Designation: B931 – 14

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*

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×.

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.

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

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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.

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 of this test method is 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.

	Average[A]	Repeatability Standard Deviation	Repeatability Limit
	$\bar{x}$	s_r	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

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[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.

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 19 mm. Evacuate the cup to minus 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 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.

X1.2.3 The metallographic structure should be free of smeared porosity. Generally at 800 to 1000×, the edge of a smeared over pore will appear as a thin gray line outlining one side of the pore, and occasionally outlining most of the pore.

X1.3 Method 2

X1.3.1 The specimen should be carefully selected so that it is representative of the region of interest. After selection, the specimen may require sectioning to provide a workable specimen. Sectioning may be made employing an abrasive or diamond wheel.

X1.3.2 Heat should be avoided to prevent occurrence of possible changes in microstructure. If slow feeds are employed, a coolant may not be necessary to avoid temperature buildups. If abrasive wheels are used, then a coolant is often necessary to avoid overheating of the specimen.

X1.3.3 If a coolant is employed, it may be retained within the pores. The lubricant must be removed prior to the preparation of the specimen for examination. This may be accomplished by using a Soxhlet extractor or an ultrasonic cleaner. The extraction condenser is the most efficient and the least expensive.

X1.3.4 Generally, specimens to be evaluated for case depth are mounted to provide edge retention. There are many kinds of mounting compounds available. Most common materials include epoxies (powder or liquid), diallyl phthalate, or Bakelite. Of these, Bakelite is sometimes preferred because it is harder and therefore provides improved edge retention. Bakelite requires equipment to apply heat and pressure, whereas the epoxies do not.

X1.3.5 After mounting, the specimen is ground to provide a flat, stress-free surface. A belt grinder is generally used first with care to prevent heating of the specimen. Grit size is dependent on the preference of the metallographer, although finer grits are preferred.

X1.3.6 The specimen is then hand ground on four emery papers, generally of 240, 320, 400, and 600 grit.

X1.3.7 Etch samples for 1 min in their normal etchant, for example, 2 % nital, to begin to open the porosity.

X1.3.8 Wet polishing follows hand grinding and etching. Several polishing media are employed including diamond paste, magnesia, alumina, etc. Grit size varies between 1 and 0.3 µm and is applied to nap-free cloths such as nylon. To remove remaining scratches and stress, a soft cloth with finer polishing compound is employed. Generally a short napped cloth is preferred. A fine 0.5 µm alumina is recommended. For best results, and to ensure complete freedom of pores from worked metal, repeat the polishing and etching procedure. Final polishing generally requires 3 to 5 min.

X1.3.9 Automated polishing equipment is also available. Automated polishing is accomplished by moving the specimen across a polishing cloth in an abrasive solution undergoing vibrating action. Cloths and abrasives available are numerous and are generally selected by experience of the metallographer.

X1.4 Two additional schemes for the preparation of sintered ferrous materials, one manual and the other automated, are discussed. The first method, basic manual preparation, has most likely been used to prepare more samples for metallographic examination than any other single method. The assumption is made that the sample has been mounted and pre-ground to give a planar surface. Vacuum impregnation with an epoxy resin is recommended for samples to be used in case depth measurement.

X1.4.1 **Basic Manual Sample Preparation**

X1.4.1.1 Grind samples using progressively finer abrasive papers.

(a) Routinely, 240, 320, 400, then 600 grit (U.S. Standard designation) SiC abrasive paper strips are used.

(b) Lubricate and cool the sample with a continuous flow of water.

(c) Rotate the sample 90° before proceeding to the next paper.

(d) Clean the surface of the sample with a soft cloth or paper towel before grinding on each paper.

NOTE X1.1—Do not progress to the next paper strip until all evidence of the previous step has been removed.

X1.4.1.2 Etching prior to polishing. This step is optional.

(a) Etch with 2 or 5 % nital prior to the first polishing step.

(b) Rinse with running water and dry with filtered, dry, compressed air.

X1.4.1.3 Coarse polish—single step.

(a) Use a slurry made of distilled or deionized water with 1 µm Al_2O_3. Polish using a Nylon cloth.

(b) Charge the cloth with the slurry at the start of the cycle and periodically as the cloth becomes dry.

(c) Pressure applied to the sample should be moderate to heavy and movement should be counter to the direction of the polishing wheel.

(d) Wash the sample with soap and water using a soft material such as cotton.

(e) Rinse with running water.

(f) Dry the surface using filtered, dry, compressed air.

(g) Repeat this step until the porosity appears to be open and the appearance of the specimen is uniform from edge to edge.

(h) Periodically clean the cloth. Keep the surface free of built-up slurry and polishing debris.

X1.4.1.4 Fine polish—single step.

(a) Use a slurry made of distilled or deionized water and 0.05 µm Al_2O_3. Polish using a soft, napped, fine, polishing cloth.

(b) Charging of the cloth, pressure applied to the sample, direction of sample movement, and cleaning of the sample are similar to the conditions used in coarse polishing.

(c) Use short polishing times to minimize rounding and relief.

(d) Perform the operations described in X1.4.1.3 *(d)*, *(e)*, and *(f)*.

X1.4.1.5 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X1.4.1.6 Remove any stains by washing with soap and water.

(a) Dry with compressed air.

X1.4.2 **Basic Automated Sample Preparation**

X1.4.2.1 Clamp or set the samples in the multi-sample holder.

(a) Try to prepare materials with similar composition and hardness at one time.

X1.4.2.2 Grind samples using progressively finer abrasive papers.

(a) Use 240, 320, 400, then 600 grit (U.S. Standard designation) SiC paper disks. The use of interrupted cut composite disks in place of most of the grinding papers is also acceptable. The disk is usually charged with 15 or 30 µm diamond spray.

(b) Cool and lubricate with a continuous flow of prepared fluid.

(c) Use pressure of 40 to 55 kPa and a time no longer than 30 s.

(d) Rinse the platen and sample before progressing to the next paper.

(e) Dry the samples using filtered, dry, compressed air.

X1.4.2.3 Etching prior to polishing. This step is optional.

(a) Etch with 2 or 5 % nital prior to the first polishing step.

(b) Rinse with running water and dry with filtered, dry compressed air.

X1.4.2.4 Coarse polish using two steps.

(a) Polish using 6 µm diamond polish on a hard cloth, that is, Nylon or chemotextile.

(b) Polishing time should be approximately 3 min, at a pressure of 40 to 55 kPa.

(c) Charge the cloth at the start of the cycle and at one minute intervals using aerosol propelled diamond spray.

(d) Ultrasonically clean the samples—do not remove from the holder.

(e) Wash polished surfaces using soap and water.

(f) Dry the surface using compressed air.

(g) Polish using 3 µm diamond polish on a second hard cloth, that is, woven or synthetic silk.

(h) Polishing time should be 2 to 3 min at a pressure of 40 to 55 kPa.

(i) Repeat steps X1.4.2.4*(c)* through *(f)* as described above.

(j) Take care not to contaminate the cloth used in the second step of coarse polishing with polish and debris from the first step.

X1.4.2.5 Fine polish.

(a) Polish using 1 µm diamond polish on a soft napped cloth.

(b) Polishing time should be 1 to 2 min.

(c) Perform steps X1.4.2.4*(c)* through *(f)* as described above (use pressure toward the low end of the range).

X1.4.2.6 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X1.4.2.7 Remove stains by washing with soap and water.

(a) Dry with compressed air.

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B931-09) that may impact the use of this standard.

(1) Added a statement on units in Section 1.
(2) Included information on the repeatability of the Test Method in Section 11.
(3) Added a statement on measurement uncertainty in Section 12.

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