Designation: B933 – 16

Standard Test Method for Microindentation Hardness of Powder Metallurgy (PM) Materials[1]

This standard is issued under the fixed designation B933; 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 the determination of the microindentation hardness of powder metallurgy (PM) materials. The test method differs from the approach used for pore-free materials in terms of the precautions required to deal with the porosity.

1.2 This procedure covers tests made with the Knoop or Vickers indenters under loads in the range from 1 to 200 gf.

1.3 Automated testing is not generally 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 9.4.

1.4 A method for converting the directly measured indentation lengths to other hardness scales, for example, HRC is described in Appendix X1.

1.5 *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

E384 Test Method for Microindentation Hardness of Materials

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

3. Terminology

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

4. Summary of Test Method

4.1 Microindentation hardness testing uses a calibrated machine to force a pyramidal-pointed diamond indenter into the surface of the test material under a known test load. The microindentation hardness value is calculated from the indenting force divided by the projected area of the resulting indentation.

NOTE 1—This test method is designed specifically for use on porous PM materials. It is intended to be a companion to Test Method E384. There are specific differences that are intentional such as restricting the applied load to a maximum of 200 gf; otherwise, the details on equipment and procedures in Test Method E384 shall be adhered to. The specific differences relate to the presence of porosity in the PM materials. Special precautions are required during sample preparation to reveal pores and heterogeneous microstructural features so that appropriate test locations may be selected.

5. Significance and Use

5.1 Microindentation hardness testing provides a measure of the hardness of the microstructural constituents of a porous material. It indicates the hardness the material would have if there were no pores present and the material was tested using macroindentation hardness methods. Loads are limited to a maximum of 200 gf to reduce the likelihood of interference from the porosity.

5.2 Microindentation hardness tests allow the evaluation of specific phases, microstructural constituents, and regions or gradients too small for macroindentation hardness testing.

6. Apparatus

6.1 *Microindentation Hardness Testing Machine,* capable of applying the required load, equipped with a Knoop or Vickers indenter, and provision for measuring the length of the diagonals of the indentation.

6.2 Apparatus requirements are summarized in method Test Method E384.

[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.

Current edition approved April 1, 2016. Published April 2016. Originally approved in 2004. Last previous edition approved in 2014 as B933 – 14. DOI: 10.1520/B0933-16.

[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 *Metallographic Etchants,* suitable for the material being tested.

8. Test Specimen

8.1 *Specimen Mounting:*

8.1.1 Sample mounting is recommended for convenience in surface preparation, edge retention, and ease of testing. The sample should be supported adequately in the mounting medium.

8.2 *Specimen Preparation:*

8.2.1 Guidelines for grinding and polishing specimens are provided in Appendix X2.

8.2.2 Care should be taken to ensure that the true area fraction of porosity is revealed throughout the entire cross section of the specimen. It is essential in surface preparation to remove all smeared metal and to identify pores clearly so that they may be avoided during testing.

8.2.3 The specimen should be lightly etched prior to microindentation hardness testing. Careful etching is necessary as heavy etching obscures features and interferes with the measurement of the diagonals of the indentation.

8.2.4 For heat treated steels, swabbing with or immersion in 2 % nital for 4 to 7 s gives an appropriate structure.

9. Procedure

9.1 Support the specimen so that its surface is perpendicular to the axis of the indenter.

9.2 Select a suitable location for testing and an appropriate load and magnification for the test. A 100 gf load is recommended for hardened materials. 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 to be tested. Magnification ranges for various indentation lengths are as follows:

Indentation Length	Magnification	
(μm)	Max	Min
<76	. . .	400
76 to 125	800	300
>125	600	200

9.3 Apply the test load.

9.4 Examine the indentation for possible sources of error such as distorted or unusually large indentations. 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.

9.5 Measure the length of the diagonals of the indentation, taking care to avoid backlash by moving only in one direction. For Knoop microindentation hardness, read the length of the larger diagonal to 0.1 μm. For Vickers microindentation hardness, measure both diagonals to the nearest 0.1 μm and calculate the average.

9.6 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.

10. Calculation or Interpretation of Results

10.1 The Knoop or Vickers microindentation hardness numbers may be calculated using the following formulae or by using tables in Test Method E384.

10.1.1 *Knoop*—Using the units of force and length commonly employed, that is, for force P in gf, and a long diagonal d in micrometres, the Knoop hardness is calculated:

$$HK = 14229\ P/d^2$$

10.1.2 *Vickers*—Using the units of force and length commonly employed, that is, for force P in gf, and the mean of the two diagonals d in micrometres, the Vickers hardness is calculated:

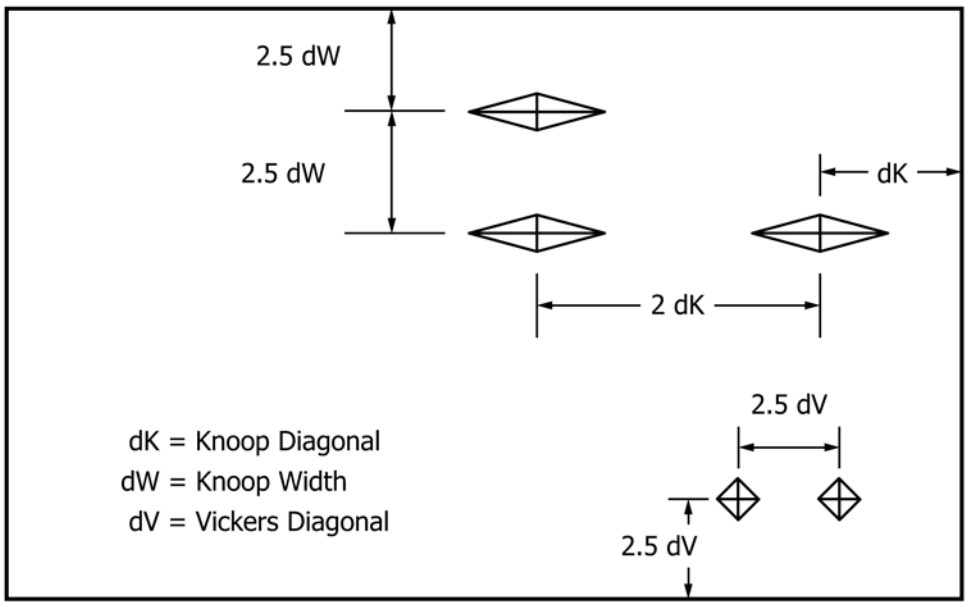

FIG. 1 Minimum Spacing Between Indentations

$$HV = 1854.4\ P/d^2$$

10.1.3 For indentation diagonals measured in millimetres, tables of *HK* and *HV* values are tabulated in Test Method E384.

11. Report

11.1 Report the following information:

11.1.1 The identification of the sample and the location at which the microindentation hardness was measured,

11.1.2 The type of indenter, Knoop or Vickers, and the load used,

11.1.3 The magnification used,

11.1.4 The identity, or description of the phase or microstructural constituent measured,

11.1.5 The type of etchant used, the duration, and method of etching, and

11.1.6 The average of at least five acceptable measurements shall be reported as the microindentation hardness of the material, microstructural constituent, or other feature measured.

11.1.7 Knoop (*HK*) or Vickers (*HV*) microindentation hardness shall be reported along with the test load used, for example, 400 *HK* 100 gf or 400 *HV* 100 gf. This is the preferred method. However, an alternative method expressing the load in kilograms force may be used in accordance with ISO, for example, 400 *HK* 0.1 or 400 *HV* 0.1. Report *HK* and *HV* values to the nearest whole number.

12. Precision and Bias

12.1 The repeatability *r* and reproducibility *R* of measurements were determined in accordance with Practice E691. Members of the Powder Metallurgy Parts Association of the Metal Powder Industries Federation conducted the interlaboratory test program. The test sample was prepared from heat treated FL-4605. One Knoop and one Vickers microindentation hardness indent was made in the surface of the test sample, and these indentations were measured by 12 participating laboratories.

12.2 The mean Knoop microindentation hardness value was 701 *HK* 100 gf with a repeatability of 22 and a reproducibility of 76. Duplicate microindentation hardness results from one laboratory should not be considered suspect at the 95 % confidence level unless they differ by more than 22. For the same test specimen, Knoop microindentation hardness results from two different laboratories should not be considered suspect at the 95 % confidence level unless they differ by more than 76.

12.3 The mean Vickers microindentation hardness value was 716 *HV* 100 gf with a repeatability of 43 and a reproducibility of 178. Duplicate microindentation hardness results from one laboratory should not be considered suspect at the 95 % confidence level unless they differ by more than 43. For the same test specimen, Vickers microindentation hardness results from two different laboratories should not be considered suspect at the 95 % confidence level unless they differ by more than 178.

13. Keywords

13.1 Knoop microindentation hardness; microindentation hardness; PM; powder metallurgy; Vickers microindentation hardness

APPENDIXES

(Nonmandatory Information)

X1. CONVERSION TO OTHER HARDNESS SCALES

X1.1 It is sometimes desired to express microindentation hardness values in terms of equivalents to other hardness scales, for example, HRC. There is no direct conversion from microindentation hardness to HRC. Approximate values can be obtained through the procedure described in this appendix.

X1.1.1 The following procedure describes a method for conversion to HRC.

X1.1.2 Obtain four or five standard HRC test blocks that span the range from the low 20's HRC to the 60's HRC.

X1.1.3 Remove a small portion from each standard test block, being careful to avoid any procedure that might affect the hardness of the test block material, and make a metallographic mount with the standardized face of the test block at the surface of the mount.

X1.1.4 Polish the specimens using standard procedures (see Appendix X2).

X1.1.5 Using either a Knoop or a Vickers indenter and a 100 gf test load (other loads might be used for a conversion to hardness scales such as HRB or HRF), make five indentations at various points in each of the standard specimens.

X1.1.6 Measure the length of the diagonals of the indentations.

X1.1.7 Prepare a graph with the filar units, micrometres, or Knoop/Vickers microindentation 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.

X1.1.8 In future tests, take any diagonal reading and use the graph to convert to HRC.

NOTE X1.1—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.

X1.1.9 *Precision of the Graphical Conversion:*

X1.1.9.1 Seven laboratories participated in an interlaboratory study. Each laboratory developed a regression line for

their own instrument. The regression line was plotted based on the results (six-reading averages) of measurements on five HRC standard test blocks with hardness ranging from 25.4 HRC to 63.2 HRC. The seven laboratories found the hardness of a circulated unknown sample to average 56.5 HRC.

X1.1.9.2 With this test method, 95 % of any future readings would be expected to repeat in a laboratory within 4.0 HRC points at this level; for six-reading averages within 1.6 HRC points. For a laboratory to duplicate any of the other laboratories, 95 % of the readings should be within 5.3 HRC; for six-reading averages within 4.5 HRC.

X2. SAMPLE PREPARATION

X2.1 The methods described in this appendix are proven practices for metallographic preparation of porous PM materials for microindentation hardness testing. 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.

X2.2 *Method 1*:

X2.2.1 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). Evacuate the cup to minus 26 in. of mercury (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).

X2.2.2 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.

X2.2.3 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.

X2.2.4 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels, swabbing with or immersion in 2 % nital for 4 to 7 s gives an appropriate structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite or fine pearlite will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.

X2.3 *Method 2*:

X2.3.1 The specimen should be carefully selected so that it is from the region of interest. After selection, the specimen may require sectioning to provide a workable specimen. Sectioning may be made employing a hacksaw, band saw, abrasive, or diamond wheel. A hacksaw is sufficient for soft materials. If harder materials are of interest, then an abrasive or diamond wheel may be required.

X2.3.2 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 build-ups. If abrasive wheels are used, then a coolant is often necessary to avoid overheating of the specimen.

X2.3.3 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.

X2.3.4 Generally, specimens to be evaluated for microindentation hardness 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.

X2.3.5 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.

X2.3.6 The specimen is then hand ground on four emery papers, generally of 240, 320, 400, and 600 grit.

X2.3.7 Etch samples for 1 min in their normal etchant, for example, 2 % nital for PM steels, to begin to open the porosity.

X2.3.8 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. A short napped cloth is generally preferred. A fine 0.05 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.

X2.3.9 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.

X2.3.10 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels, swabbing with or immersion in 2 % nital for 4 to 7 s gives an appropriate structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite or fine pearlite will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.

X2.4 Two additional schemes for the preparation of sintered ferrous materials, one manual and the other automated, are discussed. The first method, basic manual preparation, probably has 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 microindentation hardness testing.

X2.4.1 *Basic Manual Sample Preparation:*

X2.4.1.1 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 X2.1—Do not progress to the next paper strip until all evidence of the previous step has been removed.

X2.4.1.2 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.

X2.4.1.3 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.

X2.4.1.4 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 X2.4.1.3*(d)*, *(e)*, and *(f)*.

X2.4.1.5 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X2.4.1.6 Remove any stains by washing with soap and water.

(a) Dry with compressed air.

X2.4.1.7 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels, swabbing with or immersion in 2 % nital for 4 to 7 s gives an appropriate structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite or fine pearlite will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.

X2.4.2 *Basic Automated Sample Preparation:*

X2.4.2.1 Clamp or set the samples in the multi-sample holder.

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

X2.4.2.2 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.

X2.4.2.3 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.

X2.4.2.4 Coarse polish using two steps.

(a) Polish using 6 µm diamond polish on a hard cloth, for example, 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 1 min 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 X2.4.2.4*(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.

X2.4.2.5 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 X2.4.2.4*(c)* through *(f)* as described above (use pressure toward the low end of the range).

X2.4.2.6 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X2.4.2.7 Remove stains by washing with soap and water.

(a) Dry with compressed air.

X2.4.2.8 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels, swabbing with or immersion in 2 % nital for 4 to 7 s gives an appropriate structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite and fine pearlite will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B933–14) that may impact the use of this standard. (Approved April 1, 2016.)

(1) In Section 1.2, changed the maximum load permitted from 1 kgf to 200 gf.

(2) Indicated in Section 1.3 that automated testing procedures are not appropriate for porous PM materials.

(3) Added clarification about intentional differences from Test Method E384 in Note 1.

(4) Indicated in Section 5.1, Significance and Use, that loads are limited to a maximum of 200 gf.

(5) In Section 11.1.2, Report, added “and the load used.”