), to prevent alcohol from draining through the filter, and pour 2 mL of alcohol onto the test filter.

3.3.4 Increase the air pressure very slowly by gradually opening needle valve B. Watch the surface of the filter and record the backpressure on the water manometer when the first true air bubble appears. (Disregard air leaks from under the gasket.)

3.3.5 Repeat steps 3.3.2 through 3.3.4 for all test filters and average the results as inches of water to the nearest 0.1 inch (2 mm).

3.4 **Determination of breaking strength.** Use this procedure to measure the maximum force necessary to drive a 0.5 inch diameter pin through the test filter when mounted in the breaking fixture illustrated in Fig. 4. (The measured force lbf is a relative value having no direct relationship to other more conventional strength-of-materials tests.)

3.4.1 Place the sintered test filter in the breaking fixture as illustrated in Fig. 4, resting on the shoulder with the pin inserted into the top hole and contacting the filter.

3.4.2 Place the fixture in a compression testing machine and apply a force at a loading rate of 200 lbf/minute (900 N/minute).

3.4.3 Record the maximum load attained prior to fracture to the nearest 2 lbf (9 N).

3.4.4 Repeat steps 3.4.1 through 3.4.3 for each test filter and compute the average force to the nearest 2 lbf (9 N).

4. **CALCULATION**

4.1 Calculate the shrinkage from mold size for both the control standards and test filters as follows:

\[
\text{Shrinkage, } \% = \left(\frac{A - B}{A}\right) \times 100
\]

where:
A = mold I.D. (see 2.1 and Fig. 1.)
B = sintered filter specimen O.D. (see 3.1.6)

NOTE 2—This formula expresses the shrinkage as a percentage. Deletion of the factor, 100, gives the shrinkage in inch/inch (mm/mm).

5. **REPORT**

5.1 The average shrinkage from mold size of the test filter to the nearest 0.1%.

5.2 The average shrinkage from mold size of the control reference sintered simultaneously to the nearest 0.1%. (The control reference shall be identified.)

5.3 The average air permeability of the test filters as measured to the nearest 0.1 inch (2 mm) of water back pressure.

5.4 The average maximum pore diameter of the test filters as measured to the nearest 0.1 inch (2 mm) of water back pressure needed to form the first true air bubble.

5.5 The average maximum load prior to breaking of the test filters as measured to the nearest 2 lbf (9 N).

5.6 The following supplementary information should be reported for clarification:

5.6.1 Furnace temperature.
5.6.2 Time in the hot zone.
5.6.3 Type of furnace atmosphere.
5.6.4 Details of any occurrence that may have affected the results.
A1. COMPARABLE STANDARDS
ISO 4003
ISO 4022

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1. **SCOPE**

1.1 This standard covers the apparatus, test specimens, and procedures for the impact testing of PM materials.

1.2 The two methods to be included in this standard are the unnotched Charpy impact test and Izod impact test.

1.3 The values stated in inch-pound units are to be regarded as the standard. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. **APPARATUS**

2.1 The following apparatus is required.

2.1.1 The single-blow Charpy impact test shall be carried out in a pendulum-type impact machine with a capacity of at least 110 ft-lbf (150 J). This is the same apparatus as used for the simple-beam test for steel described in ASTM E23. Standard machines may be modified by using a more sensitive scale, or pendulum machines of a lower capacity where values of less than 25 ft-lbf (34 J) will be obtained.

2.1.2 The single-blow Izod impact test shall be carried out in a 110 or 120 ft-lbf (150 or 163 J) pendulum-type impact machine with a striking height of 0.866 inch (22 mm). This apparatus is the same as used in the cantilever-beam test for steel described in ASTM E23.

2.2 Gauge(s) capable of measuring the specimen to the tolerances listed in Figs. 1 and 2, to the nearest 0.001 inch (0.02 mm).

3. **TEST SPECIMEN**

3.1 The test specimens are compacted in suitable tooling capable of producing the respective specimen geometries (see Figs. 1 and 2).

3.2 The Charpy impact test specimen shall have the shape and dimensions as shown in Fig. 1.

3.3 The Izod impact test specimen shall have the shape and dimensions as shown in Fig. 2.

**NOTE 2**—The longer Izod impact test specimen (Fig. 2) may be used for the Charpy impact test.

4. **PROCEDURE**

4.1 The specimen shall be prepared in accordance with MPIF Standard 60.

4.2 The specimen shall be tested in accordance with customary procedures as set forth in ASTM E23. Unless otherwise specified, the test shall be performed at room temperature. Any absorbed energy values above 80% of the scale range of the test apparatus are inaccurate and shall be reported as approximate. The specimen shall be struck perpendicular to the direction of pressing.

**FIGURE 1:** Simple-Beam Charpy Impact Test Specimen  
Note: Permissible variations shall be as follows:  
Adjacent sides shall be at 90 deg. ± 10 min.  
Cross section dimensions ± 0.005 inch (0.125 mm)  
Length dimension ± 0.100 inch (+0.25 mm)

**FIGURE 2:** Cantilever-Beam Izod Impact Test Specimen (optional simple beam)  
Note: Permissible variations shall be as follows:  
Adjacent sides shall be 90 deg. ± 10 min.  
Cross section dimensions ± 0.005 inch (0.125 mm)  
Length dimension +0, -0.100 inch (+0.25 mm)

5. **REPORT**

5.1 Impact energy as the average of a minimum of three tests to the nearest 1 ft-lbf (1 Joule), specifying Charpy or Izod impact test.

5.2 The temperature of the test, e.g., room temperature.

5.3 The following supplementary information may be reported for clarification:

5.3.1 Chemical composition of the powder mix and alloying method used.
5.3.2 Type, brand, and percent of lubricant.
5.3.3 Density of the test specimen.
5.3.4 Apparent hardness.
5.3.5 Sintering temperature.
5.3.6 Sintering time.
5.3.7 Furnace atmosphere.
5.3.8 Final total carbon content, where applicable.
5.3.9 Other processing information, as required.
5.3.10 Any deviation from this test method.

### 6. PRECISION

6.1 The precision of this test has been determined from an interlaboratory study performed by the MPIF Standards Committee in 2010. Two materials were tested at room temperature: FC-0208 at a sintered density of 6.9 g/cm³, and SS-316L at a sintered density of 7.0 g/cm³. Each participant tested three (3) specimens of each material. ASTM Practice E691 was used to perform a statistical analysis of the data at the 95% confidence level. The results are summarized below.

<table>
<thead>
<tr>
<th>Table 1: Precision of Impact Energy Measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>FC-0208</td>
</tr>
<tr>
<td>Mean Impact Energy (ft-lbf)</td>
</tr>
<tr>
<td>r</td>
</tr>
<tr>
<td>R</td>
</tr>
<tr>
<td>Number of laboratories</td>
</tr>
</tbody>
</table>

6.2 Repeatability limits, r, are listed in Table 1. On the basis of test error alone, duplicate tests results (individual) determinations prior to averaging) in the same laboratory by the same operator on the same material should not differ by more than the value listed.

6.3 Reproducibility limits, R, are listed in Table 1. For tests conducted in two laboratories, and on the basis of test error alone, the test results (individual) determinations prior to averaging) should not differ by more than the value listed.

### Disclaimer

By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability.

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The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.
1. SCOPE
1.1 This standard covers the apparatus, test specimen and procedures for determining the bending strength or transverse rupture strength of powder metallurgy materials.
1.2 The test as described and specified herein is applicable only to materials of negligible ductility. If applied to materials showing noticeable plastic deformation (exceeds 0.02 inch [0.5 mm]) permanent deflection, the maximum load may not be equal to the break load, which could lead to misinterpretation of results. For such materials the tensile strength test should be used.
1.3 With the exception of density, for which g/cm$^3$ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM SI 10. They may be approximate and are only for information.
1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS
2.1 Gauges capable of measuring from 0 to 1,000 inch (0 to 25 mm) to the nearest 0.001 inch (0.02 mm).
2.2 A transverse rupture fixture as shown in Fig. 1.

NOTE 1—Names of manufacturers of this apparatus are listed in General Information IV.
2.3 A universal testing machine readable to within 0.1% of full-scale reading. Use the lowest testing range that can provide a measurable result.

3. TEST SPECIMEN
3.1 The test specimen shall be compacted in accordance with MPIF Standard 60 and shall have the dimensions shown in Fig. 2.

4. PROCEDURE
4.1 Measure the width and thickness of the test specimens to the nearest 0.001 inch (0.02 mm).
4.2 Locate the test specimen centrally in the transverse rupture test fixture (Fig. 1) so that the faces formed by the compacting punch faces are horizontal and one is in contact with the two, hardened supporting rods (steel or carbide, hardened to a 60 HRC minimum).
4.3 Apply a compressive force at the center of the test specimen by means of a hardened rod until rupture occurs. The direction of the applied force shall be parallel to the pressing direction of the test specimen and the no-load crosshead speed shall be no greater than 0.2 inch per minute (5 mm per minute).
4.4 Record the load at which the test specimen breaks (force required to rupture the specimen, $P$).

5. CALCULATION
5.1 Calculate the transverse rupture strength as follows:

$$TRS = \frac{3 \times P \times L}{2 \times t \times w}$$

where:
- $TRS$ = transverse rupture strength in psi or MPa
- $P$ = break load lbf (N) (as determined)
- $L$ = the distance between the supporting rods 1.000 inch (25.40 mm)
- $t$ = the thickness of the test specimen in inches (mm)
- $w$ = the width of the test specimen in inches (mm)

6. REPORT
6.1 Report the transverse rupture strength as the average of at least three (3) and preferably five (5) tests to the nearest 1,000 psi (10 MPa).
6.2 The following supplementary information may also be reported:
- 6.2.1 Chemical composition of the powder mix.
- 6.2.2 Type, brand, and percent of lubricant.
- 6.2.3 Density.
- 6.2.4 Apparent hardness.
- 6.2.5 Sintering temperature.
- 6.2.6 Sintering time.
- 6.2.7 Furnace atmosphere.
- 6.2.8 Final total carbon content where applicable.
- 6.2.9 Other processing information as required.
- 6.2.10 Any deviation from this test method.
7. PRECISION

7.1 The repeatability limit, r, and reproducibility limit, R, measurements were determined (1995) according to ASTM E691, Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods and are listed below for six materials and a range of transverse rupture strength values. On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory will be expected to exceed, r, only 5% of the time. If such a difference is found to be larger than, r, there is reason to question one or both results. Similarly, the difference in two test results obtained in different laboratories will be expected to exceed, R, only 5% of the time. If the difference is found to be larger than, R, there is reason to question one or both measurements.

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>DENSITY g/cm³</th>
<th>APPARENT HARDNESS HRB</th>
<th>TRS PSI</th>
<th># LABS</th>
<th># r psi</th>
<th># R psi</th>
</tr>
</thead>
<tbody>
<tr>
<td>F-0005-15</td>
<td>6.22</td>
<td>37 HRB</td>
<td>63,000</td>
<td>19</td>
<td>4,800</td>
<td>7,900</td>
</tr>
<tr>
<td>FC-0205-30</td>
<td>6.21</td>
<td>43 HRB</td>
<td>66,500</td>
<td>16</td>
<td>5,600</td>
<td>11,500</td>
</tr>
<tr>
<td>F-0005-25</td>
<td>6.91</td>
<td>40 HRB</td>
<td>71,000</td>
<td>12</td>
<td>5,500</td>
<td>14,000</td>
</tr>
<tr>
<td>FN-0208-35</td>
<td>6.95</td>
<td>77 HRB</td>
<td>123,500</td>
<td>19</td>
<td>6,900</td>
<td>11,600</td>
</tr>
<tr>
<td>FL4405-40</td>
<td>7.04</td>
<td>75 HRB</td>
<td>129,500</td>
<td>16</td>
<td>7,000</td>
<td>13,800</td>
</tr>
<tr>
<td>FC-0208-50</td>
<td>6.69</td>
<td>81 HRB</td>
<td>144,000</td>
<td>12</td>
<td>12,500</td>
<td>21,000</td>
</tr>
<tr>
<td>FC-0208-65HT</td>
<td>6.43</td>
<td>31 HRB</td>
<td>138,500</td>
<td>22</td>
<td>15,400</td>
<td>28,000</td>
</tr>
<tr>
<td>F-0008-65HT</td>
<td>6.68</td>
<td>35 HRB</td>
<td>148,500</td>
<td>16</td>
<td>22,000</td>
<td>31,700</td>
</tr>
<tr>
<td>FLC-4608-70HT</td>
<td>6.69</td>
<td>31 HRB</td>
<td>174,000</td>
<td>14</td>
<td>28,800</td>
<td>41,500</td>
</tr>
<tr>
<td>FN-0205-105HT</td>
<td>6.93</td>
<td>31 HRB</td>
<td>191,500</td>
<td>14</td>
<td>23,700</td>
<td>40,500</td>
</tr>
<tr>
<td>FC-0205-90HT</td>
<td>7.03</td>
<td>41 HRB</td>
<td>236,000</td>
<td>16</td>
<td>28,100</td>
<td>42,400</td>
</tr>
<tr>
<td>FLN4205-140HT</td>
<td>7.12</td>
<td>41 HRB</td>
<td>251,500</td>
<td>22</td>
<td>32,700</td>
<td>46,900</td>
</tr>
<tr>
<td>FLN24405-160HT</td>
<td>7.04</td>
<td>38 HRB</td>
<td>246,000</td>
<td>35</td>
<td>21,500</td>
<td>31,200</td>
</tr>
</tbody>
</table>

The r and R values were determined from the testing of three (3) transverse rupture test specimens by each participating laboratory.

APPENDIX

A1. COMPARABLE STANDARDS.
ASTM B528
ISO 3325
Method for
Determination of Density of Compacted or Sintered Powder Metallurgy (PM) Products

MPIF Standard 42
(Formerly included in Standards 08, 13, 16 and 35)

1. SCOPE

1.1 This standard describes a method using Archimedes’ principle for measuring the density of powder metallurgy products having surface-connected porosity. It is applicable to green, sintered and oil-impregnated specimens. The density of PM products is expressed in grams per cubic centimetre.

1.1.1 For a test method to determine the density of impermeable materials, see MPIF Standard 54.

1.2 Green Density, \( \rho_s \), is the mass per unit volume of an unsintered PM part or test specimen. Normally the green density is determined on PM parts for quality control tests and for determining the compressibility of powders and mixes.

1.3 Sintered Density, \( \rho_i \), is the mass per unit volume of a sintered, non-oil-impregnated PM part. This term is synonymous with dry density.

1.4 Oil-impregnated Density, \( \rho_o \), is the mass per unit volume of a sintered PM part impregnated with oil or other nonmetallic lubricant. This term is synonymous with wet density. Normally the density of sintered bearings is reported this way since they are supplied and used oil-impregnated.

1.5 With the exception of density, for which g/cm\(^3\) is the industry standard, SI units are used in this standard. Values in inch-pound units are shown in parentheses and result from conversions in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.6 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Analytical Balance - A precision analytical balance that will permit readings within 0.01% of the test specimen mass as shown in Table 1.

2.2 Container: A beaker or vessel, suitable for holding water, of sufficient capacity to immerse specimens of various sizes.

2.3 Test Specimen Support for Weighing in Water: Two typical arrangements are shown in Fig. 1. The suspension wire may be twisted around the test specimen or the test specimen may be supported in a wire basket.

For either arrangement, a single corrosion resistant wire, e.g. austenitic stainless steel, copper, or nichrome should be used for the basket and suspension wire. The recommended diameter of suspension wire to be used for various mass ranges is shown in Table 2.

Table 1. Balance Requirements

<table>
<thead>
<tr>
<th>Specimen Mass (g)</th>
<th>Balance Readability (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;10</td>
<td>0.0001</td>
</tr>
<tr>
<td>10 to &lt;100</td>
<td>0.001</td>
</tr>
<tr>
<td>100 to &lt;1,000</td>
<td>0.01</td>
</tr>
<tr>
<td>1,000 to &lt;10,000</td>
<td>0.1</td>
</tr>
</tbody>
</table>

Table 2. Wire Diameter Recommendations

<table>
<thead>
<tr>
<th>Specimen Mass</th>
<th>Wire Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 50 grams</td>
<td>0.12 mm [48 gauge (0.005 inch)]</td>
</tr>
<tr>
<td>50 to &lt; 200 grams</td>
<td>0.25 mm [35 gauge (0.010 inch)]</td>
</tr>
<tr>
<td>200 to &lt; 600 grams</td>
<td>0.40 mm [28 gauge (0.015 inch)]</td>
</tr>
<tr>
<td>600 grams &amp; above</td>
<td>0.50 mm [25 gauge (0.020 inch)]</td>
</tr>
</tbody>
</table>

2.4 Water: Distilled or deionized and preferably degassed water to which 0.05 to 0.1 volume % of a wetting agent has been added. The values, \( \rho_w \), shown in Table 3 should be used for the density of distilled water.

2.5 Thermometer: To determine water temperature to the nearest 1 °C (2 °F).

2.6 Impregnating oil with a kinematic viscosity of 65 cSt maximum (300 SUS) at 38 °C (100 °F). In the case of oil impregnated PM bearings or parts, match the oil that was used to impregnate the part originally.

3. TEST SPECIMEN

3.1 The test specimen shall be a PM part or a group of parts or a section of a part with a minimum mass of 1 gram.

4. DETERMINATION OF GREEN DENSITY

4.1 This procedure is used to determine the green density of unsintered specimens. In order to determine accurately the volume of the test piece by water displacement in conducting this test the specimens are oil impregnated or the pores are filled with a suitable material. The density determined is an average of the metal and any solid lubricant originally present.
Table 3. Effect of Temperature on Water Density

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>ρ_w g/cm³</th>
<th>Temperature °F</th>
<th>ρ_w g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>0.9991</td>
<td>60</td>
<td>0.9990</td>
</tr>
<tr>
<td>15.5</td>
<td>0.9990</td>
<td>61</td>
<td>0.9989</td>
</tr>
<tr>
<td>16</td>
<td>0.9989</td>
<td>62</td>
<td>0.9988</td>
</tr>
<tr>
<td>16.5</td>
<td>0.9988</td>
<td>63</td>
<td>0.9987</td>
</tr>
<tr>
<td>17</td>
<td>0.9988</td>
<td>64</td>
<td>0.9986</td>
</tr>
<tr>
<td>17.5</td>
<td>0.9987</td>
<td>65</td>
<td>0.9985</td>
</tr>
<tr>
<td>18</td>
<td>0.9986</td>
<td>66</td>
<td>0.9984</td>
</tr>
<tr>
<td>18.5</td>
<td>0.9985</td>
<td>67</td>
<td>0.9983</td>
</tr>
<tr>
<td>19</td>
<td>0.9984</td>
<td>68</td>
<td>0.9982</td>
</tr>
<tr>
<td>19.5</td>
<td>0.9983</td>
<td>69</td>
<td>0.9981</td>
</tr>
<tr>
<td>20</td>
<td>0.9982</td>
<td>70</td>
<td>0.9980</td>
</tr>
<tr>
<td>20.5</td>
<td>0.9981</td>
<td>71</td>
<td>0.9978</td>
</tr>
<tr>
<td>21</td>
<td>0.9980</td>
<td>72</td>
<td>0.9977</td>
</tr>
<tr>
<td>21.5</td>
<td>0.9979</td>
<td>73</td>
<td>0.9975</td>
</tr>
<tr>
<td>22</td>
<td>0.9978</td>
<td>74</td>
<td>0.9974</td>
</tr>
<tr>
<td>22.5</td>
<td>0.9976</td>
<td>75</td>
<td>0.9973</td>
</tr>
<tr>
<td>23</td>
<td>0.9975</td>
<td>76</td>
<td>0.9972</td>
</tr>
<tr>
<td>23.5</td>
<td>0.9974</td>
<td>77</td>
<td>0.9970</td>
</tr>
<tr>
<td>24</td>
<td>0.9973</td>
<td>78</td>
<td>0.9969</td>
</tr>
<tr>
<td>24.5</td>
<td>0.9972</td>
<td>79</td>
<td>0.9967</td>
</tr>
<tr>
<td>25</td>
<td>0.9970</td>
<td>80</td>
<td>0.9966</td>
</tr>
<tr>
<td>25.5</td>
<td>0.9969</td>
<td>81</td>
<td>0.9964</td>
</tr>
<tr>
<td>26</td>
<td>0.9968</td>
<td>82</td>
<td>0.9963</td>
</tr>
<tr>
<td>26.5</td>
<td>0.9966</td>
<td>83</td>
<td>0.9961</td>
</tr>
<tr>
<td>27</td>
<td>0.9965</td>
<td>84</td>
<td>0.9959</td>
</tr>
<tr>
<td>27.5</td>
<td>0.9964</td>
<td>85</td>
<td>0.9958</td>
</tr>
<tr>
<td>28</td>
<td>0.9962</td>
<td>86</td>
<td>0.9956</td>
</tr>
<tr>
<td>28.5</td>
<td>0.9961</td>
<td>87</td>
<td>0.9954</td>
</tr>
<tr>
<td>29</td>
<td>0.9959</td>
<td>88</td>
<td>0.9952</td>
</tr>
<tr>
<td>29.5</td>
<td>0.9958</td>
<td>*Interpolated from °C data</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>0.9956</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The values of ρ_w shown are taken from, “Metrological Handbook 145, Quality Assurance for Measurements”, 1990, NIST, pp. 9,10, and represent the values in air at one atmosphere pressure.

4.2 Determine the mass of the green test specimen in grams as indicated in Table 1. This is mass A.

4.3 Oil impregnate the part by either of the following procedures:

4.3.1 Preferred Procedure: Immerse the specimen in the impregnating oil at room temperature. Reduce the pressure over the specimen to 7 kPa (1 psi) or less for 30 minutes, then increase the pressure back to atmospheric pressure for a period of at least 30 minutes keeping the specimen immersed during the entire period.

4.3.2 Alternative Procedure: Immerse the specimen, for at least 4 hours, in the impregnating oil that has been heated to a temperature of 82 °± 5 °C (180 °± 10 °F). Cool by immersing in oil held at room temperature for at least 10 minutes.

4.4 Remove excess surface oil by wiping gently with an absorbent, lintless medium. Take care not to extract the oil absorbed within the part. Determine the mass of the oil impregnated test specimen in grams to the accuracy stated in Table 1. This is mass B.

4.5 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2a. Take care to ensure that the bridge does not restrict free movement of the balance pan. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, shield the weighing system, including the wire, from the effect of air drafts.

4.6 Suspend the test specimen support with the test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least 6 mm (0.25 inch) to minimize the effect of surface tension forces on the weighing. Take care to ensure that the test specimen and specimen support hang freely from the balance beam hook, are free of air bubbles when immersed in the water and are at the same temperature as the water and balance. Take care to ensure that the surface of the water is free of dust particles.

4.7 Weigh the test specimen and the specimen support immersed in water. This is mass C.

4.8 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before. This is mass E. Take care to ensure that the suspension support is free of air bubbles and that the suspension wire is not immersed below its normal hanging depth as a change in depth will change the measured mass.

4.9 Measure the temperature of the water to the nearest 1 °C (2 °F) and record its density ρ_w, at that temperature, from Table 3.

4.10 Calculate the green density from the following formula:

\[
\text{Green Density, } \rho_g = \frac{A \rho_w}{B - (C - E)}
\]

5. DETERMINATION OF SINTERED DENSITY

5.1 This procedure is used to determine the sintered density of PM components. This section covers sintered, pre-sintered, heat treated, and steam treated PM materials. In order to determine accurately the volume of the test piece by water displacement, the specimens are oil impregnated or the surface-connected pores are filled with a suitable material.

5.2 Determine the mass of the sintered test piece in grams as indicated in Table 1. This is mass A.

NOTE 1—Oil impregnated specimens are to be free of lubricant for determining mass A. Remove the oil in a Soxhlet apparatus using a suitable solvent, such as petroleum ether. After extraction, residual solvent shall be removed by heating specimens at 120 °C (250 °F) for 1 hour. Alternate extraction and drying shall be continued until the dry mass, A, is constant to 0.05%.

NOTE 2—A practical and fast method of oil removal for most materials is to heat the specimen in a protective
atmosphere in the temperature range of 425 to 870 °C (800 to 1,600 °F). Exercise care in the selection of temperature to avoid exceeding the melting point of any material tested. This method is applicable to sintered aluminum materials if the temperature does not exceed 540 °C (1,000 °F).

5.3 Oil impregnate the part by either of the following procedures:

5.3.1 Preferred Procedure: Immerse the specimen in the impregnating oil at room temperature. Reduce the pressure over the sample to 7 kPa (1 psi) or less for 30 minutes, then increase the pressure back to atmospheric pressure for a period of at least 30 minutes keeping the specimen immersed during the entire period.

5.3.2 Alternative Procedure: Immerse the specimen, for at least 4 hours, in the impregnating oil that has been heated to a temperature of 82 ° ± 5 °C (180 ° ± 10 °F). Cool by immersing in oil held at room temperature for at least 10 minutes.

5.4 Remove excessive oil by wiping gently with an absorbent, lintless medium. Take care not to extract oil absorbed within the part. Determine the mass of the oil impregnated test specimen in grams as indicated in Table 1. This is mass B.

5.5 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2a. Take care to ensure that the bridge does not restrict free movement of the balance pan. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, shield the suspension wire between the container of water and the bottom of the balance from the effect of air drafts.

5.6 Suspend the test specimen support with the test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least 6 mm (0.25 inch) to minimize the effect of surface tension forces on the weighing. Take care to ensure that the test specimen and specimen support hang freely from the balance beam hook, are free of air bubbles when immersed in the water and are at the same temperature as the water and balance. Take care to ensure that the surface of the water is free of dust particles.

5.7 Weigh the test specimen and the specimen support immersed in water. This is mass C.

5.8 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before. This is mass E. Take care to ensure that the suspension support is free of air bubbles and that the suspension wire is not immersed below its normal hanging depth as a change in depth will change the measured mass.

5.9 Measure the temperature of the water to the nearest 1 °C (2 °F) and record its density ρw, at that temperature, from Table 3.

5.10 Calculate the sintered density from the following formula:

\[
\text{Sintered Density, } (\rho_s) = \frac{A\rho_w}{B - (C - E)}
\]

### 6. DETERMINATION OF OIL-IMPREGNATED DENSITY

6.1 This procedure is used to determine the oil-impregnated density of PM bearings or parts. The density determined is the average of the solid material and the oil present in the voids.

NOTE 3—Ensure that the specimen is fully oil impregnated.

6.2 Oil impregnate the part by either of the following procedures:

6.2.1 Preferred Procedure: Immerse the specimen in the impregnating oil at room temperature. Reduce the pressure over the sample to 7 kPa (1 psi) or less for 30 minutes, then increase the pressure back to atmospheric pressure for a period of at least 30 minutes keeping the specimen immersed during the entire period. If the oil that had been used originally to impregnate the part had a viscosity higher than 65 cSt (300 SUS) then the oil shall be heated to a temperature of 82 ° ± 5 °C (180 ° ± 10 °F).

6.2.2 Alternative Procedure: Immerse the specimen, for at least 4 hours, in the impregnating oil that has been heated to a temperature of 82 ° ± 5 °C (180 ° ± 10 °F). Cool by immersing in oil held at room temperature for at least 10 minutes.

6.3 Remove excess surface oil by wiping gently with an absorbent, lintless medium. Take care not to extract the oil absorbed within the part.

6.4 Determine the mass of the oil impregnated test piece in grams as indicated in Table 1. This is mass B.

6.5 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2a. Take care to ensure that the bridge does not restrict free movement of the balance pan. The container of water also may be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, shield the weighing system, including the wire, from the effect of air drafts.

6.6 Suspend the test specimen support with the test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least 6 mm (0.25 inch) to minimize the effect of surface tension forces on the weighing. Take care to ensure that the test specimen and specimen support hang freely from the balance beam hook, are free of air bubbles when immersed in the water and are at the same temperature as the water and balance. Take care to ensure that the surface of the water is free of dust particles.

6.7 Weigh the test specimen and the specimen support immersed in water. This is mass C.

6.8 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before.
This is mass E. Take care to ensure that the suspension support is free of air bubbles and that the suspension wire is not immersed below its normal hanging depth as a change in depth will change the measured mass.

6.9 Measure the temperature of the water to the nearest 1 °C (2 °F) and record its density, \( \rho_w \), at that temperature from Table 3.

6.10 Calculate the oil-impregnated density from the following formula:

\[
\text{Oil – impregnated Density, } (\rho)_i = \frac{BP_w}{B - (C - E)}
\]

7. REPORT

7.1 Density as calculated from the appropriate formula to the nearest 0.01 g/cm³.

8. PRECISION

8.1 The repeatability limit, \( r \), and reproducibility limit, \( R \), were determined in accordance with ASTM E691, Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods and are listed below for ten materials and a range of density values. On the basis of test error alone, the difference in absolute value of two test results (individual determinations prior to averaging) obtained in the same laboratory will be expected to exceed \( r \) only 5% of the time. If such a difference is found to be larger than \( r \) there is reason to question one or both results. Similarly, the difference in two test results (individual determinations prior to averaging) obtained in different laboratories will be expected to exceed \( R \) only 5% of the time. If the difference is found to be greater than \( R \) there is reason to question one or both measurements.

<table>
<thead>
<tr>
<th>Table 4. Precision of Sintered (Dry) Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material</td>
</tr>
<tr>
<td>----------</td>
</tr>
<tr>
<td>FC-0205</td>
</tr>
<tr>
<td>F-0005</td>
</tr>
<tr>
<td>FN-0208</td>
</tr>
<tr>
<td>FL-4405</td>
</tr>
<tr>
<td>FC-0208</td>
</tr>
<tr>
<td>F-0008</td>
</tr>
<tr>
<td>FD-0205</td>
</tr>
<tr>
<td>FLN4-4408</td>
</tr>
<tr>
<td>FC-0205</td>
</tr>
<tr>
<td>FLN-4205</td>
</tr>
</tbody>
</table>

Notes—\( S \)=atomized powder; \( S \)=sponge powder; \( HT \)=heat treated
The \( r \) and \( R \) values were determined from the testing of three transverse rupture test specimens by each participating laboratory.

APPENDIX

A1. COMPARABLE STANDARDS

ASTM B962
ISO 2738

Disclaimer

By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability. MPIF standards are adopted in the public interest and are designed to eliminate misunderstandings between the producer and the purchaser and to assist the purchaser in selecting and obtaining the proper material for his particular product. Existence of an MPIF standard does not in any respect preclude any MPIF member or non-member from manufacturing or selling products not included in this standard or from utilizing procedures or equipment other than those included in this standard.

The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428; IEEE, 345 East 47th Street, New York, NY 10017; USA.

FIGURE 1: Methods for Holding the Test Specimen When Weighing in Water

FIGURE 2: Methods for Weighing in Water

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This standard discusses various testing procedures for determining the Rockwell apparent hardness (microindentation hardness) of powder metallurgy products at all stages of manufacturing, from green compact to fully hardened condition. It does not include testing procedures for PF (forged) products or cemented carbides or sintered friction materials.

1.2 Other hardness tests such as HV5 or HV10 and HB may be used if agreed by the parties concerned. The Vickers method, unlike Rockwell hardness testing, requires the samples to be ground and polished prior to the hardness determination. The Rockwell tests do not require this, which makes them the preferred method for testing PM products. Brinell hardness testing is rarely used by the PM industry in the United States.

1.3 Hardness tests may be used as a qualification procedure for determining the quality of PM parts.

1.4 Determination of microindentation hardness and case depth shall be performed according to MPIF Standards 51 and 52 respectively.

1.5 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Hardness Testers (mounted on a vibration-free surface).

2.1.1 Rockwell—A, B*, C, D, E, F, G, H. *The type of ball used for the Rockwell B test shall be identified when reporting the results, e.g., 72 HRBS designates a steel ball while 97 HRBW designates a tungsten carbide ball.

2.1.2 Rockwell Superficial—15T, 15N, 30N, etc.

2.2 Indenter (suitable for the scale specified)

2.3 Fixtures (for supporting the part properly)

3. TEST SPECIMEN

3.1 The specimen shall be thick enough so that markings do not appear on the surface opposite that of indentation. See ASTM E18.

3.2 The test surfaces of the specimen shall be parallel to the support since taper and curvature may give false readings. For cylindrical surfaces, see ASTM E18 for correction factors.

3.3 The surface to be tested shall be large enough so that each indentation is at least 3 diameters from any edge or previous indentation.

3.4 Test specimens shall have burr-free anvil support surfaces. This may require filing or grinding the test specimen so as to prevent projecting burrs or previous impressions from affecting results. Emery paper of 180-240 grit is acceptable for grinding.

4. PROCEDURE

4.1 Test the PM part or PM test specimen in accordance with the standard procedure for the method prescribed: ASTM E18 for Rockwell hardness.

4.2 Select the appropriate Rockwell scale for the apparent hardness of the material and the proper indenter and load for the scale used.

4.3 Test heat-treated ferrous PM parts or PM test specimens using the Rockwell C or A scales.

NOTE 1—An apparent hardness measured as HRA cannot be directly converted to HRC and vice versa.

4.4 Ensure that the anvil on which the PM part or PM test specimen rests is clean and smooth. Use the smallest diameter anvil that is large enough to prevent rocking.

4.5 Special fixtures may be required to support the PM part or test specimen properly.

4.6 Check indenters visually for deformation, chips, flatness, or any other anomaly.

4.7 Verify the hardness tester against a standard test block to ensure calibration; refer to ASTM E18.

4.8 For reporting the apparent hardness of a PM part or PM test specimen it is recommended that the average be calculated from a minimum of at least five (5) usable readings.

4.9 The producer and purchaser should agree on the hardness procedure, and the location on the PM part or PM test specimen for the test, for each product to be tested.

5. REPORT

5.1 The average of at least five (5) usable apparent hardness values to the nearest whole number in the scale mutually agreed between purchaser and producer.

5.2 Rockwell values in accordance with the current international system, e.g., 35 HRC.

5.3 The number of indentations and location for each part and the number of parts.
5.4 The density of the PM part or specimen being tested
   to the nearest 0.01 g/cm³.
5.5 The following supplementary information may also
   be reported for clarification:
5.5.1 Chemical composition of the powder mix and
   alloying method used.
5.5.2 Type, brand, and percent of lubricant.
5.5.3 Sintering temperature.
5.5.4 Sintering time.
5.5.5 Furnace atmosphere.
5.5.6 Final total carbon content, where applicable.
5.5.7 Other processing information, as required.
5.5.8 Any deviation from this test method.

### Table 1. Precision of Rockwell Apparent Hardness Readings on a Single PM Part

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>DENSITY (g/cm³)</th>
<th>NO. OF LABS</th>
<th>HARDNESS (AVERAGE)</th>
<th>REPEATABILITY (r)</th>
<th>REPRODUCIBILITY (R)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CZP-2002</td>
<td>7.92</td>
<td>9</td>
<td>82 HRH</td>
<td>1.7</td>
<td>0.8</td>
</tr>
<tr>
<td>F-0000</td>
<td>6.74</td>
<td>9</td>
<td>63 HRF</td>
<td>4.0</td>
<td>1.8</td>
</tr>
<tr>
<td>FC-0208</td>
<td>6.63</td>
<td>9</td>
<td>71 HRB</td>
<td>4.5</td>
<td>2.0</td>
</tr>
<tr>
<td>FX-2008</td>
<td>7.45</td>
<td>9</td>
<td>86 HRB</td>
<td>4.3</td>
<td>1.9</td>
</tr>
<tr>
<td>FL-4605-HT*</td>
<td>6.90</td>
<td>8</td>
<td>107 HRB</td>
<td>1.9</td>
<td>0.8</td>
</tr>
<tr>
<td>FL-4605-HT</td>
<td>6.90</td>
<td>8</td>
<td>35 HRC</td>
<td>2.2</td>
<td>1.0</td>
</tr>
<tr>
<td>FC-0208-HT</td>
<td>6.29</td>
<td>10</td>
<td>97 HRC</td>
<td>3.1</td>
<td>1.4</td>
</tr>
<tr>
<td>FC-0208-HT</td>
<td>6.29</td>
<td>10</td>
<td>19 HRC</td>
<td>4.2</td>
<td>1.9</td>
</tr>
<tr>
<td>FN-0208-HT*</td>
<td>6.89</td>
<td>10</td>
<td>105 HRC</td>
<td>2.9</td>
<td>1.3</td>
</tr>
<tr>
<td>FN-0208-HT</td>
<td>6.89</td>
<td>10</td>
<td>30 HRC</td>
<td>3.9</td>
<td>1.7</td>
</tr>
</tbody>
</table>

The repeatability limit, r, and reproducibility limit, R, readings were determined according to ASTM E691, Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods. The reproducibility limit readings, R, predict how closely one laboratory will approximate another performing Rockwell hardness testing on the same PM test specimen. If the laboratories tested two different specimens, even if they were from the same lot, then larger differences between the two specimens would be expected.

*The r and R values for readings greater than 100 HRB reflect the decreasing sensitivity of the Rockwell B scale.

### Table 2. Precision of Rockwell Apparent Hardness

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>DENSITY # LABS HARDNESS (r) (R)</th>
</tr>
</thead>
<tbody>
<tr>
<td>F-0005-15</td>
<td>6.22 24 37 HRB 2.4 9.9</td>
</tr>
<tr>
<td>F-0005-25</td>
<td>6.91 15 40 HRB 2.5 4.9</td>
</tr>
<tr>
<td>FC-0205-30</td>
<td>6.21 19 43 HRB 3.6 8.2</td>
</tr>
<tr>
<td>FL-4405-40</td>
<td>7.04 19 75 HRB 1.6 4.9</td>
</tr>
<tr>
<td>FN-0208-35</td>
<td>6.95 24 77 HRB 1.5 4.7</td>
</tr>
<tr>
<td>FC-0208-50</td>
<td>6.69 15 81 HRB 2.1 4.2</td>
</tr>
<tr>
<td>FC-0208-65HT</td>
<td>6.43 24 31 HRC 3.6 6.9</td>
</tr>
<tr>
<td>FLC-4608-70HT</td>
<td>6.69 17 31 HRC 2.1 4.3</td>
</tr>
<tr>
<td>FN-0205-105HT</td>
<td>6.93 18 31 HRC 2.1 4.0</td>
</tr>
<tr>
<td>F-0008-65HT</td>
<td>6.68 18 35 HRC 1.7 3.2</td>
</tr>
<tr>
<td>FC-0205-90HT</td>
<td>7.03 18 41 HRC 1.9 3.0</td>
</tr>
<tr>
<td>FLN-4205-140HT</td>
<td>7.12 24 41 HRC 1.5 6.2</td>
</tr>
</tbody>
</table>

Repeatability and reproducibility limit values reported from three (3) different transverse rupture test specimens, average of five (5) readings per specimen.

---

**Note:** The precision of HRB values were determined prior to the requirement for reporting the use of a steel ball versus the tungsten carbide ball and therefore are listed in Tables 1 and 2 as "HRB" only.

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1. SCOPE

1.1 This standard describes a method for determining the change in dimensions of a powder metallurgy specimen after sintering. The dimensional change is the difference, typically expressed as a percentage, between a dimension of the sintered specimen and the corresponding die dimension.

NOTE 1—The general procedure of measuring the dimensions of the compacting die or a PM compact or test piece, before and after a processing step and calculating a percent dimensional change, may be adapted for use as an internal process evaluation test to quantify green expansion, dimensional change from the size of the green part, repressing size change, heat treatment changes, or other changes in dimensions that result from a manufacturing operation.

1.2 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Compacting tool set such as that shown in Fig. 1 designed to produce the rectangular test specimen.

NOTE 2—The names of manufacturers of this apparatus are listed in General Information IV.

2.2 Universal testing machine or PM compacting press, suitable for holding the tooling and having sufficient force to apply the pressure necessary to compact the test specimen to the target green density.

2.3 Inside and outside micrometer calipers, or other suitable measuring apparatus of the size necessary to measure the long dimension of the die cavity and the sintered test specimen to the nearest 0.0001 inch (0.002 mm).

2.4 A balance readable to 0.001 g, with a minimum capacity of 200 g.

2.5 A laboratory or production sintering furnace capable of heating the specimens to a specified temperature, holding them at temperature and cooling to room temperature in a controlled atmosphere or vacuum.

3. TEST SPECIMEN

3.1 A rectangular compact having dimensions of 0.500 inch (12.70 mm) wide by 1.250 inch (31.75 mm) long by 0.250 inch (6.35 mm) thick.

3.2 The top and bottom faces of the green compact shall be parallel within 0.001 inch (0.02 mm).

3.3 The green density shall be within ± 0.02 g/cm³ of the target density.

NOTE 3—Alternative test specimens may be used to measure PM dimensional change such as the cylindrical compact used to determine compressibility, radial crushing test specimens, or the ring test specimen used for measuring magnetic properties.

4. PROCEDURE

4.1 Preliminary

4.1.1 When this test method is used to approve a shipment of metal powder, all the testing conditions, including the value of the target green density and the performance criterion of the reference lot, shall be agreed by the producer and purchaser.

4.1.2 Using the nominal dimensions of the test specimen, calculate and note the mass of powder that will be needed to produce one test specimen to the target density using the following equation:

\[ \rho_g = \frac{(0.0610) M}{w \times t \times l} \]

where:

- \( \rho_g \) = green density of the test (reference) specimen, g/cm³
- 0.0610 = conversion factor, in³ to cm³
- \( M \) = mass of green test (reference) specimen, g
- \( w \) = width of green test (reference) specimen, inch
- \( t \) = thickness of green test (reference) specimen, inch
- \( l \) = length of green test (reference) specimen, inch

4.1.3 Set up the tooling in the universal testing machine or compacting press.

4.1.4 Measure the long dimension of the die cavity \( L_0 \) to 0.0001 inch (0.002 mm) and note for later use

4.2 Powder Preparation

4.2.1 Metal powders that contain a lubricant are normally tested in the as-received condition. The method of lubrication, either die-wall or admixing, shall be agreed between the concerned parties for testing unlubricated powders.
4.2.2 When an admixed lubricant is to be used, the type, grade, and percentage shall be agreed between the concerned parties or shall follow accepted PM practice.

4.2.3 Following the procedures in MPIF Standard 01, take a composite sample of the powder to be tested (also from the reference lot of powder) of sufficient quantity to produce at least three test specimens.

4.2.4 Take three test portions from the composite sample, each of which has a mass within 0.02 g of the mass calculated in section 4.1.2.

4.3 Compacting

4.3.1 If die-wall lubrication is to be used for compacting unlubricated metal powders, it shall be applied prior to the compaction of each test piece following the procedure in MPIF Standard 60.

4.3.2 Transfer the test portion of powder that is to be tested into the die cavity.

4.3.3 Compact the test specimen, using the required pressure, to the target green density in accordance with the procedure in MPIF Standard 60.

4.3.4 Eject the test specimen from the die, determine the mass, measure the dimensions, and number the specimen.

4.3.5 Repeat to obtain three specimens of the test powder as well as three test specimens of the reference lot.

4.4 Sintering

4.4.1 When the dimensional change test is conducted to approve a metal powder lot for production use, it is desirable to use the production furnace or sintering equipment that simulates or approximates production conditions.

4.4.2 Sinter the test specimens (with three reference specimens) using the production conditions or a heating rate, sintering atmosphere, sintering time and temperature, and cooling rate suitable for the material being tested.

4.4.3 After cooling to room temperature, measure the long dimension of each sintered specimen. Record the average of three measurements of this dimension \( L_T \), and \( L_R \) for the reference specimens, for later use in the calculations.

5. CALCULATIONS

5.1 Calculate the percent absolute dimensional change for the two sets of specimens as follows:

\[
D_T, \% = \frac{[L_T - L_D]/L_D]}{100}
\]

\[
D_R, \% = \frac{[L_R - L_D]/L_D]}{100}
\]

where:

- \( D_T \) = dimensional change for the test lot of powder
- \( D_R \) = dimensional change for the reference lot of powder
- \( L_D \) = major dimension of the die
- \( L_T \) = major dimension of the test lot specimen after sintering
- \( L_R \) = major dimension of the reference lot specimen after sintering

NOTE 4—This formula expresses the dimensional change as a percentage. Deletion of the factor 100 gives the dimensional change in inch/inch (mm/mm).

5.2 Calculate the comparative dimensional change for the test lot of powder as follows:

\[
D = D_T - D_R
\]

where:

- \( D \) = comparative dimensional change

5.3 The percent absolute dimensional change of the specimens produced from the reference powder shall be within the limits agreed between the powder producer and purchaser in order to have a valid test.

6. REPORT

6.1 Dimensional change at (specified) green density.

6.1.1 The absolute dimensional change from die size for the specimens made from the test powder (\( D_T \)), as the mathematical average of the three test specimens, calculated to the nearest 0.01%, including the sign of plus (+) for growth and minus (-) for shrinkage.

6.1.2 The comparative dimensional change between the test powder and the reference powder (\( D \)), as the mathematical difference between the average absolute dimensional change from die size for the three specimens made from the test powder and those from the reference powder, as a percent to the nearest 0.01%, including the sign of (+) or (-) to indicate the direction of the deviation.

6.1.3 The green density to the nearest 0.01 g/cm³ as the average density of the three test specimens (and reference specimens).

6.2 The following supplementary information may also be reported for clarification:

6.2.1 Identification of the metal powder by brand, grade, and lot number.

6.2.2 Chemical composition of the powder mix, if other than elemental powder was being tested.

6.2.3 Type, brand, and percent of the admixed lubricant (if used), or details of the die-wall lubrication method (if used).

6.2.4 Compacting pressure.

6.2.5 Sintering temperature.

6.2.6 Sintering time, defined as the elapsed time that the test specimens are within ± 15 °F (± 8 °C) of the stated sintering temperature for iron-base and ± 10 °F (± 5 °C) for copper-base powders.

6.2.7 Furnace atmosphere.

6.2.8 Use of a boat, belt, or carrier plate in the furnace.

6.2.9 Cooling rate and the temperatures between which the rate was measured (for example 650 °C to 315 °C [1200 °F to 600 °F]).

6.2.10 Whether the specimens were tempered (tempering temperature if used).

6.2.11 Absolute dimensional change of the reference powder.

6.2.12 Sintered carbon content (for PM steels).
7. PRECISION

7.1 A precision for this test is impractical due to the wide range of material and testing conditions that may be involved. The acceptable comparative dimensional change is a matter of agreement between the parties concerned.

APPENDIX

A1. COMPARABLE STANDARDS

ASTM B610
ISO 4492

![Diagram of Tooling to Produce a Rectangular Test Specimen]

**FIGURE 1:** Example of Tooling to Produce a Rectangular Test Specimen

Note: The dimensions for the cavity shall be 0.500 ± 0.004 inch wide (12.70 ± 0.10 mm) by 1.250 ± 0.004 inch long (31.75 ± 0.10 mm). The mating parts should be fitted and lapped to a 4 microinch (N3) finish or better, to dimensions of 0.0005 inch (0.0130 mm) to 0.0010 inch (0.0250 mm) smaller than the die cavity in each dimension. The dimensions given in the drawing typify the die cavity and punch within the stated tolerance at the normal width and length. The outer ring may be AISI H-11 hardened to 45-48 HRC. The punches may be AISI A-2 or A-7 hardened to 60-62 HRC. The die insert should be die grade tungsten carbide. The shrinkage between the carbide insert and the die case typically is 0.0015-0.0025 inches per inch (0.0015-0.0025 mm per mm).

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1. **SCOPE**

1.1 This standard describes a test that determines the compressibility of metal powders by measuring the green density of a test specimen resulting from subjecting metal powder to a uniaxial compressive loading in a confining die.

1.2 Compressibility may be determined by using either of the following specimens:

1.2.1 **Specimen A:** Using a cylindrical die to produce a specimen 1 inch (25.4 mm) in diameter.

1.2.2 **Specimen B:** Using the standard rectangular transverse rupture die to produce a specimen 1.25 by 0.50 inches (31.8 by 12.7 mm).

1.3 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses. Conversion factors, with the exception of density, for which g/cm³ is the industry standard, are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. **APPARATUS**

2.1 Either of the following tool sets is required depending on the specimen selected:

2.1.1 **Specimen A:** A set of tools with a cylindrical cavity 1.0 inch (25.4 mm) in diameter. An example is shown in Fig. 1.

2.1.2 **Specimen B:** A set of tools to produce a test specimen 1.25 by 0.50 inch (31.8 by 12.7 mm) similar to that shown in Fig. 2.

2.2 A press or compression testing machine capable of applying a specified load with a minimum accuracy of ±1.0% at a rate of approximately 30 tsi/min (415 MPa/min).

NOTE 1—The alignment of the press and the construction of the tooling must be capable of producing test specimens with faces parallel within 0.001 inch (0.025 mm).

NOTE 2—Names of apparatus manufacturers are listed in General Information IV.

2.3 A balance, readable to 0.001 g with a capacity of at least 100 g.

2.4 Micrometers or other suitable measuring apparatus capable of measuring the green compacts to the nearest 0.0001 inch (0.002 mm).

3. **TEST SPECIMEN**

3.1 **Specimen A:** A cylindrical test specimen 0.270 to 0.290 inch (6.9 to 7.4 mm) in height.

3.2 **Specimen B:** A rectangular test specimen 0.240 to 0.260 inch (6.1 to 6.6 mm) thick.

4. **PROCEDURE**

4.1 Obtain, in accordance with MPIF Standard 01, a quantity of powder of sufficient mass to be split into the required number of test portions.

4.2 Compact test specimens (Specimen A or Specimen B) using the procedure in MPIF Standard 60.

4.3 Apply the compacting pressure at an approximate rate of 30 tsi/min (415 MPa/min). Release the pressure as soon as the required pressure is attained. Dwelling at pressure for as little as 10 seconds can increase iron powder compacted density by 0.3%.

4.4 Press the test portions at either a single specified pressure or a series of incremental pressures. In the latter case, the densities obtained may be illustrated as a compressibility curve of the powder, such as, a plot of density as a function of compacting pressure. Alternatively, compact the powder to a specified green density and express the compressibility as the compacting pressure used to reach that density.

4.5 Make and test a minimum of three (3) test specimens using the same conditions.

4.6 Weigh the compacted specimen and record the mass M to the nearest 0.001 g.

4.7 Use paper towel with a solvent to clean the die and the punches thoroughly after each test specimen has been compacted.
5. CALCULATIONS

5.1 Calculate the density of the test specimen as follows:

5.1.1 Specimen A: Cylindrical Test Specimen.

\[
D = 0.0777 \frac{M}{D^2 h}
\]

where:
- \(D\) = Green Density in g/cm\(^3\)
- \(M\) = Mass of test cylinder in g
- \(d\) = diameter of test cylinder in inches
- \(h\) = height of test cylinder in inches

NOTE 3—Where the test specimen is measured in mm, use the formula:

\[
D = 1273 \frac{M}{D^2 h}
\]

5.1.2 Specimen B: Transverse Rupture Test Specimen.

\[
D = 0.0610 \frac{M}{ltw}
\]

where:
- \(D\) = Green Density in g/cm\(^3\)
- \(M\) = Mass of test specimen in g
- \(l\) = length of test specimen in inches
- \(t\) = thickness of test specimen in inches
- \(w\) = width of test specimen in inches

NOTE 4—Where the test specimen is measured in mm, use the formula:

\[
D = 1000 \frac{M}{ltw}
\]

6. REPORT

6.1 Compressibility as the density in g/cm\(^3\) to the nearest 0.01 g/cm\(^3\) at a given compacting pressure. Report the arithmetic average of three test specimens (e.g. 6.50 g/cm\(^3\) at 30 tsi [415 MPa]). Alternatively, report to the nearest 0.5 tsi (10 MPa) the compacting pressure used to obtain a specified green density (e.g. 30.0 tsi [415 MPa] for a green density of 6.50 g/cm\(^3\)).

6.2 The following supplementary information should be reported for clarification purposes.

6.2.1 Identification of test specimen, i.e., Specimen A or Specimen B.

6.2.2 Identification of powder by brand, grade and lot number.

6.2.3 Chemical composition of powder mix used if other than elemental powders are being tested.

6.2.4 Method of lubrication: die-wall or admixed.

6.2.4.1 For die-wall lubrication: type or brand of lubricant.

6.2.4.2 For admixed lubricant: type or brand, and percent.

6.2.5 Identification of die wall material, i.e., whether carbide or steel.

6.3 Compressibility may also be reported in graphic form showing the density as the ordinate as a function of at least four different compacting pressures as the abscissa. Alternatively, a line may be drawn through points that represent the averages of three determinations at three different compacting pressures.

7. PRECISION AND BIAS

7.1 The following precision data were developed by ASTM Subcommittee B09.02 using the procedures contained in ASTM E691 and conducted in an interlaboratory test study (ASTM Research Report RR: B09-1002).

7.2 Precision: The repeatability limit, \(r\), is 0.025 g/cm\(^3\). On the basis of test error alone, the difference in absolute value of individual test results obtained in the same laboratory on the same material will be expected to exceed 0.025 g/cm\(^3\) only about 5% of the time.

7.3 The reproducibility limit, \(R\), for ferrous and nonferrous powders is 0.07 g/cm\(^3\). On the basis of test error alone, the difference in absolute value between individual test results obtained in two different laboratories on the same material will be expected to exceed \(R\) only about 5% of the time. Thus, if a larger difference is found, there is reason to question one or both test results.

7.4 Bias: No statement can be made about bias, because there are no accepted standard or reference powders for compressibility testing.


APPENDIX

A1. COMPARABLE STANDARDS

ASTM B331
ISO 3927
FIGURE 1: Example of Tooling to Produce a Cylindrical Test Specimen

Note: The mating parts should be fitted and lapped to a 4 microinch (N3) finish or better. The outer ring may be AISI H-11 hardened to 45-48 HRC. The punches may be AISI A-2 or A-7 hardened to 60-62 HRC. The die insert should be die grade tungsten carbide. The shrinkage between the carbide insert and the die case typically is 0.0015-0.0025 inches per inch (0.0015-0.0025 mm per mm).

FIGURE 2: Example of Tooling to Produce a Rectangular Test Specimen

Note: The dimensions for the cavity shall be 0.500 ± 0.004 inch wide (12.70 ± 0.10 mm) by 1.250 ± 0.004 inch long (31.75 ± 0.10 mm). The mating parts should be fitted and lapped to a 4 microinch (N3) finish or better, to dimensions of 0.0005 inch (0.0130 mm) to 0.0010 inch (0.0250 mm) smaller than the die cavity in each dimension. The dimensions given in the drawing typify the die cavity and punch within the stated tolerance at the normal width and length. The outer ring may be AISI H-11 hardened to 45-48 HRC. The punches may be AISI A-2 or A-7 hardened to 60-62 HRC. The die insert should be die grade tungsten carbide. The shrinkage between the carbide insert and the die case typically is 0.0015-0.0025 inches per inch (0.0015-0.0025 mm per mm).

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1. SCOPE
1.1 This standard describes a test method for measuring the maximum density reached by a metal powder when vibrated under specified conditions.
1.2 With the exception of density and mass, for which g/cm³ and g are the industry standard, SI units are used in this standard.
1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS
2.1 A balance of appropriate capacity readable to 0.01 g and capable of determining the mass indicated in Table 1 to the nearest 0.1 g.
2.2 A graduated glass cylinder of appropriate capacity and accuracy as indicated in Table 1.
2.3 A mechanical device with a stroke of 3 ± 0.2 mm capable of tapping the graduated cylinder containing the powder at a rate of 100 to 300 taps per minute so that densification can take place without any loosening of surface layers. A schematic of such a device is shown in Fig. 1.

Table 1

<table>
<thead>
<tr>
<th>Apparent Density g/cm³</th>
<th>Cylinder Capacity cm³</th>
<th>Mass of Sample g</th>
<th>Cylinder Accuracy cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 1</td>
<td>100</td>
<td>50 ± 0.2</td>
<td>0.5</td>
</tr>
<tr>
<td>1-4</td>
<td>100</td>
<td>100 ± 0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>&gt; 4</td>
<td>25</td>
<td>100 ± 0.5</td>
<td>0.2</td>
</tr>
</tbody>
</table>

3. TEST SPECIMEN
3.1 The mass of the test portion shall be based on the apparent density as indicated in Table 1.

4. PROCEDURE
4.1 Thoroughly clean and dry the inside of the graduated cylinder.
4.2 Weigh the amount of powder as indicated in Table 1.
4.3 Pour the test portion into the graduated cylinder taking care that a level surface of powder is obtained.
4.4 Place the filled cylinder in the mechanical tapping apparatus and tap until no further decrease of the volume of the powder takes place.

NOTE 2—In practice, the minimum number of taps, N, such that no further change in volume takes place would be determined. For all further tests on the same type of powder, the cylinder would be subjected to 2N taps, except where general experience and acceptance have established a specific number of taps (no less than N taps) as being satisfactory. For fine refractory metal powder, 3,000 taps has been found to be satisfactory for all sizes.
4.5 Read the volume of powder to the cylinder accuracy indicated in Table 1.

5. CALCULATION
5.1 Calculate the tap density as follows:

\[
\rho_t = \frac{m}{V}
\]

where:
\(\rho_t\) = tap density, g/cm³
\(m\) = mass of powder, g
\(V\) = volume of tapped powder, cm³

6. REPORT
6.1 Tap density to the nearest 0.1 g/cm³ for powders with apparent densities up to and including 4 g/cm³. For powders with apparent densities higher than 4 g/cm³, report to the nearest 0.2 g/cm³.

7. PRECISION
7.1 The precision of this test method is based on an interlaboratory study of ASTM B527, conducted in 2011–2012 by ASTM Committee B09 on Metal Powders and Metal Powder Products. A single laboratory with two operators analyzed three different powders for tap density. Either 10 or 20 replicates were reported for each material in this study. Except for testing in just one lab, ASTM E691 was followed...
for the study design; the details are given in ASTM Research Report No. RR: B09-1020. The precision statement was determined through statistical examination of 50 results, from a single laboratory, testing three materials. The materials were designated in the study as:
1. Cermet (TiC based), apparent density 1.02 g/cm³
2. Iron (Ancorsteel 1000), apparent density 2.92 g/cm³
3. Tungsten carbide (WC), apparent density 6.91 g/cm³

7.1.1 Test Results—The precision information presented herein has been calculated for the comparison of three materials, each of which is the mathematical average of ten or twenty individual test determinations.

7.1.2 95% Repeatability Limit (r)—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the r value for that material; r is the interval representing the critical difference between two test results for the same material, using the same mass and appropriate volumetric cylinder capacity according to Table 1. Repeatability limits are listed in Table 2. The within-laboratory repeatability limit, r, as defined by ASTM E456, is dependent on the apparent density of the tested material (see Table 2). At the 95% confidence level, duplicate tap density results from the same laboratory should not be considered to be different unless they differ by more than r.

<table>
<thead>
<tr>
<th>Material (100 g per run)</th>
<th>Cylinder Capacity cm³</th>
<th>Average</th>
<th>Repeatability Standard Deviation Sr</th>
<th>Repeatability Limit r</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cermet – 20 replicates</td>
<td>100</td>
<td>1.6</td>
<td>0.02</td>
<td>0.06</td>
</tr>
<tr>
<td>Iron – 20 replicates</td>
<td>100</td>
<td>3.7</td>
<td>0.10</td>
<td>0.29</td>
</tr>
<tr>
<td>Tungsten Carbide (WC) 10 replicates</td>
<td>25</td>
<td>8.2</td>
<td>0.07</td>
<td>0.20</td>
</tr>
</tbody>
</table>

7.1.3 The terms (“repeatability limit” and “reproducibility limit”) are used as specified in ASTM E177.

7.1.4 Any judgment in accordance with statement 7.1.2 would normally have an approximate 95% probability of being correct, however because of the limited number of participants in this study, consider the precision noted as an estimate.

7.1.5 Reproducibility Limit (R)—The reproducibility limit of this test method is being determined and will be available in December, 2019. Reproducibility results cannot be determined from a single laboratory study.

The Precision data and Figure 1 were developed by ASTM Committee B09 on Metal Powders and Metal Powder Products and are reprinted, with permission, from ASTM B527-15 Standard Test Method for Tap Density of Metal Powders and Compounds, copy-right ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

Figure 1: Example of Tapping Apparatus

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. **SCOPE**

1.1 This standard describes a procedure for determining the apparent density of both free and non-free flowing powders and premixes. The value obtained may not be the same as that obtained by other apparent density tests but is indicative of the fill characteristics of the powder or premix. See MPIF Standards 04 and 28 for other apparent density test methods.

1.2 With the exception of density, for which g/cm³ is the industry standard, SI units are used in this standard. Values in inch-pound units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. **PRINCIPLE**

2.1 This test method consists of slowly sliding a bushing partially filled with powder over a hole in a hardened steel block, collecting and weighing the powder, and calculating its apparent density.

2.2 The apparent density is an important measure of a material characteristic inherent in the powder, which is useful to the powder producers and end users in determining lot to lot consistency. Knowledge of the apparent density of the final mix as obtained with this test method is very beneficial to the PM parts fabricator for setting compression ratios for fixed fill die cavities.

2.3 This test method may be part of a purchase agreement between the powder producer and PM part producers, or it may be an internal quality control test for either party.

3. **APPARATUS**

3.1 Steel Die Block: A hardened, tempered (60 HRC minimum), ground and demagnetized tool steel block, approximately 165 by 64 mm (6.50 by 2.50 inch) and 25.400 ± 0.003 mm (1.0000 ± 0.0001 inch) in thickness, having a precise, centrally located through-hole 31.664 ± 0.003 mm (1.2466 ± 0.0001 inch) in diameter with a volume of 20.0 cm³ (1.22 inch³) (Fig. 1).

The surfaces of the sides of the die block shall be given a roughening treatment to aid in handling during use.

3.2 Bushing: A nonferrous cylindrical sleeve, preferably brass or bronze, approximately 44 mm (1.75 inch) outside diameter and 38 mm (1.50 inch) inside diameter with a height of about 38 mm (1.50 inch) long. (Fig. 1). The cylinder should be inscribed on the ID with a ring at approximately three quarters of its height to indicate 50 cm³ (3.05 inch³).

3.3 Weighing Paper: A sheet of cellophane, glazed or waxed paper measuring approximately 150 mm by 150 mm square (6.0 inch by 6.0 inch).

3.4 Balance: A balance readable to 0.001 g with a capacity of at least 200 g.

4. **TEST PORTION**

4.1 The test portion shall be obtained in accordance with MPIF Standard 01.

4.2 The test portion shall be of sufficient volume to fill the bushing to about three quarters of its height.

5. **PROCEDURE**

5.1 Clean the test block and bushing thoroughly to eliminate any loose powder particles.

5.2 Place the hardened demagnetized steel block on a sheet of preweighed or tared paper.

5.3 Place the bushing on the block—either side of the hole.

5.4 Fill the bushing slowly and carefully to three-quarters of its height with powder. (A ring on the ID of the bushing indicates the proper fill height.)

5.5 With downward pressure on the bushing, slowly slide the bushing toward the hole while twisting it about a quarter of a turn. This gives a snowplow action to the powder as it falls slowly into the hole. Continue this motion until the bushing passes the hole. Stop, and again with downward pressure on the bushing, slide it straight back over the hole to its starting position. The sliding action should be slow enough to allow for complete filling of the steel block.

5.6 Remove the steel block from the preweighed paper being careful not to tip the block and spill additional powder onto the paper.

5.7 Transfer the preweighed or tared paper, plus the powder from the cavity in the steel block, to a balance and determine the mass. Record the mass to the nearest 0.001 g.
6. **CALCULATION**

6.1 Calculate the apparent density as follows:

Arnold Apparent Density

\[
\rho_A = \frac{m}{V} = \frac{m}{20}
\]

where:

\[
\rho_A = \text{Arnold apparent density, g/cm}^3
\]

\[
m = \text{mass of powder, g}
\]

\[
V = \text{volume of center hole, 20 cm}^3
\]

7. **REPORT**

7.1 Apparent density to the nearest 0.01 g/cm³. To minimize confusion with other test methods, report as Arnold Density, g/cm³.

8. **PRECISION**

8.1 Precision has been calculated from the test data determined in an ASTM Subcommittee B09.02 interlaboratory study of Arnold apparent density. The testing was performed in 1991 by eight laboratories. Each of the laboratories made three tests on seven powder samples. ASTM practice E691 was followed for the details of the testing and calculations are given in MPPA Research Report No. MPPA R-48-00.

8.1.1 The powders tested and their Arnold apparent densities are listed in Table 1.

8.1.2 The 95% repeatability limit, r, (within a laboratory) is 0.08 g/cm³.

8.1.3 The 95% reproducibility limit, R, (between laboratories) is 0.17 g/cm³.

8.1.4 Duplicate results from the same laboratory should be considered acceptable at the 95% confidence level unless they differ by more than r, the repeatability interval.

8.1.5 Duplicate results from two different laboratories should be considered acceptable at the 95% confidence level unless they differ by more than R, the reproducibility interval.

**APPENDIX**

A1. **COMPARABLE STANDARD**

ASTM B703

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**Table 1. Powders Tested and Their Arnold Apparent Densities**

<table>
<thead>
<tr>
<th>Powder</th>
<th>Arnold Apparent Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sponge iron</td>
<td>2.62</td>
</tr>
<tr>
<td>Atomized iron</td>
<td>3.25</td>
</tr>
<tr>
<td>Atomized iron (lubricated)</td>
<td>3.35</td>
</tr>
<tr>
<td>Bronze premix (lubricated)</td>
<td>3.51</td>
</tr>
<tr>
<td>Brass (lubricated)</td>
<td>3.88</td>
</tr>
<tr>
<td>Tin</td>
<td>4.26</td>
</tr>
<tr>
<td>Spherical bronze</td>
<td>5.10</td>
</tr>
</tbody>
</table>
FIGURE 1: Arnold Apparent Density Meter

Dimensions shown in millimetres [inches].

Note: Break all edges except noted. 0.8 x 45° [0.031] grind all over.

Material: Tool steel, hardened tempered 60HRC min. Demagnetized.

PART NO. 1

PART NO. 2

Material: Brass

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1. **SCOPE**

1.1 This standard describes recommended test methods for evaluating the performance of copper-base infiltrating powders. The infiltrant is placed in contact with a part, usually of iron-base composition, and both components are heated to a temperature above the melting point of the infiltrant. Infiltration may be simultaneous with sintering (one-step infiltration) or in a separate step after sintering (two-step infiltration).

1.2 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. **DEFINITIONS**

2.1 Infiltration—a process of filling the surface-connected pores of a sintered or unsintered PM compact with a metal or alloy of lower melting point.

2.2 Efficiency—the ratio, in percent, of the amount of infiltrant absorbed by the part, to the amount of infiltrant originally used.

2.3 Erosion—the rapid solution of the base metal at the contact area between the part and the molten infiltrant, where the infiltrant flows into the part causing pits, channels and coarse surface porosity.

2.4 Residue—material that remains on the surface of the part after infiltration.

2.5 Adherent Residue—material that remains on the surface of the part after infiltration, that cannot be readily removed by tapping, lightly brushing or wiping.

2.6 Infiltrant Loading Density—infilitrant mass per unit area of contact between infiltrant and part.

3. **APPARATUS**

3.1 Furnace: A muffle type furnace or equivalent having a heating chamber with a delubrication zone, capable of maintaining a temperature of 2050 ± 15 °F (1120 ± 8 °C) in the hot zone should be used. It must have a reducing atmosphere in the hot zone while maintaining a non-oxidizing atmosphere in the cooling chamber.

3.2 A balance readable to 0.001 g with a minimum capacity of 100 g.

3.3 Micrometers or other suitable measuring apparatus capable of measuring to 0.0001 inch (0.002 mm).

4. **TEST SPECIMENS**

4.1 **Preferred Test Specimen:** To evaluate an infiltrating powder, the preferred compacted base-powder specimen to be infiltrated shall be 1 inch (25.4 mm) dia. by 0.5 inch (12.7 mm) long. The composition of the compacted base-powder specimen, including the type of material and the green density, shall be a matter of agreement between the concerned parties.

4.2 The infiltrant compact shall be a cylinder nominally 0.5 inch (12.7 mm) in diameter compacted from a copper or copper-base infiltrant mix. Normally, the infiltrant mix will also contain a dry lubricant. The type or composition of the infiltrant and the mass and green density of the infiltrant compact shall be a matter of agreement between the parties concerned.

4.2.1 When placed on the compacted base-powder specimen, the recommended infiltrant compact has a contact area of 0.196 square inches (1.265 cm²). Since the mass per unit area of infiltrant running through the contact area can affect the test results, the infiltrant loading density should be a matter of agreement between parties and should be reported.

4.3 **Alternative Test Specimen:** An alternative compacted base-powder test specimen is the transverse rupture specimen (TRS) 1.25 inch (31.8 mm) by 0.5 inch (12.7 mm) by 0.25 inch (6.4 mm) as described in MPIF Standard 41. The advantage of using this specimen is that the transverse rupture strength can be determined. The infiltrant compact shall be a cylinder nominally 0.5 inch (12.7 mm) in diameter compacted from a copper or copper-base infiltrant mix as covered in Section 4.2.

NOTE 1—The TRS bar has a different mass compared with the cylindrical compact. This will affect the mass of the infiltrant compact and the infiltrant loading density. The different loading density may affect the measured infiltration efficiency and the surface appearance of the infiltrated compact.
4.4 Alternative compacted base-powder specimens may be any structural part of composition and sintered or green density that is acceptable to the parties concerned. Likewise, the infiltrant compact may be any available shape that is acceptable to the parties concerned with the composition, lubricant, mass and green density a matter of agreement between the parties concerned. The masses of the compacted base-powder specimens and infiltrant compacts shall not vary more than ± 0.5% from the mean. The densities of the compacted base-powder specimens and infiltrant compacts, as calculated from their dimensions and masses, shall not vary more than ± 0.5% from the mean.

5. PROCEDURE

5.1 Weigh the compacted base-powder specimens and record the mass to 0.001 g. Measure their dimensions to the nearest 0.0001 inch (0.002 mm). A minimum of three specimens shall be prepared for each infiltrant being evaluated.

5.1.1 Determine the density of the compacted base-powder specimens by calculation from the mass and the dimensions of the specimens or by weighing in air and water as described in MPIF Standard 42. If Standard 42 is used to determine the density, do not use any specimen that was used for displacement density testing for subsequent infiltration.

5.1.2 Determine the uniformity of the compacted base-powder specimens by measuring the mass and thickness. The mass shall not vary more than ± 0.5% from the mean. The density, as calculated from the dimensions and mass of the specimen, shall not vary more than ± 0.5% from the mean.

5.1.3 Place the compacted base-powder specimens on a suitable tray or container for transporting the specimens through the infiltrating furnace. The interested parties should agree upon the type of tray or container. Avoid materials that will react with either the compacted base-powder specimen or the infiltrant. Position the compacted base-powder specimens with one of the surfaces that was formed by a punch face surface facing up.

5.2 Center the weighed infiltrant compact, defined as mass S, on the upward facing punch surface of the compacted base-powder specimen. Space the specimens well apart so that they are not touching.

5.2.1 Determine the uniformity of the infiltrant compacts by determining their mass. The mass of any compact shall not vary more than ± 0.5% from the mean. In addition, the uniformity of the infiltrant compacts shall be determined by measuring their density. The density of any compact, as calculated from the dimensions and mass of the compact, shall not vary more than ± 0.5% from the mean.

5.2.2 Process a reference infiltrating powder, which has been agreed to by the parties concerned, at the same time as the infiltrant under test. A comparison between the reference infiltrant and the infiltrant under test will serve to verify that the test conditions, particularly the furnace conditions and atmosphere, are those expected.

5.2.3 Process a minimum of three, green, pre-weighed compacted base-powder specimens through the infiltrating furnace cycle without an infiltrant compact in contact. The temperature in the hot zone of the furnace should be 2050 ± 15 °F (1120 ± 8 °C) and there should be a reducing atmosphere in the hot zone and a non-oxidizing atmosphere in the cooling chamber. After the furnace treatment, determine the average mass of these specimens, identified as B. This value permits an adjustment to the mass of the original compacted base-powder specimen to account for mass lost in the furnace treatment resulting from reducible oxides, decarburization, volatilization of lubricant and loss of other volatiles.

5.3 Observe and note the appearance of the infiltrated specimens as each tray is removed from the furnace.

5.3.1 Weigh each infiltrated specimen plus any residue and record the mass to 0.001 g. Take precautions to include any loose residue that may fall off. Assign any loose residue lying on the tray to this mass, if it can be established that it has definitely come from this particular specimen and not from one of its neighbors. This mass is identified as T.

5.3.2 Remove all loose residue by inverting the infiltrated specimens and reweigh them. Record the mass to 0.001 g. In the case of a residue-free powder, weigh the infiltrated specimen as is. Those infiltrants that produce a slightly adherent “button” will require a scraping action to remove this form of residue before the specimens are weighed. This mass is defined as F.

5.3.3 Examine the infiltrated specimen surfaces, especially the surface that has been in contact with the infiltrant compact, using approximately 10X magnification. Record the presence of adherent residue and/or erosion. The amount of adherent residue and/or erosion allowable should be established by agreement between the parties concerned.

5.4 Measure the dimensions of the infiltrated specimens to the nearest 0.0001 inch (0.002 mm).

5.4.1 Determine the dimensional change of the infiltrated specimens from die size according to MPIF Standard 44.

5.5 Determine the density of the infiltrated specimen, either by calculation from its mass and dimensions, or as described in MPIF Standard 42.

5.6 When transverse rupture strength specimens are used, determine the transverse rupture strength of the infiltrated specimens using the procedure described in MPIF Standard 41.
5.7 Determine the apparent hardness of the infiltrated specimens using procedures described in MPIF Standard 43. The locations for hardness determination shall be a matter of agreement between the parties concerned.

6. CALCULATIONS

6.1 Calculate the infiltrant loading density by dividing the infiltrant mass \( S \) by the cross-sectional area of the infiltrant compact.

6.2 Calculate the gross infiltration efficiency by the following method:

\[
\text{Efficiency, \%} = \left( \frac{F-B}{S} \right) \times 100
\]

where:
\( F = \) mass of infiltrated specimen, in grams, after residue has been removed
\( B = \) mass of sintered, uninfilitrated specimen, in grams (see section 5.2.3)
\( S = \) mass of infiltrant compact, in grams

6.2.1 Calculate the average efficiency from the results of a minimum of three tests.

6.3 Calculate the loose residue amount as follows:

\[
\text{Residue, \%} = \left( \frac{T-F}{S} \right) \times 100
\]

where:
\( T = \) mass of infiltrated specimen plus all residue in grams
\( F = \) mass of infiltrated specimen, in grams, after residue has been removed
\( S = \) mass of infiltrant compact, in grams

6.3.1 Calculate the average residue amount from the results of a minimum of three tests.

7. REPORT

7.1 Specific infiltration conditions, including the use of one-step or two-step infiltration, shall be a matter of agreement between the parties concerned. It is suggested that the following infiltration data be determined and recorded:

7.1.1 Heat-up time and rate, cooling time and rate, and time at temperature.

7.1.2 Range of furnace temperatures during infiltration.

7.1.3 Furnace atmosphere, including moisture content at atmosphere inlet to furnace or sampled from hot zone, and the rate of flow with respect to the cross-section of the furnace hearth.

7.2 Chemical composition, mass and the density of the compacted base-powder specimen.

7.3 Type or composition, mass and green density of the infiltrant compact used.

7.4 Infiltrant loading density.

7.5 Appearance of infiltrated specimens (presence of adherent residue and erosion).

7.6 Average efficiency to the nearest 0.1%

7.7 Average residue amount to the nearest 0.1%

7.8 Average dimensional change.

7.9 Average infiltrated density.

7.10 Average transverse rupture strength, if tested.

7.11 Average apparent hardness.

APPENDIX

A1. COMPARABLE STANDARD
ISO 14168
1. SCOPE
1.1 This standard describes the preparation and evaluation of tension test specimens to be used in determining tensile properties of sintered and heat treated metal injection molded materials.
1.2 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.
1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS
2.1 Feedstock mixer
2.2 Injection molding machine
2.3 Debinding equipment
2.4 Sintering furnace
2.5 Tensile testing machine
2.6 Gauges capable of measuring from 0 to 1 inch (0 to 25 mm) to an accuracy of 0.0005 inch (0.01 mm).
2.7 Extensometer

3. TEST SPECIMEN
3.1 Fig. 1 shows tool dimensions for the large tensile test specimen. Fig. 2 shows tool dimensions for the small tensile test specimen.
3.2 Test specimen size to be used is dependent upon the material being tested and capacity of tensile testing machine.
NOTE 1—Expected gauge length to diameter ratio of the large, as-processed specimen is 5.3 and for the small, as-processed specimen is 8.0.

4. PROCEDURE
4.1 Specific processes such as feedstock mixing, injection molding, debinding, sintering and heat treating, are left to individual discretion. Processing shall be carried out in a manner that produces specimens free from defects that would yield inconsistent test results.
4.2 Tests should be carried out on a minimum of 3 specimens.
4.3 Recognized standards for testing parameters and property determination are described in ASTM E8.
4.4 Gauge length to be at least four times the diameter of the specimen.
NOTE 2—Grip ends of tensile specimens have holes for linking pins to pass through them. These linking pins connect the specimen to the testing machine. It is recommended that heat treated alloy steel be used for the linking pins. Care should be taken to avoid point contact between the pins and inside diameter of the holes. An example of a design for tensile grips is shown in Fig. 3. Other methods of gripping may exist.

5. REPORT
5.1 Test specimen size (large or small).
5.2 Chemical composition of material.
5.3 Processing condition, e.g. as-sintered or heat treated.
5.4 Density, g/cm³.
5.5 Average ultimate tensile strength to the nearest 100 psi (1 MPa).
5.6 Average yield strength (0.2% offset) to the nearest 100 psi (1 MPa).
5.7 Average percentage elongation in 1 inch (25 mm) to the nearest 1%.
5.8 Other processing information, as required.

6. PRECISION
6.1 As a specimen preparation procedure that does not include actual performance of a test, no precision statement is required.

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By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability.
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The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System.” Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.
FIGURE 1: Large MIM Tensile Test Specimen Tool Dimensions
Dimensions shown in inches [millimetres]

NOTES:
1) GATE, MFG. I.D., AND EJECTOR LOCATION OPTIONAL BUT SHALL NOT BE IN THE GAGE LENGTH OR DATUM [A].
2) NO DRAFT PERMITTED. OPTIONAL: .257 [6.53]["].
DIA. AFTER SINTERING.
3) FINAL DIMENSIONS OF SINTERED SPECIMEN WILL VARY WITH SHRINKAGE.
4) GEOMETRIC TOLERANCING REQUIREMENTS APPLY AS-SINTERED
   APPLIES TO GAGE LENGTH.
5) INTERPRET DRAWING PER ANSI/ASME Y14.5.M.
6) DIMENSIONS IN PRINTED TEXT ARE IN MILLIMETRES.

FIGURE 2: Small MIM Tensile Test Specimen Tool Dimensions
Dimensions shown in inches [millimetres]

NOTES:
1) GATE, MFG. I.D., AND EJECTOR LOCATION OPTIONAL BUT SHALL NOT BE IN THE GAGE LENGTH OR DATUM [A].
2) NO DRAFT PERMITTED. OPTIONAL: .196 [4.97]["].
DIA. AFTER SINTERING.
3) FINAL DIMENSIONS OF SINTERED SPECIMEN WILL VARY WITH SHRINKAGE.
4) GEOMETRIC TOLERANCING REQUIREMENTS APPLY AS-SINTERED
   APPLIES TO GAGE LENGTH.
5) INTERPRET DRAWING PER ANSI/ASME Y14.5.M.
6) DIMENSIONS IN PRINTED TEXT ARE IN MILLIMETRES.
FIGURE 3: Example of Tensile Grips (not mandatory)
Dimensions shown in inches [millimetres]

NOTES:
1) THE DESIGN AND/OR USE OF THESE GRIPS ARE OPTIONAL.
2) INTERPRET DRAWING PER ANS/ASME Y14.5M.
3) DIMENSIONS IN BRACKETS ([ ]) ARE IN MILLIMETRES.
1. SCOPE

1.1 This standard covers the determination of the microindentation hardness of PM materials. The procedure differs from that applied to pore-free material in terms of the precautions required to deal with the porosity. This procedure covers tests made with the Knoop or Vickers indentors under test loads in the range from 1 to 200 gf.

1.2 Automated testing is generally not suitable for use with porous PM materials, because acceptable indentations require avoiding placing indentations in the immediate vicinity of a pore, a condition not guaranteed with automated placement of the indentations. Any automated testing shall allow for review of indentations post-test to reject any distorted or unusually large indentations in accordance with Section 4.2.5.

1.3 A method of conversion from the directly measured indentor lengths to other hardness scales, e.g. HRC, is described.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. PRINCIPLES

2.1 Hardness tests consist of forcing an indentor into a test specimen under a prescribed load and carefully controlled conditions. The greater the indentor penetration, the softer the material. In Rockwell hardness testing, one directly measures the depth of penetration and assigns a hardness number to that penetration, e.g. an HRC number. In Vickers and Knoop microindentation hardness testing, a pyramidal-pointed diamond is forced into the test specimen. The hardness is calculated as the indenting force divided by the measured projected area of the resulting indentation.

2.2 Microindentation hardness testing provides the hardness of the fully dense regions in a porous material. It indicates the hardness that the material would have if there were no pores.

2.3 Microindentation hardness tests allow the evaluation of specific phases, microstructural constituents and regions or gradients too small for apparent hardness testing.

NOTE 1—Apparent (macroscopic) hardness testing, e.g. with the Rockwell “C” indentor, shows the composite hardness of pores and fully dense regions. The apparent hardness is lower than would be observed if there were no pores.

2.4 The Knoop hardness number is calculated by dividing the applied load in kilograms force by the projected area of the indentation in square millimetres, computed from the measurement of the long diagonal. Because of the unequal angles between the four intersecting faces, (172 °30´ and 130°), the use of the Knoop indentor results in a shallow, elongated indentation.

2.5 The Vickers hardness number is calculated by dividing the applied load in kilograms force by the projected area of the indentation in square millimetres, computed from the mean of the measured diagonals of the indentations as it uses a square based pyramidal diamond point with an included angle of 136 °.

3. APPARATUS

3.1 A microindentation hardness testing machine capable of applying the required load, equipped with a Knoop or Vickers indentor and a provision for measuring the length of the indentation diagonals, with a precision of 4 microinch (0.1 micrometre).

4. PROCEDURE

4.1 Specimen Mounting and Preparation - Guidelines for these procedures are in Appendix A.

4.1.1 Carefully section and mount the test specimen in a suitable medium to allow for ease of handling and polishing.

4.1.2 Polish and lightly etch the specimen to reveal the phases present, as necessary.

NOTE 2—Careful etching is necessary as heavy etching obscures features and interferes with the measurement of the diagonals of the indentation.

4.1.3 Care should be taken so that the true area fraction of the porosity is revealed throughout the entire cross section.

4.2 Specimen Testing

4.2.1 Support the surface of the specimen so that it is perpendicular to the axis of the indentor.
4.2.2 Locate a suitable and desired location for testing. Space the indentations so that the distance between them or the edge of the specimen satisfies the requirements shown in Fig. 1 (as per ASTM E384).

4.2.3 Select a suitable load and magnification for the test. A 100 gf load is recommended for hardened steel. Lower loads may be used for softer materials or when small regions need to be tested. For the best precision use the highest load compatible with the feature being tested. Magnification ranges for indentation length are as follows:

<table>
<thead>
<tr>
<th>Indentation Length</th>
<th>Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Micrometres</td>
<td>Max</td>
</tr>
<tr>
<td>&lt;76</td>
<td>400</td>
</tr>
<tr>
<td>76 – 125</td>
<td>800</td>
</tr>
<tr>
<td>&gt;125</td>
<td>600</td>
</tr>
</tbody>
</table>

4.2.4 Apply test load.

4.2.5 Examine the indentation for possible defects. The two sections of each diagonal should agree within 20% of each other. Discard any distorted or unusually large indentations. Unusually large indentations sometimes occur due to the presence of pores directly under the indentation.

4.2.6 Measure the length of indentation diagonal, taking care to avoid backlash by moving only in one direction. For Knoop, read the larger diagonal length to 0.1 micrometres. For Vickers, read both diagonals to the equivalent of 0.1 micrometres and calculate the average.

4.2.7 Make additional indentations. Space the indentations, so that adjacent tests do not interfere with each other. The minimum spacing between tests is illustrated in Fig. 1.

4.2.8 Calculations - The Knoop or Vickers numbers may be calculated using the following formulas in this section or tables in ASTM E384.

a. Knoop - Using the units of force and length commonly employed, i.e. for force P in gf, and a long diagonal d in micrometres, the Knoop hardness is calculated:

\[ HK = 14229 \frac{P}{d^2} \]

b. Vickers - Using the units of force and length commonly employed, i.e. for force P in gf, and the mean of the two diagonals d in micrometres, the Vickers hardness is calculated:

\[ HV = 1854.4 \frac{P}{d^2} \]

c. For indentor diagonals measured in millimetres, tables of HK and HV values are tabulated in ASTM E384.

5. CONVERSION TO OTHER HARDNESS SCALES

5.1 It is often desired to express microindentation hardness values in other hardness scales, e.g. HRC. There is no direct conversion from microindentation hardness to HRC. Approximate values can be obtained through a procedure described in Appendix B.

6. REPORT

6.1 Identify the test specimen and the location in the test specimen where the hardness was measured.

6.2 Average the hardness number from a minimum of five readings. Knoop (HK) or Vickers (HV) microindentation hardness shall be reported along with the test load used, e.g., 400 HK 100 gf or 400 HV 100 gf. However, an alternative method, expressing the load in kilograms force may be used in accordance with ISO, e.g., 400 HK 0.1 or 400 HV 0.1. Report HK or HV values to the nearest whole number.

6.3 Magnification.

6.4 Identity of phase measured, or description of phase measured.

6.5 For HRC and other converted measurements, whether conversion was made using a procedure other than described in Appendix B.

6.6 The following supplemental information may also be reported:

6.6.1 Material and processing conditions.

7. PRECISION

7.1 The repeatability limit, r, and reproducibility limit, R, measurements were determined according to ASTM E691, Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods and values are listed below for ten materials and a range of densities. On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory will be expected to exceed, r, only 5% of the time.
If such a difference is found to be larger than, \( r \), there is reason to question one or both results. Similarly, the difference in two test results obtained in different laboratories will be expected to exceed, \( R \), only 5% of the time. If the difference is found to be greater than, \( R \), there is reason to question one or both measurements.

### Table 1. Precision of Knoop and Vickers Microindentation Hardness

<table>
<thead>
<tr>
<th>Material</th>
<th>Density g/cm(^3)</th>
<th># of Labs</th>
<th>Hardness</th>
<th>( r )</th>
<th>( R )</th>
</tr>
</thead>
<tbody>
<tr>
<td>FC-0205</td>
<td>6.39</td>
<td>17</td>
<td>169 HV</td>
<td>42</td>
<td>92</td>
</tr>
<tr>
<td>FN-0205</td>
<td>6.93</td>
<td>17</td>
<td>211 HV</td>
<td>44</td>
<td>111</td>
</tr>
<tr>
<td>FL-4405</td>
<td>6.67</td>
<td>15</td>
<td>258 HV</td>
<td>31</td>
<td>63</td>
</tr>
<tr>
<td>FLN2-4405</td>
<td>6.98</td>
<td>15</td>
<td>268 HV</td>
<td>29</td>
<td>53</td>
</tr>
<tr>
<td>FN-0200</td>
<td>6.67</td>
<td>16</td>
<td>118 HK</td>
<td>17</td>
<td>48</td>
</tr>
<tr>
<td>FD-0208</td>
<td>6.74</td>
<td>16</td>
<td>301 HK</td>
<td>45</td>
<td>103</td>
</tr>
<tr>
<td>FL-4905-HT</td>
<td>6.91</td>
<td>14</td>
<td>721 HV</td>
<td>65</td>
<td>123</td>
</tr>
<tr>
<td>FLC-4905-HT</td>
<td>6.98</td>
<td>14</td>
<td>759 HV</td>
<td>70</td>
<td>140</td>
</tr>
<tr>
<td>FLC-4808-HT</td>
<td>6.70</td>
<td>14</td>
<td>778 HK</td>
<td>51</td>
<td>217</td>
</tr>
<tr>
<td>FL-4808-HT</td>
<td>6.96</td>
<td>14</td>
<td>753 HK</td>
<td>45</td>
<td>240</td>
</tr>
</tbody>
</table>

Repeatability and reproducibility values reported from one specimen, average of three (3) readings per specimen.

NOTE: Precision data determined prior to the requirement for reporting the average of five readings.

### APPENDIX A

**A1.** The method described in this appendix for specimen mounting and preparation is a proven practice. It is recognized that other procedures or materials used in preparation of a sample may be equally as good and used on the basis of availability and preference of individual laboratories.

**A2.** **Specimen Preparation**

**A2.1** Careful preparation and polishing of the specimen is necessary in order to make accurate measurement of diagonal lengths. In porous materials, pores easily become smeared during cutting and grinding. Polishing correctly will open these smeared pores thereby producing an accurate structure.

**A2.2** If pores are left smeared, the indentor will encounter these hidden pores, necessitating extra measurements.

**A3.** **Mounting and Grinding**

**A3.1** Remove a suitable specimen from the test piece, by cutting, using sufficient coolant to protect the specimen from overheating. Excessive heating during cut-off may temper the specimen yielding inaccurate hardness measurements.

**A3.2** Remove any residual coolant or cut-off fluid from the specimen using Soxhlet extraction.

**A3.3** Vacuum impregnate the specimen with epoxy resin and then mount in this same epoxy (e.g., Struers Calofix or Buehler Epoxide). The resin will fill the porosity in the specimen thereby helping to prevent smearing of the porosity and pick up of corrosive chemicals during etching.

**NOTE 3—**The mounting media must be strong enough to support the test specimen under load without deflection.

**A3.4** After curing the epoxy, grind the specimen successively on 240, 400, and 600 mesh wet SiC paper, preferably using a rotating wheel.

**A4.** **Manual Polishing**

**A4.1** Rough polish by hand for 8 to 12 minutes on long napped cloth (e.g. Struers felt cloth) using 1 micrometre alumina at 250 rpm. This procedure removes smeared material and exaggerates the pore area.

**A4.2** The second polishing step returns the pores to their true area fraction. Polish for 4 minutes at 125 rpm using medium napped cloth (e.g. Struers MOL cloth) and 1 micrometre diamond paste.

**A4.3** Final polish for 20-30 seconds on a long napped cloth (e.g. Buehler Microcloth) using 0.05 micrometre de-agglomerated alumina, at 125 rpm.

**A5.** **Automated Polishing**

**A5.1** Rough polish using an automated polisher with 30 N of force per specimen, 3 micrometre diamond paste and 10 minutes of polishing on Buehler Trident cloth or Struers DAC cloth. This should give a very flat surface, out to the edge of the test specimen.

**A5.2** Final polish using an automated polisher for 30 seconds to 1 minute at 3-5 N of force per specimen, 0.05 micrometre alumina suspension, on a long napped cloth such as Buehler Microcloth.

**A6.** **Etching**

**A6.1** Careful etching is necessary as heavy etching obscures phases and changes the microindentation hardness values by interfering with the measurement of the diagonals.

**A6.2** For heat treated steels about 4-7 seconds immersion in 2% nital gives an appropriate structure. Rinse in alcohol and blow dry with clean air. Martensite will be very light and the dark etching non-martensitic transformation products (fine pearlite or upper bainite) will be evident by contrast.

**A6.3** Multi-phase materials should be tested in the lightly etched condition.

### APPENDIX B

**B1.** The following procedure describes a method to convert microindentation hardness values to HRC.

**B2.** **Use of HRC Standard Test Blocks** - Obtain 4 or 5 standard HRC test blocks spanning the range from the low 20’s to the 60’s HRC. Remove a small portion of each block and mount with the standardized face at the surface of the mount and polish the specimens using standard procedures. Using either a Knoop or Vickers indentor, make five (5) sets of indentations at various points in each of the standard specimens. Measure the length in filar units or micrometres for each indentation.

**B3.** **Graphical Conversion** - Prepare a graph with the filar units, micrometres, or Knoop/Vickers micro-indentation hardness number on the y-axis (ordinate)
and HRC on the x-axis (abscissa). Plot all measured diagonals and using regression analysis (regression of y on x), construct a best-fit curve to the data points. In future tests take any diagonal reading and use the graph to convert to HRC.

NOTE 4—The graph that is constructed applies to the specific instrument used for the microindentation hardness test, the test load used, and the person performing the test. A separate graph needs to be plotted for each operator, each test instrument, and for each load used for microindentation hardness testing.

B4. Rockwell C Hardness Conversion Precision - In an interlaboratory study done in 1994 by the Powder Metallurgy Parts Association (PMPA) Standards Committee members of the Metal Powder Industries Federation, using a hardened prealloyed FL-4605 material at 6.8 g/cm³, and 56 average HRC microindentation hardness, the reproducibility between laboratories, looking at the identical specimen, but making their own hardness indentations, was 5.3 HRC points. This means that two laboratories, each making only one measurement, should expect that 95% of the time, their results would agree within 5.3 HRC points; for the average of six readings the reproducibility should be 4.5 HRC points. Within a single laboratory, with one operator, repeatability of single measurements should be within 4.0 HRC points. In any one laboratory, repeatability for the averages of 6 readings should be 1.6 HRC points. These tests were done using the 100 gf load on Knoop indentors. Conversion to HRC was as described above, B1.-B3., using pieces from the same standard HRC blocks.

APPENDIX C

C1. COMPARABLE STANDARDS
ASTM E384
ASTM B933

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The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.
Determination of Effective Case Depth of Ferrous Powder Metallurgy (PM) Products

MPIF Standard 52
(Formerly included in Standard 37)

1. SCOPE

1.1 This standard describes the requirements and inspection methods for measuring the effective case depth of ferrous powder metallurgy products.

1.2 In order to determine effective case depth the procedures for determining the microindentation hardness of powder metallurgy materials, as described in MPIF Standard 51 shall be followed.

1.3 The values stated in inch-pound units are to be regarded as the standard. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. DEFINITION

2.1 Effective Case Depth - shall be defined as that portion of the case that has a microindentation hardness equivalent to or harder than a specified value.

3. APPARATUS

3.1 A microindentation hardness testing machine capable of applying the required load, equipped with a Knoop or Vickers indentor and a provision for measuring the length of the indentations with a precision of 4 micro-inch (0.1 micrometre). The instrument shall have a means for measuring the distance from the surface of the part to the site of the hardness impression with a precision of 0.001 inch (0.02 mm).

4. PROCEDURE

4.1 Prepare the sample for microindentation hardness testing according to the procedures described in MPIF Standard 51. The section to be tested shall be taken perpendicular to the hardened surface.

4.2 Measure the microindentation hardness at increasing depth from the surface using a test load of 100 gf.

4.2.1 Microindentation hardness impressions should not be placed in soft regions such as copper or the centers of nickel-rich austenite regions. Randomly encountered fine pearlite in the martensite should not be excluded as a measurement location. Space the measurements so that the adjacent indentations satisfy the minimum spacing requirements described in MPIF Standard 51.

4.3 Plot the microindentation hardness traverse measurements on a graph of microindentation hardness versus distance from the surface of the test specimen. Plot definition will dictate the number of readings; particularly in the critical region of effective case depth. The effective case depth shall be determined from this graph as the depth from the surface at which the microindentation hardness measurement falls below the equivalent of 50 HRC unless otherwise specified.

4.4 The microindentation hardness value equivalent to 50 HRC shall be determined using the procedure in Appendix B of MPIF Standard 51.

NOTE 1—An alternative method may be used in the case of routine quality control testing.

(1) When a minimum effective case depth is specified, measure the microindentation hardness at a distance from the surface of the test specimen that is equal to or greater than the specified minimum depth. At least five indentations are required. If the average microindentation hardness at this depth is equal to or greater than the effective case depth hardness required, the test specimen meets the specified requirement.

(2) When a maximum effective case depth is specified, measure the microindentation hardness at a distance from the surface of the test specimen that is less than the specified maximum depth in order to confirm the test specimen has been case hardened. At least five indentations are required. The average microindentation hardness shall be equal to or greater than that specified for the effective case hardness. If it is, then measure the microindentation hardness at a distance from the surface of the test specimen that is equal to the specified maximum depth. At least five indentations are required. If the average microindentation hardness is less than the value specified for the effective case hardness then the maximum effective case depth specification is met. If the microindentation hardness value is greater than or equal to the specified value, the maximum effective case depth is exceeded.
5. REPORT

5.1 Type of material and process used to produce the case.

5.1.1 Microindentation hardness measurements versus depth from the surface of the part or whether one of the alternative methods was used to determine compliance with a minimum or maximum effective case depth.

5.1.2 Effective case depth to the nearest 0.005 inch (0.1 mm).

5.1.3 Case microstructure.

5.1.4 Method of microindentation hardness measurement, such as HK or HV and the load applied.

APPENDIX

A1. COMPARABLE STANDARDS

ASTM B934
ISO 4507
SAE J423

6. PRECISION

6.1 The repeatability, $r$, and reproducibility, $R$, limits were determined (1993) according to ASTM E691, Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods. The test sample was prepared from FL-4605 (no graphite added) at 7.1 g/cm$^3$ and then case carburized to develop the desired case/core relationship. The effective case depth was determined at 50 HRC after conversion from direct microindentation hardness values by 7 participating laboratories.

The mean case depth was 0.030 inch (0.7 mm) with a repeatability limit, $r$, of 0.005 inch (0.1 mm) and a reproducibility limit, $R$, of 0.020 inch (0.5 mm). Duplicate results from the same laboratory should not be considered suspect at the 95% confidence level unless they differ by more than 0.005 inch (0.1 mm). For the same test specimen, test results from two different laboratories should not be considered suspect at the 95% confidence level unless they differ by more than 0.020 inch (0.5 mm).

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The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10: “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.

This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE
1.1 This standard describes a method for measuring the internal volume of the apparent density cup used in MPIF Standards 04 and 28.
1.2 The cup, particularly its rim, may become worn during usage and it is recommended that the volume of the cup be checked periodically (at least once per year) in order to ensure it complies with the specified volume of 25 ± 0.03 cm³.
1.3 The values in SI units are to be regarded as the standard. The inch-pound units in parentheses were converted in accordance with IEEE/ASTM SI 10. They may be approximate and are only for information.
1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS
2.1 Density cup: A cylindrical cup (Fig. 1) capacity of 25 ± 0.03 cm³ with an inside diameter of 28 ± 0.5 mm.
2.2 Balance: A balance readable to 0.0001 g, with a capacity of at least 200 g.
2.3 Microscope slide: a transparent slide at least 40 mm wide.
2.4 Water: Distilled or deionized water, boiled 5 minutes.
2.5 Alcohol: Reagent grade ethyl alcohol.
2.6 Wire: A wire not exceeding 2.5 mm (0.10 inch) in diameter by 150 mm (6 inch) long.
2.7 Thermometer: A thermometer capable of measuring the temperature of the water to the nearest 1°C.

3. PROCEDURE
3.1 Boil at least 150 mL of distilled or deionized water for 5 minutes to remove dissolved air. Cool to room temperature and handle so as to avoid the introduction of air bubbles.
3.1.1 Repeat the determination at least three times and calculate the average of the three measurements.
3.2 Thoroughly clean the interior of the density cup by rubbing with a soft, lint-free fabric wetted with reagent grade ethyl alcohol. Ensure that the cup is thoroughly dry before proceeding.
3.3 Weigh the empty density cup along with a clean, clear microscope slide. This is the tare mass T.
3.4 Fill the density cup with the prepared water until the water overflows.
3.5 Dislodge any air bubbles inside the density cup with a clean wire.
3.6 Slide the clean, clear microscope slide horizontally across the rim of the density cup, spilling the excess water over the sides of the cup, and center the microscope slide on top of the cup.
3.7 If any air bubbles appear, remove the microscope slide and repeat the preparation steps beginning at Section 3.4.
3.8 Dry the outside of the density cup and all the exposed surfaces of the microscope slide with paper toweling. Ensure that the absorbency of the toweling does not wick water from inside the cup.
3.9 Weigh the density cup filled with water, including the microscope slide cover. This is the gross mass G.
3.10 Measure the temperature of the water to the nearest 1°C and determine the density of the water, \( \rho_w \), from Table 1.

4. CALCULATIONS
4.1 Calculate the mass of water contained in the density cup from the following formula:

\[
M = G - T
\]

where:
\( M \) = mass of water in the density cup, g
\( G \) = gross mass of density cup, plus water, plus microscope slide, g
\( T \) = tare mass of density cup plus microscope slide, g

4.2 Calculate the volume of the density cup, V from the relationship:

\[
V = \frac{M}{\rho_w}
\]

where:
\( V \) = volume of the density cup, cm³
\( M \) = mass of water in the density cup, g
\( \rho_w \) = density of the water from Table 1, g/cm³
5. REPORT

5.1 Volume of the density cup (average of at least three determinations) to the nearest 0.01 cm³.

6. PRECISION

6.1 The repeatability limit, r, and reproducibility limit, R, of this test method were determined in 2014 by an interlaboratory study in which eight laboratories participated. Six laboratories supplied single data sets while one provided two sets and another three, making a total of eleven data sets. Three density cups were circulated to participants and three repetitions were performed for each cup. The data were analyzed using ASTM E691 and the results are summarized in Research Report: MPPA-R-53-2014.

6.2 The repeatability limit, r, was found to be 0.02 cm³. Duplicate results from the same laboratory (individual determinations prior to averaging) should be considered acceptable at the 95% confidence level unless they differ by more than r, the repeatability interval.

6.3 The reproducibility, R, was found to be 0.04 cm³. Duplicate results from two different laboratories should be considered acceptable at the 95% confidence level unless they differ by more than R, the reproducibility interval.

APPENDIX

A1. COMPARABLE STANDARD
ASTM B873

FIGURE 1: Density Cup (25 ± 0.03 cm³)

Table 1. Effect of Temperature on Water Density

<table>
<thead>
<tr>
<th>Temperature °C</th>
<th>( \rho_w ) g/cm³</th>
<th>Temperature °F</th>
<th>( \rho_w ) g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>0.9991</td>
<td>60</td>
<td>0.9990</td>
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<tr>
<td>15.5</td>
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</table>

The values of \( \rho_w \) shown are taken from, “Metrological Handbook 145, Quality Assurance for Measurements”, 1990, NIST, pp. 9, 10, and represent the values in air at one atmosphere pressure.

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1. SCOPE

1.1 This standard describes a procedure for determining the density of powder metallurgy (PM) materials, including cemented carbides that generally contain less than two percent porosity. The method is based on Archimedes’ principle.

NOTE 1—A test specimen that gains mass when immersed in water indicates the specimen contains surface-connected porosity. Unsealed surface porosity will absorb water and cause density values higher than the true value. If this problem occurs, this method is not applicable and Standard 42 should be used instead.

1.2 With the exception of density, for which g/cm³ is the industry standard, SI units are used in this standard. Values in inch-pound units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.3 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Analytical Balance: A precision analytical balance that will permit readings within 0.01% of the test specimen mass as shown in Table 1.

2.2 Water: Distilled or deionized water to which 0.05 to 0.1 volume percent of a wetting agent has been added to reduce the effects of surface tension.

NOTE 2—Degassing the water by evacuation, boiling or ultrasonic agitation helps to prevent air bubbles from collecting on the test specimen and specimen support when immersed in water.

2.3 Container: A glass beaker or other suitable transparent container should be used to contain the water.

2.4 Test Specimen Support for Weighing in Water - Two typical arrangements are shown in Figure 1. The suspension wire may be twisted around the test specimen or the test specimen may be supported in a wire basket that is attached to the suspension wire. For either arrangement, a single corrosion resistant wire, e.g. austenitic stainless steel, copper, or nichrome shall be used for the basket and suspension wire. The recommended diameter of suspension wire to be used for various mass ranges is shown in Table 2.

Table 2. Wire Diameter Recommendations

<table>
<thead>
<tr>
<th>Specimen Mass (g)</th>
<th>Wire Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 to &lt; 50 grams</td>
<td>0.12 mm [48 gauge] (0.005 inch)</td>
</tr>
<tr>
<td>50 to &lt; 200 grams</td>
<td>0.25 mm [35 gauge] (0.010 inch)</td>
</tr>
<tr>
<td>200 to &lt; 600 grams</td>
<td>0.40 mm [28 gauge] (0.015 inch)</td>
</tr>
<tr>
<td>600 grams &amp; above</td>
<td>0.50 mm [25 gauge] (0.020 inch)</td>
</tr>
</tbody>
</table>

NOTE 5—For the most precise density determinations it is important that the mass and volume of all supporting wires immersed in water be minimized.

2.5 Thermometer - A thermometer with an accuracy of 0.2 °C (0.5 °F) to measure the temperature of the water.

3. TEST SPECIMEN

3.1 For the highest precision, the test specimen shall be a PM part or a section of a part with a minimum mass of 5.0 g. If less precision can be tolerated, several test specimens may be used to reach the minimum mass, provided each test specimen has a mass of not less than 1.0 g.

NOTE 6—For metal injection molded (MIM) parts of less than 1.0 g several parts may be used to reach the minimum mass.
4. **PROCEDURE**

4.1 Clean test specimen surfaces of all adhering foreign materials such as dirt, grease, oil, oxide scale, metal powders or assembly materials. For cut specimens, avoid rough surfaces to which an air bubble can adhere. A 100-grit sanding or abrasive grinding is recommended to remove all rough surfaces.

4.2 Determine the mass of the test specimen in grams as indicated in Table 1. This is mass A. It is important that the test specimen, balance, water and surrounding air be at a uniform temperature when the weighing is performed.

NOTE 7—For the most precise density determination, duplicate weighings should be made for all mass determinations. The balance should be adjusted to zero prior to each weighing. Duplicate mass determinations should be averaged before calculating the density.

NOTE 8—For improved reproducibility, the balance should be calibrated periodically with a standard mass that is approximately equal to the test specimen mass.

4.3 Support the container of water over the pan of the balance using a suitable bridge as shown in Figure 2a. Ensure that the bridge does not touch the balance pan. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose (See Figure 2b). If this arrangement is used, it is important to shield the suspension wire between the container of water and the bottom of the balance from air drafts.

4.4 Suspend the test specimen support with the test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least 6 mm (0.25 inch) to minimize the effect of surface tension forces on the weighing. Care should be taken to ensure that the test specimen and specimen support hang freely from the balance beam hook, are free of air bubbles when immersed in the water and are at the same temperature as the water and balance. Ensure that the surface of the water is free of dust particles.

4.5 Weigh the test specimen and the specimen support immersed in water. This is mass B. The formation of bubbles on the surface of the specimen may indicate that the surface is permeable. If the mass of the specimen increases during the weighing, the specimen is not impermeable. If the mass of specimen increases by more than 0.01% of mass A during the weighing in water, MPIF Standard 42 should be used.

4.6 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before. This is mass C. Ensure that the suspension support is free of air bubbles and that the suspension wire is not immersed below its normal hanging depth as a change in depth will change the measured mass.

NOTE 9—Some balances are capable of being tared. This automatically removes the necessity of reweighing the specimen support every time. In this case, tare the specimen support alone, immersed in water to the same depth as with the specimen, before weighing the specimen support and specimen immersed in water. The mass of the specimen support and specimen immersed in water is mass F, which replaces mass B minus mass C.

4.7 Measure the temperature of the water to the nearest 0.5 °C (1 °F) and record its density \( \rho_w \), at that temperature, from Table 3.

### Table 3. Effect of Temperature on Water Density

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>( \rho_w ) (g/cm³)</th>
<th>Temperature (°F)</th>
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</table>

*The values of \( \rho_w \) shown are taken from, “Metrological Handbook 145, Quality Assurance for Measurements”, 1990, NIST, pp. 9.10, and represent the values in air at one atmosphere pressure.*
5. CALCULATION

5.1 Calculate the density as follows:

\[ D = \frac{\text{Mass}}{\text{Volume}} = \frac{(A \cdot \rho_w)}{(A - (B - C))} = \frac{(A \cdot \rho_w)}{(A - F)} \]

where:
- \( D \) = density of test specimen, g/cm\(^3\)
- \( A \) = mass of test specimen in air, g
- \( B \) = apparent mass of test specimen and specimen support in water, g
- \( C \) = mass of specimen support immersed in water, g
- \( F \) = mass of test specimen in water with mass of specimen support tared, g
- \( \rho_w \) = density of water, g/cm\(^3\)

6. REPORT

6.1 Density rounded to the nearest 0.01 g/cm\(^3\).

7. PRECISION

7.1 The following precision data were developed using the procedures contained in ASTM B311-86. The results of the interlaboratory study that was conducted are summarized in a Research Report, RR B09-1014, on file at ASTM Headquarters.

7.2 The repeatability limit, \( r \), and reproducibility limit, \( R \), were determined at the 95% confidence level.

7.3 Duplicate results (individual determinations prior to averaging) from the same laboratory should not be considered suspect at the 95% confidence level unless they differ by more than \( r \).

7.4 Test results (individual determinations prior to averaging) from two different laboratories should not be considered suspect at the 95% confidence level unless they differ by more than \( R \).

<table>
<thead>
<tr>
<th>Material Density (g/cm(^3))</th>
<th>Specimen Mass (g)</th>
<th>Repeatability (( r ))</th>
<th>Reproducibility (( R ))</th>
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APPENDIX

A.1 COMPARABLE STANDARDS

ASTM B311

ISO 3369

“Figures reprinted, with permission, from ASTM B963 - 08 Standard Test Methods for Oil Content, Oil-Impregnation Efficiency, and Interconnected Porosity of Sintered Powder Metallurgy (PM) Products Using Archimedes’ Principle, copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.”

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This standard covers the apparatus, test specimens and procedures for the radial crush testing of PM materials. This is a destructive test using a hollow, cylindrical specimen.

1.2 The test as described and specified herein is applicable only to materials of negligible ductility. If applied to a material showing noticeable plastic deformation before fracture, the test results should be used only for comparison of similar materials.

1.3 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion from IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Gauge(s) capable of measuring the specimen to the nearest 0.001 inch (0.02 mm).

2.2 Compression testing machine readable to 0.1% of the full-scale reading. Use the lowest range that can produce a measurable result.

2.3 Loading plates—ground, flat, hardened steel of a hardness greater than the microindentation hardness of the material being tested and large enough to encompass the entire length of the specimen.

3. TEST SPECIMEN

3.1 The specimen preferably should be pressed in tooling suitable to produce a ring-shaped specimen where, t, the wall thickness [(D-d)/2] is less than 30% of the test specimen outer diameter. If a test specimen geometry is selected where the wall thickness is greater than 30% of the specimen outer diameter, the test results may only be used as a guide for comparison with similar materials.

The preferred dimensions for this specimen are:
- Outer diameter, D 0.80-2.00 inch (20-51 mm)
- Inner diameter, d 0.50-1.00 inch (13-25 mm)
- Length, L 0.25-1.00 inch (6-25 mm)

3.2 Flanged bearings shall be tested by cutting off the flange and compressing the two sections separately.

3.3 Spherical bearings shall be machined to plain cylindrical shape.

NOTE 1—The specimen may be machined from a part or blank; however, the test result may be different from a pressed specimen.

NOTE 2—For specimen dimensions other than those listed above, the tolerances should be agreed between the concerned parties.

4. PROCEDURE

4.1 Prepare the test specimen per MPIF Standard 60 or machine the test specimen from a suitable PM product.

4.2 Measure the outside diameter, inside diameter, and the length of the test specimen to the nearest 0.001 inch (0.02 mm).

4.3 The test specimen diameters shall be round within 0.003 inch per inch (0.003 mm per mm).

4.4 Clean any surface oil or other contaminants from the test specimen and the flat surfaces of the loading plates.

4.5 Position the test specimen in the central region of the lower loading plate in the compression testing machine.

4.6 Bring the upper loading plate into contact with the test specimen and slowly apply the diametrical load at a constant crosshead closure rate that does not exceed 0.2 inch/min (5.0 mm/min)—see Fig. 1.

4.7 Use a scale on the compression testing machine that has a precision of 0.1% or better of the crushing load and record, to the precision listed in Table 1, the load at which the test specimen fractures or the first reading at which the applied load drops.

<table>
<thead>
<tr>
<th>Inch-pound (lbf)</th>
<th>SI (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(D – t)/L²</td>
<td>Record Break Load to</td>
</tr>
<tr>
<td>&lt; 100</td>
<td>± 10</td>
</tr>
<tr>
<td>&gt; 100</td>
<td>± 1</td>
</tr>
</tbody>
</table>

Table 1. Precision for the Measurement of the Crushing Load


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5. CALCULATION

5.1 Calculate the radial crush strength, K from the following equation:

\[ K = \frac{P}{L} \left[ \frac{D + d}{2} \right] \]  

where:

- K = radial crush strength, psi (MPa)
- P = radial crush force, pounds force (N)
- D = outside diameter, inch (mm)
- d = inside diameter, inch (mm)
- L = length, inch (mm)
- t = wall thickness, inch (mm)

6. REPORT

6.1 Radial crush strength shall be reported to the nearest 1,000 psi (10 MPa), as the average value from at least three (3) but preferably five (5) test specimens.

6.2 The following supplemental information may also be reported:

6.2.1 Chemical composition of the powder mix.

6.2.2 Type, brand, and percent of lubricant.

6.2.3 Density.

6.2.4 Apparent hardness.

6.2.5 Sintering temperature.

6.2.6 Sintering time.

6.2.7 Furnace atmosphere.

6.2.8 Final total carbon content, where applicable.

6.2.9 Other processing information, as required.

6.2.10 Any deviation from this test method.

7. PRECISION

7.1 The repeatability limit, r, and reproducibility limit, R, measurements were determined according to ASTM E691, Practice of Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods and are listed below for three materials and a range of radial crush strength values. On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory will be expected to exceed, r, only 5% of the time. If such a difference is found to be larger than, r, there is reason to question one or both results. Similarly, the difference in two test results obtained in different laboratories will be expected to exceed, R, only 5% of the time. If the difference is found to be larger than, R, there is reason to question one or both measurements.

### Table 2.

<table>
<thead>
<tr>
<th>Material</th>
<th>Dry Density g/cm³</th>
<th>D inch</th>
<th>d inch</th>
<th>L inch</th>
<th># labs</th>
<th>Radial Crush Strength psi</th>
<th>r psi</th>
<th>R psi</th>
</tr>
</thead>
<tbody>
<tr>
<td>CTG-1001-K23</td>
<td>6.56</td>
<td>1.00</td>
<td>0.75</td>
<td>0.75</td>
<td>9</td>
<td>31,000</td>
<td>2,200</td>
<td>3,300</td>
</tr>
<tr>
<td>FC-1000-K20</td>
<td>5.96</td>
<td>1.00</td>
<td>0.75</td>
<td>0.75</td>
<td>9</td>
<td>58,000</td>
<td>4,900</td>
<td>6,600</td>
</tr>
<tr>
<td>FC-0208-50</td>
<td>6.76</td>
<td>2.00</td>
<td>0.875</td>
<td>0.75</td>
<td>10</td>
<td>114,000</td>
<td>6,900</td>
<td>6,900</td>
</tr>
</tbody>
</table>

### APPENDIX

A1. COMPARABLE STANDARDS

- ASTM B939
- ISO 2739

![FIGURE 1: Schematic of the Arrangement for Loading the Test Specimen](image)

**Disclaimer**

By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability. MPIF-standards are adopted in the public interest and are designed to eliminate misunderstandings between the producer and the purchaser and to assist the purchaser in selecting and obtaining the proper material for his particular product. Existence of an MPIF standard does not in any respect preclude any MPIF member or non-member from manufacturing or selling products not included in this standard or from utilizing procedures or equipment other than those included in this standard.

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1. SCOPE

1.1 This method applies to ferrous materials which have an endurance limit. The endurance limit is that stress below which the material will not fail in $10^7$ cycles.

1.1.1 The method may be used to measure the fatigue strength of nonferrous materials which do not have an endurance limit.

1.2 This standard uses the staircase method to analyze the data and determine statistically the mean endurance limit as well as estimating the standard deviation and the stresses for which 10% and 90% of test bars would survive for $10^7$ cycles at the 50% confidence level. See Appendix.

1.3 The specimens may be as-sintered or heat treated.

1.4 This test method uses an unnotched test specimen and prescribes its design. A notched test specimen could be used, but the shape and location of the notch must be reported with the data.

1.5 In a rotating beam machine, the specimen is gripped between two collets and subjected to a bending stress as it rotates. During each revolution, the stress in the outer surface of the reduced diameter gauge region alternates from tension to compression and back to tension, $R = -1$.

NOTE 1—There is no precise relationship between rotating beam endurance data and axial fatigue data. A correlation analysis between axial fatigue limit and rotating beam fatigue limit found the axial fatigue limit to be 80–90% of the rotating beam fatigue limit.

1.6 With the exception of density, for which $g/cm^3$ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.7 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 A calibrated rotating beam fatigue apparatus with a loading capacity of at least 200 lbf·in (22 N·m) is recommended for ferrous materials.

2.1.1 Appropriate collets to grip the specimen.

2.1.2 Rotational speed variable up to at least 10,000 revolutions per minute with a counter up to 500,000,000 cycles.

2.1.3 Automatic switch to turn off test machine when the sample breaks.

2.1.4 Dial gauge indicator and stand, readable to 0.0001 inch (0.002 mm).

2.1.5 Instrument to measure revolutions per minute

3. TEST SPECIMEN

3.1 A recommended test specimen configuration is shown in Fig. 1. The preparation sequence is slightly different for as-sintered or heat-treated test specimens.

3.2 A minimum of 25 test specimens are recommended to obtain the endurance limit by the staircase method.

As-Sintered Ferrous and Non-Ferrous Materials

3.3 The test specimens shall be machined from pressed and sintered compacts of sufficient size to give a bar that is 0.40–0.43 inch by 0.40–0.43 inch (10–11 mm by 10–11 mm) in cross-section and at least 3 inches (75 mm) long. The compacts shall be prepared in accordance with MPIF Standard 60.

NOTE 2—Test specimens may also be machined from as-sintered PM parts provided they are of sufficient size.

3.3.1 Turn the bars to a 0.390 inch (9.90 mm) diameter on centers.

3.3.2 Machine the bar to achieve a 1.375 inch (34.93 mm) radius, with a 0.200 inch (5.08 mm) diameter at the center of the test specimen removing about 0.015 inch (0.375 mm) of material per pass.

3.3.3 Radius the bar to about a 0.189 inch (4.80 mm) diameter at the center of the test specimen removing material at a rate of no more than 0.0002 inch (0.005 mm) per pass.

3.3.4 Machine the end regions of the test specimen to the final 0.375 inch (9.52 mm) diameter.

3.3.5 Polish the central section of the test specimen (longitudinal direction) to a diameter of 0.188 inch (4.78 mm) and a surface finish of 8 microinch (0.2 μm) Ra or better.
NOTE 3—The surface finish may be achieved by mechanically polishing in a longitudinal direction on all sides using successively #240, #400, #500 and #600 grades of emery paper. Extreme caution should be used in polishing to ensure that material is being properly removed rather than merely smeared to produce a smooth surface. This is a particular danger in soft materials as material can be smeared over tool marks, thereby creating a potentially undesirable influence on crack initiation during testing.

NOTE 4—The test section shall be free of nicks, dents, and scratches, and circumferential tool marks visible at 20x magnification.

3.3.6 Stress relieve the test specimens at 350 °F (180 °C), or some other agreed upon temperature, for 1 h.

**Quench-Hardened and Tempered Ferrous Materials**

3.4 The test specimens shall be machined from pressed and sintered compacts of sufficient size to give a bar that is 0.40–0.43 inch by 0.40–0.43 inch (10–11 mm by 10–11 mm) in cross-section and at least 3 inches (75 mm) long. The compacts shall be prepared in accordance with MPIF Standard 60.

NOTE 5—Test specimens may be machined from heat-treated PM parts provided they are of sufficient size. Test specimens, cut from a heat-treated PM part and machined to the specified test piece dimensions, should be stress relieved at 350 °F (180 °C) or some other agreed upon temperature, for 1 h.

3.4.1 Turn the bars to a 0.390 inch (9.90 mm) diameter on centers.

3.4.2 Austenitize at the appropriate temperature for the alloy and the carbon content of the material, and quench into an agreed appropriate, agitated quenchant.

NOTE 6—The specimens should be suspended vertically during quenching to minimize distortion.

3.4.3 Temper for 1 h at the temperature recommended for the material.

3.4.4 Machine the bar to achieve a 1.375 inch (34.93 mm) radius, with a 0.200 inch (5.08 mm) diameter at the center of the test specimen removing about 0.015 inch (0.375 mm) of material per pass.

NOTE 7—Use coolant during machining to prevent overheating of the test specimen.

3.4.5 Radius the bar to about a 0.189 inch (4.80 mm) diameter at the center of the test specimen removing material at a rate of no more than 0.0002 inch (0.005 mm) per pass.

3.4.6 Machine the end regions of the test specimen to the final 0.375 inch (9.52 mm) diameter.

3.4.7 Polish the central section of the test specimen (longitudinal direction) to a diameter of 0.188 inch (4.78 mm) and a surface finish of 8 microinch (0.2 μm) Ra or better.

NOTE 8—The surface finish may be achieved by mechanically polishing in a longitudinal direction on all sides using successively #240, #400, #500, and #600 grades of emery paper. Extreme caution should be used in polishing to ensure that material is being properly removed rather than merely smeared to produce a smooth surface.

NOTE 9—The test section shall be free of nicks, dents, and scratches, and circumferential tool marks visible at 20x magnification.

3.4.8 Stress relieve the test specimens at 350 °F (180 °C), or some other agreed upon temperature, for 1 h.

**Sinter-Hardened Ferrous Materials**

3.5 The test specimens shall be machined from pressed and pre-sintered compacts of sufficient size to cut a bar that is 0.40–0.43 inch by 0.40–0.43 inch (10–11 mm by 10–11 mm) in cross-section and at least 3 inches (75 mm) long. The compacts shall be prepared in accordance with MPIF Standard 60.

NOTE 10—Test specimens may be machined from sinter-hardened PM parts provided they are of sufficient size. Test specimens, cut from a sinter-hardened PM part and machined to the specified test piece dimensions, should be stress relieved at 350 °F (180 °C) or some other agreed upon temperature, for 1 h.

3.5.1 Pre-sinter the compacts at 1,500–1,600 °F (815–870 °C) in an inert or reducing atmosphere for 15–20 minutes, unless otherwise agreed.

3.5.2 Turn the bar to a 0.390 inch (9.90 mm) diameter on centers.

3.5.3 Sinter harden the machined, pre-sintered bars using the appropriate conditions for the material.

3.5.4 Temper for 1 h at the temperature recommended for the material.

3.5.5 Machine the bar to achieve a 1.375 inch (34.93 mm) radius, with a 0.200 inch (5.08 mm) diameter at the center of the test specimen removing about 0.015 inch (0.375 mm) of material per pass.

NOTE 11—Use coolant during machining to prevent overheating of the test specimen.

3.5.6 Radius the bar to about a 0.189 inch (4.80 mm) diameter at the center of the test specimen removing material at a rate of no more than 0.0002 inch (0.005 mm) per pass.

3.5.7 Machine the end regions of the test specimen to the final 0.375 inch (9.52 mm) diameter.

3.5.8 Polish the central section of the test specimen (longitudinal direction) to a diameter of 0.188 inch (4.78 mm) and a surface finish of 8 microinch (0.2 μm) Ra or better.

NOTE 12—The surface finish may be achieved by mechanically polishing in a longitudinal direction on all sides using successively #240, #400, #500 and #600 grades of emery paper. Extreme caution should be used in polishing to ensure that material is being properly removed rather than merely smeared to produce a smooth surface.

NOTE 13—The test section shall be free of nicks, dents, and scratches, and circumferential tool marks visible at 20x magnification.
3.5.9 Stress relieve the test specimens at 350 °F (180 °C), or some other agreed upon temperature, for 1 h.

4. PROCEDURE

4.1 Follow the instructions provided by the machine manufacturer while taking care to measure the concentricity on the inserted test specimen to the tolerances required. The recommended rotational speed of the machine is 10,000 rpm so that 10⁷ cycles can be accomplished in about 16 hours. The rotational speed shall not exceed 11,000 rpm.

4.2 To determine a stress level to begin the testing, a value of the fatigue endurance limit for standardized materials can be found in MPIF Standard 35-SP, “Materials Standards for PM Structural Parts”. For other materials, an estimate of 0.3 to 0.5 times the known tensile strength of the material is recommended. It is generally best to start at a higher stress level until the turn-around (change from failure to run out) is found.

4.3 Calculate the bending moment to be applied by the test machine, using the machine manufacturer’s formula relating stress to bending moment, and from the estimate of the endurance limit and the diameter of the test specimen. An example of one such formula is:

\[ M = \pi \sigma_s D^3/32 \]

where:
- \( M \) = moment arm setting
- \( \sigma_s \) = applied stress (at reduced section)
- \( D \) = bar diameter

4.4 In the staircase method of testing, if the first specimen fails in less than 10⁷ cycles, the stress for the subsequent specimen is decreased by a certain step, d. Conversely, if the specimen survives a stress, the next specimen is tested at a stress higher by the same step.

4.5 The choice of step size, d, depends on the standard deviation, s, of the applied stress, which may not be known at the outset (see equation in Appendix, A17). The step size, d, should be between 0.5s and 2s. For sintered steels with tensile strengths less than 100,000 psi (689 MPa), a step size of 1,500 psi (10 MPa), is a reasonable choice, if no other data are available. For tensile strengths over 100,000 psi (689 MPa), a step size of 3,000 psi (20 MPa) is reasonable. To conserve specimens, a step size of 3,000 psi (20 MPa) to 6,000 psi (40 MPa) may be used until a run out is found.

NOTE 14—Too large a step size results in successive tests bouncing between run out and failure while too small a step size results in a wandering staircase curve.

4.6 The data for failures and run-outs should be evaluated by the staircase procedure as shown in Table 1 and described in the Appendix. The step size, the applied stresses, and the number of failures and run-outs at each stress level are used to calculate the mean endurance limit and the 10% and 90% survival stresses.

4.7 Disregard the specimen if failure is outside the gauge length and retest a new specimen at the same stress.

4.8 Do not reuse specimens that do not fail.

5. REPORT

5.1 The mean endurance limit (50% survival) and the 10% and 90% survival stresses to the nearest 1,000 psi (10 MPa).

5.2 The following supplemental information may also be reported:

5.2.1 Chemical composition of the powder mix and alloying method used.

5.2.2 Type, brand, and percent of lubricant.

5.2.3 Density (as-sintered or heat-treated).

5.2.4 Apparent hardness.

5.2.5 Sintering temperature.

5.2.6 Sintering time.

5.2.7 Furnace atmosphere.

5.2.8 Final total carbon content, where applicable.

5.2.9 Other processing information, as required such as the heat-treatment conditions or the cooling rate and tempering conditions for sinter-hardened materials.

5.2.10 Any deviation from this test method.

6. PRECISION

6.1 Precision is affected by error in the determination of stress from the specimen load geometry, the repeatability of application of stress during operation of the test process, variation of fatigue properties from specimen to specimen (due to material and to specimen preparation), statistical analysis of the test data is not available for PM materials at this time. An analysis of the staircase method itself is available in SAE Technical Paper No. 2005-01-0803. The method provides a good estimate of the mean value.

APPENDIX

A1. Statistical Analysis of Fatigue Data by the Staircase Method (See Table 1).

A2. \( \sigma_s \) = the stress amplitude in the rotating beam test. The stress amplitudes for the series of tests are separated by a fixed amount, d, expressed in 10⁴ psi.

A3. For all tests run, enter values of \( \sigma_s \) in column 1, in decreasing order of stress, from top to bottom.

A4. Under column heading 2, sample number, indicate if the sample has failed, using an X, or did not fail before 10⁷ cycles, an O. Enter the number of test cycles for each specimen at the bottom of Table 1.

A5. Columns 3 and 4, indicate the total number of failures (column 3) and no failure (column 4) for each stress amplitude level shown in column 1.

A6. Add up the numbers in column 3 and column 4 and place at the bottom of the column.

A7. Whichever column, 3 or 4, has the lower total sum, is called the “Min.” column. Mark that name at the bottom of the appropriate column.
A8. $\sigma_{ao}$ = the lowest stress amplitude in column 1 for which there is a corresponding X or O in the designated Min. column, 3 or 4. In the Min. column, find the cell with the lowest $\sigma_a$ value that has an X or O, and then move across to column 1 to find $\sigma_{ao}$.

A9. $d$ = the step or increment in stress between adjacent stress levels, $\sigma_a$.

A10. Fill in column 5, $z$ = order number of the step, $z = 0$ applies to the step at the level of $\sigma_{ao}$. $z = 1$ applies at the next higher step, $z = 2$ applies at the second higher step, etc.

A11. Fill in column 6, $f$, frequency, from the event totals shown in the Min. column, at the same stress level, $\sigma_a$.

A12. Calculate $z * f$, and $z^2 * f$, columns 7 and 8 respectively.

A13. Calculate the sum of column 6, = N.

A14. Calculate the sum of column 7, = A.

A15. Calculate the sum of column 8, = B.

A16. Calculate $\sigma_A$ = endurance limit, reversed rotational bending, qualified by the percentage success, e.g. $\sigma_{A,50}$.

$\sigma_{A,50} = \sigma_{ao} + d(A/N \pm 0.5)$ (50% pass $\sigma_A$ @ 10⁷ cycles)

where:
+ if Minimum column is 4
– if Minimum column is 3

A17. $s$ = standard deviation of the applied stress $\sigma_A$, recorded by X or O in the minimum value column, in 10⁷ psi.

$s = 1.62 \times d \left[ \frac{(NB-A^2)}{N^2} + 0.029 \right]$  

This equation provides an approximate value for the standard deviation and is valid only when $(NB-A^2)/N^2 > 0.3$. If $(NB-A)/N^2 < 0.3$, set $s = 0.75 \times d$.

A18. Select the t value from the table. N is the number of test bars from the minimum column, i.e., the sum of column 6. Use t to calculate the upper and lower survival stress limits.

A19. $\sigma_{A,10} = \sigma_A + (t * s)$  

$\sigma_{A,10}$ is a 50% confidence estimate that 10% of the test bars will pass 10⁷ cycles.

$\sigma_{A,90} = \sigma_A - (t * s)$  

$\sigma_{A,90}$ is a 50% confidence estimate that 90% of the test bars will pass 10⁷ cycles.
Table 1.
Calculation of the Endurance Limit by the Staircase Method, (1,2,3)

Samples, Sample Identification: ____________________

<table>
<thead>
<tr>
<th>1</th>
<th>(X = Failure)</th>
<th>2</th>
<th>(O = No Failure)</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
<td>7</td>
<td>8</td>
<td>9</td>
<td>10</td>
</tr>
</tbody>
</table>

\[ \sigma_{ao} = 10^3 \text{ psi} \]
\[ \sigma_A = \sigma_{ao} + d \left[ \frac{A}{N} \pm 0.5^* \right] = 10^3 \text{ psi} \]

*Use: + if minimum value comes from column 4,
- if minimum value comes from column 3,
either, if value in column 3 = value in column 4.

The minimum value column is the one with lowest sum at the bottom.

\[ s = 1.62 \times d \left[ \frac{(NB-A^2)}{N^2 + 0.029} \right] = 10^3 \text{ psi} \]
\[ \sigma_A 10\% = \sigma_A + (t \times s) = 10^3 \text{ psi} \]

\[ \sigma_{A, 50} = \sigma_{ao} + d \left[ \frac{A/N \pm 0.5^*}{N} \right] = \text{ } 10^3 \text{ psi} \]
\[ \sigma_A 90\% = \sigma_A - (t \times s) = \text{ } 10^3 \text{ psi} \]
\[ \sigma_A = \text{ alternating stress amplitude, } 10^3 \text{ psi} \]
\[ f = \text{ same frequency as in the minimum column (col. 3 or 4) } \]
\[ \sigma_{ao} = \text{ lowest stress amplitude, } 10^3 \text{ psi, from the column called minimum, i.e., the lowest stress where a result, } X \text{ or } O, \text{ shows in minimum value column. } \]
\[ \sigma_A = \text{ endurance limit, reversed rotational bending, qualified by the percentage success, } 10^3 \text{ psi} \]
\[ d = \text{ step size, } 10^3 \text{ psi} \]
\[ z = \text{ order number of step, } z = O \text{ at } \sigma_{ao} \]
\[ z = 1 \text{ at next step above } \sigma_{ao}, \text{ etc. } \]
\[ s = \text{ standard deviation of the applied stresses, } \sigma_a \text{ recorded by } X \text{ or } O \text{ in minimum value column. } \]
\[ t = \text{ } t \text{ value from table (for only an upper or lower limit) } \]
\[ N = \text{ the number of test bars whose results show in the minimum value column, i.e., the sum of column 6. } \]
\[ A = \text{ sum of column 7 } \]
\[ B = \text{ sum of column 8 } \]
\[ df = \text{ degrees of freedom } = (N-1) \]


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### Table 2. Example of Calculation

**Calculation of the Endurance Limit by the Staircase Method**

**Samples, Sample Identification:**

<table>
<thead>
<tr>
<th>1</th>
<th>(X = Failure)</th>
<th>2</th>
<th>(O = No Failure)</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>Y</td>
<td>C</td>
<td>L</td>
<td>E</td>
<td>S</td>
<td>X</td>
<td>O</td>
<td>X</td>
<td>O</td>
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<tr>
<td>762,000</td>
<td>1,120,000</td>
<td>907,000</td>
<td>12,060,000</td>
<td>10,920,000</td>
<td>5,100,000</td>
<td>892,000</td>
<td>1,364,000</td>
<td>10,811,000</td>
<td>890,000</td>
</tr>
<tr>
<td>11,642,000</td>
<td>10,778,000</td>
<td>979,000</td>
<td>11,642,000</td>
<td>10,778,000</td>
<td>979,000</td>
<td>11,642,000</td>
<td>10,778,000</td>
<td>979,000</td>
<td>11,642,000</td>
</tr>
</tbody>
</table>

**Insert the sums of columns 3, 4, 6, 7, 8 --------->**

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<td>FAILURE</td>
<td>STEP</td>
<td>ORDER</td>
</tr>
<tr>
<td>A</td>
<td>B</td>
<td>N</td>
<td></td>
</tr>
</tbody>
</table>

\[
\begin{align*}
\sigma_0 &= 36 \times 10^3 \text{ psi} \\
\sigma_A &= \sigma_0 + d [A/N \pm 0.5^*] = 39.5 \times 10^3 \text{ psi} \\
\sigma_A, 90% &= \sigma_A - (t \times s) = 35.3 \times 10^3 \text{ psi} \\
\sigma_A, 50% &= \sigma_0 + d [A/N \pm 0.5^*] = 39.5 \times 10^3 \text{ psi} \\
\sigma_A, 10% &= \sigma_A + (t \times s) = 43.7 \times 10^3 \text{ psi} \\
\end{align*}
\]

\[s = 1.62 \times d [(N\cdot B - A^2) / N^2 + 0.029] = 2.84 \times 10^3 \text{ psi}
\]

| Student's t, 90%  
(1/2) | N | df | t |
<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>3</td>
<td>1.638</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>1.533</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>5</td>
<td>1.476</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>6</td>
<td>1.440</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>7</td>
<td>1.415</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>8</td>
<td>1.397</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>9</td>
<td>1.383</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>10</td>
<td>1.372</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>11</td>
<td>1.363</td>
<td></td>
</tr>
</tbody>
</table>

---

FIGURE 1: Recommended Test Specimen Configuration

NOTES:
(a) Diameters (D) 0.375 inch (9.52 mm) and (d) 0.188 inch (4.76 mm) to be concentric within 0.001 inch (0.025 mm)
(b) Test section shall be free of nicks, dents and scratches, and circumferential tool marks visible at 20X magnification. The gauge region of the ferrous specimen may be finished by longitudinal polishing to a surface finish of 8 microinch (0.2 µm) Ra or better.
(c) Diameter, d < D/2

<table>
<thead>
<tr>
<th>Diameter D</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>R</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.375 inch</td>
<td>1.000 inch</td>
<td>2.955 inch</td>
<td>1.000 inch</td>
<td>1.375 inch</td>
</tr>
<tr>
<td>9.52 mm</td>
<td>25.40 mm</td>
<td>75.00 mm</td>
<td>25.40 mm</td>
<td>34.93 mm</td>
</tr>
</tbody>
</table>

Tolerances A and B ± 0.200 inch (0.500 mm), C ± 0.050 inch (0.127 mm), D ± 0.001 inch (0.025 mm), R is reference.

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
**1. SCOPE**

1.1 This standard describes a method for measuring the oil content, surface-connected porosity and oil-impregnation efficiency of powder metallurgy products having surface-connected porosity.

1.2 Oil content as received, \((P_i)\), is the percentage of oil by volume of a sintered PM part.

1.3 Surface-connected porosity, \((P)\), is the percentage of surface-connected porosity by volume of a sintered PM part determined by the amount of oil impregnation.

**NOTE 1—The surface-connected porosity \((P)\) is the same as the fully impregnated oil content \((P)\).**

1.4 Oil-impregnation efficiency, \((I_o)\), is calculated by dividing the as-received oil content \((P_i)\) by the fully impregnated oil content \((P)\) and expressing the result as a percentage.

1.5 With the exception of density, for which g/cm\(^3\) is the industry standard, SI units are used in this standard. Values in inch-pound units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.6 **This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.**

**2. APPARATUS**

2.1 Analytical balance: A precision analytical balance that will permit readings within 0.01% of the test specimen mass as shown in Table 1. NOTE: It is important to shield the balance pan, beakers and suspension wires from the effects of air drafts.

2.2 Container: A beaker or vessel, suitable for holding water, of sufficient volume to immerse specimens of various sizes.

2.3 Thermometer: To determine the water temperature to the nearest 1 °C (2 °F).

2.4 Soxhlet extractor with oil solvent.

2.5 Impregnation oil of known density. Match the oil that was used to originally impregnate the part. If it is not impregnated, then use an oil that has been found suitable [Kinematic viscosity of 65 cSt maximum (300 SUS) at 38 °C (100 °F)].

**Table 1. Balance Requirements**

<table>
<thead>
<tr>
<th>Specimen Mass (g)</th>
<th>Balance Readability (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;10</td>
<td>0.0001</td>
</tr>
<tr>
<td>10 to &lt;100</td>
<td>0.001</td>
</tr>
<tr>
<td>100 to &lt;1,000</td>
<td>0.01</td>
</tr>
<tr>
<td>1,000 to &lt;10,000</td>
<td>0.1</td>
</tr>
</tbody>
</table>

2.6 Test Specimen Support for Weighing in Water: Two typical arrangements are shown in Fig. 1. The suspension wire may be twisted around the test specimen or the test specimen may be supported in a wire basket. For either arrangement, a single corrosion resistant wire, e.g. austenitic stainless steel, copper or nichrome should be used for the basket and the suspension wire. The recommended diameter of suspension wire to be used for various mass ranges is shown in Table 2.

<table>
<thead>
<tr>
<th>Specimen Mass</th>
<th>Wire Diameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 50 grams</td>
<td>0.12 mm [48 gauge] (0.005 inch)</td>
</tr>
<tr>
<td>50 to &lt; 200 grams</td>
<td>0.25 mm [35 gauge] (0.010 inch)</td>
</tr>
<tr>
<td>200 to &lt; 600 grams</td>
<td>0.40 mm [28 gauge] (0.015 inch)</td>
</tr>
<tr>
<td>600 grams &amp; above</td>
<td>0.50 mm [25 gauge] (0.020 inch)</td>
</tr>
</tbody>
</table>

2.7 Water: Distilled or deionized and preferably degassed water to which 0.05 to 0.1 volume % of a wetting agent has been added. The values, \(p_w\), shown in Table 3 should be used for the density of distilled water.

**3. TEST SPECIMEN**

3.1 The test specimen shall be a PM part or group of parts or a section of a part with a minimum mass of 1 g.

**4. DETERMINATION OF OIL CONTENT**

4.1 This procedure is used to determine the oil content of PM components.

4.2 Determine the mass of the as-received test specimen in grams as indicated in Table 1. This is mass \(J\).
4.3 In order to determine the volume of the test piece by water displacement accurately, the specimens are oil impregnated or the pores are filled with a suitable material. Determine the mass of the fully impregnated test specimen. To determine the mass of the fully impregnated test specimen, oil impregnate by either of the following methods:

4.3.1 Preferred Procedure: Immerse the specimen in oil at room temperature. Reduce the pressure over the specimen to 7 kPa (1 psi) or less for 30 minutes, then increase the pressure back to atmospheric pressure for a period of at least 30 minutes keeping the specimen immersed during the entire period. For higher viscosity oil, the oil can be heated.

4.3.2 Alternative Procedure: Immerse the specimen in oil at a temperature of 82 °C +/- 5 °C (180 °F +/- 10 °F) for at least 4 hours. Cool by immersing in oil at room temperature for at least 10 minutes.

4.4 Remove excess oil by wiping gently with an absorbent, lintless material. Take care not to extract the oil absorbed within the part. Determine the mass of the fully impregnated test specimen in grams as indicated in Table 1. This is mass B.

4.5 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2a. Take care to ensure that the bridge does not restrict the free movement of the balance pan. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, it is important to shield the suspension wire between the container of water and the bottom of the balance from the effect of air drafts.

4.6 Suspend the test specimen support with the test specimen from the beam hook of the balance. The water should cover any wire twists and the suspension support basket by at least 6 mm (0.25 in.) to minimize the effect of surface tension forces on the weighing. Take care to ensure that the test specimen and specimen support hang freely from the balance beam hook, are free from air bubbles when immersed in the water and are at the same temperature as the water and balance. Take care to ensure that the surface of the water is free of dust particles.

4.7 Weigh the test specimen and the specimen support immersed in water. This is mass C.

4.8 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before. This is mass E. Take care to ensure that the specimen support is free from air bubbles when immersed in the water and that the suspension wire is not immersed below its normal hanging depth as a change in the depth will change the measured mass.

4.9 Measure the temperature of the water to the nearest 1 °C (2 °F) and record its density ρ_w, at that temperature, from Table 3.

4.10 Determine the mass of the dry test specimen in grams as indicated in Table 1. This is mass A. To determine this mass, remove the oil from the test specimen by either of the following procedures:

4.10.1 Remove the oil in a Soxhlet apparatus using a suitable solvent such as petroleum ether. After extraction, residual solvent shall be removed by heating the test specimens to 20 °C (36 °F) above the boiling point of the solvent. Alternate extraction and drying shall be continued until the dry mass, A, is constant within 0.05%. For large, high density parts, an aggressive solvent may be required.

4.10.2 A practical and fast method of oil removal for most materials is by heating the test specimens in a protective atmosphere in the temperature range of 425 °C to 870 °C (800 °F to 1,600 °F). This method which results in values in close agreement with those obtained using the Soxhlet apparatus may be used if

---

**Table 3. Effect of Temperature on Water Density**

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>ρ_w (g/cm³)</th>
<th>Temperature (°F)</th>
<th>ρ_w (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>15</td>
<td>0.9991</td>
<td>60</td>
<td>0.9990</td>
</tr>
<tr>
<td>15.5</td>
<td>0.9990</td>
<td>61</td>
<td>0.9989</td>
</tr>
<tr>
<td>16</td>
<td>0.9989</td>
<td>62</td>
<td>0.9988</td>
</tr>
<tr>
<td>16.5</td>
<td>0.9988</td>
<td>63</td>
<td>0.9987</td>
</tr>
<tr>
<td>17</td>
<td>0.9988</td>
<td>64</td>
<td>0.9986</td>
</tr>
<tr>
<td>17.5</td>
<td>0.9987</td>
<td>65</td>
<td>0.9985</td>
</tr>
<tr>
<td>18</td>
<td>0.9986</td>
<td>66</td>
<td>0.9984</td>
</tr>
<tr>
<td>18.5</td>
<td>0.9985</td>
<td>67</td>
<td>0.9983</td>
</tr>
<tr>
<td>19</td>
<td>0.9984</td>
<td>68</td>
<td>0.9982</td>
</tr>
<tr>
<td>19.5</td>
<td>0.9983</td>
<td>69</td>
<td>0.9981</td>
</tr>
<tr>
<td>20</td>
<td>0.9982</td>
<td>70</td>
<td>0.9980</td>
</tr>
<tr>
<td>20.5</td>
<td>0.9981</td>
<td>71</td>
<td>0.9978</td>
</tr>
<tr>
<td>21</td>
<td>0.9980</td>
<td>72</td>
<td>0.9977</td>
</tr>
<tr>
<td>21.5</td>
<td>0.9979</td>
<td>73</td>
<td>0.9975</td>
</tr>
<tr>
<td>22</td>
<td>0.9978</td>
<td>74</td>
<td>0.9974</td>
</tr>
<tr>
<td>22.5</td>
<td>0.9976</td>
<td>75</td>
<td>0.9973</td>
</tr>
<tr>
<td>23</td>
<td>0.9975</td>
<td>76</td>
<td>0.9972</td>
</tr>
<tr>
<td>23.5</td>
<td>0.9974</td>
<td>77</td>
<td>0.9970</td>
</tr>
<tr>
<td>24</td>
<td>0.9973</td>
<td>78</td>
<td>0.9969</td>
</tr>
<tr>
<td>24.5</td>
<td>0.9972</td>
<td>79</td>
<td>0.9967</td>
</tr>
<tr>
<td>25</td>
<td>0.9970</td>
<td>80</td>
<td>0.9966</td>
</tr>
<tr>
<td>25.5</td>
<td>0.9969</td>
<td>81</td>
<td>0.9964</td>
</tr>
<tr>
<td>26</td>
<td>0.9968</td>
<td>82</td>
<td>0.9963</td>
</tr>
<tr>
<td>26.5</td>
<td>0.9966</td>
<td>83</td>
<td>0.9961</td>
</tr>
<tr>
<td>27</td>
<td>0.9965</td>
<td>84</td>
<td>0.9959</td>
</tr>
<tr>
<td>27.5</td>
<td>0.9964</td>
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<td>0.9958</td>
</tr>
<tr>
<td>28</td>
<td>0.9962</td>
<td>86</td>
<td>0.9956</td>
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<td>28.5</td>
<td>0.9961</td>
<td></td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>0.9959</td>
<td></td>
<td></td>
</tr>
<tr>
<td>29.5</td>
<td>0.9958</td>
<td>*Interpolated from °C data</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>0.9956</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The values of ρ_w shown are taken from, “Metrological Handbook 145, Quality Assurance for Measurements”, 1990, NIST, pp. 9,10, and represent the values in air at one atmosphere pressure.
agreed upon by all parties. Exercise care in the selection of temperature to avoid exceeding the melting point of any material tested. This method is applicable to sintered aluminum materials if the temperature does not exceed 540 °C (1,000 °F).

4.11 Calculate the oil content by volume as-received from the following formula:

\[ P_1 = \left( \frac{I-A}{B-(C-E)} \right) \rho_o \times 100 \]

where:
- \( P_1 \) = Oil content of the as-sintered bearing, in volume%
- \( J \) = mass of the as-received, oil-impregnated specimen in air, g
- \( A \) = mass of the oil-free specimen in air, g
- \( B \) = mass of the fully impregnated specimen in air, g
- \( C \) = mass of the fully oil-impregnated specimen suspended and immersed in water, g
- \( \rho_o \) = density of as-received oil at the temperature of test, in g/cm³
- \( E \) = mass of test specimen support in water, g
- \( \rho_w \) = density of distilled water at the temperature of test

5. DETERMINATION OF SURFACE-CONNECTED POROSITY

5.1 This procedure is used to determine the surface-connected porosity of PM components.

5.2 Determine the mass of the oil-free test specimen (mass A), the fully impregnated test specimen (mass B), the mass of the oil impregnated specimen and test specimen support immersed in water (mass C), the mass of the test specimen support in water (mass E), the density of the oil (\( \rho_o \)) and the density of water at the immersion temperature (\( \rho_w \)) as detailed in the previous section (section 4).

5.3 Calculate the surface-connected porosity by volume, %, from the following formula:

\[ P = \left( \frac{B-A}{(B-(C-E))} \right) \rho_o \times 100 \]

where:
- \( P \) = surface-connected porosity by volume, %
- \( \rho_o \) = density of the as-received oil, g/cm³
- \( \rho_w \) = density of water at the immersion temperatures, g/cm³
- \( A \) = mass of the oil-free specimen in air, g
- \( B \) = mass of fully oil-impregnated specimen in air, g
- \( C \) = mass of fully oil-impregnated specimen and test specimen support immersed in water, g
- \( E \) = mass of test specimen support in water, g

6. DETERMINATION OF OIL- IMPREGNATION EFFICIENCY

6.1 Oil-impregnation efficiency, \( I_E \), is defined as the % of open porosity filled with oil and is calculated from the following formula:

\[ I_E = \frac{P_1}{P} \times 100 \]

where:
- \( I_E \) = oil-impregnation efficiency
- \( P_1 \) = oil content of the as-received bearing in volume %
- \( P \) = fully impregnated oil content in volume %

NOTE 2—The fully impregnated oil content in volume % is the same as the surface-connected porosity in volume %

7. REPORT

7.1 Oil content, as calculated from the appropriate formula, to the nearest 1%.

7.2 Surface-connected porosity, as calculated from the appropriate formula, to the nearest 1%.

7.3 Oil-impregnation efficiency, as calculated from the appropriate formula, to the nearest 1%.

8. PRECISION

<table>
<thead>
<tr>
<th>Bronze (CT-1000)</th>
<th>Sintered Density (g/cm³)</th>
<th>Iron Graphite (FG-0008)</th>
<th>Sintered Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Received Oil Content (%)</td>
<td>( P_1 ) average</td>
<td>r</td>
<td>27.9</td>
</tr>
<tr>
<td>Fully Impregnated Oil Content (%)</td>
<td>( P ) average</td>
<td>r</td>
<td>28.9</td>
</tr>
<tr>
<td>Oil Impregnation Efficiency (%)</td>
<td>( P/I ) average</td>
<td>r</td>
<td>96.4</td>
</tr>
</tbody>
</table>

These data, from thirteen laboratories, are based on an inter-laboratory study conducted by the MPIF Standards Committee in 2010. The data were analyzed statistically using ASTM E691 to determine the repeatability limit, \( r \), and the reproducibility limit, \( R \), at the 95% confidence level. Duplicate tests in the same laboratory (individual determinations prior to averaging) by the same operator should not be considered different unless they exceed \( r \). Tests of the same PM structural parts or bearings (individual determinations prior to averaging) conducted by two laboratories should not be considered different unless they differ by more than \( R \).

APPENDIX

A1. COMPARABLE STANDARDS
ASTM B963
ISO 2738
FIGURE 1: Methods for Holding the Test Specimen When Weighing in Water

FIGURE 2: Methods for Weighing in Water

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1. SCOPE

1.1 This standard describes a method for measuring the surface finish of powder metallurgy products at all stages of manufacturing, from green compact to fully hardened.

1.2 This method filters out the influence of open surface porosity on surface finish.

1.3 The values stated in inch-pound units are to be regarded as the standard. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Surface finish measuring instrument.

2.2 Stylus with chisel point, 0.050 inch (1.27 mm) length and 0.0004 inch ± 30% (0.010 mm ± 30%) tip radius as shown in Fig. 1. The chisel tip shall be oriented so that its long edge is perpendicular to the direction of travel of the probe.

2.3 If a conical stylus is used, then filtering software shall also be used to minimize the influence of open surface porosity.

NOTE 1—Names of manufacturers of this apparatus are listed in General Information IV.

3. TEST SPECIMEN

3.1 The test surface shall be clean and free of any oil, dirt, debris or foreign material.

3.2 Sufficient surface area shall be available to permit multiple traverses by the measuring instrument.

3.3 The test surface shall be flat over a sufficient length (per instrument instructions) to allow proper movement of the stylus.

4. PROCEDURE

4.1 The surface finish parameter to be measured should be agreed upon by the parties concerned; for example, Rₐ, Rₐ, Rₕ. In addition, the traverse length to be used should also be agreed upon.

NOTE 2—Rₐ is the arithmetic average value of the filtered roughness profile determined from deviations about the centerline within the evaluation length lₑ as shown in Fig. 2. Rₕ is the maximum peak-to-valley height over the tested length (absolute value between the highest peak and lowest valley) as shown in Fig. 3. Rₚ is the ten-point height, or the absolute value of the five highest peaks and five lowest valleys over the evaluation length as shown in Fig. 4. Rₚ is also known as the ISO 10 point height parameter.

4.2 The PM parts manufacturer and the purchaser shall agree on the desired location and direction for the surface finish measurement. A minimum of three traverses at different locations is recommended.

4.3 Place the surface finish instrument in a position suitable for measuring the test sample.

4.4 Prior to testing, the calibration of the instrument should be checked over the surface finish range expected for the test sample.

4.5 Place the test sample under the stylus and then lower the stylus to the measuring position per the instrument instructions.

4.6 Measure the surface finish of the test surface.

5. REPORT

5.1 Report the surface finish to the nearest whole number, in microinches, as required by the specification. Unless otherwise indicated the surface finish shall be Rₐ (average surface roughness), as illustrated in Fig. 2. Depending on the type of instrument being used, other surface finish measures may also be reported.

5.2 Report the direction of measurement with respect to the pressing direction.

6. PRECISION

6.1 The precision of this test has been determined from an interlaboratory study performed in 2007 in which 11 MPIF laboratories participated.

6.2 The repeatability limit, r, and reproducibility limit, R, measurements listed in Table 1 were determined according to ASTM E691, Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods.
6.3 FLC2-4808 sinter-hardened transverse rupture test specimens with a sintered density of 6.94 g/cm³ and an apparent hardness of 45 HRC were used in the study. Each lab received a single TRS sample from this batch.

6.4 On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory will be expected to exceed, \( r \), only 5% of the time. If such a difference is found to be larger than, \( r \), there is reason to question one or both results. Similarly, the difference in two test results obtained in different laboratories will be expected to exceed, \( R \), only 5% of the time. If the difference is found to be larger than, \( R \), there is reason to question one or both measurements.

6.5 The analysis is based on three measurements per surface using a length of travel that varied from 0.098 inch to 0.5 inch. Some labs used the recommended chisel stylus, other used the conventional full radius stylus point and one lab provided data using both types of stylus.

<table>
<thead>
<tr>
<th>Surface Tested</th>
<th>Type of Stylus</th>
<th>Surface Finish</th>
<th>( r )</th>
<th>( R )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Punch Face</td>
<td>Chisel</td>
<td>66 (1.65)</td>
<td>8 (0.20)</td>
<td>19 (0.48)</td>
</tr>
<tr>
<td>Die Face</td>
<td>Chisel</td>
<td>26 (0.65)</td>
<td>29 (0.73)</td>
<td>40 (1.00)</td>
</tr>
<tr>
<td>Punch Face</td>
<td>Conical</td>
<td>70 (1.75)</td>
<td>38 (0.95)</td>
<td>56 (1.40)</td>
</tr>
<tr>
<td>Die Face</td>
<td>Conical</td>
<td>36 (0.90)</td>
<td>14 (0.35)</td>
<td>52 (1.30)</td>
</tr>
</tbody>
</table>

**APPENDIX**

A1. **COMPARABLE STANDARD**

ASTM B946

**FIGURE 1:** Chisel Stylus for Surface Finish Measurement with Tip Orientation vs. Direction of Travel

Note - The stylus is chisel shaped and has a standard radius on the edge and 0.050 inch (1.27 mm) wide so that it will not drop into the porosity of the PM surface, in effect filtering out the influence of the surface pore cavi ties on surface finish.

**FIGURE 2:** \( R_a \) Arithmetic Mean Roughness Value

Note - The arithmetic average value of filtered roughness profile determined from deviations about the centerline within the evaluation length \( l_e \).

**FIGURE 3:** \( R_t \) Maximum Peak-to-Valley Height

Note - The maximum peak-to-valley height of the filtered profile over the evaluation length \( l_v \), irrespective of the sampling lengths \( l_a \).

**FIGURE 4:** \( R_z \) ISO Ten-Point Height

Note - The average height difference between the five highest peaks and five lowest valleys contained within a chosen evaluation length.

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1. SCOPE

1.1 This standard covers the apparatus, test specimen, and procedure for the impact testing of sintered or heat-treated MIM materials.

1.2 The method used in this standard is the Charpy impact test.

1.3 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 The single-blow Charpy impact test shall be carried out in a pendulum-type impact machine with a capacity of at least 240 ft-lbf (325 J). This is the same apparatus as used for the simple-beam test for steel described in ASTM E23. The impact energies measured in testing shall not exceed 80% of the machine’s maximum capacity.

2.2 Gauge(s) capable of measuring the specimen to the tolerances listed in Fig. 1, to the nearest 0.001 inch (0.02 mm).

3. TEST SPECIMEN

3.1 The test specimen shall have the shape and dimensions as shown in Fig. 1. The gate shall be located within 0.197 inch (5 mm) of either end of the part to minimize its effect on the center section of the specimen. Machining of the specimens is allowable.

4. PROCEDURE

4.1 A minimum of 3 specimens shall be tested in accordance with procedures set forth in ASTM E23. Unless otherwise specified, the test shall be performed at room temperature. Any absorbed energy values above 80% of the scale range of the test apparatus are inaccurate and shall be reported as approximate.

5. REPORT

5.1 Impact energy to the nearest 1 ft-lbf (1 Joule).

5.2 The temperature of the test, e.g. room temperature.

5.3 The following supplementary information may be reported for clarification purposes:

5.3.1 Chemical composition (identification) of alloy and production lot number.

5.3.2 Density of the test specimen, g/cm³.

5.3.3 Final total carbon content, where applicable.

5.3.4 Heat treatment condition and hardness of the specimens, if applicable.

5.3.5 Other processing information, as required.

5.3.6 Any deviation from this test method.

6. PRECISION

6.1 Precision of this test method has not been determined.

APPENDIX

A1. COMPARABLE STANDARDS

<table>
<thead>
<tr>
<th>Standard</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM E23</td>
<td>Simple-beam test for steel</td>
</tr>
<tr>
<td>ISO 5754</td>
<td>Test for steel</td>
</tr>
</tbody>
</table>

FIGURE 1: Charpy Impact Test Specimen

Note: Permissible variations shall be as follows:

- Adjacent sides shall be at 90 deg. ± 10 minutes
- Cross section dimension: ± 0.005 inch (0.125 mm)
- Length of specimen: ± 0.100 inch (2.5 mm)
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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This standard describes the method and apparatus for the preparation of uniaxially compacted powder metallurgy test specimens.

1.2 The values stated in inch-pound units are to be regarded as the standard. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Compacting press or compression testing machine of sufficient capacity to compact the test specimens to the desired green density.

2.2 Punches and dies for producing the specimens. See the following MPIF Standards for a description of the typical tooling or specimen geometry: Std. 10 for tensile specimens, Std. 40 for impact specimens, Std. 41 for transverse rupture specimens and Std. 55 for radial crush specimens.

2.3 Balance, readable to 0.001 g, of a capacity suitable for determining the mass of the compacted test specimen.

2.4 Micrometers or other suitable measuring apparatus capable of measuring the compacted test specimens to the nearest 0.001 inch (0.02 mm).

NOTE 1—Names of manufacturers of this apparatus are listed in General Information IV.

3. TEST SPECIMEN

3.1 Processing PM test specimens using production conditions is the most reliable method to generate PM material property data. Test specimens may be machined from PM parts or blanks provided they are of a suitable size to produce a test piece of the standard dimensions.

4. PROCEDURE

4.1 Obtain test portions from the powder lot that is to be tested following the procedures in MPIF Standard 01. The approximate mass can be estimated by calculating the volume of the test specimen from its dimensions and multiplying this by the required density (the mass required for flat, unmachined tensile bars may require some preliminary tests and density determination in accordance with MPIF Standard 42, due to the shape of the bars).

4.2 Test specimens are produced by uniaxially compacting a test portion of the powder using double-action pressing. Information on the compaction tooling required is presented in the relevant test method standard.

4.3 Lubrication is required to assist the ejection of the compacted test specimen from the die. Either die-wall lubrication or powder with admixed lubricant may be used.

NOTE 2—The results of testing may vary based on the type of lubrication used and the parties involved shall agree upon lubrication methods.

4.4 Die-wall lubrication method

4.4.1 The die-wall lubrication method is typically used to test the properties of base powders with no other ingredients.

4.4.2 Lightly coat the die wall using a lubricant-solvent suspension (e.g., 100 g of a lubricant such as a synthetic wax per litre of acetone or ethyl alcohol) or a die-release aerosol.

NOTE 3—This suspension is flammable and should be used in a suitably ventilated area.

4.4.3 Allow the adherent lubricant to dry on the die walls. A new die-wall coating shall be applied for the compaction of each test specimen.

4.5 Powder lubrication method

4.5.1 Thoroughly mix a solid lubricant (typically a synthetic wax or metal stearate) with the base powder and other ingredients, such as copper powder, graphite, etc., if required.

4.5.2 A lubricant mass of 0.5–1.0 wt.% is typical for ferrous, copper, and aluminum powder-based mixes while 2.0 wt.% is typical for tungsten-based mixes.

4.5.3 Test specimens may be made from pre-weighed test portions or by adjusting the fill level of the die cavity.
4.6 Compaction

4.6.1 Laboratory Tooling

4.6.1.1 Insert the lower punch into the die cavity and position the die containing the lower punch on the lower press platen so that the die is supported on spacer blocks or springs and the lower punch is at the desired filling height. If spacer blocks are used, follow the sequence in Figure 1.

4.6.1.2 Pour the test portion of the powder into the die cavity taking care to ensure that the powder is uniformly and evenly distributed.

4.6.1.3 Insert the upper punch and apply and then release a pre-compacting pressure of approximately 5,000 psi (35 MPa).

4.6.1.4 Remove the spacer blocks that have supported the die (If the die is supported on springs, the pre-compacting step is not needed).

4.6.1.5 Apply the final compacting pressure (typically 60,000 to 120,000 psi [415 to 830 MPa]) depending on the compressibility of the powder mix and the desired green density of the test specimen.

NOTE 4—In special cases, where the results may be affected by the rate at which pressure is applied, a rate not exceeding 60,000 psi per minute (415 MPa per minute) is recommended.

4.6.1.6 Release the compacting pressure as soon as the maximum pressure is attained because pressure dwells of as little as 10 s can increase the green density of the test specimen by 0.3%.

4.6.1.7 Place spacer blocks between the top of the die and the upper press platen. The ejection spacer blocks should be longer than the combined lengths of the upper punch and the compacted test specimen.

4.6.1.8 If possible, remove the upper punch by hand. If not possible, apply pressure so that the ejection spacer blocks push the die down.

4.6.1.9 Remove the upper punch when it clears the die. Continue to eject the green test specimen until it can be picked off the lower punch.

4.6.1.10 Deburr each test specimen carefully with fine emery paper and measure the dimensions to the nearest 0.001 inch (0.02 mm). Parallelism shall be within 0.002 inches per inch (0.002 mm per mm). Determine the green density of the test specimen by dividing the mass of the test specimen by its volume calculated from the dimensions of the specimen. Alternatively, the density may be determined in accordance with MPIF Standard 42 using an extra test specimen prepared using the same conditions.

4.6.1.11 Repeat steps 4.6.1.2 through 4.6.1.9 to obtain the desired number of test specimens. When compacting multiple test specimens, care should be taken to ensure that the green densities are held to +/- 0.02 g/cm³ of the agreed target density.

4.6.2 Production tooling

4.6.2.1 When test specimens are compacted in a tool set adapted to a production compaction press, the die filling, pressing, and ejection steps are all controlled by the programmed motions of the press. When using production equipment, it is usually necessary for the mix to contain an admixed lubricant. A large number of test specimens can be made quite rapidly when using a production press.

5. REPORT

5.1 Chemical composition of the powder mix and alloying method used.

5.2 The method of lubrication.

5.3 The type, brand, and amount of lubricant.

5.4 The individual and averaged green densities of the test specimens.

6. PRECISION

6.1 As a specimen preparation procedure that does not include actual performance of a test no precision statement is required.

APPENDIX

A.1 COMPARABLE STANDARD

ASTM B925

FIGURE 1: Sequence of Operations to Produce a Green Test Specimen in a Manually Operated Laboratory Tool Set

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1. SCOPE

1.1 This standard describes a method for determining the compressive yield strength of powder metallurgy materials and not the complete compressive stress-strain curve.

1.2 This method is applicable to PM materials other than composite structures such as cemented carbides that may exhibit non-linear stress-strain behavior.

1.3 The values stated in inch-pound units are to be regarded as the standard. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI 10. They may be approximate and are only for information.

1.4 This standard may involve hazardous materials, operations or equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Universal testing machine, of suitable capacity, to load the specimen in compression. The machine shall meet the requirements of ASTM E4. An extensometer that meets the requirements of grade B-2 per ASTM E83 shall be used to measure strain.

2.2 Gauge(s) suitable for measuring the test specimen dimensions; typically a micrometer, accurate to 0.0001 inch (0.002 mm) and a dial (or electronic) indicator accurate to 0.0001 inch (0.002 mm).

2.3 Surface finish measuring instrument per MPIF Standard 58.

3. TEST SPECIMEN

3.1 The specimen should be nominally 0.375 inch (9.5 mm) in diameter and 1.05 inch (26.7 mm) high. The tolerance on the diameter is ± 0.002 inch (0.05 mm) and for the height ± 0.010 inch (0.25 mm).

3.2 The specimen shall be machined from a suitable test specimen. The Charpy impact specimen is sufficiently oversize (see MPIF Standard 40) to allow the compressive yield specimen to be machined from it. This method will provide a test specimen with its axis perpendicular to the pressing direction.

NOTE 1—Due to the 1.05 inch (26.7 mm) length of the test specimen, a significant density variation can be expected if the specimen is compacted parallel to this direction.

3.3 The end faces of the cylindrical test specimen shall be flat and parallel within 0.0005 inch (0.0125 mm), perpendicular to the specimen axis within 0.0005 inch (0.012 mm) and have a surface finish of 16 rms (0.4 micrometres), or better.

4. PROCEDURE

4.1 The specimen shall be machined from a rectangular bar such as a Charpy impact specimen that has been prepared in accordance with MPIF Standard 60. Measure the diameter of the test specimen to the nearest 0.001 inch (0.02 mm).

4.2 Place the test specimen on one of its flat faces in the center of the platen. Care should be taken to ensure the contact surfaces are always harder than the test specimen. Platen surfaces shall be finished to a surface finish of 16 rms (0.4 micrometres), or better. High-pressure lubricant may be required to reduce “barreling” of the specimen under load.

4.3 Close the top platen so as to make light contact with the test specimen. Engage the extensometer; typically with a 0.5 inch (12.7 mm) gauge length.

4.4 Load the test specimen at a strain rate of less than 0.005 inch/inch/minute (0.005 mm/mm/minute). Continue applying the load until the stress-strain curve has safely passed the 0.1 % offset strain mark. Stop loading and then unload.

NOTE 2—if the instrument does not determine stress-strain values, refer to load-extension as the measured value.

4.5 Assuming a straight-line path for the initial portion of the stress-strain curve, draw a line parallel to this initial portion using a 0.1% offset from zero strain. The line drawn intersects the stress-strain curve at the 0.1% yield point, and the yield load may be determined. In the event the initial portion of the stress-strain curve is not straight, extend the initial unload portion of the curve to the strain axis and then draw a line through the 0.1% offset parallel to this unload line until it intersects the stress-strain curve. This intersection is the 0.1% compressive yield point.
4.6 Calculate the 0.1% compressive yield strength in psi (MPa) by dividing the compressive yield load in pounds (N) by the initial test specimen cross-sectional area, in inches² (mm²).

5. REPORT

5.1 Compressive yield strength as the average of at least three (3) but preferably five (5) tests to the nearest 1,000 psi (10 MPa).

5.1.1 Method used—extensometer or load-extension.

5.2 The following supplemental information may also be reported:

5.2.1 Chemical composition of the powder mix.

5.2.2 Type, brand, and percent of lubricant.

5.2.3 Density.

5.2.4 Apparent hardness.

5.2.5 Sintering temperature.

5.2.6 Sintering time.

5.2.7 Furnace atmosphere.

5.2.8 Final total carbon content where applicable.

5.2.9 Other processing information as required.

5.2.10 Any deviation from this test method.

6. PRECISION

6.1 The following data were developed by an interlaboratory study involving ten (10) participants using the procedures outlined in MPIF Standard 61. The results of the study are summarized in MPIF Research Report R-61-2015.

6.2 Five test specimens of each of the following materials, machined from rectangular compacts were supplied to each of the participating laboratories:

- FC-0208 (as-sintered)—green density of 6.9 g/cm³
- FY-4500 (as-sintered)—green density of 7.0 g/cm³
- FC-0208 HT (heat treated)—green density of 6.9 g/cm³
- FL-4405 HT (heat treated)—green density of 7.0 g/cm³

6.3 The resulting data were evaluated using ASTM E691 to determine the repeatability, r, and reproducibility limit, R, of the data. The results of the analysis are shown in Table 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Average Yield Strength (psi)</th>
<th>Repeatability (r)</th>
<th>Reproducibility (R)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FC-0208</td>
<td>60,186</td>
<td>7,190</td>
<td>11,818</td>
</tr>
<tr>
<td>FY-4500</td>
<td>34,898</td>
<td>5,037</td>
<td>5,888</td>
</tr>
<tr>
<td>FC-0208 HT</td>
<td>154,156</td>
<td>12,304*</td>
<td>25,673*</td>
</tr>
<tr>
<td>FL-4405 HT</td>
<td>150,540</td>
<td>11,978*</td>
<td>16,984*</td>
</tr>
</tbody>
</table>

* Data from two of the ten laboratories were excluded as their average values were either statistically too high or too low.

6.4 Duplicate results (individual determinations prior to averaging) from the same laboratory should be considered acceptable at the 95% confidence level unless they differ by more than r, the repeatability interval.

6.5 Duplicate results (individual determinations prior to averaging) from two different laboratories should be considered acceptable at the 95% confidence level unless they differ by more than R, the reproducibility interval.

APPENDIX

A1. COMPARABLE STANDARD

ASTM E9

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1. SCOPE

1.1 This standard covers a procedure for determining the corrosion resistance of sintered or heat treated MIM grades of stainless steel materials immersed in a 2% sulfuric acid solution.

1.2 Immersion of MIM stainless steel in 2% H₂SO₄ solution may result in the gradual dissolution of the metal with time. Corrosion resistance is measured in terms of loss of mass in a specified time. The loss of mass is expressed in g/dm² per day to normalize for the surface area.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 400 ml covered beakers or other corrosion resistant containers for each specimen being tested.

2.2 Balance suitable for reading to 0.0001 g, and weighing accurately to within 0.001 g.

2.3 Distilled water.

2.4 Reagent grade (95.0-98.0 %) concentrated H₂SO₄.

NOTE 1—Reagent grade: Conforms to the Specifications of the Committee on Analytical Reagents of the American Chemical Society, found in Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC.

2.5 Methyl alcohol or ethyl alcohol.

2.6 Drying oven capable of 110 °C and accurate to ± 5 °C, with non-corroding racks.

2.7 Timer capable of measuring 60 minute intervals.

2.8 Gauges capable of measuring from 0 to 64 mm to the nearest 0.013 mm.

2.9 Non-corroding tongs.

NOTE 2—Stainless steel or plastic tongs may be used.

2.10 Glass beads (4 mm typical diameter is recommended).

2.11 Tap water for rinsing specimens.

3. TEST SPECIMEN

3.1 The recommended test specimen is the un-machined half thickness Charpy bar approximately 5 mm by 10 mm by 55 mm for convenience in molding and measuring the surface area (see MPIF Standard 59).

3.2 Other convenient shapes may also be used.

3.3 Sample condition should be representative of production part manufacturing processes (e.g. machining, surface passivation).

4. PROCEDURE

4.1 Remove any surface grease or debris, using an alcohol rinse on the specimen surface. Evaporate alcohol to dryness.

4.2 Calculate the surface area of the test specimen from the measured dimensions.

4.3 Weigh the specimen to the nearest 0.001 g.

4.4 Prepare a 2 ± 0.1% by mass H₂SO₄ solution in distilled water taking care to add the acid to the water. Fill beakers to 350 ± 25 ml.

4.5 Using tongs, place one specimen in each beaker, minimizing the contact area with the bottom of the beaker an example of which is shown in Figure 1. Solution must be at least 6 mm above the specimen.

4.6 Cover the beaker to prevent evaporation, e.g. with a watch glass or plastic wrap.

4.7 A minimum of 3 test specimens is recommended for each material and/or condition.

4.8 Leave the specimens undisturbed in the solution for the duration of the test, e.g. 24 hours, 96 hours, 500 hours, or 1,000 hours. Parties should agree on duration. Room temperature should be maintained at 22 ± 2 °C.

4.9 At the conclusion of the test, remove the specimen with tongs, rinse in tap water, and rinse in alcohol.

4.10 Dry the specimen on a non-corroding rack or pan at 110 ± 5 °C for 45 ± 5 minutes.

4.11 Cool to room temperature.

4.12 Weigh the test specimen to the nearest 0.001 g.

5. CALCULATIONS

5.1 Calculate the loss of mass in grams (g).

5.2 Express specimen surface area in square decimeters (dm²).

5.3 Calculate the loss of mass in g/dm² per day.
6. REPORT

6.1 MIM grade or chemical composition.
6.2 Description of specimen (approximate dimensions and surface conditions, machined, passivated, etc.).
6.3 Duration of test.
6.4 Loss of mass in g/dm² per day for each specimen, to the nearest 0.001 g/dm² per day.
6.5 Average loss of mass in g/dm² per day for each alloy or other grouping, to the nearest 0.001 g/dm² per day.
6.6 Any unusual occurrences.

7. PRECISION

7.1 The precision of this test method has not been determined.

FIGURE 1: Example of Corrosion Testing Apparatus

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The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; "Standard for Use of the International System of Units (SI): The Modern Metric System". Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.

This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6892. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This standard covers a procedure for determining the density of green (as-molded), sintered or heat-treated MIM components using a gas pycnometer.

NOTE 1—Any open porosity will not be included as part of measured volume. The density obtained by the gas pycnometer method will typically be higher than the density obtained by water displacement (see MPIF Standard 42).

NOTE 2—This is a generic method that describes the basic steps involved to obtain a final result. For further specific details, the manufacturers’ operating instructions are to be reviewed.

1.2 With the exception of density, for which g/cm$^3$ is the industry standard, SI units are used in this standard.

1.3 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Balance suitable for weighing accurately to within 0.001 g (readable to 0.0001 g) and having a capacity of at least 100 g.

2.2 Gas pycnometer.

2.3 Helium, 99.9 % minimum purity. Other gases may be substituted for helium. Refer to equipment manufacturers’ suggestions.

NOTE 3—Names of manufacturers of this apparatus are listed in General Information IV.

3. SAMPLE PREPARATION

3.1 Sample(s) to be tested must be clean, dry and free of any foreign matter.

4. PROCEDURE

NOTE 3—The following procedure is intended as a generic description of density testing with a gas pycnometer. The manufacturer’s operating instructions for the pycnometer in use will supersede these instructions.

4.1 Test specimen may be comprised of a component, or multiple components or sections of a component, suitable to achieve a minimum of 10 %, but ideally greater than 60 %, of the maximum volume of the particular holder being used.

4.2 Weigh test specimen to the nearest 0.001 g and input mass of specimen into the pycnometer.

4.3 Place the pre-weighed test specimen into the holder.

4.4 Place the specimen and holder into the sample chamber.

4.5 Seal the sample chamber and initiate the test. The pycnometer measures the volume of the specimen and calculates the density from the sample mass entered in grams (g) divided by the volume in cubic centimetres (cm$^3$). Repeat the testing until a run precision of 0.04 % of the cell volume is achieved in five consecutive runs, and record the average of these five values. For example, if the cell volume is 2.9123 cm$^3$, the five runs would have to be within a total variation of 0.0012 cm$^3$ (0.0004 by 2.9123 = 0.0012 cm$^3$). If the run precision is not achieved, restart the test.

5. REPORT

5.1 Specimen identification - MIM grade or chemical composition.

5.2 Density to the nearest 0.001 g/cm$^3$.

6. PRECISION

6.1 Precision of this test method has not been determined.

APPENDIX

A1. COMPARABLE STANDARDS

ASTM D2638

ASTM D4892
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1. SCOPE

1.1 This standard includes definitions of those terms that are necessary for the interpretation and application of metal injection molding and other related standards. The purpose of this standard is to ensure consistency of usage, proper understanding, and precise interpretation.

Acicular Powder - Needle shaped particles.
Activated Sintering - A process by which the rate of sintering is significantly increased by means other than changing time or temperature, e.g., addition of a constituent to the powder, or the atmosphere, or thermal cycling.
Agglomerate - Several particles adhering together.
Air Classification - The separation of powder into particle size fractions by means of an air stream of controlled velocity.
Alloy Powder - See Pre-alloyed Powder.
Angle of Repose - The basal angle of a pile formed by a powder when freely poured under specified conditions onto a horizontal surface.
Apparent Density - The mass per unit volume of powder, usually expressed as grams per cubic centimetre, determined by a specified method.
Apparent Hardness - The hardness of a MIM material (including the effects of porosity), measured using macrohardness equipment. Not to be confused with microindentation hardness.
Atomization - The dispersion of a molten metal into particles by a rapidly moving gas or liquid stream or by mechanical means.
Atomized Metal Powder - Metal powder produced by the dispersion of a molten metal by a rapidly moving gas or liquid stream, or by mechanical means.
Average Injection Velocity - The mean value of the velocity of the feedstock flow front within a single or a group of identical cavities during the injection time that is calculated from the shot volume and injection time. The average injection velocity is calculated as follows:

\[ V_{av} = \frac{V_s}{t_i \times A_c \times n} \]

where:
- \( V_{av} \) = average injection velocity, mm/s;
- \( V_s \) = shot volume, mm\(^3\);
- \( t_i \) = injection time, s;
- \( A_c \) = cross section of the cavity, mm\(^2\);
- \( n \) = number of cavities

Binder - A mixture of polymers and/or other materials that when combined with metal powders enables the mixture to flow during the injection molding process. The binder imparts green strength to the MIM part and allows it to retain its molded shape.
Binder Removal - A chemical or thermal extraction of binder from a part. Synonymous with Debinding.
Blank - A near-net shape MIM component requiring machining or some other operation to give it its final shape.
Bleedout - See Exudation.
Blending - The thorough intermingling of powders or feedstock of the same nominal composition (not to be confused with mixing).
Blister - An imperfection, a rounded elevation on the surface of a part, with boundaries that may be more or less sharply defined, somewhat resembling in shape a blister on the human skin.
Bloom - A visible exudation or efflorescence on the surface of a material.
Blowing Agent - A compounding ingredient used to produce gas by chemical or thermal action, or both, in manufacture of hollow or cellular articles.
Bridging - The formation of arched cavities in a powder or feedstock mass.
Brown Density - The mass per unit volume of a brown MIM part.
Brown Part - A MIM part that has had a majority of the binder (first stage) removed. Synonymous with debound part.
Bulk Density - The density of a powder under non-specified conditions, for example, in a shipping container.
Burn Off - That stage of thermal processing referring to the time and temperature necessary to remove the binder ingredients used to assist the forming of a MIM part.
Carbonyl Powder - A metal powder prepared by the thermal decomposition of a metal carbonyl.

Cavity - The hollow space of a mold that forms one part.

Cemented Carbides - A solid and coherent mass made by molding and sintering a mixture of powders of one or more metallic carbides and a much smaller amount of a metal, such as cobalt, to serve as a metallic binder.

Ceramic Injection Molding - A process in which a ceramic component is shaped by injection molding a ceramic feedstock to form a green part. The binder is removed from the part and the resultant brown part is then densified by sintering. The final result is a dense ceramic part formed without machining.

Chemically Precipitated Metal Powder - Powder produced by the replacement of one metal from a solution of its salts by the addition of another element higher in the electrochemical series, or by other reducing agents.

CIM - The acronym for ceramic injection molding.

Classification - Separation of a powder into fractions according to particle size.

Closed Pore - A pore not connected with the surface.

Coin or Coining - The pressing of a sintered part to create a new surface configuration (not be confused with Size or Sizing).

Cold Slug - A solidified piece of feedstock that is pushed ahead of the feedstock flow fronts.

Comminuted Powder - A powder produced by mechanical disintegration of solid metal.

Composite Powder - A powder in which each particle consists of two or more distinct materials.

Compounding - Process whereby the metal powders and molten binder constituents are combined to produce a feedstock.

Cooling Time - In molding, the time interval from the end of the hold phase until the mold opens.

Continuous Sintering - Presintering, or sintering, in such a manner that the objects are advanced through the furnace at a fixed rate by manual or mechanical means.

Crazing - Apparent fine cracks at or under the surface of a part.

Creep - The time-dependent part of strain resulting from stress.

Cross-Product Contamination - The unintentional mixing of powders with distinct differences in either physical characteristics or chemical compositions or both.

Cut - See Fraction.

Cycle Time - The total time used to carry out a complete sequence of operations.

Debinding - A step between molding and sintering where the majority of the binder used in molding is extracted by heat, solvent, a catalyst or other techniques.

Dendritic Powder - Particles, usually of electrolytic origin, having the typical pine tree structure.

Density - The mass per unit volume, usually expressed in grams per cubic centimetre.

Density Ratio - The ratio of the determined density of a part to the pore-free density of metal of the same composition, usually expressed as a percentage.

Disintegration - The reduction of massive material to powder.

Dimensional Change - See Shrinkage.

Dispersion-Strengthened Material - A material consisting of a metal and a finely dispersed, substantially insoluble, metallic or nonmetallic phase.

Drag - Distortion caused by the frictional effects of the setter on the part during sintering.

Electrolytic Powder - Powder produced by electrolytic deposition or the pulverization of an electro-deposit.

Elemental Powder - Powder of a single chemical species such as iron, nickel, titanium, copper or cobalt, with no alloying ingredients.

Endothermic Atmosphere (Gas) - A reducing gas atmosphere used in secondary heat treatment and produced by the reaction of a hydrocarbon fuel gas and air over a catalyst with the aid of an external heat source. It is low in carbon dioxide and water vapor with relatively large percentages of hydrogen and carbon monoxide. Maximum combustibles approximately 60%.

Equi-axed Powder - Particles having approximately equidimensional, non-spherical shapes.

Exudation - The action by which all or a portion of the low melting constituent of a part is forced to the surface during molding or thermal processing.

Feedstock - In metal injection molding (MIM), a moldable mixture of metal powder and binder.

Flake Powder - Flat or scale-like particles whose thickness is small compared with the other dimensions.

Flash - Excess feedstock that has flowed outside the mold cavity forming thin, sheet-like protrusions on the part. This defect occurs most commonly along parting lines.

Fraction - That portion of a powder sample that lies between two stated particle sizes. Synonymous with Cut.

Fully Dense Material - A material completely free of porosity and voids.

Gas Atomized Powder - A powder formed by the disintegration of a melt stream by a gas expansion nozzle. The particles solidify during free flight after atomization.

Gas Classification - The separation of powder into particle size fractions by means of a gas stream of controlled velocity.

Gate - The interface in the flow channel between the runner and the mold cavity.

Green - As-molded; for example: green molded part, green density, green strength.

Green Density - The mass per unit volume of an as-molded part.
Hold Pressure - The melt pressure, or the pressure applied by the molding machine during the hold time interval.

Hot Isostatic Pressing (HIP) - In MIM, subjecting a sintered part, at elevated temperature, to nominally equal pressure from every direction, with the intent of decreasing porosity.

Hydrogen Loss - The loss in mass of metal powder caused by heating a representative sample for a specified time and temperature in a purified hydrogen atmosphere - broadly a measure of the oxygen content of the sample, when applied to materials containing only such oxides as are reducible with hydrogen and no hydride forming material.

Hydrogen Reduced Powder - Powder produced by the hydrogen reduction of a metal oxide.

Infiltration - A process of filling the pores of a pre-sintered MIM part having interconnected porosity with a metal or alloy of lower melting point.

Injection Molding - The process of forming a material by forcing it, in a fluid state and under pressure, through a runner system (sprue, runner, gate[s]) into the cavity of a closed mold.

Injection Time - The time interval from the beginning of screw forward movement until switching over to hold pressure.

Interconnected Porosity - A network of contiguous pores in and extending to the surface of a sintered part. Usually applied to PM materials where the interconnected porosity is determined by impregnating the specimens with oil.

Irregular Powder - Powder having particles lacking symmetry.

Jetting - A condition that arises during the rapid filling of an injection mold where the feedstock shoots across the mold and fills back toward the gate.

Knit-line – A mark on, or location in a molded part formed by the union of two or more streams of feedstock flowing together. Synonymous with Weld-line.

Liquid Phase Sintering - Sintering of a MIM part under conditions where a liquid phase is present during part of the sintering cycle.

Magnetic Permeability - A measure of the ease of a material to be magnetized.

Master Alloy Powder - A pre-alloyed powder of high concentration of alloy content designed to be diluted when mixed with a base powder to produce the desired composition.

Matrix Metal - The continuous phase of a polyphase alloy or mechanical mixture; the physically continuous metallic constituent in which separate particles of another constituent are embedded.

Mechanically Alloyed Powder - A composite powder produced by mechanically incorporating other constituents which are generally insoluble within the deformable particles of the matrix metal.

Melt Pressure - The pressure of the molten feedstock generated by the injection molding machine.

Melt Temperature - The temperature of the feedstock at the nozzle during injection molding.

Metal Injection Molding (MIM) - A process whereby a metal component is shaped by injection molding a feedstock to form a green part. The binder is removed from the part and the resultant brown part is then densified by sintering. The final result is a dense metal part formed without machining.

Metal Powder - Discrete particles of elemental metals or alloys normally within the size range of 0.1 to 1000 micrometres.

Micro-MIM - A process to produce MIM parts that typically weigh less than 0.01 grams.

Milling - The mechanical treatment of metal powder, or metal powder mixtures, as in a ball mill, to alter the size or shape of the individual particles, or to coat one component of the mixture with another.

MIM - The acronym representing metal injection molding. See Metal Injection Molding.

MIM Part - A part made by metal injection molding.

Minus Sieve - The portion of a powder sample which passes through a standard sieve of specified number. See Plus Sieve.

Mixed Powder - A powder made by mixing two or more powders as uniformly as possible. The constituent powders will differ in chemical composition and/or in particle size and/or shape.

Mixing - The thorough intermingling of various metal powders and/or various binder constituents.

Mold - The components making up the confining form in which a feedstock is molded. The parts of the mold may be some or all of the following: cavities, gates, runners and sprues.

Molding - See Injection Molding.

Mold Open Time - The time interval from the instant the mold begins to open until it is closed again.

Mold Temperature - The mean temperature of the mold cavity surface measured after the system has obtained thermal equilibrium and immediately after opening the mold.

Neck Formation - The development of a neck-like bond between particles during sintering.

Net Shape - A part that meets the final density and dimensional requirements in the as-sintered state.

Nitrogen Alloying - The transfer of nitrogen from a furnace atmosphere to powder or a MIM part, in such a way as to increase the nitrogen content of the material within controlled limits.

Nodular Powder - Irregular particles having knotted, rounded, or similar shapes.

Open Pore - A pore connected with the surface.

Oversize Factor - The numerical scaling factor that is applied to all part design dimensions in order to specify targeted mold cavity dimensions.
Oversize Powder - Particles coarser than the maximum permitted by a given particle size specification.

Oxide Network - Continuous or discontinuous oxides that follow prior particle boundaries.

Packing Material - Any material in which MIM parts are embedded during the debinding, pre-sintering or sintering operation.

Particle Size - The controlling lineal dimension of an individual particle as determined by analysis with sieves or other suitable means.

Particle Size Distribution - The percentage by mass, or by number of each fraction into which a powder sample has been classified with respect to sieve number or micrometres. (Preferred usage: "Particle size distribution by mass" or "particle size distribution by frequency.")

Plasticizer - A binder constituent incorporated into a feedstock to increase its workability, flexibility, or flow properties.

Platelet Powder - Flat particles of metal powder having considerable thickness (as compared to flake powder).

Plus Sieve - The portion of a powder sample retained on a standard sieve of specified number. See Minus Sieve.

PIM - The acronym representing powder injection molding. See Powder Injection Molding.

PM - The acronym representing powder metallurgy. Used as PM Part, PM Product, PM Process, etc.

PM Part - A shaped object that has been formed from metal powders and bonded by heating below the melting point of the major constituent. A structural or mechanical component made by the powder metallurgy process.

Polymer - A substance consisting of molecules characterized by the repetition (neglecting ends, branch junctions and other minor irregularities) of one or more types of monomeric units.

Pore - An inherent or induced cavity within a particle or within a part.

Pore-Forming Material - A substance included in a powder mixture that produces a desired porosity in a sintered part.

Pore-Free Density - Density of a material without pores.

Porosity - The amount of pores (voids) expressed as a percentage of the total volume of the MIM part.

Powder Flow Meter - An instrument for measuring the rate of flow of a powder according to a specified procedure.

Powder Injection Molding - A process in which a component is shaped by injection molding a feedstock. The binder is removed from the part and the resultant brown part is then densified by sintering. The final part is a dense part formed without machining. The term powder injection molding encompasses a broad range of materials, including metals, ceramics, intermetallics, etc. See Metal Injection Molding.

Powder Metallurgy - The production and utilization of metal powders.

Powder Metallurgy Part - See PM Part.

Pre-Alloyed Powder - A metallic powder composed of two or more elements which are alloyed in the powder manufacturing process, and in which the particles are of the same nominal composition throughout. Synonymous with Alloy-Powder.

Presintering - The heating of a part at a temperature below the normal final sintering temperature, usually to increase the ease of handling or to remove binder constituents prior to sintering.

Pulverization - The reduction in size of metal powder by mechanical means.

Reduced Metal Powder - Metal powder produced, without melting, by the chemical reduction of metal oxides or other compounds.

Relative Density - See Density Ratio.

Release Agent - A material added to a compound or applied to the mold cavity, or both, to reduce adherence of parts to the mold surface.

Runner - (1) The secondary feed channel in an injection mold that runs from the inner end of the sprue to the cavity gate.

Relative Density - See Density Ratio.

Screen Analysis - See Sieve Analysis.

Segregation or Separation - The undesirable separation of one or more components of a feedstock or alloy.

Setter - A substrate that parts are placed on for processing.

Shrink Factor - See Oversize Factor.

Shrinkage - A decrease in dimensions of a part occurring during processing.

Short Shot or Short - An imperfection in a MIM part due to an incompletely filled out condition.

Shot Volume - The total hollow space of a mold part due to cavity or cavities, runner(s), and sprue.

Shrink Mark - See Sink.

Sink - A depression in the surface of a molded part where it has retracted from the mold.

Sieve Analysis - Particle size distribution; usually expressed as the mass percentage retained upon each of a series of standard sieves of decreasing size and the percentage passed by the sieve of finest size. Synonymous with Screen Analysis.

Sieve Classification - The separation of powder into particle size ranges by the use of a series of graded sieves.

Sieve Fraction - That portion of a powder sample which passes through a standard sieve of specified number and is retained by some finer sieve of specified number.

Sintering - The metallurgical bonding of particles in a MIM part resulting from a thermal treatment at a temperature below the melting point of the main constituent.

Sintering time - The total elapsed time during which a part/specimen is within (+/-) a specified percentage of the stated sintering temperature.
Size or Sizing - To press a sintered MIM part to decrease the variation of the specific dimension and/or to secure the desired size.

Slumping - The lack of shape retention of a molded part, during subsequent processing, due to the effect of gravity.

Solids Loading - The relative volume of metal powder in a feedstock designed for metal injection molding. Expressed as a volume percent, e.g. 65% solids loading.

Solid-State Sintering - Sintering of a part without formation of a liquid phase.

Specific Surface Area - The total surface area of the powder particles per unit mass of powder.

Sprue - (1) The primary feed channel that runs from the outer face of an injection mold to the runner.
- (2) The piece formed in a primary feed channel or sprue.

Spherical Powder - Powder consisting of round or globular shaped particles.

Straightening - A sizing operation in which a part is returned to its original or desired configuration. Typically done to achieve flatness or perpendicularity.

Superfines - The portion of a powder composed of particles which are smaller than a specified size, currently less than 10 micrometres. Powders used for MIM are often in this size range.

Tap Density - The apparent density of a powder obtained when the receptacle is tapped or vibrated during loading under specified conditions.

Thermoplastic - Capable of being repeatedly softened by heating and hardened by cooling through a temperature range characteristic of the plastic, and that in the softened state can be shaped by flow into articles by molding or extrusion.

Thermoset - A plastic that, after having been cured by heat or other means, is substantially infusible and insoluble.

Tool - See Mold.

Viscosity - The property of resistance to flow exhibited by a feedstock.

Void - A pore or hole in a MIM part formed during molding or thermal processing.

Warpage - Distortion which may occur in a part during processing.

Water Atomized Powder - A powder formed by the disintegration of a melt stream with high pressure water.

Weld-line - See Knit-line.

Wrought - A material fabricated by conventional fusion metallurgy techniques with a final stage of plastic deformation to improve mechanical properties.

APPENDIX

A1. Recommended Particle Size Data for Certification of Powders for Metal Injection Molding (MIM)

A2. When purchasing powder for MIM feedstock production, it is suggested that the powder supplier report data to adequately reflect the particle size distribution of the powder batch. This normally consists of the 10th, 50th, and 90th percentile on a volume distribution of the powder particles. It is common to measure these data using laser particle size analysis equipment. Care should be taken when comparing powders from various suppliers due to differences in powder production techniques and particle size testing equipment. Even when similar equipment types are used, for example two laser particle size analyzers from the same equipment supplier, slight differences can be seen in the data that are generated.

A3. COMPARABLE STANDARDS:

ASTM B243
ASTM D883
MPIF 09

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1. SCOPE

1.1 This standard describes a method for preparing and determining the hardenability of powder metallurgy steels.

1.2 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations or equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 End-quench hardenability test system per ASTM A255.

2.2 Furnace capable of a temperature of at least 1,800 °F (980 °C) with an inert atmosphere.

NOTE 1 — Inserting the Jominy bar in a rectangular cavity in a graphite block and heating in air has also been shown to be an acceptable practice.

3. TEST SPECIMEN

3.1 Generally, the Jominy test bar shall be machined from an oversized, sintered, compact to the dimensions specified in ASTM A255.

3.1.1 Turn one end of the compact to form the supporting collar of the Jominy bar.

3.1.2 Holding the bar by this collar, turn the square section of the bar, place a center point in the end opposite the collar, and then turn the bar to a 1.000 inch (25.4 mm) diameter.

3.1.3 Cut the Jominy bar to length—generally 4.0 inch (100 mm).

3.2 An inert atmosphere shall be used in the furnace to protect the test specimen from any possible source of oxygen that would decarburize the material. Since carbon is a major contributor to hardenability, maintaining an accurate surface carbon content is critical to the success of the test.

4. PROCEDURE

4.1 Conduct the hardenability test according to the procedures described in ASTM A255.

4.1.1 Ensure that the austenitizing temperature is appropriate for the material being tested.

4.2 Take apparent hardness measurements in steps of 1/16 inch (1.6 mm) for the first 16 sixteenths of an inch (25.4 mm), then 18, 20, 22, 24, 28, and 32 sixteenths of an inch from the water quenched end of the test bar. Continue to take apparent hardness measurements to a distance of 56 sixteenths of an inch from the water quenched end of the Jominy bar spacing the measurements at 1/4 inch intervals from a distance of 32 sixteenths onwards.

4.3 Measure the apparent hardness value on the Rockwell A scale unless the Rockwell C scale is specifically agreed upon by the supplier and the customer.

5. REPORT

5.1 The hardenability curve, plotting measured apparent hardness values versus distance from the water quenched end of the test bar in sixteenths of an inch increments for the first 32 sixteenths of an inch and at the increments listed in Section 4.2 thereafter.

5.2 The hardenability index, Jₖₜ (Jₖₜ is the distance from the quenched end of the test bar where the apparent hardness is 65 HRA) for the PM steel in sixteenths of an inch.

5.3 The density of the Jominy bar at the water-quenched end of the test bar.

5.4 Measure the carbon content of the test bar—take drillings at the slow-cooled end of the test bar for this, drilling through one or both of the ground, flat surfaces that were used for apparent hardness determination.

5.5 The following supplemental information may also be reported:

5.5.1 Chemical composition of the powder mix.

5.5.2 Type, brand, and percent of lubricant.

5.5.3 Density.

5.5.4 Apparent hardness.

5.5.5 Sintering temperature.

5.5.6 Sintering time.

5.5.7 Furnace atmosphere.

5.5.8 Final total carbon content where applicable.

5.5.9 Other processing information as required.

5.5.10 Any deviation from this test method.
6. PRECISION

6.1 Precision is not available at this time.

APPENDIX

A1. COMPARABLE STANDARD
ASTM A255

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1. SCOPE

1.1 This standard describes a method for preparing the specimen used to determine the total carbon content of powder metallurgy materials. This method is NOT applicable to cemented carbides. Types of carbon possibly present in PM materials are also described.

1.2 With the exception of density, for which g/cm³ is the industry standard, this standard uses inch-pound units. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM SI 10. They may be approximate and are only for information.

1.3 This standard may involve hazardous materials, operations or equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 Carbon analysis instrument and accelerator that conforms to the requirements of ASTM E1019.

2.2 A balance, readable to 0.0001 g, for determining the mass of the approximately 1 g test portion.

2.3 Appropriate standard reference material.

2.4 Equipment or device to create drillings, chips or other small form of the sample for testing.

3. TEST SPECIMEN

3.1 This standard only applies to PM materials in which the carbon is uniformly distributed and present in the forms listed in Table 1. In those materials where the carbon is not uniformly distributed, such as case hardened steel parts, the method of selecting the test specimen shall be agreed upon between the producer and the purchaser.

3.2 The presence of carbonaceous contaminants, either on the surface of the sample or in the pores, interferes with the determination of the carbon content (see Table 2).

3.3 The test specimen shall be a PM part, a portion of a PM part or test material in another form such as powder, green, sintered or heat treated product. The sample mass shall be sufficient to meet the requirements of the test instrument.

Table 1. Forms of Carbon Determined by Analytical Methods

<table>
<thead>
<tr>
<th>Form of Carbon</th>
<th>Typical Material</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Combined carbon</td>
<td>Steel</td>
<td>Determined by metallographic comparison with referenced microstructures (1)</td>
</tr>
<tr>
<td>Free carbon</td>
<td>Bronze and steel bearings; material impregnated with graphite containing oil</td>
<td>Determined as the difference between total carbon and combined carbon</td>
</tr>
<tr>
<td>Total carbon</td>
<td></td>
<td>Determined by direct combustion of the specimen</td>
</tr>
</tbody>
</table>

(1) Metallographic estimate is possible only with material systems that exhibit a ferrite-pearlite microstructure.

Table 2. Alternative Sources of Carbonaceous Material & Feasibility of Carbon Determination

<table>
<thead>
<tr>
<th>Carbonaceous Material</th>
<th>Origin</th>
<th>Feasibility</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid or semi-liquid organic substance on the surface or in the pores</td>
<td>Sizing lubricant, quenching oil, impregnating lubricant, cutting fluid, grease</td>
<td>Carbon determination only feasible when these organics are totally removed by method described in Section 4.</td>
</tr>
<tr>
<td>Waxes and wax-like substances</td>
<td>Sizing lubricant, corrosion protection</td>
<td></td>
</tr>
<tr>
<td>Deposits of carbon on parts</td>
<td>Sintering, heat treatment</td>
<td></td>
</tr>
<tr>
<td>Solid organic substances, such as plastic impregnate in the pores</td>
<td>Sealant material</td>
<td>In general not possible to eliminate these substances; carbon determination not possible (2)</td>
</tr>
</tbody>
</table>

(2) Combined carbon may be determined by metallographic comparison with referenced microstructures for ferrite-pearlite.

3.4 For alloy systems where a metallographic estimate is not feasible for determining the combined carbon content, the total carbon content provides an estimate of the combined carbon, assuming no evidence of free graphite is found in the microstructure.
4. PROCEDURE

4.1 Alternative sources of carbonaceous materials (see Table 2 for description) that affect the carbon determination shall be removed from the material to be analyzed as follows:

4.1.1 Remove surface carbon deposits (such as paint, powder coatings, etc.) by mechanical methods such as grinding, abrasive polishing, etc.

4.1.2 Reduce the test specimen or part to a mass appropriate for the analysis instrument (the approximate mass of the test portion is 1 g or less, depending on the carbon content of the material or the equipment being used). This is typically performed by crushing in a mortar using a non-contaminating material or by using a suitable machining operation (drilling, milling, or turning, etc.). Take care to avoid overheating, oxidation, contamination or loss of any free graphite particles during machining or subsequent handling.

4.1.3 Heat-treated steels are sometimes hard to drill. If the specimens cannot be drilled or broken into smaller pieces by crushing in a mortar, a fixed bolt cutter, a cut-off or wafering saw may be used to cut small sections from the specimen or PM part. These may be reduced in size to obtain the appropriate mass for testing.

4.1.4 Soxhlet Extraction Procedure: Remove impregnated organic substances from the pores or on the surface (non-sealant materials, see Table 2) with an appropriate solvent such as petroleum ether. After extraction, remove the residual solvent by heating the test portion to 36 °F (20 °C) above the boiling point of the solvent. Continue to alternate extraction and drying steps until the dry mass is constant within 0.05%. For high-density parts a more aggressive solvent may be required.

4.1.5 If the impregnating fluid contains colloidal graphite, it is possible that not all of these particles can be extracted by the Soxhlet apparatus. Remove additional colloidal graphite, if necessary, using the procedure in either 4.1.7 or 4.1.8.

4.1.6 If there is no concern for the subsequent metallurgical properties of the test portion, the organic substances may be removed using the procedure in either 4.1.7 or 4.1.8.

4.1.7 Alternatives to Soxhlet Extraction Procedure: Heat the section or test portion to a temperature of 1,000 ± 20 °F (540 ± 10 °C) for 15–30 minutes in air. Remove the section or test portion from the heat and let it cool. This method is very effective for removing quench oils, machining coolant and other hydrocarbon fluids.

NOTE 1—A common industry practice to remove oil is to pass parts through a sintering furnace.

4.1.8 Another method that has proven to be effective for removing residual quench oil, machining coolant, and other hydrocarbon fluids when there is no concern for the metallurgical properties of the specimen is to heat a section of appropriate mass, cut from the test specimen or PM part, in the tip of the blue inner flame of a Bunsen burner for about 1 minute. Remove the section or test portion from the heat and let it cool.

4.2 Determine the mass of the actual test portion and record it to the nearest 0.0001 g.

4.3 Measure the total carbon content of the fully prepared test portion using the procedure described in ASTM E1019.

5. REPORT

5.1 Report the total carbon content to the nearest 0.01%.

5.2 The following supplemental information may be reported for clarification:

5.2.1 Chemical composition of the powder mix.

5.2.2 Sintered density.

5.2.3 Sintering temperature.

5.2.4 Sintering time.

5.2.5 Furnace atmosphere.

5.2.6 Other processing information as required.

6. PRECISION

6.1 The repeatability limit, r, and reproducibility limit, R, measurements listed in Table 3 were determined in 2008 according to ASTM E691, Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods.

6.2 As-sintered transverse rupture test specimens were received by a series of PM laboratories that participated in a quality assurance interlaboratory test program. The laboratories were instructed to take three separate drillings from the specimen. Different materials and densities were used for the specimens.

6.3 On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory will be expected to exceed r only 5% of the time. If such a difference is found to be larger than r, there is reason to question one or both results. Similarly, the difference in two test results obtained in different laboratories will be expected to exceed R only 5% of the time. If the difference is found to be larger than R there is reason to question one or both measurements.

6.4 The analysis is based on three measurements per specimen.
Table 3. Precision of Total Carbon Content Measurements

<table>
<thead>
<tr>
<th># of Labs Participating</th>
<th>Material</th>
<th>Density g/cm³</th>
<th>Total Carbon Content, %</th>
<th>r</th>
<th>R</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>FL-4205</td>
<td>7.09</td>
<td>0.36</td>
<td>0.02</td>
<td>0.05</td>
</tr>
<tr>
<td>18</td>
<td>FL-4005</td>
<td>6.71</td>
<td>0.49</td>
<td>0.03</td>
<td>0.07</td>
</tr>
<tr>
<td>18</td>
<td>FL-4000</td>
<td>7.04</td>
<td>0.18</td>
<td>0.01</td>
<td>0.03</td>
</tr>
<tr>
<td>18</td>
<td>FLN2-4405</td>
<td>6.76</td>
<td>0.46</td>
<td>0.01</td>
<td>0.04</td>
</tr>
<tr>
<td>18</td>
<td>FC-0205</td>
<td>6.39</td>
<td>0.43</td>
<td>0.02</td>
<td>0.05</td>
</tr>
<tr>
<td>18</td>
<td>FN-0205</td>
<td>6.93</td>
<td>0.47</td>
<td>0.02</td>
<td>0.04</td>
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<tr>
<td>20</td>
<td>FN-0200</td>
<td>6.67</td>
<td>0.09</td>
<td>0.02</td>
<td>0.05</td>
</tr>
<tr>
<td>20</td>
<td>FD-0208</td>
<td>6.74</td>
<td>0.77</td>
<td>0.03</td>
<td>0.07</td>
</tr>
<tr>
<td>17</td>
<td>FC-0205</td>
<td>6.29</td>
<td>0.59</td>
<td>0.01</td>
<td>0.04</td>
</tr>
<tr>
<td>17</td>
<td>SS-434</td>
<td>7.01</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
</tbody>
</table>

APPENDIX

A1. COMPARABLE STANDARD
ISO 7625

Disclaimer

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-8692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This guide describes the procedures appropriate for the sample preparation of powders, premixes and PM parts for the chemical analysis of metallic elements. This guide does not cover the determination of iron in ferrous materials.

1.2 For the determination of the total carbon content of sintered powder metallurgy (PM) materials refer to MPIF Standard 66.

1.3 Only prealloyed powders and sintered materials made from prealloyed powders are chemically homogeneous. Prealloyed powders or PM parts made from prealloyed powders are considered chemically homogenous and may be analyzed using similar procedures to those used for wrought materials, with the exception of the removal of admixed lubricants from premixed materials, or sizing or quenching oils from PM parts. Admixed, diffusion-alloyed and hybrid PM materials are not chemically homogenous, so special precautions are needed when preparing these powders or parts made from such powders for chemical analysis. Larger test portions are generally required compared with those used for wrought materials.

1.4 Because metal injection molded (MIM) parts are made from very fine particle size powders and are generally high temperature sintered, the materials tend to be homogeneous; even when, the parts are not made from prealloyed powders.

1.5 This standard may involve hazardous materials, operations or equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. APPARATUS

2.1 The test instrument(s) used to determine the chemical composition of PM materials are specific to the analytic method selected. These include:

- Inductively coupled plasma—optical emission spectrometer (ICP-OES)
- Atomic absorption spectrometer (AA)
- Optical emission spectrometer (OES)
- Glow discharge spectrometer (GDS)
- X-ray fluorescence spectrometer (XRF)

2.2 For the actual determinations follow the instructions of the instrument makers and the procedures outlined in the relevant ASTM standard.

3. REFERENCE STANDARDS

3.1 The chemical composition of PM materials tends to differ considerably from that of most wrought or cast materials for which there are appropriate primary reference standards.

3.2 Secondary reference materials may be developed for use with PM materials. This will be particularly necessary for XRF, GDS, or OES techniques.

3.3 Calibration standards for the test instrument should be matrix matched to the solutions used for analysis in order to account for signal coming from the dissolution materials. This means that reference standards shall be digested with the same acids used to dissolve the test samples.

3.4 Calibration standards should also match the approximate concentrations of the analytes in the test samples.

3.5 If there are no appropriate reference materials, manufacturing standards should be considered.

4. TEST SPECIMENS (TEST PORTIONS)

4.1 ICP and AA test methods use a solution made by dissolving the test portion and pumping the resulting solution into the analyzer

4.2 OES, GDS and XRF use powder forged test samples, solid sections cut from PM parts, or melt buttons from the powder or PM part.

NOTE 1—Careful selection of the mold material and melting/cooling process are necessary to ensure a homogeneous test surface. Some alloys phase separate during cooling and may need to be quenched. Some elements have low vapor pressure, so heating times should be minimized.

4.3 For OES and GDS analysis, the melt button should be analyzed at multiple locations across the surface. Consistent results will show that the button was prepared properly.
## 5. PROCEDURE

### A. Powders and Premixes

5.1 Sample the powder or premix in accordance with MPIF Standard 01. The use of a sample splitter to obtain a representative sample for admixed, diffusion alloyed, and hybrid PM materials is imperative.

5.2 Take a 5 g test portion from the composite sample for chemical analysis of ferrous materials using ICP or AA test methods.

For nonferrous powders smaller test portions may be acceptable. The test portion size should be agreed by the concerned parties.

#### NOTE 2

—It is often better to sinter a TRS bar and then dissolve 5 g of the test bar for chemical analysis. In the case of a lubricated premix this eliminates the need to delubricate the material before testing it. It also leads to a more uniform distribution of the alloy additions prior to taking the test portion for chemical analysis.

5.3 If the chemical analysis is to be performed by OES, GDS or XRF, prepare a powder forged test sample or melt button. If necessary, grind the surface flat prior to analysis.

5.4 Prepare the surface of the forged test specimen or melt button according to the analytical technique and material type (OES, GDS, and XRF test methods require the samples to be ground flat; the surface finish depends on the analytical technique). The reference materials and the samples should be prepared in the same manner.

5.5 Wherever possible, make multiple determinations.

### B. PM Parts

5.6 Many PM parts contain residual oil from sizing, rust preventative operations, or heat treatment processes. It is therefore strongly recommended that parts for chemical analysis be cleaned prior to having portions extracted for testing.

5.6.1 Remove surface contaminants by mechanical methods such as grinding, abrasive polishing, etc. Ultrasonic cleaning may be used to remove residual abrasive media.

5.6.2 Remove surface oils and oils from surface-connected pores through Soxhlet extraction with an appropriate solvent, such as petroleum ether. After extraction, remove solvent by heating at least 20 °C (36 °F) above the boiling point of the solvent. Multiple iterations of extraction and drying may be necessary to remove all of the oils. When completely clean, the dry mass should not vary more than 0.05%. For large, high density parts a more aggressive solvent may be required.

5.6.3 An alternative to solvent extraction is to heat the sample to 540 ± 10 °C (1000 ± 20 °F) for 15–30 minutes to burn off any organic material. Longer times may be required for large or low density parts. Remove the specimen from the heat and allow it to cool prior to testing. This method is very effective for removing quench oil, machining coolant and other hydrocarbons. Do not use this method if the sample will react or change in air at temperature.

5.7 Section a ferrous PM part to obtain at least a 5 g sample for either ICP or AA analysis. A smaller test portion may be used if the ferrous PM part is known to have been made from a prealloyed powder. For ICP and AA, thoroughly rinse during filtration (this is particularly important for resin impregnated parts or parts with high carbon contents). For nonferrous PM parts smaller test portions may be acceptable. The test portion size should be agreed by the concerned parties.

5.8 If the chemical analysis is to be performed using OES, GDS or XRF, make a melt button from the cut section or sections of the part and grind the surface to the appropriate flatness.

5.9 Wherever possible, make multiple determinations.

## 6. REPORT

6.1 The sample history—state if the sample was received for analysis as a powder or part, lubricated/unlubricated, sintered, heat treated or as-compacted.

6.2 The sampling protocol.

6.3 The test method.

6.4 The average of multiple tests, if possible. Indicate the sample size.

6.5 The composition for the particular element in question to the appropriate precision.

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1. **SCOPE**

1.1 This guide describes procedures appropriate for checking the thermal profile of a continuous mesh-belt sintering furnace.

1.2 This standard may involve hazardous materials, operations or equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limits prior to use.

2. **APPARATUS**

2.1 Thermocouples suitable for the temperature range covered, of sufficient length to traverse the entire length of the sintering furnace.

2.2 PM parts or steel blocks with pre-drilled holes into which the thermocouples may be inserted.

NOTE 1—Profiles may be developed for PM parts of a specific size by inserting thermocouples directly into the PM parts or into steel blocks of a size and shape that simulate the PM part. Alternatively, the same size PM part or steel block may be used each time the furnace is profiled. The thermocouples should be fixed in the PM parts or steel blocks in a consistent manner for the best repeatability.

2.3 Temperature resistant wire, such as thermocouple wire or Inconel wire for attaching the thermocouples to the sintering belt.

2.4 Temperature recording device, capable of recording temperature readings from the individual thermocouples at time intervals from 1 second up to 10 seconds. The temperature recording device should be calibrated in accordance with the manufacturer’s recommendations.

2.5 Masking tape or something similar, to indicate event locations on the thermocouple sheath (only one thermocouple needs to be marked when a multiple thermocouple profile is being performed).

3. **PROCEDURE**

3.1 **Identification of the Event Marker Locations**

3.1.1 Obtain event locations by physically measuring the furnace to identify critical transitions.

3.1.2 Event locations are locations such as (see Figure 1):

- the start of the furnace hood
- the entrance to the muffle
- the entrance to the preheat (de-lubrication) section
- the location of the first control thermocouple in the preheat section
- the location of subsequent control thermocouples in the preheat section
- the exit from the preheat section
- the entrance to the high-heat section
- the first high-heat section control thermocouple
- subsequent high-heat section control thermocouples
- the exit from the high-heat section
- the first cooling section or the convective cooling section
- subsequent cooling sections or convective cooling sections
- the entrance to the first curtain box
- the entrance to subsequent curtain boxes
- the exit from the furnace

3.1.3 Verify the locations, if using previous thermal profile sheets for the furnace to determine the event locations.

3.2 **Procedure for Preparing the Thermocouples**

3.2.1 Carefully unroll and straighten the thermocouples.

3.2.2 Lay out the thermocouples marking the left, center, and the right thermocouple.

3.2.3 Mark the event locations on at least one of the thermocouples using the masking tape (see Section 3.1.2 for the description of event locations).

3.2.4 Place the end of each thermocouple into either the PM parts or steel blocks with pre-drilled holes, and secure them in position with temperature resistant wire. The procedure used should be documented and the same procedure should be used each time to ensure consistency.

3.2.5 Place the thermocouple/PM part assemblies on the sintering belt and secure them in position so that they will not change position on the belt during the measurement of the thermal profile (for example, tie each thermocouple to the belt in three places approximately 20 cm (8 inch) apart, with a short length of wire). Thermocouple placement for a three-thermocouple profile shall be at the center of the belt and 3 to 5 cm (1 to 2 inches) from each edge.
FIGURE 1: Sintering Furnace Schematic with Examples of Event Marker Locations

NOTE 2—It is not necessary to run a three-thermocouple profile each time. Generally, a single thermocouple profile, with the thermocouple located at the center of the sintering belt, is sufficient for routine profiling. A three-thermocouple profile may be run when starting up a furnace for the first time, when any major changes have been made to the furnace, or if a problem of thermal consistency across the belt is suspected.

3.3 Procedure for Running the Thermal Profile

3.3.1 Measure the belt speed directly to verify the speed or indirectly from the belt-drive roller. For consistency, use the same belt speed each time if you are using the same size PM part or steel block. When determining the profile for different size PM parts or steel blocks, ensure that the correct speed is set for the part in question.

3.3.2 Verify and document that the set points for the various furnace zones are correct and that the process values are in line with the set points (i.e. that the conditions are nominally stable and the furnace is in a steady-state condition).

3.3.3 Verify and record the atmosphere flow rates.

3.3.4 For a furnace that has convective cooling, verify and record that the fan speed is correct.

3.3.5 Measure and record the outlet water temperature and water flow rate if available, for each cooling jacket.

3.3.6 When the profile is being run with a loaded furnace, make sure that the loading pattern for the parts being sintered is correct (the same one that is normally used for the parts in question) both ahead of and behind the thermocouple/PM part assemblies. Record the part mass and the mass per hour being sintered. Alternatively, the profile may be determined for an empty furnace.

3.3.7 Connect the thermocouple or thermocouples to the temperature-recording device.

3.3.8 Generally, the thermal profile is run from the front of the furnace.

3.3.9 Record the time at which each event marker enters the furnace. This provides a check on the belt speed.

3.3.10 For a high-heat set point of 1120 °C (2050 °F) for example, record the following:
- the time when 1090 °C (2000 °F) is reached,
- the length of time within ± 5 °C (± 10 °F) of the set point,
- when the temperature drops below ± 5 °C (± 10 °F) of the set point,
- when the temperature drops below 1090 °C (2000 °F),
- the maximum temperature observed.
A similar procedure may be followed using a different high heat set point if, for example, the furnace is being profiled for sintering a non-ferrous material.

3.3.11 The profile is complete when the thermocouple/PM part assembly tip exits the furnace.

3.3.12 Detach the thermocouple/s from the sintering belt and from the temperature-recording device and pull the thermocouple/s back through the sintering furnace. It is advisable to store the thermocouples in a manner that will not damage their integrity.

3.3.13 Use caution when handling the thermocouples as they may still be hot.

3.3.14 For MGO type (magnesium oxide insulated) thermocouples with wires of less than 3.264 mm (0.1285 inch), the cooling rate is determined by running the cooling profile from the back of the furnace. The thermal profile can be run from the back of the furnace by feeding a wire through the furnace and then attaching it to the end of the thermocouple and pulling the thermocouple back through the furnace to the charge end of the furnace. Tie the thermocouple down to the belt as described in Section 3.2.5. Start to record the temperature when the tip of the thermocouple/PM part assembly enters the high-heat region of the furnace and continue recording until the recorded temperature is below 100 °C (212 °F).
4. DISPLAYING THE THERMAL PROFILE

4.1 Plot the recorded data with time on the x-axis and temperature on the y-axis.

4.2 Determine the time in the effective high-heat region. For a high-heat set point of 1120 °C (2050 °F) this might be for example:
- the total time above 1090 °C (2000 °F), or
- the total time ± 15 °C (± 25 °F) of the set point.

4.3 For sinter hardening, report the cooling rate as the rate of cooling, for example, between the temperatures of 650 °C to 315 °C (1200 °F to 600 °F). Calculate the cooling rate from the recorded data between the two set temperatures, using linear regression analysis to determine the best fit to the data.

NOTE 3—These temperatures are used because they are in the vicinity of the “nose” of the continuous cooling transformation curves for typical sinter-hardenable PM materials.

4.4 Report the type of thermocouple and wire gauge used along with a description of the part or fixture used.

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1. **SCOPE**

1.1 This guide describes procedures appropriate for measuring the porosity of powder metallurgy products using light optical microscopy and automated image analysis.

1.2 The porosity is expressed as an area percentage.

1.3 Automated image analysis is useful in determining local differences in porosity that may result from compaction, sintering, or other operations such as infiltration, repressing, or surface densification.

1.4 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. **APPARATUS**

2.1 Automated metallographic grinding and polishing equipment.

2.2 A high-quality reflected light microscope fitted with bright-field objective lenses of suitable magnifications.

2.3 The movement of the microscope stage should be programmable and automated for movement in the x and y directions. A feature for automated focus in the z direction is highly recommended.

2.4 An automated system for checking the light intensity is also highly recommended.

2.5 A high-quality digital camera linked to an automated image analysis system.

2.6 Computer for data storage and analysis.

3. **SAMPLE PREPARATION**

3.1 Sample mounting is required to facilitate automated grinding and polishing procedures.

3.2 The sample shall be of sufficient size to permit the measurement of enough fields of view at the required magnification. More than one section may be used if needed in order to examine sufficient fields. This is particularly useful where specific small regions are of interest.

3.3 Automated grinding and polishing procedures similar to those outlined in ASTM B931 and B933 are needed in order to produce metallographic samples in which the true nature of the porosity is revealed.

3.4 Resin impregnation of the sample to help retain the true shape of the porosity is highly recommended.

3.5 The highest quality sample preparation is needed in order to obtain meaningful results.

3.6 Samples for porosity determination are examined in the as-polished, unetched condition.

4. **IMAGE ANALYSIS PROCEDURE**

4.1 Place the polished specimen on the microscope stage.

4.2 Program the computer to indicate the position of the first field to be examined and the pattern to be followed for subsequent fields. Make sure that there is no overlapping of the fields that are examined.

4.3 Adjust the magnification so that the pore edges are clearly defined. Higher magnifications are suggested in order to maximize the resolution of the pore edges, thereby minimizing measurement variability.

4.4 Adjust the threshold setting to detect the pores without causing enlargement of the larger pores. The use of higher magnifications makes the porosity measurement less dependent on the threshold setting – see Appendix A.

**NOTE 1**—The threshold setting is a gray-scale window that ranges from black for the pores to a dark gray at the pore edges. The gray-scale level of the metallic matrix should be nearly white to ensure consistency in the gray level of the pores and pore edges.

4.5 The number of fields that need to be measured will be determined by the relative accuracy required for the porosity measurements and the size of the region(s) of interest.

**NOTE 2**—As this is an automated image analysis procedure, having to examine a large number of fields is not an issue.

5. **REPORT**

5.1 Details of the sample being examined such as the material and processing used.

5.2 The region of interest of the PM part in which the porosity is being determined. (include a cross-section drawing of the part indicating this region wherever possible).

5.3 The microscope and automated image analyzer settings (magnification, number of fields examined, threshold setting etc.).

5.4 The area percent porosity to the nearest whole number.
A1. The Influence of Image Analysis Variables**

To determine the influence of image analysis variables, a study examined the effect of changing the magnification and the threshold setting on the area percent porosity that was measured. The study also looked at the field-to-field scatter at two magnifications using a fixed threshold setting.

The sample used was an as-sintered copper steel (FC-0208). At each magnification, an automated square grid stage pattern using 5 by 5 fields was selected, with each pattern starting from the same location. The area tested was reduced as the magnification was increased (doubling the magnification reduced the field area to $\frac{1}{4}$). The threshold level was increased over a total range of 20 points at each magnification with testing performed at gray scale increments that were 5 points apart.

For the field variation tests, the sampled areas came from a stripe taken across the center of the samples in the long direction of the section, using the same starting point. The area of the traverse at 200x was half that of the traverse at 100x because twice the number of fields were examined at the 200x magnification. Both traverses were run at the same threshold setting. The amount of reflection from the sample surface was kept constant by adjusting the lamp intensity as the magnification was changed.

1. Magnification/Threshold Setting (Detection Level) Tests

The amount of scatter around the mean porosity level, between the lowest and highest fields, is least at the lower magnification and becomes greater as the magnification is increased (see Figures A.1 – A.3). As expected, an increase in the threshold setting correlates to an increase in the area percent porosity detected. As the magnification is increased, the slope of the line decreases. In addition, for a given threshold setting, the area percent porosity measured increases as the magnification decreases. The effect of the threshold setting is greatest at the lowest magnification – the area percent porosity is 15.6% at the lowest threshold setting whereas it is 18.7% at the highest threshold setting (a relative difference of about 0.25 g/cm$^3$).

The effect of the detection level setting decreases as the magnification is increased since increasing the magnification increases the resolution of the system. At a magnification of 200x the area percent porosity ranges from 14.1% at the lowest threshold setting to 15.1% at the highest (a relative difference of less than 0.1 g/cm$^3$).

2. Field-to-Field Variation Tests

The amount of variation in the area percent porosity (scatter of points around the mean) is much greater at the higher magnification (see Figures A.4 and A.5). At a magnification of 200x, large differences in area percent porosity can be observed in neighboring fields (the largest difference is > 8% between fields 31 and 32). In contrast, at a magnification of 100x, the greatest difference between neighboring fields is slightly greater than 3% (fields 2 and 3).
3. Conclusions
   a. The effect of the threshold setting is more severe at lower magnifications but field-to-field variation is less.
   b. Higher magnifications are more forgiving to minor differences in threshold setting adjustments but more fields must be examined to compensate for the greater field-to-field variation and to assess the regions of interest accurately.
   c. Higher magnification levels require more time to perform an analysis because more fields need to be measured to provide information for the same sample area. Nevertheless, this is not a problem for automated image analysis (the increase in time is only system/computer time – the preparation, set-up time etc. is the same).

**This study and analysis is courtesy of Tom Murphy of Hoeganaes Corporation.**

### APPENDIX B

#### B1. COMPARABLE STANDARD

ASTM E1245

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Disclaimer

By publication of these standards no position is taken with respect to the validity of any patent rights in connection therewith, and the Metal Powder Industries Federation does not undertake to insure anyone utilizing the standards against liability for infringement of any Letters Patent nor assume any such liability.

MPIF standards are adopted in the public interest and are designed to eliminate misunderstandings between the producer and the purchaser and to assist the purchaser in selecting and obtaining the proper material for his particular product. Existence of an MPIF standard does not in any respect preclude any MPIF member or non-member from manufacturing or selling products not included in this standard or from utilizing procedures or equipment other than those included in this standard.

The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; “Standard for Use of the International System of Units (SI): The Modern Metric System”. Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.

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This Standard, prepared by the Metal Powder Industries Federation, is subject to periodic revision. Suggestions for revision should be addressed to the Metal Powder Industries Federation, 105 College Road East, Princeton, N.J. 08540-6692. Users of Standards are cautioned to secure the latest editions. Complete edition of standards may be obtained from the Federation at the above address.
1. SCOPE

1.1 This guide describes procedures for preparing samples of ferrous powder metallurgy (PM) materials for metallographic evaluation using cross-sectional analysis. Procedures for the preparation of copper-based and aluminum PM materials are covered in separate standards.

1.2 This guide does not cover procedures for the preparation of samples of cemented carbides.

1.3 Because most PM materials are porous, care needs to be taken to ensure that the true level of porosity is revealed and that smeared metal does not remain in the pores after grinding and polishing. Powder forged and metal injection molded samples that do not contain high levels of porosity may be prepared in a manner similar to that used for wrought materials.

1.4 The methods described in this guide are proven practices for the metallographic preparation of porous PM materials. It is recognized that other procedures or materials used in the preparation of a sample may be equally effective and can be used based on material availability and preferences of individual laboratories.

1.5 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. REAGENTS

2.1 Reagent grade nitric acid (concentrated)

2.2 Reagent grade denatured alcohol (histological grade)

2.3 Reagent grade picric acid

3. PROCEDURE

3.1 Sectioning

3.1.1 Sectioning defines the plane (cross-section) to be analyzed. It is one of the most important steps in the sample preparation sequence as more damage can be inflicted on the sample during sectioning than any other step if done incorrectly.

3.1.2 Large samples or those with a complex shape may require a reduction in size by pre-sectioning. Non-precision hacksaws or abrasive cut-off saws can be used for this preliminary size reduction. Pre-sectioning cuts should be made far enough away from the area of interest that there is no risk of damage to the sample.

3.1.3 Choose the wheel abrasive and binder type to match the material that is being cut (for example, the blade may be made from SiC, Al₂O₃, c-BN, or have a diamond impregnated rim). Coolant should be used to minimize heating so that the microstructure of the sample is not altered. It is very important to control the pressure exerted on the sample by the blade as excessive pressure can distort the material and increase the possibility of thermal damage. It is equally important to control both the approach and cutting speed. Excessive approach speed may cause distortion of the material and excessive cutting speed may cause thermal damage.

3.1.4 After any pre-sectioning, make final cut(s) using a precision cut-off (wafering) saw to minimize damage to the sample. If the sample is to be put into a metallographic mount (Section 3.2), the sample should be small enough that any sharp outer features are far enough away from the edges to avoid cracking of the mount.

NOTE 1—Use a fast setting liquid epoxy or other protective liquid or protective coating (e.g., electroless nickel plating) to preserve coatings, surface layers, or the edges of specimens. If the surface is porous or irregular, use vacuum impregnation, if possible, to fill the surface-connected porosity.

NOTE 2—When sectioning a part that has a coating, apply the pressure into the coating, forcing the coating towards the substrate. If the surface is round, rotate the specimen into the blade of the wafering saw.

3.1.5 PM parts often contain residual fluids such as quench oils, cutting fluids, oxide inhibitors, etc. These should be removed before the cross-section is mounted.

3.1.5.1 Soxhlet extraction is often used to remove residual fluids. The technique entails repeated washing of the sample with an appropriate solvent using an apparatus known as a Soxhlet extractor—an example is illustrated in Figure 1.

3.1.5.2 Heating to 100 °C in an oven in air is often sufficient to force liquids from the surface-connected porosity.
3.2 Mounting

3.2.1 Many cross-sections are too small or complex in shape to be prepared in the as-sectioned condition. Grinding and polishing is usually easier and safer if the sections are held in a carrier mount. This also standardizes the size and shape of the sample, which is useful for automated or semi-automated preparation, where multiple mounts are prepared concurrently. In addition, the mount aids in creating a planar surface during grinding/polishing, helps maintain the integrity of edges, and offers protection for delicate, friable, complex sections where the sample might be damaged during grinding and/or polishing. Mounting also helps the preparation of cross-sections of powder particles and small cross-sections.

3.2.2 Thermosetting (diallyl phthalate, epoxy powder, phenolics, and Bakelite), thermoplastic (acrylics, e.g., poly-methyl methacrylate [Lucite®]), or castable materials (epoxy) may be used for mounting the specimens. Castable materials fill porosity and cause less specimen damage than pressure-assisted mounting.

3.2.3 When mounting thin cross-sections, use spring-type clips to stabilize the surface for mounting.

3.3 Grinding and Polishing

3.3.1 Grinding and polishing remove the damaged surface region (damage caused during sectioning) from the sample incrementally using progressively finer abrasive papers, until the true microstructure is reached.

3.3.2 Each successive step must remove the deformation introduced in the previous step. The amount of material removal is affected by the times, pressures, speeds, abrasive concentrations, and coolant used.

3.3.3 Manual Sample Preparation

3.3.3.1 Grind samples using progressively finer abrasive papers. For example, 240, 320, 400, then 600 grit (U.S. Standard designation) SiC abrasive papers.

3.3.3.2 Lubricate and cool the sample with a continuous flow of water.

NOTE 3—Do not use grinding papers for more than 30 seconds to ensure a known grit size paper is being used.

3.3.3.3 Rinse the surface of the sample thoroughly between grinding stages. Rotate the sample 90° before proceeding to the next paper so that the sample can be visually inspected to ensure that the grinding scratches from the previous step are completely eliminated.

NOTE 4—Do not progress to the next grit size grinding paper until all evidence of the previous step has been removed (i.e., the appearance of the new scratch pattern is uniform).

3.3.3.4 An acid etching step (e.g., with 2 or 5 vol.% nital for steel samples – see Section 4.4) prior to the first polishing step is optional. This step helps open the porosity and removes smeared metal chemically rather than mechanically.

3.3.3.5 Rinse the sample thoroughly with running water, rinse with alcohol, and dry with filtered, dry, compressed air.

3.3.3.6 Coarse polish on a hard cloth such as Nylon using a slurry made from distilled or deionized water with 1 μm Al₃O₃ (de-agglomerated). Charge the cloth with slurry at the start of the cycle and periodically as the cloth becomes dry or free of polish. Use moderate to heavy pressure and move the sample in a direction counter to that of the rotation of the polishing wheel. Wash the sample with soap and warm water using a soft material such as cotton and rinse thoroughly with running water. Rinse with alcohol and dry the surface of the sample with filtered, dry, compressed air. Repeat this step until the porosity appears to be open and the appearance of the specimen is uniform from edge to edge. Periodically, clean the Nylon cloth. Keep the surface free of built-up slurry and polishing debris.

3.3.3.7 Fine polish on a soft, napped polishing cloth using a slurry made from distilled or deionized water with 0.05 μm Al₃O₃ (de-agglomerated). Charging of the cloth, the pressure applied to the sample, movement of the sample during polishing, and cleaning of the sample are similar to the procedures used during coarse polishing. Use short polishing times to minimize rounding at pore and sample edges and relief where localized differences in hardness are present. Wash the sample with soap and warm water using a soft material such as cotton and rinse thoroughly with running warm water. Rinse with alcohol and dry the surface of the sample with filtered, dry, compressed air. Drying in a vacuum chamber can be helpful to remove entrapped moisture. Remove any stains by washing with soap and warm water and dry with filtered, dry, compressed air.

NOTE 5—Ultrasonic cleaning, between the coarse and fine polishing steps and after the fine step will help remove polish and debris from the pores.

3.3.4 Automated Sample Preparation

3.3.4.1 Clamp or set the mounted samples in the multi-sample holder in a balanced manner. Group materials with similar chemical composition and hardness so that grinding and polishing occur uniformly on all samples.

3.3.4.2 Grind the samples using progressively finer abrasive papers; for example, 240, 320, 400, then 600 grit (U.S. Standard designation) SiC paper disks. Alternatively, grind the samples using a 120 grit SiC paper disk followed by an interrupted cut grinding disk with 15 μm diamond spray. Do not use grinding papers for more than 30 seconds to ensure a known grit size paper is being used.

NOTE 6—The initial grinding is used to remove the damage caused by sectioning and to establish the plane for further preparation. The preparation of soft ferritic or austenitic materials may be accomplished by starting with a finer grit size abrasive (e.g., 320 or 400 grit). This depends on the extent of prior damage to the surface and how planar the surface is.
3.3.4.3 Apply pressure appropriate for the material (e.g., 40–55 kPa for steel samples) while cooling the samples with a continuous flow of prepared fluid. This should be repeated until evidence of the prior grinding step is removed. Thoroughly rinse the platen and samples between each grinding step. Dry with filtered, dry, compressed air. If the holder is of a type where the mounts are clamped in place, they shall remain clamped during cleaning to maintain parallelism of the sample surfaces. For these holders, clean the holder and the mounts together.

3.3.4.4 An acid etching step prior to the first coarse polishing step is optional (e.g., with 2 or 5 vol.% nital for steel samples). This helps to open the porosity and removes smeared metal.

3.3.4.5 Rinse the sample thoroughly with running warm water, rinse with alcohol, and dry with filtered, dry, compressed air.

NOTE 7—Ultrasonic cleaning, between grinding and the first coarse polishing step will help remove debris from the pores.

3.3.4.6 Coarse polish using two steps. First, polish using a coarse polishing agent (e.g., 6 μm diamond polish for steel samples on a hard cloth such as Nylon or chemotextile) and use a time and pressure combination appropriate for the material (e.g., for approximately 3 minutes using a pressure of 40–55 kPa for steel samples). Charge the cloth at the start of the cycle and periodically thereafter.

3.3.4.7 Clean the samples in an ultrasonic bath. If the holder is of a type where the mounts are clamped in place, they shall remain clamped during cleaning to maintain parallelism of the sample surfaces. For these holders, clean the holder and the mounts together. Wash the polished surfaces using soap and warm water, then thoroughly rinse with running water. Rinse with alcohol and dry with filtered, dry, compressed air.

3.3.4.8 Next, polish on a second, hard cloth (woven or synthetic silk) using a finer polishing agent and appropriate equipment settings (e.g., 3 μm diamond polish for about two to three minutes using a pressure of 40–55 kPa for steel samples). Charge the cloth at the start of the cycle and periodically thereafter.

3.3.4.9 Clean the samples in an ultrasonic bath. If the holder is of a type where the mounts are clamped in place, they shall remain clamped during cleaning to maintain parallelism of the sample surfaces. For these holders, clean the holder and the mounts together. Wash the polished surfaces using soap and warm water then thoroughly rinse with running water and dry with filtered, dry, compressed air.

3.3.4.10 Fine polish—Polish using a fine polishing agent and appropriate equipment settings (e.g., 1 μm diamond polish using a soft, napped cloth for about one to two minutes applying a pressure of 40–45 kPa for steel samples). Charge the cloth at the start of the cycle and periodically thereafter. It may be necessary to apply lubricant more frequently with this step than with the others.

3.3.4.11 Wash the polished samples with soap and warm water using a soft material such as cotton and rinse thoroughly with running warm water. Dry the surfaces of the samples with filtered, dry, compressed air then remove the mounts from the holder. Drying them in a vacuum chamber can help remove entrapped moisture. Remove any stains by washing with soap and warm water and dry with filtered, dry, compressed air.

NOTE 8—Samples should be examined after each polishing step to ensure elimination of scratches, the revealing of porosity, and uniformity in the appearance of the sample. As pores are opened, the reflectivity of the surface changes. Do not remove the mounts from the sample holder until after the final polishing step has been completed satisfactorily. Repeat the polishing step in question if the appearance of the surface is not uniform and free of scratches.

4. ETCHING

4.1 Samples are generally examined initially in the as-polished condition. This reveals the size, size distribution, and shape of any porosity that is present. Color can often be used to reveal other features such as unmelted or free copper in PM steels.

4.2 To reveal the underlying microstructure (the phases and transformation products present in the material) the samples need to be chemically etched or stained (tint etched). This involves either selective material removal or the deposition of thin, chemical layers on the surface of the sample and may be a single- or multiple-step procedure.

4.3 Specific etchants and chemical stains have been formulated to reveal particular transformation products in various alloys. Nital and picral are the most common etchants for iron and steel. Samples should be freshly prepared for etching as passivation films may form over time that inhibit or prevent etching.

4.4 Nital consists of a mixture of nitric acid and alcohol. The most common concentration for iron and low-alloy steels is 2 vol.% nitric acid in alcohol, although higher or lower concentrations may be used for some alloys. Nital reveals ferrite grain boundaries along with other transformation products.

CAUTION—Mixtures above 10 vol.% are unstable and mixtures >5 vol.% concentration should not be stored in sealed containers as they can become explosive.

4.5 Picral consists of a mixture of picric acid and alcohol. The most common concentration used is 4 wt.% picric acid in alcohol. Picral is preferred for structures that contain ferrite and carbides (e.g., pearlite and bainite) but is not effective at revealing grain boundaries in ferrite or differences in microstructural orientation.

CAUTION—Picric acid is a solid and is stored as a water-based slurry. It must not be allowed to dry as it becomes extremely explosive.
4.6 Mixtures of nital and picral are often used to obtain the benefit of both. A combination of 2 vol.% nital + 4 wt.% picral is common.

4.7 Nital or combinations of nital and picral are best applied using a squeegee bottle to pour the chemical onto the surface of the sample. The sample should be gently rocked during the etching procedure. After sufficient etching time, thoroughly rinse the sample with running warm water, rinse with alcohol, and dry with filtered, dry, compressed air.

NOTE 9—If immersing samples in a bath of nital or combinations of nital and picral, change the contents of the container frequently. Alcohol is volatile and the concentration of the etchant will change over time. In addition, chemical residues in the bath from the etching process will interfere with proper etching of the samples.

5. REFERENCES

5.1 Additional information on metallographic sample preparation techniques for ferrous PM materials may be found in the following:


FIGURE 1: Example of the Soxhlet apparatus

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1.2 This guide does not cover procedures for the preparation of samples of cemented carbides.

1.3 Because most PM materials are porous, care needs to be taken to ensure that the true level of porosity is revealed and that smeared metal does not remain in the pores after grinding and polishing. Powder forged and metal injection molded samples that do not contain high levels of porosity may be prepared in a manner similar to that used for wrought materials.

1.4 The methods described in this guide are proven practices for the metallographic preparation of porous PM materials. It is recognized that other procedures or materials used in the preparation of a sample may be equally effective and can be used based on material availability and preferences of individual laboratories.

1.5 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. REAGENTS

2.1 Distilled water
2.2 Anhydrous ferric chloride (FeCl₃)
2.3 Reagent grade hydrochloric acid (HCl)
2.4 Potassium dichromate (K₂Cr₂O₇), Certified ACS Grade
2.5 Saturated sodium chloride (NaCl) solution
2.6 Reagent grade sulfuric acid (H₂SO₄)
2.7 Reagent grade hydrogen peroxide (H₂O₂)
2.8 Reagent grade ammonium hydroxide (NH₄OH)
2.9 Copper ammonium chloride dihydrate (NH₄)₂(CuCl₂)
2.10 Reagent grade denatured alcohol (histological grade)

3. PROCEDURE

3.1 Sectioning

3.1.1 Sectioning defines the plane (cross-section) to be analyzed. It is one of the most important steps in the sample preparation sequence as more damage can be inflicted on the sample during sectioning than any other step if done incorrectly.

3.1.2 Large samples or those with a complex shape may require a reduction in size by pre-sectioning. Non-precision hacksaws or abrasive cut-off saws can be used for this preliminary size reduction. Pre-sectioning cuts should be made far enough away from the area of interest that there is no risk of damage to the sample.

3.1.3 Choose the wheel abrasive and binder type to match the material that is being cut (for example, the blade may be made from SiC, Al₂O₃, c-BN, or have a diamond impregnated rim). Coolant should be used to minimize heating so that the microstructure of the sample is not altered. It is very important to control the pressure exerted on the sample by the blade as excessive pressure can distort the material and increase the possibility of thermal damage. It is equally important to control both the approach and cutting speed. Excessive approach speed may cause distortion of the material and excessive cutting speed may cause thermal damage.

3.1.4 After any pre-sectioning, make final cut(s) using a precision cut-off (wafering) saw to minimize damage to the sample. If the sample is to be put into a metallographic mount (Section 3.2) the sample should be small enough that any sharp outer features are far enough away from the edges to avoid cracking of the mount.

NOTE 1—Use a fast setting liquid epoxy or other protective liquid or protective coating (e.g., electroless nickel plating) to preserve coatings, surface layers, or the edges of specimens. If the surface is porous or irregular, use vacuum impregnation, if possible, to fill the interconnected surface porosity.

NOTE 2—When sectioning a part that has a coating, apply the pressure into the coating, forcing the coating towards the substrate. If the surface is round, rotate the specimen into the blade of the wafering saw.
3.1.5 PM parts often contain residual fluids such as quench oils, cutting fluids, oxide inhibitors, etc. These should be removed before the cross-section is mounted.

3.1.5.1 Soxhlet extraction is often used to remove residual fluids. The technique entails repeated washing of the sample with an appropriate solvent using an apparatus known as a Soxhlet extractor—an example is illustrated in Figure 1.

3.1.5.2 Heating to 100 °C in an oven in air is often sufficient to force liquids from the surface-connected porosity.

3.2 Mounting

3.2.1 Many cross-sections are too small or complex in shape to be prepared in the as-sectioned condition. Grinding and polishing is usually easier and safer if the sections are held in a carrier mount. This also standardizes the size and shape of the sample, which is useful for automated or semi-automated preparation, where multiple mounts are prepared concurrently. In addition, the mount aids in creating a planar surface during grinding/polishing, helps maintain the integrity of edges, and offers protection for delicate, friable, complex sections where the sample might be damaged during grinding and/or polishing. Mounting also helps the preparation of cross-sections of powder particles and small cross-sections.

3.2.2 Thermosetting (diallyl phthalate, epoxy powder, phenolics, and Bakelite), thermoplastic (acrylics, e.g., poly-methyl methacrylate) [Lucite®], or castable materials (epoxy) may be used for mounting the specimens. Castable materials fill porosity and cause less specimen damage than pressure-assisted mounting.

3.2.3 When mounting thin cross-sections, use spring-type clips to stabilize the surface for mounting.

3.3 Grinding and Polishing

3.3.1 Grinding and polishing remove the damaged surface region (damage caused during sectioning) from the sample incrementally using progressively finer abrasives, until the true microstructure is reached.

3.3.2 Each successive step must remove the deformation introduced in the previous step. The amount of material removal is affected by the times, pressures, speeds, abrasive concentrations, and coolant used.

3.3.3 Manual Sample Preparation

3.3.3.1 Grind samples using progressively finer abrasive papers. For example, 240, 320, 400, 600, 800 then 1200 grit (U.S. Standard designation) SiC abrasive papers. Do not use excessive pressure, as this can impregnate SiC into soft materials.

3.3.3.2 Lubricate and cool the sample with a continuous flow of water.

NOTE 3—Do not use grinding papers for more than 30 seconds to ensure a known grit size paper is being used.

3.3.3.3 Rinse the surface of the sample thoroughly between grinding stages. Rotate the sample 90° before proceeding to the next paper so that the sample can be visually inspected to ensure that the grinding scratches from the previous step are completely eliminated.

NOTE 4—Do not progress to the next grinding paper until all evidence of the previous step has been removed (i.e., the appearance of the new scratch pattern is uniform).

3.3.3.4 An acid etching step prior to the first polishing step is optional. This step helps open the porosity and removes smeared metal chemically rather than mechanically. For copper-based materials, a suitable etchant is an acidic ferric chloride solution (5 g FeCl₃ + 30 mL HCl +120 mL H₂O).

3.3.3.5 Rinse the sample thoroughly with running water, rinse with alcohol, and dry with filtered, dry, compressed air.

3.3.3.6 Coarse polish on a hard cloth such as Nylon using a slurry made from distilled or deionized water with 0.3 μm Al₂O₃ (de-agglomerated). Charge the cloth with slurry at the start of the cycle and periodically as the cloth becomes dry or free of polish. Use moderate to heavy pressure and move the sample in a direction counter to that of the rotation of the polishing wheel. Wash the sample with running water, rinse with alcohol, and dry the surface of the sample with filtered, dry, compressed air. Repeat this step until the porosity appears to be open and the appearance of the specimen is uniform from edge to edge. Periodically, clean the Nylon cloth. Keep the surface free of built-up slurry and polishing debris.

3.3.3.7 Fine polish on a soft, napped polishing cloth using a slurry made from distilled or deionized water with 0.05 μm Al₂O₃ (de-agglomerated). Charging of the cloth, the pressure applied to the sample, movement of the sample during polishing, and cleaning of the sample are similar to the procedures used during coarse polishing. Use short polishing times to minimize rounding at pore and sample edges and relief where localized differences in hardness are present. Wash the sample thoroughly with water, rinse with alcohol, and dry the surface of the sample with filtered, dry, compressed air. Drying in a vacuum chamber can be helpful to remove entrapped moisture. Remove any stains by washing with soap and warm water and dry with filtered, dry, compressed air.

NOTE 5—Ultrasonic cleaning, between the coarse and fine polishing steps and after the fine step will help remove polish and debris from the pores.

3.3.4 Automated Sample Preparation

3.3.4.1 Clamp or set the mounted samples in the multi-sample holder in a balanced manner. Group materials with similar chemical composition and hardness so that grinding and polishing occur uniformly on all samples.

3.3.4.2 Grind the samples using progressively finer abrasive papers; for example, 240, 380, 400, 600, 800 then 1200 grit (U.S. Standard designation) SiC paper disks. Do not use grinding papers for more than 30 seconds to ensure a known grit size paper is being used.
NOTE 6—The initial grinding is used to remove damage caused by sectioning and to establish the plane for further preparation.

3.3.4.3 Apply pressure suitable for the material for an appropriate amount of time, while cooling the samples with a continuous flow of prepared fluid. This should be repeated until evidence of the prior grinding step is removed. Table 1 outlines an example of the grinding sequence and parameters for copper-based PM parts. The exact parameters will depend on the equipment being used and the condition of the sample.

3.3.4.4 Thoroughly rinse the platen and samples between each grinding step. Dry with filtered, dry, compressed air. If the holder is of a type where the mounts are clamped in place, they shall remain clamped during cleaning to maintain parallelism of the sample surfaces. For these holders, clean the holder and the mounts together.

3.3.4.5 An acid etching step prior to the first coarse polishing step is optional. This helps to open the porosity and removes smeared metal. For copper-based materials, a suitable etchant is an acidic ferric chloride solution (5 g FeCl$_3$ + 30 mL HCl + 120 mL H$_2$O).

3.3.4.6 Rinse the sample thoroughly with running water, rinse with alcohol, and dry with filtered, dry, compressed air.

NOTE 7: Ultrasonic cleaning, between grinding and the first polishing step is optional. This helps to open the porosity and removes smeared metal. For copper-based materials, a suitable etchant is an acidic ferric chloride solution (5 g FeCl$_3$ + 30 mL HCl + 120 mL H$_2$O).

3.3.4.7 Polish using a polishing agent on a hard cloth such as Nylon or chemotextile and use a time and pressure combination appropriate for the material. Table 2 outlines an example of the polishing parameters for copper-based PM parts. The exact parameters will depend on the equipment being used and the condition of the sample. Charge the cloth at the start of the cycle and periodically thereafter.

3.3.4.8 Between polishing steps, clean the samples in an ultrasonic bath. If the holder is of a type where the mounts are clamped in place, they shall remain clamped during cleaning to maintain parallelism of the sample surfaces. For these holders, clean the holder and the mounts together. Wash the polished surfaces using soap and warm water then thoroughly rinse with running water. Rinse with alcohol and dry with filtered, dry, compressed air.

### Table 1. Example of Grinding Sequence and Parameters for Copper-Based Materials

<table>
<thead>
<tr>
<th>Grinding Paper</th>
<th>Platen Speed (rpm)</th>
<th>Sample Direction (vs. platen)</th>
<th>Sample Speed (rpm)</th>
<th>Pressure (kPa)</th>
<th>Time (min)</th>
<th>Water</th>
</tr>
</thead>
<tbody>
<tr>
<td>380 grit</td>
<td>150</td>
<td>Counter</td>
<td>150</td>
<td>23</td>
<td>3</td>
<td>On</td>
</tr>
<tr>
<td>600 grit</td>
<td>150</td>
<td>Counter</td>
<td>150</td>
<td>23</td>
<td>3</td>
<td>On</td>
</tr>
<tr>
<td>1200 grit</td>
<td>150</td>
<td>Counter</td>
<td>150</td>
<td>16</td>
<td>2x3</td>
<td>On</td>
</tr>
</tbody>
</table>

NOTE 8—Samples should be examined after each polishing step to ensure elimination of scratches, the revealing of porosity, and uniformity in the appearance of the sample. As pores are opened, the light reflectivity of the surface changes. Do not remove the mounts from the sample holder until after the final polishing step has been completed satisfactorily. Repeat the polishing step if the appearance of the surface is not uniform and free of scratches.

4. ETCHING

4.1 Samples are generally examined initially in the as-polished condition. This reveals the size, size distribution, and the shape of the porosity that is present. Any features, such as copper-rich or tin-rich areas in a sintered bronze PM part, are visible at this stage due to their characteristic color. Color could possibly be of use to reveal other features.

4.2 To reveal the underlying microstructure (the phases and transformation products present in the material) the samples need to be chemically etched or stained (tint etched). This involves either selective material removal or the deposition of thin, chemical layers on the surface of the sample and may be a single- or multiple-step procedure.

4.3 Etchants and chemical stains have been specifically formulated to reveal particular transformation products in a particular material—see information on etchant selection and formulation in the references. Samples should be freshly prepared for etching as passivation films may form over time that inhibit or prevent etching.

4.4 The etching protocol (time and method of application) should be defined for the material and the microstructural feature(s) of interest. After sufficient etching time, thoroughly rinse the sample with running warm water, rinse with alcohol, and dry with filtered, dry, compressed air.
5. REFERENCES

5.1 Additional information on metallographic sample preparation techniques for copper-base PM materials may be found in the following:


FIGURE 1: Example of the Soxhlet apparatus

1. Stirrer bar/anti-bumping granules
2. Still pot (extraction pot)
3. Distillation path
4. Soxhlet Thimble
5. Extraction solid (residue solid)
6. Syphon arm inlet
7. Syphon arm outlet
8. Expansion adapter
9. Condenser
10. Cooling water in
11. Cooling water out

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1. **SCOPE**

1.1 This guide describes procedures for preparing samples of aluminum powder metallurgy (PM) materials for metallographic evaluation using cross-sectional analysis. Procedures for the preparation of ferrous and copper-based PM materials are covered in separate standards.

1.2 This guide does not cover procedures for the preparation of samples of cemented carbides.

1.3 Because most PM materials are porous, care needs to be taken to ensure that the true level of porosity is revealed and that smeared metal does not remain in the pores after grinding and polishing. Powder forged and metal injection molded samples that do not contain high levels of porosity may be prepared in a manner similar to that used for wrought materials.

1.4 The methods described in this guide are proven practices for the metallographic preparation of porous PM materials. It is recognized that other procedures or materials used in the preparation of a sample may be equally effective and can be used based on material availability and preferences of individual laboratories.

1.5 This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2. **REAGENTS**

2.1 Reagent grade hydrofluoric acid (concentrated)

2.2 Reagent grade nitric acid (concentrated)

2.3 Reagent grade hydrochloric acid (concentrated)

2.4 Reagent grade denatured alcohol (histological grade)

3. **PROCEDURE**

3.1 **Sectioning**

3.1.1 Sectioning defines the plane (cross-section) to be analyzed. It is one of the most important steps in the sample preparation sequence as more damage can be inflicted on the sample during sectioning than any other step if done incorrectly.

3.1.2 Large samples or those with a complex shape may require a reduction in size by pre-sectioning. Non-precision hacksaws or abrasive cut-off saws can be used for this preliminary size reduction. Pre-sectioning cuts should be made far enough away from the area of interest that there is no risk of damage to the sample.

3.1.3 Choose the wheel abrasive and binder type to match the material that is being cut (for example, the blade may be made from SiC, Al$_2$O$_3$, c-BN, or have a diamond impregnated rim). Coolant should be used to minimize heating so that the microstructure of the sample is not altered. It is very important to control the pressure exerted on the sample by the blade as excessive pressure can distort the material and increase the possibility of thermal damage. It is equally important to control both the approach and cutting speed. Excessive approach speed may cause distortion of the material and excessive cutting speed may cause thermal damage.

3.1.4 After any pre-sectioning, make final cut(s) using a precision cut-off (wafering) saw to minimize damage to the sample. If the sample is to be put into a metallographic mount (Section 3.2), the sample should be small enough that any sharp outer features are far enough away from the edges to avoid cracking of the mount.

**NOTE 1**—Use a fast setting liquid epoxy or other protective liquid or protective coating (e.g., electroless nickel plating) to preserve coatings, surface layers, or the edges of specimens. If the surface is porous or irregular, use vacuum impregnation, if possible, to fill the interconnected surface porosity.

**NOTE 2**—When sectioning a part that has a coating, apply the pressure into the coating, forcing the coating towards the substrate. If the surface is round, rotate the specimen into the blade of the wafering saw.

3.1.5 PM parts often contain residual fluids such as quench oils, cutting fluids, oxide inhibitors, etc. These should be removed before the cross-section is mounted.

3.1.5.1 Soxhlet extraction is often used to remove residual fluids. The technique entails repeated washing of the sample with an appropriate solvent using an apparatus known as a Soxhlet extractor—an example is illustrated in Figure 1.

3.1.5.2 Heating to 100 °C in an oven in air is often sufficient to force liquids from the surface-connected porosity.
3.2 Mounting
3.2.1 Many cross-sections are too small or complex in shape to be prepared in the as-sectioned condition. Grinding and polishing is usually easier and safer if the sections are held in a carrier mount. This also standardizes the size and shape of the sample, which is useful for automated or semi-automated preparation, where multiple mounts are prepared concurrently. In addition, the mount aids in creating a planar surface during grinding/polishing, helps maintain the integrity of edges, and offers protection for delicate, friable, complex sections where the sample might be damaged during grinding and/or polishing. Mounting also helps the preparation of cross-sections of powder particles and small cross-sections.

3.2.2 Thermosetting (diallyl phthalate, epoxy powder, phenolics, and Bakelite), thermoplastic (acrylics, e.g., poly-methyl methacrylate [Lucite®]), or castable materials (epoxy) may be used for mounting the specimens. Castable materials fill porosity and cause less specimen damage than pressure-assisted mounting.

3.2.3 When mounting thin cross-sections, use spring-type clips to stabilize the surface for mounting.

3.3 Grinding and Polishing
3.3.1 Grinding and polishing remove the damaged surface region (damage caused during sectioning) from the sample incrementally using progressively finer abrasives, until the true microstructure is reached.

3.3.2 Each successive step must remove the deformation introduced in the previous step. The amount of material removal is affected by the times, pressures, speeds, abrasive concentrations, and coolant used.

3.3.3 Automated Sample Preparation
3.3.3.1 Clamp or set the mounted samples in the multi-sample holder in a balanced manner. Group materials with similar chemical composition and hardness so that grinding and polishing occur uniformly on all samples.

3.3.3.2 Coarse grind the samples using a pressure of 16 kPa for 45 seconds at 300 rpm using 320 grit (U.S. Standard designation) SiC paper disks with running water.

NOTE 3—The initial grinding is used to remove the damage caused by sectioning and to establish the plane for further preparation.

3.3.3.3 Rinse the sample thoroughly with running warm water, rinse with alcohol, and dry with filtered, dry, compressed air.

NOTE 4—Ultrasonic cleaning, between grinding and the first coarse polishing step will help remove debris from the pores.

3.3.3.4 Coarse polish on a composite disc (MD-Largo pad or equivalent) using a pressure of 20 kPa for 7 minutes at 150 rpm using a 9 μm diamond suspension. Charge the cloth at the start of the cycle and periodically thereafter.

3.3.5 Clean the samples in an ultrasonic bath. If the holder is of a type where the mounts are clamped in place, they shall remain clamped during cleaning to maintain parallelism of the sample surfaces. For these holders, clean the holder and the mounts together. Wash the polished surfaces using soap and warm water then thoroughly rinse with running water. Rinse with alcohol and dry with filtered, dry, compressed air.

3.3.6 Next, fine polish on a satin woven acetate cloth (MD-Dac pad or equivalent) using a pressure of 20 kPa for 5 minutes at 150 rpm using a 3 μm diamond suspension. Charge the cloth at the start of the cycle and periodically thereafter.

3.3.7 Clean the samples in an ultrasonic bath. If the holder is of a type where the mounts are clamped in place, they shall remain clamped during cleaning to maintain parallelism of the sample surfaces. For these holders, clean the holder and the mounts together. Wash the polished surfaces using soap and warm water then thoroughly rinse with running water and dry with filtered, dry, compressed air.

3.3.8 Finally polish on a satin woven acetate cloth (MD-Dac pad or equivalent) using a pressure of 20 kPa for 3 minutes at 150 rpm using a colloidal silica suspension. Turn on water halfway through the cycle. Charge the pad at the start of the cycle and periodically thereafter.

3.3.9 Wash the polished samples with soap and warm water using a soft material such as cotton and rinse thoroughly with running warm water. Dry the surfaces of the samples with filtered, dry, compressed air then remove the mounts from the holder. Drying them in a vacuum chamber can help remove entrapped moisture. Remove any stains by washing with soap and warm water and dry with filtered, dry, compressed air.

NOTE 5—Samples should be examined after each polishing step to ensure elimination of scratches, the revealing of porosity, and uniformity in the appearance of the sample. As pores are opened, the reflectivity of the surface changes. Do not remove the mounts from the sample holder until after the final polishing step has been completed satisfactorily. Repeat the polishing step in question if the appearance of the surface is not uniform and free of scratches.

4. ETCHING
4.1 Samples are generally examined initially in the as-polished condition. This reveals the size, size distribution, and shape of any porosity that is present.

4.2 To reveal the underlying microstructure (the phases and transformation products present in the material) the samples need to be chemically etched or stained (tint etched). This involves either selective material removal or the deposition of thin, chemical layers on the surface of the sample and may be a single- or multiple-step procedure.
4.3 Either immerse or swab the polished sample with Keller’s reagent (190 mL distilled water + 5 mL HNO₃ + 3 mL HCl + 2 mL HF) until the mirror-like finish begins to cloud over. After sufficient etching time, thoroughly rinse the sample with running warm water, rinse with alcohol, and dry with filtered, dry, compressed air.

NOTE 6—Alternative etchants may also be used – see references in Section 5.

5. REFERENCES

5.1 Additional information on metallographic sample preparation techniques for aluminum PM materials may be found in the following:


FIGURE 1: Example of the Soxhlet apparatus

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