Designation: B 721 – 91 (Reapproved 1999)

Standard Test Method for Microhardness and Case Depth of Powder Metallurgy (P/M) Parts[1]

This standard is issued under the fixed designation B 721; 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 determination of the microhardness of powder metallurgy (P/M) parts and applications of microhardness test results to methods for determination of the case depth. Technique for measurement of case depth of P/M parts by observation is also outlined.

1.2 *Part A:* Microhardness Measurement—This procedure covers test methods to determine the microhardness of P/M parts with the Knoop (HK) or the Vickers (HV) indenters. Procedures for surface preparation of the P/M material prior to microhardness measurement are included.

1.3 *Part B:* Case Depth Measurement—Procedures and methods for determination of both effective case depth and observed case depth for P/M parts are included. The principles of Part A on Microhardness Measurement are utilized to measure case depth.

1.4 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are for information only.

1.5 *This standard does not purport to address 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:*

B 243 Terminology of Powder Metallurgy[2]

E 384 Test Method for Microhardness of Materials[3]

3. Terminology

3.1 Definitions of powder metallurgy (P/M) terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material section of Vol. 02.05 of the *Annual Book of ASTM Standards.*

4. Summary of Test Method

4.1 *Part A:* Microhardness is measured by using a calibrated machine to force a diamond indenter of specific geometry, under a known test load, into the surface of the test material.

4.1.1 Ordinarily, the impression of the indenter is measured optically and correlated with available tables to obtain a value in the desired hardness scale; or, the optical measurement can be used to calculate a hardness number.

NOTE 1—This test method is designed specifically for use on P/M parts. It is intended to be a companion to Test Method E 384. There are specific differences that are intentional; otherwise the details on equipment and procedures in Test Method E 384 shall be adhered to.

4.1.2 A new technique, on direct HRC equivalent, is recommended as part of the test method. It involves constructing a plot of microhardness equivalent (HRC) versus the measured diagonal length of the impression made in the material by the indenter. This master plot can then be used to obtain an HRC equivalent directly from the optical measurement.

4.2 *Part B:* Case Depth is defined as the distance from the surface of a part to a point below the surface where:

4.2.1 There is a drop in hardness below some prescribed level,

4.2.2 There is a divergence from a linear decrease in hardness as a function of the distance from the surface, or

4.2.3 There is an easily seen transition in metallurgical structure.

5. Significance and Use

5.1 Especially for P/M materials, microhardness is useful for determination of the actual hardness of the metal matrix. Also, in most metallic materials, microhardness can be used to differentiate between metallurgical phases and non-metallic inclusions. Of particular interest, microhardness tests can be used to determine the actual hardness of the case in surface hardened materials and to help define the useful thickness of such cases.

5.2 Cases, hardened layers, are used on P/M parts and metal parts produced by other methods to provide required properties economically. Proper engineering function of the case requires proper hardness and a specified thickness.

6. Test Specimens

6.1 *Specimen Mounting*:

6.1.1 Mounting is recommended for convenience in surface preparation, edge retention, and testing. The specimen should be adequately supported in the mounting medium.

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[1] This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Productsand is the direct responsibility of Subcommittee B09.05on Structural Parts.

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

[3] *Annual Book of ASTM Standards*, Vol 03.01.

6.1.2 Edge retention is important to proper depth measurement of the case. The mounting material must be selected to provide good edge retention and sufficient rigidity so that no movement of the specimen can occur during the application of load.

6.1.3 At sample densities below 6.6 g/cm^3 it is advisable to vacuum impregnate the specimen with a suitable resin or epoxy to support the structure.

7. Surface Preparation

7.1 Surface preparation is critical to obtaining sound microhardness measurements. With the inherent porosity of most P/M materials it is essential in surface preparation to remove all smeared metal and clearly identify the pores so that they can be avoided during placement of the hardness drops and enhance optical determination that the indent is completely contained within solid structure. Recommended procedures for surface preparation are presented in Appendix X1.

8. Procedure

8.1 *Part A—Microhardness Measurement*:

8.1.1 A microindentation hardness test is made using a calibrated machine to force a diamond indenter of specific geometry, under a test load of 100 gf (0.9807 N) into the surface of the test material and to measure the diagonal or diagonals optically. Optional loads are acceptable only upon agreement between customer and producer.

8.1.1.1 Test Method E 384 presents requirements of the testing machine and optical system for hardness indent measurement.

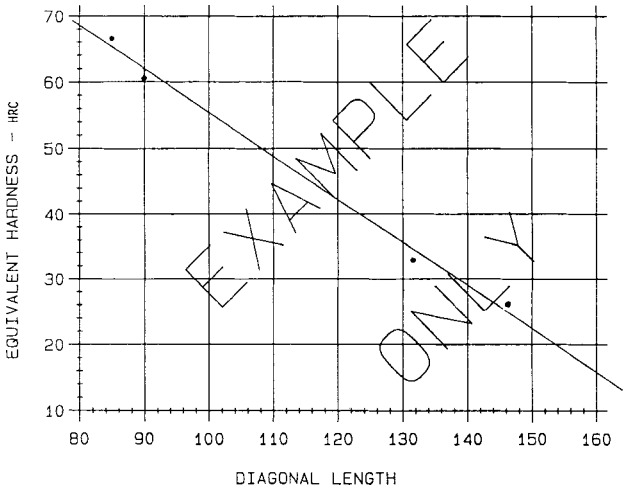
8.1.2 A direct HRC equivalent hardness value is obtained by measurement of the 100 gf (0.9807 N) Knoop or Vickers diagonal length and selecting the corresponding HRC value from an appropriate plot of HRC versus filar unit diagonal length. It is assumed that the indentation is an imprint of the undeformed indenter.

8.1.3 The HRC microhardness versus Knoop or Vickers filar unit diagonal length graph must be individually constructed. Four HRC standard blocks selected over the range 20 to 65 HRC are metallographically mounted, measured with the selected microhardness indenter at 100 gf (0.9807 *N*) and the appropriate diagonal measurement graphed in terms of HRC value. A straight line is drawn between these points and future measurements read directly in HRC value from intersection of the indent diagonal value with the constructed line. (Fig. 1 is an example of this graph.)

8.2 *Part B—Case Depth Measurement*:

8.2.1 The test procedure covers determination of case depth in powder metal parts utilizing the prescribed principles presented in Part A when the measurements are taken on a properly prepared section of the part where that section's geometric relationship is 90 ± 5° to the surface of the part.

8.2.2 Case depth will be defined as either effective or observed case depth. Effective case depth is determined by measurement of microhardness at a series of known distances from the part surface to a designated hardness level. Observed case depth is determined by measurement of the distance from the surface of the metallographically observed case to core transition zone structure.



NOTE 1—Actual curve must be developed by the user laboratory.

FIG. 1 Example of HRC Equivalent Microhardness versus Diagonal Length of the Microhardness Indent

8.2.3 *Effective Case Depth Procedure*:

8.2.3.1 At a determined distance from the part surface, make a minimum of three acceptable measurements. Repeat this procedure at incremental distances from the part surface maintaining a distance between impressions of at least 2.5 times the width of the smallest diagonal. Measurements obviously deformed due to underlying void should be discarded. Indentations should not be placed in soft phases such as copper or the centers of nickel-rich austenite regions. Randomly encountered fine pearlite in the martensite should not be excluded as a measurement location. A lightly etched surface is helpful in defining these regions, such as etching for about 6 to 7 s in 2 % Nital.

8.2.3.2 Effective case depth will be determined by the distance from the surface, beyond which, the hardness is below 50 HRC or an agreed upon value. When the hardness versus depth relationship is graphed, effective case depth will be at the divergence point in the linear microhardness to surface depth relationship indicated in Fig. 2.

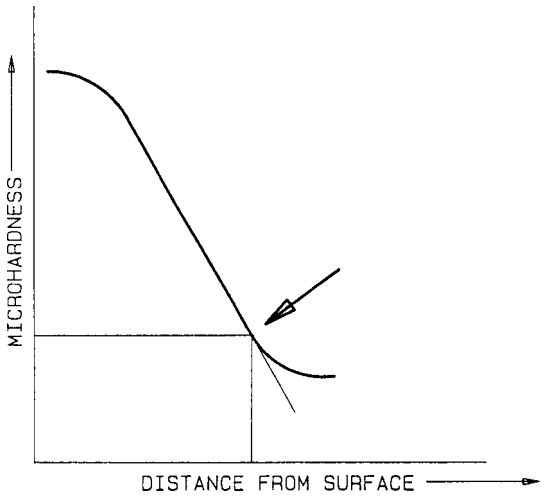


FIG. 2 Linear Relationship of Edge Distance versus Case Microhardness

8.2.3.3 Effective case depth determined by variance from a customer-producer agreed upon value, which is often 50 HRC, will be the distance from the part surface to the point where the microhardness falls below that specified value on a graph of hardness versus depth relationship. The microhardness will be the average value of three acceptable impressions and the case depth the average distance those impressions lie from the part surface.

8.2.3.4 Effective case depth determined by divergence in the linear microhardness to surface distance relationship will be resolved by plotting the average value of three acceptable impressions taken at incremental distance from the part surface versus the average distance of the three averaged impressions from the surface. The effective case depth determined by this technique will be the second point at which the microhardness diverges from the linear relationship with surface depth as illustrated in Fig. 2. A vertical line from that point of divergence to the *x* axis of the plot will determine the case depth and a horizontal line from the point of divergence to the *y* axis will determine the microhardness of the case hardened structure.

8.2.4 *Observed Case Depth Procedure*:

8.2.4.1 In those materials where a metallurgically determined transition zone between case and core structure can be resolved at magnifications of 50 to 100× the case depth will be determined by measurement of the distance of the part surface to the beginning edge of that zone. The beginning of the transition from case to core is characterized by the appearance of fine pearlite colonies mixed in with the martensite. Using a visual estimate, the distance in from the surface where approximately 5 % of the area is fine pearlite shall be defined as the case depth.

9. Report

9.1 The report shall include:

9.1.1 The method of microhardness measurement: HK, HV, HRC/HK, or HRC/HV. In each case, the load used in testing shall be expressed as a subscript, for example, HK_{100}.

9.1.2 The method of case depth measurement.

9.1.3 The case depth values.

9.1.3.1 The effective case depth is the distance from the part surface at which the measured value falls below the specified microhardness value or the microhardness value and distance from the part surface that divergence from the linear microhardness to surface distance relationship occurs.

9.1.3.2 For observed case depth the distance from the part surface up to, but not including the case to core transition zone, the microhardness of the case measured at the case edge of that transition zone, and the magnification at which the measurement was taken.

10. Precision and Bias

10.1 *Precision*—Using individual regression lines based upon six-reading averages for each of five HRC test blocks ranging from 25.4 HRC to 63.2 HRC seven laboratories found values of a circulated unknown to average 56.5 HRC. With this 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 HRC.

10.2 *Bias*—No bias can be defined since there is no way to define true hardness, and therefore any innate deviation from true hardness.

APPENDIX

(Nonmandatory Information)

X1. SAMPLE PREPARATION

X1.1 The methods described in this Appendix are proven practices for metallographic preparation of a microhardness sample. 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 cut-off fluid, using Soxhlet extraction if needed. The samples are then vacuum impregnated with, and mounted in, epoxy resin, to fill porosity and to prevent pick-up of etchants. Using a sample cup or holder to form the mount, a ¾-in. (19 mm) deep layer of epoxy resin is poured over the sample in the cup. The cup is evacuated to − 26 in. Hg (100 Torr) and held at that pressure for 10 min. Ambient air pressure is restored, forcing the resin into most of the sample. Curing can be done at room temperature or accelerated at 50°C.

X1.2.2 The cured mounts are ground on 240, 400, and 600 mesh wet SiC paper, on a rotating wheel, with the polishing direction changed 90° after each paper. Samples are etched for 1 min. in their normal etchant, for example, 2 % Nital, to begin to open the porosity. Rough polishing opens smeared pores: 8 to 12 min total on 1 µm alumina (Al_2O_3), long napped cloth (for example, Struers felt cloth), 250 r/min, 300 g load, automatic polisher. This polishing opens and exaggerates the pores. The pores are then returned to their true area fraction of porosity by polishing for 4 min at 125 r/min on shorter nap cloth (for example, Struers MOL cloth), with 1 µm diamond paste. Final polishing is done for 20 to 30 s on 0.05 µm deagglomerated alumina, long napped cloth (for example, Buehler Microcloth), 125 r/min, 75 g load, automatic 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 grey 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 a hacksaw, band saw, abrasive, or diamond wheel. For soft materials a hacksaw is sufficient; however, if harder materials are of interest, then an abrasive or diamond wheel may be required.

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 build-ups. If abrasive wheels are used, then often a coolant is necessary to prevent heating of the specimen.

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

X1.3.4 Generally, specimens to be evaluated for microhardness are mounted to provide edge retention.[4] There are many kinds of mounting compounds available. Most common materials include epoxide or bakelite. Of the two, bakelite is preferred because it is harder and therefore provides improved edge retention. Bakelite requires equipment to apply heat and pressure, whereas the epoxides 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 upon 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.[4]

X1.3.7 Hand grinding is followed by wet polishing. 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.

X1.3.8 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.

[4] For more specific details concerning specimen mounting and grinding procedures, consult Kehl, "The Principles of Metallurgical Laboratory Practice," *ASM Handbook*, American Society of Metals, or other texts containing instructions on prescribed metallographic practice.