Operation of Microhardness Testers

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 personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: general physical and chemical analysis in support of metallurgy examinations. The following procedures outline the basic process for Knoop or Vickers microhardness testing using equipment with a digital filar optical measuring system and automated hardness calculation capability.

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

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 adjustment of the test loads and/or the indenter shape must be chosen to accommodate these requirements.

For accurate and precise measurement, the test surface presented to the indenter must be flat, smooth and perpendicular to the loading axis. The specimen should be ground and polished using methods that do not impart high temperatures or result in cold work that could locally alter the hardness of the material. (See the CU Metallurgy SOP *Procedure for Metallographic Examinations*.)

5 Equipment/Materials/Reagents

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

b. 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

c. Specimen fixture or fixturing material, e.g., beeswax or clay or universal specimen holder

6 Standards and Controls

Adequate instrument performance is demonstrated using CRM test blocks. CRMs will be tested and the results recorded every time the instrument is powered up for use or the load or indenter is changed. CRM test blocks must be certified for the type (indenter and load) of test performed in order to assure that the indenter is seated properly, contains no defects and the load application is reliable. At least one CRM in the expected hardness range of the sample must be tested prior to sample analysis. Additional blocks may be tested at the examiner's discretion.

7 Calibration and Verification

The instrument is calibrated annually by a certified and licensed service provider that meets the LOM requirements. Prior to microhardness testing of case samples, the user will verify the instrument performance using one or more CRM test blocks. The performance check procedure can be found in 9 Procedure.

8 Sampling

Whole components, or sections thereof, are typically presented to the indenter for microhardness testing. 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.

Sampling of items examined under this protocol is determined by the nature of the evidence received and can consist of multiple items or one or multiple regions of interest on one item. If large numbers of physically indistinguishable samples are received for testing, a sampling plan may be employed. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be documented in case notes.

9 Procedure

9.1 Instrument Performance Check

a. Affix the appropriate CRM test block onto the specimen stage with wax, clay or a sample fixture.

b. 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.

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

d. Select the test load to be applied as indicated by the CRM test block certificate (typically 500 grams), and select the appropriate diamond indenter (Knoop or Vickers) for the test being performed.

e. 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.

f. 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.

g. 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.

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

h. Repeat the test two more times. Adjust the sample position away from any previous indentations using the stage micrometers to avoid any plastically deformed or damaged material.

i. Ensure that the microhardness readings are consistent with the hardness range reported on the CRM certification sheet. Alternatively, see section16 References (ASTM E384) to calculate the error E and repeatability R for comparison to the maximum tolerances given. If consistent hardness readings are not obtained, wipe the specimen holder surface and the back of sample with a clean rag to remove any debris from the standard. Ensure that the sample surface is perpendicular to the indenter loading axis. If efforts to alleviate inconsistent readings are unsuccessful, the instrument must be serviced and recalibrated by a certified and licensed service provider that meets the FBI Laboratory Operations Manual (LOM) requirements.

j. Record the date, operator, test type (Knoop or Vickers), test load, measured values, performance check result and other appropriate information in the instrument log. Record the hardness, load used and test type in the case notes.

9.2 Sample Testing

a. Samples should have a good quality surface finish so that a clearly defined indentation may be obtained. Polishing is recommended if feasible. Small components may be mounted in polymer to accommodate polishing. (See the CU-Metallurgy SOP *Procedure for Metallographic Examinations*.)

b. The surface being tested must be perpendicular to the direction of indenter motion and the back surface must be securely supported. 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. For example, a difference in length of greater than ~10% in the two portions of the long diagonal to either side of the short diagonal of a Knoop test requires leveling the sample surface.

c. For a Vickers indentation, 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 before proceeding. If leveling the specimen does not correct the problem, the indenter should be replaced and the performance check repeated prior to proceeding.

d. Affix the test sample onto the specimen stage with wax, clay or a sample fixture. Assure that the manner of mounting will support the applied test load. Test as in steps 9.1.c through 9.1.h Performance Check, and record the microhardness measurements in the case notes.

e. If comparative statistics are required, take additional readings in the areas of interest to provide typically 5-10 readings per area.

10 Instrumental Conditions

The instrument must not be subject to vibration when the test is underway. The indenter must be seated securely and free of foreign material. Indenters must be free of nicks or broken edges. Damaged indenters must be replaced, the new indenter reseated securely, and the performance check repeated.

The test load and indenter type used are dependent upon the hardness and shape of the material in the test region.

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 and hard, brittle materials.

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.

11 Decision Criteria

The performance check is acceptable when the microhardness 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 allowable tolerances (see 9.1.i Instrument Performance Check). If the performance check remains unsuccessful after assuring that the test block and indenter are sound, then the instrument must be serviced and re-calibrated by a certified and licensed service provider that meets the LOM requirements.

Variability in microhardness measurements made on a single sample beyond that demonstrated by the verification CRM test block is significant. If this occurs, the results should not be averaged but should be evaluated individually or as a range for the area tested.

When evaluating whether sets of test readings from different locations on a single specimen or from different specimens are statistically distinguishable, a Student t-test of the local means will be employed.

12 Calculations

12.1 Quantitative Analysis

a. Microhardness values are automatically calculated by the instrument software. These readings, and their associated measurement uncertainty, may be used to report a range or series of measurements, e.g., the decrease of microhardness from the surface to the interior of a casehardened specimen.

   i. 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).

   ii. 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. To report averaged microhardness measurements collected over a broader area, collect at least five values. Calculate and report the mean and expanded measurement uncertainty.

   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 microhardness value.

c. General correlation to a macrohardness number, e.g. Rockwell or Brinell, can be read from appropriate conversion charts (see 14 Limitations).

12.2 Comparative Analysis

Where quantitative data from two specimens are being compared, a pooled, two-tailed, Student-t test statistic of the sample means is typically used for the comparison. Two samples are deemed to be “indistinguishable” in the property under consideration if the two samples differ by less than the preselected critical t value ($t_{critical}$). The critical t value is typically chosen so that a value of $\alpha = 0.05$ can be achieved for the analysis and is determined by the degrees of freedom

associated with the measurements. An $\alpha = 0.05$ means there is a 5.0% chance of incorrectly rejecting a match between two samples when one actually exists.

To perform this test, the means and variances of each sample are determined as follows:

The mean value: $\bar{x}_a = \frac{\sum_{i=1}^{n_a} x_i}{n_a}$ where $\bar{x}_a$ is the average value of the measurements on sample "a",

$\sum x_i$ is the sum of the individual measurements and n_a is the number of measurements made on that sample. The variance of the individual measurement values from sample "a" is given by:

$$s_a^2 = \frac{\sum_{i=1}^{n_a} (x_i - \bar{x})^2}{n_a - 1}$$

The mean and variance of the data from sample "b" are calculated in the analogous manner.

The pooled sample variance is then calculated as: $s_p^2 = \frac{(n_a - 1)s_a^2 + (n_b - 1)s_b^2}{(n_a + n_b - 2)}$

A standard two-tailed statistical test of the two sample means is performed.

If $\left| \frac{(\bar{x}_a - \bar{x}_b)}{\left(\sqrt{s_p^2 (\frac{1}{n_a} + \frac{1}{n_b})} \right)} \right| > t_{critical}$ for any point of comparison, the samples are concluded to have a statistically significant difference. If not, the samples are concluded to be indistinguishable.

Typically five or more measurements per sample are used for performing comparisons.

13 Measurement Uncertainty

In the event that it is necessary to calculate the expanded uncertainty of a measurement, it will be done in accord with the Chemistry Unit *Procedures for Estimating Measurement Uncertainty*. Each time measurement uncertainty is calculated and reported, the repeatability component(s) will be updated. Often the variation present in a part production run, or allowed in a part specification, is substantially larger than the uncertainty contribution from the measuring instrument. In these cases, instrument measurement uncertainties will not be reported because they are considered negligible.

Quantitative data are sometimes used for comparative purposes. Expanded uncertainty should not be used for these inter-comparisons because it increases the probability that two samples will appear to be analytically indistinguishable and therefore increases the likelihood of type II errors (false inclusion).

Uncertainties are calculated for the diagonal measurements in microns. The formulas given in section 12 Calculations can be used to convert these measurements into equivalent microhardness values.

14 Limitations

The guidelines for appropriate specimen dimensions stated above are not to be substituted for the sound engineering judgment of a trained operator. Extremely soft materials may flow non-uniformly, preventing accurate microhardness measurement of even large, flat specimens.
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.
All conversion tables of hardness scales, including those in section 16 References, are based on the assumption that the metal tested is homogeneous to a depth several times greater than the indentation. If not, different loads and indenter shapes would penetrate, or encounter the resistance of, metal of varying hardness depending upon the indentation depth.

Conversion tables relating hardness values measured on different scales are only approximate and never mathematically exact due to the difference in cold-working response of a material to the indenter shape and load applied. Nevertheless, conversion is of considerable value when comparing different hardness scales in a general way.

15 Safety

Standard safety precautions, such as wearing protective gloves, should be observed when handling evidentiary materials. Assemblies or components that have electrical or mechanical hazards may require special precautions to disassemble and prepare specimens for microhardness testing.

This instrument SOP has the following specific safety requirements:

- Wear safety glasses when making microhardness indentations
- During specimen preparation, wear personal protective gear and use engineering controls that are appropriate for the task being performed

If additional guidance is required, contact the Laboratory Health and Safety Group.

16 References

ASM Handbook, Volume 8, Mechanical Testing and Evaluation, ASM International, Metals Park, OH, 1992

Chandler, H., ed., *Hardness Testing*, 2nd ed., ASM International, Materials Park, OH, 1999, or latest revision

ASTM Method E384, *Standard Test Method for Microindentation Hardness of Materials*, ASTM International, West Conshohocken, PA, latest revision

ASTM Method E140: *Standard Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness*, ASTM International, West Conshohocken, PA, latest revision

Chemistry Unit Quality Assurance and Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision

Rev. #	Issue Date	History
3	02/05/2014	Section 4 updated to amend requirements for indentation spacing. Section 9.1 updated to include requirement to test CRM block JH97 when performing verification of the tester. Text amended to require verification measurements to be recorded in the instrument verification log. Section 9.2 spelling correction made. Subsection e deleted. Language in section 10 updated. Section 11 updated to include requirement to include expanded estimate of uncertainty when reporting hardness values. Paragraph from section 10 relocated to section 11. Section 12 includes minor language updates. Formula for sample variance simplified. Typographical error in formula for variance of mean corrected. Formula for calculation of Knoop hardness added. Section 13 has been rewritten to better reflect requirements on measurement uncertainty. Section 16. References updated.
4	12/21/2018	Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 15 and is now designated Metal 702. Added personnel to section 2. Added relation between test type and feature size to section 3. Made minor editorial corrections throughout document. Added reference to CU Metallurgy SOPs in sections 4 and 8. Added item to section 5. Removed reference to specific CRM in sections 6 and 8. Revised sampling statement in section 8. Revised section 9 to clarify procedure. Separated quantitative and comparative calculations in 12. Added α and $t_{critical}$ selection process to 12. Updated sections 13 and 15. Updated reference section 16.

Approval

Redacted - Signatures on File

Metallurgy Technical Leader _ Date: 12/20/2018

Chemistry Unit Chief _ Date: 12/20/2018

QA Approval

Quality Manager _ Date: 12/20/2018