

Designation: B311 - 17

# Standard Test Method for Density of Powder Metallurgy (PM) Materials Containing Less Than Two Percent Porosity<sup>1</sup>

This standard is issued under the fixed designation B311; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

# 1. Scope\*

1.1 This test method covers the determination of density for powder metallurgy (PM) materials containing less than two percent porosity and for cemented carbides. This test method is based on the water displacement method.

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. This test method is not applicable if this problem occurs.

- 1.2 With the exception of the values for density and the mass used to determine density, for which the use of the gram per cubic centimetre (g/cm³) and gram (g) units is the long-standing industry practice, the values in SI units are to be regarded as standard.
- 1.3 This standard does not purport to address all of the safety concerns, 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.
- 1.4 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:<sup>2</sup>

B243 Terminology of Powder Metallurgy

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

# E456 Terminology Relating to Quality and Statistics

# 3. Terminology

3.1 Definitions of powder metallurgy (PM) terms can be found in Terminology B243. Additional descriptive material is available in the Related Material section of Vol. 02.05 of the *Annual Book of ASTM Standards*.

#### 4. Summary of Test Method

4.1 Using an analytical balance, the test specimen is first weighed in air and then in water. The density is determined by calculation using Archimedes' principle.

# 5. Significance and Use

- 5.1 For PM materials containing less than two percent porosity, a density measurement may be used to determine if the part has been densified, either overall or in a critical region, to the degree required for the intended application. Density alone cannot be used for evaluating the degree of densification because chemical composition and heat treatment affect the pore-free density.
- 5.2 For cemented carbides, a density measurement is normally used to determine if there is any significant deviation in composition of the carbide grade. For straight tungsten carbide-cobalt grades, the relationship is straightforward. For complex carbide grades (for example, grades containing tantalum carbide or titanium carbide, or both, in addition to tungsten carbide-cobalt), the situation is more complicated. If the measured density is beyond the specified limits, the composition is outside of the specified limits. A measured density within the specified limits does not ensure correct composition; compensation between two or more constituents could result in the expected density with the wrong composition. Density alone cannot be used for evaluating a cemented carbide grade.

# 6. Apparatus

6.1 Analytical Balance—precision single-pan analytical balance that will permit readings within 0.01% of the test specimen mass. The analytical balance shall be supported in a

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.11 on Near Full Density Powder Metallurgy Materials.

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<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



manner to eliminate mechanical vibrations and be shielded from air drafts. See Table 1.

6.2 Weighing Liquid—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.

6.3 *Water Container*—A glass beaker or other suitable transparent container should be used to contain the water.

Note 3—A transparent container makes it easier to see air bubbles adhering to the test specimen and specimen support when immersed in water.

Note 4—For the most precise density determination, the water container should be of a size that the level of the water does not rise more than 2.5 mm when the test specimen is lowered into the water.

6.4 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 that is attached to the suspension wire. For either arrangement, a single corrosion resistant wire—for example, austenitic stainless steel, copper, nichrome—shall be used for the basket and suspension wire. For the maximum recommended diameter of suspension wire to be used for various mass ranges see Table 2.

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.

6.5 *Thermometer*—A thermometer with an accuracy of 0.2 °C to measure the temperature of the water.

#### 7. Preparation of Test Specimens

7.1 A complete part or a section of a part may be used for the test specimen. For the highest precision, the test specimen shall have 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.

7.2 All test specimen surfaces shall be thoroughly cleaned of all adhering foreign materials, such as, dirt, grease, oil, oxide scale, metal powders or assembly materials. For cut specimens, care must be used to avoid rough surfaces to which an air bubble can adhere. A 100-grit sanding or abrasive grinding is recommended to remove all rough surfaces.

#### 8. Procedure

8.1 Weigh the test specimen in air using an analytical balance. This is mass A. This and all subsequent weighings shall be to 0.01% of the test specimen mass.

**TABLE 1 Balance Readability** 

Mass, g	Balance Readable to, g
less than 10 10 to less than 100 100 to less than 1000 1000 to less than 10 000	0.0001 0.001 0.01 0.1
1000 to 1033 than 10 000	0.1

**TABLE 2 Maximum Recommended Wire Diameter** 

Mass, g	Wire Diameter, mm
less than 50	0.12
50 to less than 200 200 to less than 600	0.25 0.40
600 and greater	0.50

It is important that the test specimen, analytical balance and surrounding air be at a uniform temperature when the weighing is performed.

Note 6—For improved reproducibility, the analytical balance should be periodically calibrated with a standard mass that is approximately equal to the test specimen mass.

8.2 Support the container of water over the pan of the balance using a suitable bridge as shown in Fig. 2. 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 air drafts.

8.3 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 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 where immersed in the water and are at the same temperature as the water and balance. Care should also be taken to ensure the surface of the water is free of dust particles.

8.4 Weigh the test specimen and specimen support immersed in water. This is mass B.

8.5 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before. This is mass C. Care should be taken 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.

Note 7—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

8.6 Measure the temperature of the water to the nearest 0.5 °C and record its density E, at that temperature, from Table 3.

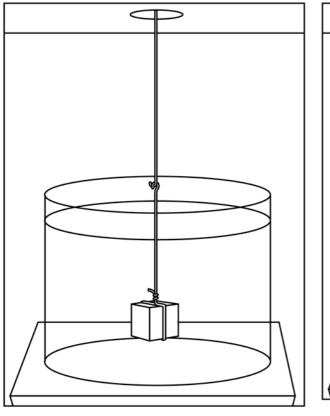
# 9. Calculation

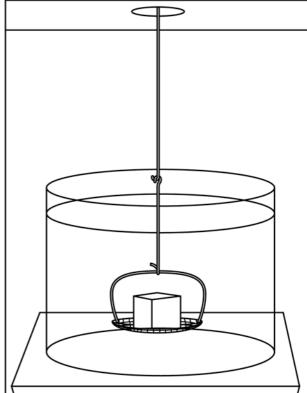
9.1 Calculate the density as follows:

Density = 
$$D = \text{Mass/Volume}$$
 (1)

$$D = A / \frac{\left[A - \left(B - C\right)\right]}{E} \tag{2}$$

$$D = (A \times E)/(A - B + C) = (A \times E)/(A - F)$$
(3)





(a) Twisted wire arrangement

**(b)** Basket support arrangement

FIG. 1 Methods for Holding the Test Specimen When Weighing in Water

## where:

 $D = \text{density of test specimen, g/cm}^3$ ,

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, and

 $E = \text{density of water in g/cm}^3$ .

#### 10. Report

10.1 Report the density rounded to the nearest 0.01 g/cm<sup>3</sup>.

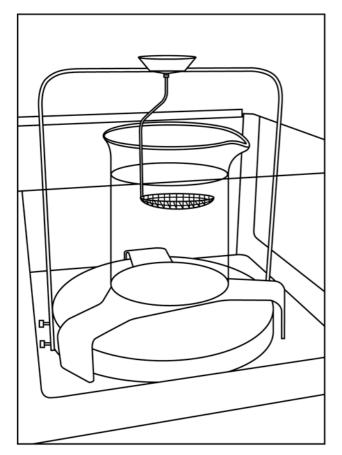
# 11. Precision and Bias

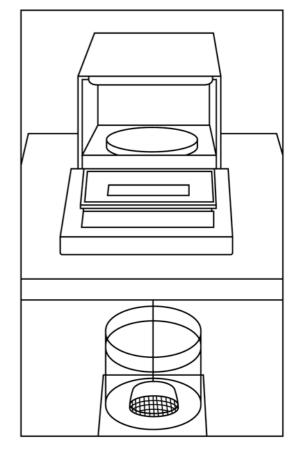
- 11.1 Interlaboratory Test Program—An interlaboratory study of the Density Test Method was run in 1990. Eleven test laboratories tested eighteen different materials. The design of the study followed Practice E691 and a within-between analysis of the data are given in Research Report RR:B09-1014<sup>3</sup>.
- 11.2 *Test Results*—The precision information presented herein has been calculated for the results from eleven laboratories and for duplicate results for each of eighteen materials tested.

# 11.3 Precision:

- 11.3.1 95% Repeatability Limit (within a laboratory)—The within-laboratory repeatability limit, r, as defined by Terminology E456, is estimated to be 0.025 g/cm<sup>3</sup>. At the 95% confidence level, duplicate density test results from the same laboratory should not be considered different unless they differ by more than r.
- 11.3.2 95% Reproducibility Limit (between laboratories)—The between laboratories reproducibility limit, R, as defined by Terminology E456, is estimated to be 0.03 g/cm<sup>3</sup> for test specimens with a mass of greater than 5 g. For test specimens with a mass of from 1 to 5 g it is estimated to be 0.05 g/cm<sup>3</sup>. At the 95% confidence level, duplicate density test results from different laboratories should not be considered different unless they differ by more than R.
- 11.4 *Bias*—No information can be presented on the bias of the procedure in Test Method B311 for measuring Density because no material having an accepted reference value is available.
- 11.5 *Measurement Uncertainty*—The precision of Test Method B311 shall be considered by those performing the test when reporting Density test results.

<sup>&</sup>lt;sup>3</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1014. Contact ASTM Customer Service at service@astm.org.





(a) Beaker support above balance pan

(b) Weighing arrangement below the balance pan

FIG. 2 Methods for Weighing in Water

TABLE 3 Density of Air-Free Water <sup>A</sup>	
Temperature (°C)	Density (g/cm <sup>3</sup> )
18.0	0.9986
18.5	0.9985
19.0	0.9984
19.5	0.9983
20.0	0.9982
20.5	0.9981
21.0	0.9980
21.5	0.9979
22.0	0.9978
22.5	0.9976
23.0	0.9975
23.5	0.9974
24.0	0.9973
24.5	0.9972
25.0	0.9970
25.5	0.9969
26.0	0.9968
26.5	0.9966
27.0	0.9965
27.5	0.9964
28.0	0.9962
28.5	0.9961
29.0	0.9959
29.5	0.9958
30.0	0.9956

<sup>&</sup>lt;sup>A</sup>Metrological Handbook 145, "Quality Assurance for Measurements," National Institute of Standards and Technology, 1990, p. 9.10.

# 12. Keywords

12.1 cemented carbides; density; hard metals; metal injection molded (MIM) parts; powder metallurgy (PM); powder forged (PF) parts; powder metallurgy



#### SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B311 - 13) that may impact the use of this standard. (Approved April 1, 2017.)

(1) Changed the heading of the right-hand column of Table 1 from "Balance Sensitivity, g" to "Balance Readable to, g."

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