Designation: B795 – 13

Standard Test Method for Determining the Percentage of Alloyed or Unalloyed Iron Contamination Present in Powder Forged (PF) Steel Materials[1]

This standard is issued under the fixed designation B795; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers a metallographic procedure for determining the percentage of alloyed or unalloyed iron contamination present in powder forged low-alloy steel materials and the percentage of alloyed iron contamination in powder forged iron and carbon steel materials.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B243 Terminology of Powder Metallurgy

E3 Guide for Preparation of Metallographic Specimens

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy terms can be found in Terminology B243. Additional descriptive information is available in the Related Material Section of Vol 02.05 of the *Annual Book of ASTM Standards*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *cross-product contamination*—the unintentional mixing of powders with distinct differences in chemical composition.

4. Summary of Test Method

4.1 A section representing the core region is taken from the powder forged material and prepared for metallographic examination.

4.2 The polished and etched sample is examined microscopically at a magnification of 100× and a systematic point count made of features with etching characteristics different from that of the matrix.

4.3 The amount of contaminant is reported as a percentage to the nearest 0.1 %.

5. Significance and Use

5.1 Cross-product contamination occurs whenever alloy steel powders are processed in the same equipment as iron powders.

5.2 Unalloyed iron particles, because they may not harden upon heat treatment, are a potential source of soft spots in low-alloy steel parts.

5.3 Alloyed iron particles, having higher hardenability than an iron or carbon steel matrix, are a potential source of hard spots.

5.4 Hard or soft spots may cause problems in service or machining.

5.5 The results of the tests may be used to qualify parts for shipment in accordance with guidelines agreed between purchaser and manufacturer or to check the suitability of mixes for use in powder forging.

6. Apparatus

6.1 Equipment for the metallographic preparation of test specimens.

6.2 A metallographic microscope permitting observation and measurement at a magnification of 100×.

[1] This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Productsand is the direct responsibility of Subcommittee B09.11 on Near Full Density Powder Metallurgy Materials.

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[2] 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.

***A Summary of Changes section appears at the end of this standard**

7. Sampling

7.1 Take a metallographic specimen from the powder forged material. The polished surface of the specimen should be not less than that required to superimpose 2500 grid points at a magnification of 100×. Multiple sections are permitted in order to obtain the necessary area for measurement on small parts or test pieces.

7.2 The polished surface shall be parallel to the direction of forging, that is, parallel to the direction of travel of the forging punch, or as specified in the contract or purchase order, and shall represent an area away from the surface of the material.

8. Procedure

8.1 *Preparation of Specimens:*

8.1.1 *Polishing*—In polishing the specimens, it is highly important that the polished surface be free from artifacts and debris. It is recommended that the procedures described in Practice E3 be followed. Automated grinding and polishing procedures are recommended.

8.1.2 *Etching*—Lightly etch the freshly polished specimen with 2 % nital (2 mL nitric acid, 98 mL ethyl alcohol). Next, etch the polished and lightly etched specimen by immersion in a freshly prepared aqueous solution containing 3 g potassium metabisulfite and 10 g sodium thiosulfate per 100 mL. Rinse the specimen in running water, then rinse with low residue alcohol and dry with a blast of dry air.

8.1.2.1 The etching time will depend on alloy type, carbon content, and microstructure. The greater the alloy content, the slower the etching rate; the greater the carbon content, the faster the etching rate.

8.1.2.2 A good contrast is developed between the matrix and the contaminant because of a combination of etching and staining. The areas containing the highest alloy content are the least affected. Unalloyed iron will become darkened in a low-alloy matrix and low-alloy particles will remain light in an iron or carbon steel matrix. In a low-alloy matrix, contaminant particles of another low-alloy powder can be distinguished from unalloyed iron contamination because the particles etch differently (see Fig. 1 and Fig. 2).

8.2 *Examination*—Superimpose a grid of between 100 and 250 systematically placed points upon a 100× magnified image (that is, a field of view) of the polished and etched specimen. Count and record the number of grid points falling upon contaminant particles; if necessary, a separate count may be kept to distinguish between alloy contamination and unalloyed iron contamination in low-alloy steel materials, or, types of alloy contaminant in iron or carbon steel materials. (See Note 1.) Counting of randomly selected discrete fields should be continued until at least 2500 grid points have been superimposed on the specimen. The total number of points falling on contaminant particles for all fields counted shall be divided by the total number of grid points superimposed and multiplied by 100 to determine the area percentage of contamination.

NOTE 1—Any grid point that falls on a contaminant particle boundary should be counted as one half. To avoid bias, questionable points should be counted as one half.

9. Report

9.1 Report the area percentage of contaminant to the nearest 0.1 %.

10. Precision and Bias

10.1 *Precision:*

10.1.1 The precision of this test method is based on an intralaboratory study of Test Method B795, conducted in 2012. A single laboratory participated in this study, testing one material for alloy contamination and iron contamination. Every "test result" represents an individual determination. The laboratory reported fifteen replicate test results for each analysis. Except for the use of only one laboratory, Practice E691 was

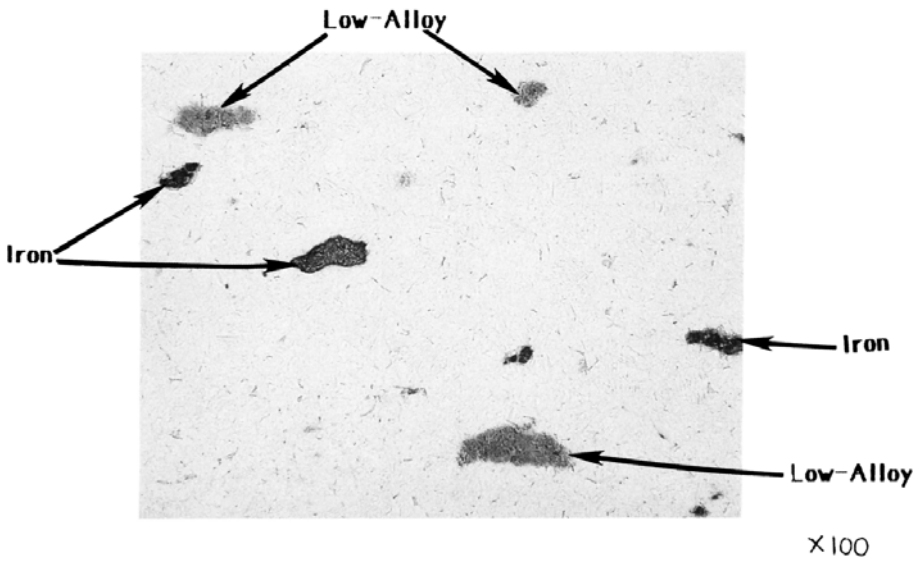

FIG. 1 Illustration of Iron and Low-Alloy Contaminants in PF-4650

FIG. 2 Illustration of Low-Alloy Contaminant in PF-1060

followed for the design and analysis of the data; the details are given in ASTM Research Report No. B09-1019.[3]

TABLE 1 Alloy Contamination (%)

	Average $\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r
A	1.3	0.26	0.73

TABLE 2 Iron Contamination (units)

	Average $\bar{x}$	Repeatability Standard Deviation S_r	Repeatability Limit r
B	1.2	0.20	0.56

10.1.2 *Repeatability (r)*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

10.1.2.1 Repeatability can be interpreted as maximum difference between two results, obtained under repeatability conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

10.1.2.2 Repeatability limits are listed in Table 1 and Table 2.

10.1.3 *Reproducibility (R)*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run and in the normal and correct operation of the test method, exceed the following values only in one case in 20.

10.1.3.1 Reproducibility can be interpreted as maximum difference between two results, obtained under reproducibility conditions, that is accepted as plausible due to random causes under normal and correct operation of the test method.

10.1.3.2 Reproducibility limits cannot be calculated from a single laboratory's results.

10.1.4 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

10.1.5 Any judgment in accordance with statement 10.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results essentially guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit as a general guide, and the associated probability of 95 % as only a rough indicator of what can be expected.

10.1.6 The precision statement was determined through statistical examination of 30 results, from a single laboratory, on the two materials described below. 15 repetitions were performed for each material. The reproducibility of this test method is being determined and will be available on or before December 2018.[3]

10.1.6.1 Material A : Ancorsteel 1000B + 0.22 % graphite + 1 % Ancorsteel 737 SH

10.1.6.2 Material B : Ancorsteel 737 SH + 0.22 % graphite + 1 % Ancorsteel 1000B

10.2 *Bias:*

10.2.1 No information can be presented on the bias of the procedure in Test Method B795 for measuring the percentage of alloyed or unalloyed contamination present in powder-forged steel materials because no material having an accepted reference value is available.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1019. Contact ASTM Customer Service at service@astm.org.

11. Measurement Uncertainty

11.1 The precision of Test Method B795 shall be considered by those performing the test when reporting the percentage of alloyed or unalloyed contamination present in powder forged steel materials.

12. Keywords

12.1 cross-product contamination; powder forging (PF); powder forged (PF) parts and test specimens; powder forged (PF) steels

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B795- 07) that may impact the use of this standard.

(1) Revised statement on units in Section 1.2.
(2) Added repeatability data in Section 10.1.
(3) Added a statement on measurement uncertainty in Section 11.
(4) Added a statement on bias in Section 10.2.