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Standard Test Method for Metallographically Estimating the Observed Case Depth of Ferrous Powder Metallurgy (PM) Parts[1]

This standard is issued under the fixed designation B931; 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~~ Scope*

1.1 A metallographic method is described for estimating the observed case depth of ferrous powder metallurgy (PM) parts. This method may be used for all types of hardened cases where there is a discernible difference between the microstructure of the hardened surface and that of the interior of the part.

1.2 With the exception of the values for grit size for which the U.S. standard designation is the industry standard, the values stated 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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B243 Terminology of Powder Metallurgy

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

E407 Practice for Microetching Metals and Alloys

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy (PM) 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 The metallographically estimated observed case depth is defined as the distance from the surface of the part to the point where, at a magnification of 100X, there is a discernible difference in the microstucture of the material.

4. Summary of Test Method

4.1 The powder metallurgy part is sectioned and the surface prepared for metallographic evaluation. The metallographic specimen is etched and the distance is measured from the surface of the part to the point at which a discernible difference in the microstructure of the material is observed.

5. Significance and Use

5.1 The engineering function of many PM parts may require an exterior portion of the part to have a hardened layer. Where case hardening produces a distinct transition in the microstructure, metallographic estimation of the observed case depth may be used to check the depth to which the surface has been hardened.

6. Apparatus

6.1 Equipment for the metallographic preparation of test specimens—see Appendix X1.

6.2 *Metallographic Microscope,* permitting observation and measurement at a magnification of 100×.

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[1] 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 B09.05 on Structural Parts.

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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. Reagents and Materials

7.1 Etchants such as 2 to 5 % nital, nital/picral combinations, or other suitable etchants. For more information on suitable etchants refer to Practice E407.

8. Test Specimens

8.1 Cut a test specimen from the PM part, perpendicular to the hardened surface at a specified location, being careful to avoid any cutting or grinding procedure that would affect the original microstructure.

8.2 Mounting of the test specimen is recommended for convenience in surface preparation and edge retention. Edge retention is important for proper measurement of the observed case depth.

9. Procedure

9.1 Grind and polish the test specimen using methods such as those summarized in Appendix X1.

9.2 Etch the specimen with etchants such as 2 to 5 % nital or nital/picral combinations.

9.2.1 *Observed Case Depth:*

9.2.1.1 Examine the surface region of the part at a magnification of 100×.

9.2.1.2 Measure the distance from the surface of the part to the point where there is a discernible difference in the microstructure of the material.

NOTE 1—The nature and amount of intermediate transformation products will depend on the material being heat treated, its density, and the type of surface hardening treatment being used. The sharpness of the change in the microstructure at the point of transition will therefore vary. The microstructure expected at this transition point should be agreed between the producer and user of the part. Magnifications higher than 100× may be used to check the microstructure of the part in the region of the transition zone. However, the metallographic estimate of the observed case depth shall be made at a magnification of 100×.

10. Report

10.1 Report the following information:

10.1.1 The type of material and case measured,

10.1.2 The type of etchant used,

10.1.3 The location of the measurement, and

10.1.4 The metallographically estimated observed case depth to the nearest 0.1 mm.

11. Precision and Bias

11.1 The precision ~~that can be expected through the use~~ of this test method is ~~currently under review by Subcommittee B09.05 on Structural Parts.~~based on an intralaboratory study of ASTM B931, Standard Test Method for Metallographically Estimating the Observed Case Depth of Ferrous Powder Metallurgy (PM) Parts, conducted in 2013. A single laboratory participated in this study, testing two different induction-hardened PM parts. Every "test result" represents an individual determination. The laboratory reported 40 replicate test results for each of the materials. Except for the use of only one laboratory, Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. B09-1021[3].

11.1.1 *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.

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

11.1.1.2 Repeatability limits are listed in Table.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1021. Contact ASTM Customer Service at service@astm.org.

	Average[A] $\bar{x}$	Repeatability Standard Deviation s_r	Repeatability Limit r
Sprocket A	880	39.2	110
Sprocket B	560	42.3	120

[A] The average of the laboratories' calculated averages.

11.1.2 *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, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

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

11.1.2.2 Reproducibility limits cannot be calculated from a single laboratory's results. The reproducibility of this test method is being determined and will be available on or before December 2018.

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

11.1.4 Any judgment in accordance with statement 11.1.1 would normally have an approximate 95% probability of being correct. The precision statistics obtained in this ILS must not, however, 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.

11.2 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

11.3 The precision statement was determined through statistical examination of 80 results, from a single laboratory, on two different PM parts described below:

PM sprocket A: induction-hardened case depth of approximately 900 µm

PM sprocket B: induction-hardened case depth of approximately 500 µm

12. Measurement Uncertainty

12.1 The precision of Test Method B931 shall be considered by those performing the test when reporting metallographically estimated case depth results.

13. Keywords

13.1 case depth; observed case depth; PM; powder metallurgy

APPENDIX

(Nonmandatory Information)

X1. SAMPLE PREPARATION

X1.1 The methods described in this appendix are proven practices for metallographic preparation of porous PM materials. It is recognized that other procedures or materials used in preparation of a sample may be equally as good and can be used on the basis of availability and preference of individual laboratories.

X1.2 Method 1

X1.2.1 The porous samples should be free of oil or coolant. Remove any oil using Soxhlet extraction. Mount and vacuum impregnate samples with epoxy resin, to fill porosity and to prevent the pickup of etchants. Use a sample cup or holder to form the mount. Pour epoxy resin over the sample in the cup to a total depth of about ~~0.75 in (19 mm).~~ 19 mm. Evacuate the cup to minus ~~26 in. of mercury (88 kPa)~~ 88 kPa and hold at that pressure for 10 min. Then restore ambient air pressure to force the resin into most of the sample. Cure at room temperature or at ~~122 °F (50 °C).~~ 50 °C.

X1.2.2 Grind on 240, 400, and 600 grit wet SiC paper, on a rotating wheel, and change the polishing direction 90° after each paper. Etch samples for 1 min in their normal etchant, for example, 2 % nital, to begin to open the porosity. Rough polishing for 8 to 12 min total on 1 µm alumina (Al_2O_3), long napped cloth (for example Struers felt cloth), at 250 rpm, and 300 gf load, using an automated polisher opens smeared pores. This rough polishing opens and exaggerates the pores. To return the pores to their true area fraction, polish for 4 min at 125 rpm on a shorter nap cloth (for example Struers MOL cloth), with 1 µm diamond paste. Final polishing is done for 20 to 30 s using 0.05 µm deagglomerated alumina, and a long napped cloth (for example, Buehler Microcloth), at 125 rpm, and 75 gf load, on an automated polisher. Polishing may also be done by hand for the times indicated. The first two polishings require moderate pressure and the final polish requires light pressure.