Microhardness Testing

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1 Introduction

Microhardness testing is an indentation hardness method that can aid in evaluating production characteristics, degradation (during service or post-service exposure), and the uniformity of the material throughout a component. Common applications include determining:

- local hardness in a limited region (such as case depth, multiple layers, or surface degradation)
- hardness of metals embedded in a secondary material (metallographic mounts)
- hardness of parts too thin or too small to register valid results via standard (macro) indentation testing,

2 Scope

This document applies to case working personnel using the associated instrument in support of metallurgy examinations.

This document outlines the basic process for Knoop or Vickers microhardness testing using equipment with a digital filar optical measuring system and automated hardness calculation capability. Metallographic preparation of specimens is detailed in METAL-450.

3 Principle

To measure microhardness, a diamond indenter of specified shape is pressed, under load of up to 1000 grams, into a polished metal surface. The indenter shape is specific to the type of test performed, Knoop or Vickers. The size of the resulting impression is measured microscopically and related through the appropriate correlation equations to hardness scales. Since the Vickers and Knoop indenters are shaped differently, the choice of test type will depend on the shape of the feature(s) to be characterized.

4 Specimens

Microhardness specimens are typically sectioned, ground, and polished. Such destructive testing must be approved by the contributor prior to specimen preparation.

At a minimum, a specimen subjected to microhardness testing must be large enough to receive three hardness indentations, all of which are sufficiently spaced to prevent interactions between the measurements. The specimen thickness must exceed two times the depth of the indentations for a homogeneous material and potentially more for unfavorably oriented material. An appropriate combination of the test loads and the indenter shape must be chosen to accommodate these requirements.

5 Equipment

- Microhardness tester with integral diamond indenters, magnifying objectives, optical filar measuring system, and automated hardness calculation

- Certified Reference Material (CRM) test block(s) appropriate for the parameters being used, i.e., only use Vickers hardness standards with the Vickers diamond indenter and Knoop hardness standards with the Knoop diamond indenter
- Specimen fixture or fixturing material, e.g., beeswax or clay or universal specimen holder

6 STANDARDS AND CONTROLS

Adequate instrument performance is verified using at least one CRM test block(s) every time the instrument is powered up for use or the indenter or test scale is changed. CRM blocks must be certified for the scale (indenter and load) of the test to be performed in order to assure the indenter is seated properly, it contains no defects, and the load application is reliable.

7 SAMPLING

Whole components, or sections thereof, are typically presented to the indenter for microhardness testing. Parts are often sectioned, mounted in polymer, ground, and polished to test a particular region of interest. Microhardness readings should be interpreted to apply only to that material in the immediate vicinity of the indentation. Since cold work and thermal gradients during heat treatment can alter hardness, regions of a component with differing thickness, geometry and/or surface treatment should be examined separately.

8 PROCEDURE

8.1 Specimen Preparation

A. The test surface must be flat and have a good quality surface finish so that the edges of test indentations can clearly be observed. Follow steps for sectioning, mounting, grinding, and polishing in METAL-450 as needed. Polishing to a 1 μm finish typically produces a satisfactory surface.

B. Features of interest may be exposed by etching (e.g., weld heat affected zone or case-hardened region.) Follow steps for etching in METAL-450.

C. The specimen must be supported such that the test surface is perpendicular to the load axis.
   1. If the specimen is to be mounted by placing it directly on the instrument stage, the back of the test specimen (the support surface) must be free from debris and parallel to the test surface.
   2. A universal specimen holder can be used to present the flat test surface of an irregularly shaped part to the indenter in the correct orientation.

8.2 Instrument Operation

8.2.1 Indentation Placement

A. Align a region of interest of the test specimen beneath the indenter.

1. Flat test surfaces must be perpendicular to the axis of load application.

2. Assure that the indentation will be placed not less than two and one half indentation widths from any edge or change in part geometry.

3. Assure that multiple indentations on a single surface are separated by at least three indentation widths to avoid interference by plastically deformed or damaged material.

B. Secure the specimen on the platen or in a gripping fixture in a manner that will support the applied test load perpendicular to the load axis and not allow the specimen to move during the load application. Beeswax or clay can be used to secure the side of a specimen to the platen but must not be present between the platen and the back of the specimen.

8.2.2 *Data Acquisition*

A. Focus the surface region to be tested with the objective lens that will be used for measuring the indentation, typically 5X, 10X or 50X magnification. Adjust the illumination to permit comfortable viewing.

B. Assure the ocular filars are sharply in focus. Adjust the ocular if necessary, then refocus on the specimen surface.

C. Select the test load to be applied and select the appropriate diamond indenter (Knoop or Vickers) for the test being performed.

D. Apply the test load. Never attempt to rotate the turret during a test as it will damage the instrument. When the test is complete, the measurement objective moves over the specimen automatically.

E. Observe the location of the indentation in the field and refocus if necessary. The indentation should be straight, symmetrical, and approximately centered on the ocular filar. If it is not, the test is invalid and must be repeated on a location sufficiently spaced from this indentation. If a second indentation is not straight and symmetrical, check the specimen mounting to assure a flat, perpendicular plane is presented to the indenter and the underside is well supported.

1. Significant deviations from perpendicular are readily detected by a lack of symmetry in the indentation. If this is observed, the sample must be refinished prior to proceeding or the back of the sample mount must be ground flat and parallel with the test surface.

i. For a Knoop test, a difference in length of greater than ~10% in the two portions of the long diagonal to either side of the short diagonal requires leveling the sample surface.

ii. For a Vickers test, if one half of either diagonal is more than 5% longer than the other, or if all four corners of the indentation do not focus simultaneously, then the sample surface requires leveling.

iii. If leveling the specimen does not correct the problem, the indenter should be replaced and the performance check repeated prior to proceeding.

F. Place both filars at one vertex (end point) of the indentation (long indentation axis for Knoop) side-by-side with no space between them and zero the measurement scale. Adjust the measurement filar to the opposite vertex of the indentation. To mitigate errors from any rotational slack in the dial, always move the filar into position from the same direction of dial rotation. Press the button on the ocular to accept the length value. For Vickers testing, rotate the filar ocular 90 degrees, and measure the second diagonal. The tester will automatically compute and display the indentation length(s) and the hardness on the digital read-out.

G. Calculations for determining Knoop or Vickers hardness number from the linear indentation dimensions are performed by the instrument software.

8.3 Verify Instrument Performance

A. Select an appropriate combination of indenter type (Knoop or Vickers) and load for the evidentiary specimens to be tested from the instrument control panel. The test load and indenter type used are dependent upon the hardness and shape of the material in the test region.

1. The Knoop indenter's geometry creates indentations of accurately measurable lengths with light testing loads (1000 g or less). The indenter is very sensitive to surface flatness and perpendicularity to the indenter movement and to surface finish. The penetration depth of a Knoop diamond indent is only about one-thirtieth of the longer diagonal length. This shallow indentation makes it well-suited for measuring the hardness of thin plating layers, case hardening, thin metal and foils, decarburized regions in steels, and hard, brittle materials.

2. Because the indentation area is smaller, the Vickers test is better suited for testing microscopic particles. It can be used when the region of interest is too small to accommodate the elongated Knoop indentation. Microhardness Vickers indentations are limited to loads of 1000 g or less.

3. For the same indenter on the same material, lighter loads produce smaller indentations. Select a load and indenter combination that produces an indentation that is large enough to measure in the ocular, but small enough to be within the region of interest on the microstructure. This determination

can be done iteratively by making several preliminary indentations on the test specimen.

B. Select a CRM test block for the test scale of interest.

C. Set the instrument to apply the selected test type (Knoop or Vickers) and load.

D. Make one indentation to seat the specimen. (Do not record this value.)

E. Perform three indentations and record the values in the instrument logbook. Adjust the test position away from the center of any previous indentations by at least three indentation widths to avoid interference by plastically deformed or damaged material.

   1. If all three hardness values are within the hardness range reported on the CRM certification sheet, record the PASS status in the instrument logbook. Certified hardness ranges and uncertainties are reported on certificates of analysis in hardness number units and/or in diagonal lengths (μm). The equations in section 9 Calculations can be used to calculate hardness number from diagonal length(s).

   2. If any of the three hardness values fall outside of the hardness range reported on the CRM certification sheet:

      i. Wipe the support contact surfaces (sample base, platen, grip prongs, etc.) with a clean cloth to remove any debris from the standard and retest to verify.

      ii. If any of the retest values fall outside of the hardness range reported on the CRM certification sheet, replace the indenter and re-verify.

      iii. Calculate the average of the final three microhardness values.

         a. If the average falls outside of the hardness range reported on the CRM certification sheet, the instrument must be serviced and recalibrated by a service provider that meets the FBI Laboratory requirements. Record the FAIL status in the logbook.

         b. If the average falls within the hardness range reported on the CRM certification sheet, calculate the error, E, and repeatability, R, for comparison to the maximum tolerances allowed per ASTM E384.

1. If E and R are within the ASTM E384 tolerance, record the PASS status in the logbook.

2. If either E or R are outside of the ASTM E384 tolerance, record the FAIL status in the logbook. The instrument must be serviced and recalibrated by a service provider that meets the FBI Laboratory requirements.

F. Record the date, operator, test type (Knoop or Vickers), test load, measured values, and performance check result in the instrument log.

8.4 Specimen Testing

A. Align the prepared specimen in the instrument and acquire a minimum of three hardness measurements.

1. If the measured hardness is outside of the scale that was initially verified, re-verify the instrument using the appropriate scale with appropriate test block(s).

2. If comparative statistics are required, acquire additional readings in the areas of interest. Typically, 5 to 10 measurements per area will provide sufficient data for statistical comparisons.

B. Record the hardness values, load used, and test type in the case notes.

9 Calculations

9.1 Quantitative Analysis

A. Microhardness values are automatically calculated by the instrument software. These readings, and their associated measurement uncertainty, are used to report a range or series of measurements, e.g., the decrease of microhardness from the surface to the interior of a case-hardened specimen. The following equations can be used to relate diagonal measurements to hardness values:

1. The hardness number for Knoop (HK) is calculated as:

$$HK = 14.229 \times (P/d^2)$$

where:

d is the length of the long diagonal in microns (µm) and

P is the test load (force) in grams-force (gf).

2. The hardness number for Vickers (HV) is calculated as:

$$HV = 1854.4 \times (P/d^2)$$

where:

d is the mean diagonal length in microns (µm) and

P is the test load (force) in grams-force (gf).

B. Mean is calculated as: $\bar{x} = \frac{\sum_{i=1}^{n} x_i}{n}$, where $\sum_{i=1}^{n} x_i$ is the sum of the measurements, n is the number of measurements, and $\bar{x}$ is the mean hardness value.

C. Estimate measurement uncertainty as detailed in CHEM-100. Include the repeatability component of the evidentiary specimen.

9.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed Student's t-test statistic of the sample means will be used for the comparison, as detailed in METAL-210, Calculations section. Typically, five to ten measurements per sample are used for performing comparisons.

9.3 Hardness Conversion Estimate

ASTM E140 provides tables correlating hardness values between scales. The tables are material specific. To report an estimated conversion, report the original measurement and scale used, as well as the converted value and scale. Reference ASTM E140 and the table in the report.

10 Measurement Uncertainty

Quantitative data from this procedure are sometimes used for comparative purposes. Expanded measurement uncertainty should not be used for these inter-comparisons because it increases the probability two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion). Instead, the variances associated with the samples and with data acquisition are accommodated by the statistical comparison.

When quantitative data are compared to a particular specification or when quantitative measurements are reported, the estimated measurement uncertainty will be calculated in accordance with CHEM-100.

Measurement uncertainty is expected to be significantly influenced by composition, heat treatment, part geometry, and other factors. Since these factors are specimen dependent, the measurement uncertainty will be calculated on a case-by-case basis. When an average of multiple measurements is reported, the measurement uncertainty will include the repeatability of the evidentiary specimen hardness values.

Measurement uncertainty will not be calculated for converted hardness values due to the approximate nature of the conversions.

11 ACCEPTANCE CRITERIA

11.1 Calibration

The system is calibrated annually by a service provider that meets the FBI Laboratory certification requirements. Calibration is demonstrated by the service provider for the Knoop and Vickers scales.

11.2 Performance Check

Prior to the first use of a microhardness tester to make a significant measurement on a given day, the user will verify the calibration on the appropriate test scale and enter the verification results in the instrument logbook. See 8.3 Verify Instrument Performance. Verification is acceptable when the hardness values determined by the instrument on a given CRM fall within the range of values indicated on its certificate or if calculated E and R are within the maximum tolerances allowed. If the verification remains unsuccessful after assuring that the test block and indenter are sound, then the instrument must be serviced and re-calibrated by a service provider that meets the FBI Laboratory requirements.

11.3 Quantitative Analysis Conclusions

Variability in hardness measurements may be metallurgically significant (e.g., resulting from grain size differences, heat affected zones, or case-hardened regions.) If this is observed, the results should not be averaged but should be reported individually or as a range for the area tested.

11.4 Comparative Analysis Conclusions

When evaluating whether the mean hardness from different locations on a single specimen or from different specimens are statistically distinguishable, a Student's t-test of the local means will be employed.

12 LIMITATIONS

A. Extremely soft materials may flow non-uniformly, preventing accurate microhardness measurement of even large, flat specimens.

B. For loads of 100 g or less, a high-quality metallographic polish is required. The hardness readings obtained are dependent upon load if below 500 g for the Knoop test and below 100 g for the Vickers test and are therefore generally used for comparative purposes only.

C. Conversion tables relating hardness values measured on different scales are only approximate, thus conversions are only reported as estimates.

13 SAFETY

A. Wear safety glasses when making hardness indentations.

B. Wear personal protective gear and use engineering controls that are appropriate for the task being performed when preparing specimens.

14 REFERENCES

- CHEM-100, Quality Assurance and Operations Manual, FBI Laboratory, Chemistry Unit, latest revision
- METAL-210, Examinations for Association or Origin, Chemistry Unit, latest revision
- METAL-450, Metallography, Chemistry Unit, latest revision
- ASTM E140: Standard Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, and Scleroscope Hardness, ASTM International, West Conshohoken, PA, latest revision
- ASTM Method E384, Standard Test Method for Microindentation Hardness of Materials, ASTM International, West Conshohocken, PA, latest revision
- ASM Handbook Volume 8: Mechanical Testing and Evaluation, ASM International, Materials Park, OH, latest revision

15 REVISION HISTORY

Revision	Issued	Changes
05	09/30/2022	Revised to comply with new formatting requirements. Distributed information from previous Instrumental Conditions and Decision Criteria sections into Acceptance Criteria and Procedure sections. Removed informational references.