METAL POWDER INDUSTRIES FEDERATION

Method for

Determination of Microindentation Hardness of Powder Metallurgy Materials

MPIF Standard 51
Issued 1993, Revised 1994, 2005, 2010
(Formerly included in Standard 37)

STANDARD 51

1. SCOPE

1.1 This standard covers the determination of the microindentation hardness of PM materials. The procedure differs from that applied to pore-free material in terms of the precautions required to deal with the porosity. This procedure covers tests made with the Knoop or Vickers indentors under test loads in the range from 1 to 1000 gf.

1.2 A method of conversion from the directly measured indentor lengths to other hardness scales, e.g. HRC, is described.

1.3 *This standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the potential safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. PRINCIPLES

2.1 Hardness tests consist of forcing an indentor into a test specimen under a prescribed load and carefully controlled conditions. The greater the indentor penetration, the softer the material. In Rockwell hardness testing, one directly measures the depth of penetration and assigns a hardness number to that penetration, e.g. an HRC number. In Vickers and Knoop microindentation hardness testing, a pyramidal-pointed diamond is forced into the test specimen. The hardness is calculated as the indenting force divided by the measured projected area of the resulting indentation.

2.2 Microindentation hardness testing provides the hardness of the fully dense regions in a porous material. It indicates the hardness that the material would have if there were no pores.

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

NOTE 1—Apparent (macroscopic) hardness testing, e.g. with the Rockwell "C" indentor, shows the composite hardness of pores and fully dense regions. The apparent hardness is lower than would be observed if there were no pores.

2.4 The Knoop hardness number is calculated by dividing the applied load in kilograms force by the projected area of the indentation in square millimeters, computed from the measurement of the long diagonal. Because of the unequal angles between the four intersecting faces, (172 °30′ and 130 °), the use of the Knoop indentor results in a shallow, elongated indentation.

2.5 The Vickers hardness number is calculated by dividing the applied load in kilograms force by the projected area of the indentation in square millimeters, computed from the mean of the measured diagonals of the indentations as it uses a square based pyramidal diamond point with an included angle of 136 °.

3. APPARATUS

3.1 A microindentation hardness testing machine capable of applying the required load, equipped with a Knoop or Vickers indentor and a provision for measuring the length of the indentation diagonals, with a precision of 4 microinch (0.1 micrometer).

4. PROCEDURE

4.1 **Specimen Mounting and Preparation** - Guidelines for these procedures are in Appendix A.

4.1.1 Carefully section and mount the test specimen in a suitable medium to allow for ease of handling and polishing.

4.1.2 Polish and lightly etch the specimen to reveal the phases present, as necessary.

NOTE 2—Careful etching is necessary as heavy etching obscures features and interferes with the measurement of the diagonals of the indentation.

4.1.3 Care should be taken so that the true area fraction of the porosity is revealed throughout the entire cross section.

4.2 **Specimen Testing**

4.2.1 Support the surface of the specimen so that it is perpendicular to the axis of the indentor.

4.2.2 Locate a suitable and desired location for testing. Space the indentations so that the distance between them or the edge of the specimen satisfies the requirements shown in Fig. 1 (as per ASTM E384).

4.2.3 Select a suitable load and magnification for the test. A 100 gf load is recommended for hardened steel.

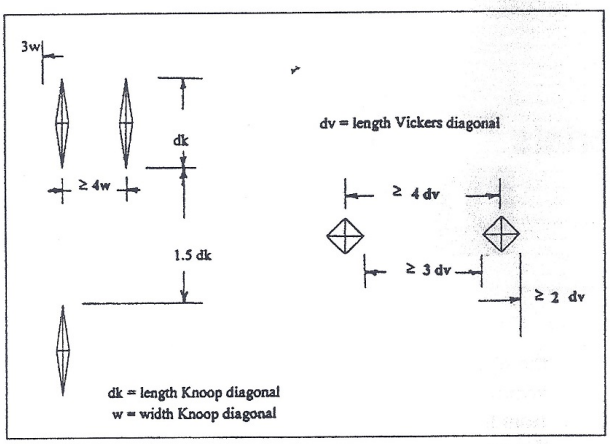

FIGURE 1: Closest Permitted Spacing for Knoop and Vickers Indentations

Figure 1 is reprinted, with permission, from E384-99el Standard Test Method for Microindentation Hardness of Materials, *copyright ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428*

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 being tested. Magnification ranges for indentation length are as follows:

Indentation Length Micrometers	Magnification Max	Min
<76		400
76 – 125	800	300
>125	600	200

4.2.4 Apply test load.

4.2.5 Examine the indentation for possible defects. 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.

4.2.6 Measure the length of indentation diagonal, taking care to avoid backlash by moving only in one direction. For Knoop, read the larger diagonal length to 0.1 micrometers. For Vickers, read both diagonals to the equivalent of 0.1 micrometers and calculate the average.

4.2.7 Discard an individual value if, by including that value, the microindentation hardness range of the other values (the difference between the highest and lowest values in the set of numbers) is more than doubled. In all cases of a discarded value, make a replacement. For example, consider the following five values: 300, 370, 330 and 450. The range for this set of five numbers is 150 (i.e. 450-300). If the value of 450 is excluded, the range becomes 70 (i.e. 370-300). Including the value of 450 more than doubles the range (70) of the other remaining values. The 450 should be discarded and replaced with a new, usable measurement.

4.2.8 Calculations - The Knoop or Vickers numbers may be calculated using the following formulas in this section or tables in ASTM E384.

a. Knoop - Using the units of force and length commonly employed, i.e. for force P in gf, and a long diagonal d in micrometers, the Knoop hardness is calculated:

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

b. Vickers - Using the units of force and length commonly employed, i.e. for force P in gf, and the mean of the two diagonals d in micrometers, the Vickers hardness is calculated:

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

c. For indentor diagonals measured in millimeters, tables of HK and HV values are tabulated in ASTM E384.

5. CONVERSION TO OTHER HARDNESS SCALES

5.1 It is often desired to express microindentation hardness values in other hardness scales, e.g. HRC. There is no direct conversion from microindentation hardness to HRC. Approximate values can be obtained through a procedure described in Appendix B.

6. REPORT

6.1 Identify the test specimen and the location in the test specimen where the hardness was measured.

6.2 Average the hardness number from a minimum of five readings. Knoop (HK) or Vickers (HV) microindentation hardness shall be reported along with the test load used, e.g., 400 HK 100 gf or 400 HV 100 gf. However, an alternative method, expressing the load in kilograms force may be used in accordance with ISO, e.g., 400 HK 0.1 or 400 HV 0.1. Report HK or HV values to the nearest whole number.

6.3 Magnification.

6.4 Identity of phase measured, or description of phase measured.

6.5 For HRC and other converted measurements, whether conversion was made using a procedure other than described in Appendix B.

6.6 The following supplemental information may also be reported:

6.6.1 Material and processing conditions.

7. PRECISION

7.1 The repeatability (r) and reproducibility (R) measurements were determined according to ASTM E691,

Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods and are listed below for ten materials and a range of density values. On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory will be expected to exceed (r) only 5% of the time. If such a difference is found to be larger than (r) there is reason to question one or both results. Similarly, the difference in two test results obtained in different laboratories will be expected to exceed (R) only 5% of the time. If the difference is found to be greater than (R) there is reason to question one or both measurements.

TABLE 1. Precision of Knoop and Vickers Microindentation Hardness

Material	Density g/cm³	# of Labs	Hardness	(r)	(R)
FC-0205	6.39	17	169 HV	42	92
FN-0205	6.93	17	211 HV	44	111
FL-4405	6.67	15	258 HV	31	63
FLN2-4405	6.98	15	268 HV	29	53
FN-0200	6.67	16	118 HK	17	48
FD-0208	6.74	16	301 HK	45	103
FL-4905-HT	6.91	14	721 HV	65	123
FLC-4905-HT	6.98	14	759 HV	70	140
FLC-4808-HT	6.70	14	778 HK	51	217
FL-4808-HT	6.96	14	753 HK	45	240

Repeatability and reproducibility values reported from one specimen, average of three (3) readings per specimen.

APPENDIX A

A1. The method described in this appendix for specimen mounting and preparation is a proven practice. It is recognized that other procedures or materials used in preparation of a sample may be equally as good and used on the basis of availability and preference of individual laboratories.

A2. **Specimen Preparation**

A2.1 Careful preparation and polishing of the specimen is necessary in order to make accurate measurement of diagonal lengths. In porous materials, pores easily become smeared during cutting and grinding. Polishing correctly will open these smeared pores thereby producing an accurate structure.

A2.2 If pores are left smeared, the indentor will encounter these hidden pores, necessitating extra measurements.

A3. **Mounting and Grinding**

A3.1 Remove a suitable specimen from the test piece, by cutting, using sufficient coolant to protect the specimen from overheating. Excessive heating during cut-off may temper the specimen yielding inaccurate hardness measurements.

A3.2 Remove any residual coolant or cut-off fluid from the specimen using Soxhlet extraction.

A3.3 Vacuum impregnate the specimen with epoxy resin and then mount in this same epoxy (e.g., Struers Caldofix or Buehler Epoxide). The resin will fill the porosity in the specimen thereby helping to prevent smearing of the porosity and pick up of corrosive chemicals during etching.

NOTE 3—The mounting media must be strong enough to support the test specimen under load without deflection.

A3.4 After curing the epoxy, grind the specimen successively on 240, 400, and 600 mesh wet SiC paper, preferably using a rotating wheel.

A4. **Manual Polishing**

A4.1 Rough polish by hand for 12 minutes on long napped cloth (e.g. Struers felt cloth) using 1 micrometer alumina at 250 rpm. This procedure removes smeared material and exaggerates the pore area.

A4.2 The second polishing step returns the pores to their true area fraction. Polish for 4 minutes at 125 rpm using medium napped cloth (e.g Struers MOL cloth) and 1 micrometer diamond paste.

A4.3 Final polish for 20-30 seconds on a long napped cloth (e.g Buehler Microcloth) using 0.05 micrometer deagglomerated alumina, at 125 rpm.

A5. **Automated Polishing**

A5.1 Rough polish using an automated polisher with 30 N of force per specimen, 3 micrometer diamond paste and 10 minutes of polishing on Buehler Trident cloth or Struers DAC cloth. This should give a very flat surface, out to the edge of the test specimen.

A5.2 Final polish using an automated polisher for 30 seconds to 1 minute at 3-5 N of force per specimen, 0.05 micrometer alumina suspension, on a long napped cloth such as Buehler Microcloth.

A6. **Etching**

A6.1 Careful etching is necessary as heavy etching obscures phases and changes the microindentation hardness values by interfering with the measurement of the diagonals.

A6.2 For heat treated steels about 4-7 seconds immersion in 2% nital gives an appropriate structure. Rince in alcohol and blow dry with clean air. Martensite will be very light and the dark etching non-martensitic transformation products (fine pearlite or upper bainite) will be evident by contrast.

A6.3 Multi-phase materials should be tested in the lightly etched condition.

APPENDIX B

B1. The following procedure describes a method to convert microindentation hardness values to HRC.

B2. **Use of HRC Standard Test Blocks** - Obtain 4 or 5 standard HRC test blocks spanning the range from the low 20's to the 60's HRC. Remove a small portion of

each block and mount with the standardized face at the surface of the mount and polish the specimens using standard procedures. Using either a Knoop or Vickers indentor, make five (5) sets of indentations at various points in each of the standard specimens. Measure the length in filar units or micrometers for each indentation.

B3. **Graphical Conversion** - 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. In future tests take any diagonal reading and use the graph to convert to HRC.

NOTE 4—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.

B4. **Rockwell C Hardness Conversion Precision** - In an interlaboratory study done in 1994 by the Powder Metallurgy Parts Association (PMPA) Standards Committee members of the Metal Powder Industries Federation, using a hardened prealloyed FL-4605 material at 6.8 g/cm^3, and 56 average HRC microindentation hardness, the reproducibility between laboratories, looking at the identical specimen, but making their own hardness indentations, was 5.3 HRC points. This means that two laboratories, each making only one measurement, should expect that 95% of the time, their results would agree within 5.3 HRC points; for the average of six readings the reproducibility should be 4.5 HRC points. Within a single laboratory, with one operator, repeatability of single measurements should be within 4.0 HRC points. In any one laboratory, repeatability for the averages of 6 readings should be 1.6 HRC points. These tests were done using the 100 gf load on Knoop indentors. Conversion to HRC was as described above, B1.-B3., using pieces from the same standard HRC blocks.

APPENDIX C

C1. COMPARABLE STANDARDS
ASTM E384
ASTM B933

The metric system conversion factors used in this standard are in accordance with IEEE/ASTM SI 10; "Standard for Use of the International System of Units (SI): The Modern Metric System". Recognized as an American National Standard (ANSI), the standard is published by the following organizations: ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; and Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, NY 10017, USA.