Designation: ~~B933 – 09~~ B933 – 14

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

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

E384 Test Method for Knoop and Vickers 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; 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.

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

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[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 B09.05 on Structural Parts.

Current edition approved ~~April 1, 2009~~April 1, 2014. Published ~~April 2009~~April 2014. Originally approved in 2004. Last previous edition approved in ~~2004~~2009 as B933-~~04.~~09. DOI: ~~10.1520/B0933-09.~~10.1520/B0933-14.

[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**

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.

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. ~~A100~~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 (μm)	Magnification	
	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.

~~9.7 Discard any value if by including this value the hardness range of the other points is more than doubled. In all cases of a discarded value, make a replacement.~~

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:

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

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

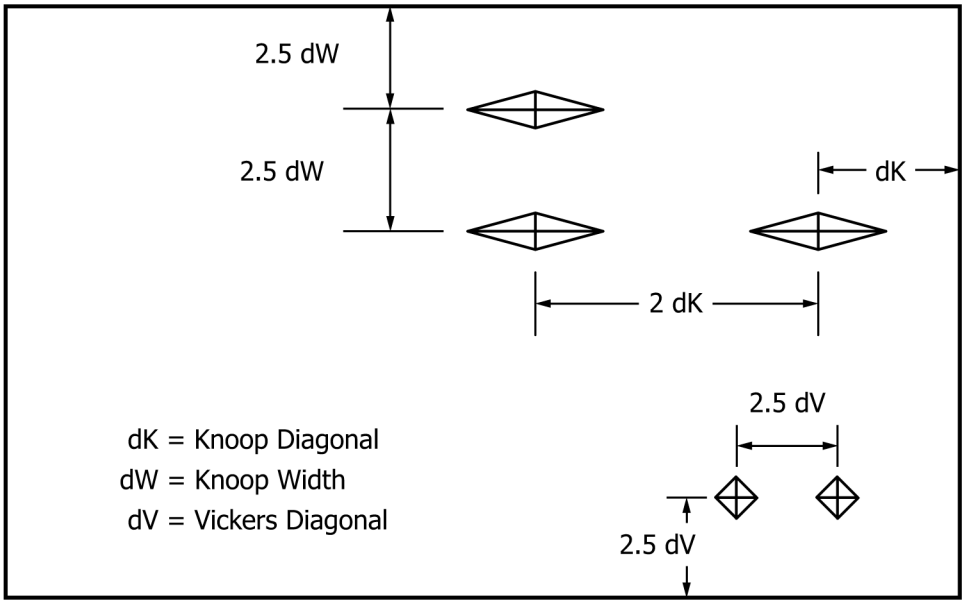

FIG. 1 Minimum Spacing Between Indentations

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,

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 ~~2.2~~4.5 HRC.