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TECH NOTES

An Introduction to Microindentation Methods

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Hardness-Perhaps Our Oldest Test

At a time when companies are seeking to quantify and document virtually every process, a renewed interest has appeared in some of the old standbys such as hardness testing. Hardness testing is a proven quality control and inspection tool; it can be used to quickly determine if an incoming product meets specification or if a heat treatment was properly done. Hardness, as a mechanical property, is best defined as resistance to penetration or permanent deformation. The application of the hardness value, however, is specific to different professions: to a mechanical engineer it relates to the wear resistance; to a design engineer it relates to the flow stress, to a mineralogist it relates to the scratch resistance, and to a machinist it relates to the cutting rate.

Historically, hardness tests were first performed by scratching the specimen with various standard substances. One of the earliest forms of scratch testing dates back to Réaumur in 1722. His scale of testing consisted of a scratching tool in the form of a long bar which increased in hardness from one end to the other. The degree of hardness was determined by the position on the tool that the specimen being tested would scratch.

In 1895, a more advanced form of scratch testing was devised by Osmond and Troost. They used hardened steel scribes to make scratches across a hardened case to core region on a steel specimen. Comparing the resulting scratch with the microstructure, they were able to show that the presence of retained austenite resulted in a decreased hardness. This was the first known application of hardness testing to case hardened steels. Scratch tests are seldom used today because they are subjective and difficult to quantify.

The first true indentation hardness test was devised by Johann Brinell in 1900. In the Brinell test, a load is applied to the surface of a test piece with a hardened steel ball indenter typically 10mm in diameter. The hardness number is determined by measuring the diameter of the indentation using a simple magnifier with an eyepiece containing a graduated scale. The hardness value is calculated by dividing the load by the surface area of the indent.

La Gris performed one of the first microindentation hardness tests in 1911. He used a 0.74mm diameter spherical indenter with a load of 750 grams to place a mark on the specimen. A microscope was used to measure the resulting indentation.

The next efforts were directed toward devising other indenters; in particular, those made from diamond to accommodate the testing of fully hardened steels. In 1919, the Rockwell test was introduced and it has become the most common hardness test in use today in the US. This test can be conducted rapidly because the depth of the indentation is detected by the instrument rather than the operator measuring the indentation.

The BUEHLER® MICROMET® 2100 Series of Microhardness Testers can perform precision microindentation hardness tests on a wide range of engineered materials. These state of the art testers assure accurate Vickers and Knoop hardness testing throughout their load range. The MICROMET's parallel leaf spring design and dead weight loading mechanism eliminate indenter wander and skipping. The MICROMET 2103 and 2104 digital models offer advanced data storage, automatic hardness conversions and statistical data analysis capabilities including mean, maximum and minimum values and standard deviation.

For the Vickers test, developed in 1925, a square-based diamond indentor was chosen with an angle of 136° between opposite faces in order to obtain hardness numbers similar in magnitude to Brinell numbers. Tangents drawn to the Brinell ball at the impression edges meet below the center of the impression at an angle of 136°. Thus, the Vickers hardness values were approximately equal to HB values over the range of the Brinell test. This fact gave the Vickers test rapid acceptance in industry.

Development of the light load Vickers test in 1932 and the Knoop test by the National Bureau of Standards in 1939 has made microindentation testing a routine procedure. Both of these tests use precisely shaped diamond indenters and various loads to determine the hardness of a wide variety of materials. The term microhardness is commonly used in place of microindentation hardness; however, it can be misleading because the term micro is intended to describe the indentation size and not the magnitude of hardness. Microindentation hardness testing provides information on the hardness characteristics that cannot be revealed with other tests such as Brinell or Rockwell.

More recent developments target the need to test small microelectronic components and thin films along with finer phase dispersions in composite materials. Nanoindentation hardness testing is becoming more common with load forces less than 1 gram being applied. However, this does require more stringent environmental control of factors such as vibration, airflow, and temperature. Also, the indentations are typically too small to be measured with standard light optical microscopes and instead require electron optical devices.

Static Indentation Testing

In the U. S., static indentation hardness testing is typically divided into two classes: macro and micro. These designations refer to the load force applied and the resulting size of the indentation. A macro test implies that a load force of 1kg or heavier was applied while a micro test employs loads under 1kg. Another way to subdivide indentation hardness methods is based on the method of measurement. Brinell, Vickers, and Knoop values are derived from a measurement of the indenter impression size whereas Rockwell values are based on the indenters penetration depth.

Selection of a Hardness Testing Method

The following list represents many of the factors to consider when determining the most applicable hardness testing method.

- Hardness range of test material
- Size and shape of the component
- Degree of flatness
- Surface condition
- Degree of homogeneity
- Effect of indentation marks
- Work environment
- Number of identical components to be tested

Microindentation Hardness

Selection criteria as they relate to microindentation testing are as follows:

Hardness range of test material – A wide range of material types and hardnesses can be evaluated. Additional criteria are necessary when working with plastics, transparent materials, and some ceramics.

Size and shape of the component – Most microindentation hardness testers have a limited specimen size capacity. Typically there is a height restriction with the acceptable length determined by the support fixture.

Degree of flatness – The flatter the specimen surface, the more accurate the results due to the complete contact of the indenter and the surface. The indenter must be perpendicular to the surface. Correction factors are available for some curvatures; however, their application is limited.

Surface condition – A fairly smooth surface of a 3µm finish or better is recommended. The depth of surface damage becomes more important as the load force applied decreases resulting in a shallower indentation. In addition, a certain degree of reflectivity is needed to accurately determine the outer corners of the indentation.

Degree of homogeneity – Due to the relatively small indentations created during a microindentation hardness test, the degree of homogeneity is often revealed with this type of testing. For this reason, in order to obtain an average hardness value of a material, at least five indentations should be made, measured and the results averaged.

Effect of indentation marks – The indentations created on the surface of the component are often difficult to see without the aid of a microscope. These indentations are relatively insignificant in size when compared with the indentations which result from a macrohardness test.

Work environment – Of particular importance is the level of vibration in the work area. The source of vibration can range from the forging press across the way to a delivery dock across the hall. Vibrations can enlarge the indent resulting in an underestimation of the true material hardness. Even drumming your fingers on the table can affect light load test values substantially!

Number of identical components to be tested – For a quick test of the bulk hardness, a Rockwell test is typically used. However, if the application requires a microindentation hardness test, operators who routinely check a large volume of parts will benefit from automation. Systems are available today which enable the operator to set up the test parameters and then walk away while the tests are conducted and measurements are accomplished. Look for more information on the OMNIMET® MHT later in this issue.

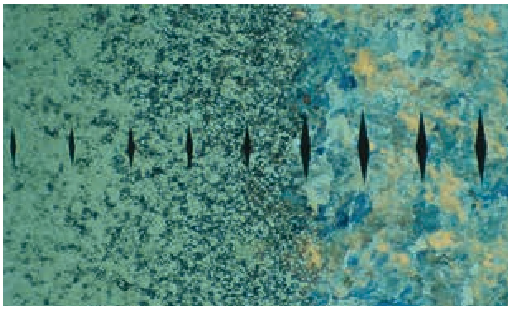
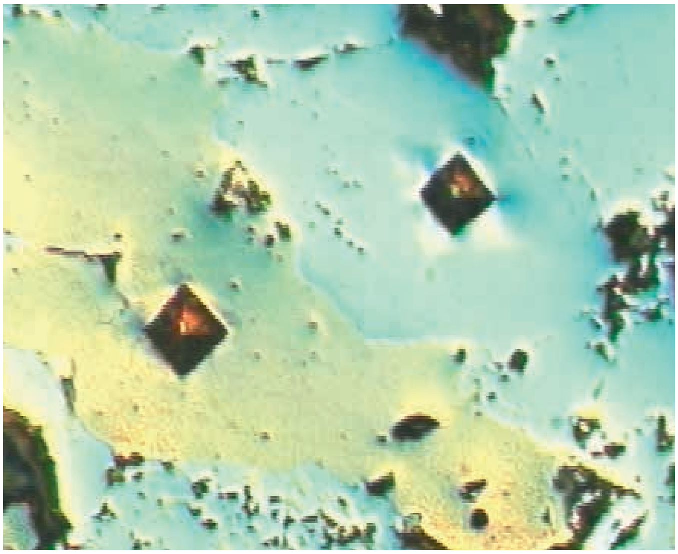


Figure 1: Knoop microindentation hardness impressions made across the case and core of a hardened steel specimen. 2% nital etch, DIC – 100x.

Applications

In the case of microindentation hardness testing, the following applications are typically of interest:

- Measuring the hardness of precision components and product forms like foil or wire that are too thin or too small to be measured by bulk test methods.
- Monitoring carburizing, nitriding, or other surface modification operations by hardness traverses taken on crosssections. (Figure 1)
- Measuring the hardness of individual microconstituents. (Figure 2)
- Measuring at the edge of a test piece to detect surface conditions such as decarburization.



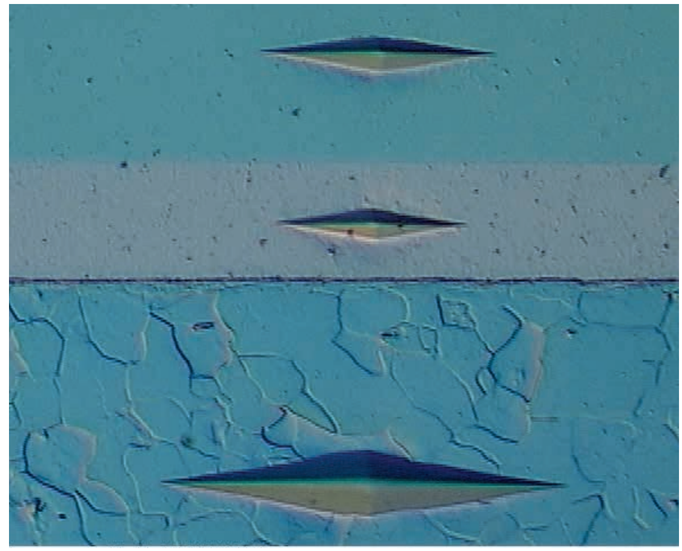


Figure 2 (left): Vickers microindentation hardness impressions in a copper infiltrated steel powder metallurgy specimen. DIC – 200x. Figure 3 (right): Knoop indentations positioned in nickel and copper coatings as well as the steel base material. While a 50g load provided properly sized (width vs. spacing) indents in the nickel and the steel, a 10g load produced an oversized indent (i.e., the spacing between the indent and the interface is <2.5 times the indent width) in the copper layer. 2% nital etch, DIC – 250x.

- Measuring the hardness of surface layers such as plated or bonded layers. (Figure 3)

Why do some people use Vickers indenters while others use Knoop?

Vickers

The Vickers hardness test follows the Brinell principle in that an indenter of specific shape is pressed into the test material, the load is removed, the diagonals of the resulting indentation are measured, and the hardness number is calculated by dividing the load by the surface area of the indentation. The indenter is made of diamond and is in the form of a squarebased pyramid with an angle of 136° between faces. The facets are highly polished, free from surface imperfections, and the point is sharp. The depth of indentation is about oneseventh of the average diagonal length.

Knoop

The methodology of the Knoop hardness test is similar to the Vickers test with the exception of the indenter shape. A standard Knoop indenter is a rhombic-based pyramid which produces an indentation that has an ideal ratio between long and short diagonals of approximately 7 to 1. The depth of indentation is about one-thirtieth of the length of the long diagonal.

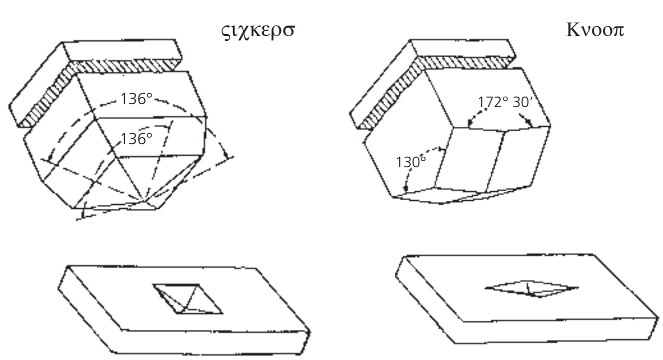


Figure 4: Diagrams of two diamond indenters, Vickers and Knoop. The Vickers indenter has two identical 136° angles to form a pyramidal indentation; the Knoop indenter has two unique angles to form an elongated indentation.

- Because the Vickers indenter penetrates deeper into the specimen than the Knoop indenter at the same load, the Vickers test is less sensitive to surface conditions.
- A Knoop indenter is often selected for testing surface defects due to the limited penetration depth.
- Both of the diagonal lengths of the Vickers indentation are measured and averaged. This results in a value that many operators have more confidence in.
- The shape of the Knoop indenter makes it desirable for placement in coatings and elongated microconstituents. In addition, the shorter diagonal enables the operator to place the indents closer together to detect rapid changes in hardness observed in some case hardened components.
- Vickers test results vary little with the test load (except below 100gf) which increases the relevance of conversion to other test scales, in comparison to Knoop test results.

Standard Test Method

Below is an example of the routine typically followed by the operator. However, if a semi- or fully-automated system is used, some of the following steps may be combined. n Place standard test block or specimen in the vise secured to the stage.

- Focus with low power objective; select area of interest.
- Focus with high power objective; adjust illumination.
- Select area for the indentation by adjusting the x and y stage controls.
- Select the appropriate load force.
- Set timer to the required dwell time, typically 15 seconds.
- Rotate turret to indenter position.
- Apply the load.
- Rotate turret back to the higher objective.
- Measure the length of the long diagonal (Knoop) or both diagonals (Vickers).
- Calculate the hardness value (formula and calculator or tables).

Knoop Formula:

HK = P/AP = P/d2 c = 14229P/d2

where:

HK: Knoop hardness number

P: test load, gf
A_P: projected area of indent, mm2
c: indenter constant relating AP to d2
d: length of the longer diagonal, µm

Vickers Formula:
$HV = P/A_S = 2P \sin(\alpha/2)/d^2 = 1854.4P/d^2$
HV: Vickers hardness number
P: test load, gf
A_S: surface area of indent, mm2
α: face angle of the indent, 136°
d: mean of indent diagonals, µm

- Report the hardness value, the type of indenter used, load force applied, dwell time, magnification used for measurement, and any unusual conditions encountered during the test.

Reporting of a hardness value is typically abbreviated using the following notation:

400 HK200 . The hardness value is followed by HK for a Knoop measurement or HV for a Vickers measurement while the load is reported in grams-force by subscript notation according to ASTM E 384. The same value reported using ISO notation, however, would appear as follows: 400 HK 0,2.

Troubleshooting Your Results

The possible sources of significant error are found in three areas as summarized in the table below.

Tester	Test Piece	Test Opetation
Cleanliness	Mounting	Vibration
Leveling	Polishing	Load Selection
Indenter	Etching	Test Block Check
Indenter/ Objective Alignment	Fixturing/ Vise Selection	Alignment of Filar Lines

The Tester should be protected against dust and fumes that are often present in a laboratory environment. The simplest protection is to cover the equipment when it is not in use. Regular maintenance, including professional cleaning, alignment, and calibration are recommended if the tester is expected to perform at its best. The location of the tester should be selected such that the tester is level or can be easily leveled by adjusting the supporting feet.

Because both Vickers and Knoop tests may be made on the same instrument in some laboratories, the indenters will be handled frequently. Before conducting a test, make certain the correct indenter is in place and aligned. Check the condition of the indenter at regular intervals to determine if it is cracked or dirty. Typically, these faults will be noticeable when looking at the indentation. The indenter needs to be aligned to have the indentation appear at the correct angle. The indenter is often marked and inserted such that the identifying mark aligns with a mark or feature on the tester. In addition, if the indentation is not found within the center of the field, it is necessary to align the objective. Most objectives are centered by adjusting three set screws.

The Test Piece surface condition is critical because non-representative conditions can affect the readings. Unlike macrohardness testing, test specimens are usually encapsulated in a mounting resin for support and edge retention. The mounted specimen must be metallographically prepared to produce a flat deformation-free surface. The test specimen must be fixtured securely on the stage of the tester so that it will not move while testing is in progress. The stage vise must also permit leveling of the specimen such that the test surface is perpendicular to the axis of the indenter. The best way to insure this condition is by using a self-leveling vise. In this device, the specimen is forced with screw pressure from below against a surface(s) that establish the proper test plane.

The Test Operation can be a significant source of error if the operating conditions and environment are taken for granted. The tester must be located in a room where it will not be affected by vibration produced
by the operation of other nearby equipment. The lighter the test load, the more significant this will be. If vibrations are a problem, isolate the tester from all other equipment by placing it on a separate table.

If this is not enough, an isolation platform may be required.

Microindentation hardness testers are designed to control the operating parameters needed to meet the test specifications. However, to obtain accurate and repeatable tests, the correct test conditions must be set. Choose the load appropriate for the material you are testing and select the dwell time for the specific test according to ASTM or other relevant standards. When all the parameters are set, indirect verification can be accomplished. Place five indentations in a calibrated test block and compare the measured results with the values found on the certificate accompanying the block. Guidelines for machine repeatability are present in ASTM E 384 which take into account the load applied and the hardness of the calibrated test block. The operator should also be instructed as to the correct use of optical measuring devices. In most testers, there are at least two objective lenses, a low power one (10x) for location of the test area and a high power one (40x) for measuring the indentations. To obtain the maximum accuracy, correct positioning of the indentation relative to the filar lines is important.

Tech-Tips

Question: How much specimen preparation is necessary for microindentation hardness testing?

Answer: The degree of surface finish required varies with the load force applied and magnification used in the testing. In general, as the test load decreases, the surface finish requirements become more stringent. Light load tests will be affected by residual damage, so it is necessary to remove all of the damage produced by cutting, grinding, polishing, etc. Further, the surface must be flat. While a 3µm finish is often sufficient when applying heavier loads, in all cases the perimeter of the indentation must be clearly defined in the field of the microscope.

Additionally, it is necessary to consider the heat the specimen can be subjected to when mounting and the possible heat or distortion that can be introduced when grinding or polishing if improper methods are used. For some materials, hot compression mounting may not be an option. Edge retention, which is critical for measuring case depth and surface treatments, can be enhanced with the selection of higher hardness mounting compounds such as EPOMET®. Additionally, the use of an automated polishing head has been shown to improve specimen flatness in comparison to most hand polishing methods.

Question: What is the best load force to use for my application?

Answer: In general, use the highest possible load to produce the largest indent within the restrictions of the application. The error introduced when measuring the diagonals has a greater influence on the calculated hardness as the indentation decreases in size, particularly for indentations < 20µm in diameter. Also, with lighter load applications, thelaboratory environment can have a greater influence on the accuracy of the test.

Some of the limitations to consider are:

- Size of the indent in comparison to the feature of interest.
- Spacing between indentations should be a minimum of 2.5 times the width of the diagonal.
- Ability to accurately measure the indent. If too light of a load is applied, it may be difficult to measure the indent (20µm limit is recommended by ASTM E 384).

When new materials are being tested, some experimentation with the load force is often required to obtain optimum conditions.

Question: How come my results and those from another facility are different?

Answer: The first step is to use statistical analysis to see if the difference is statistically significant. Next, determine any differences in the test method. The two most common problems relate to either a different load being applied or a converted value Some people assume that the hardness number is independent of the load force. However, in the case of microindentation hardness testing, tests performed with loads of less than 500g with the Knoop indenter and less than 100g with the Vickers indenter are a function of magnitude of the test load. Some of the factors that explain the dependence relate to errors in determination of indent size, differences in elastic recovery, and influences of the work environment such as vibrations or large temperature differences. It is important to report the details of the test as well as the final hardness value.

In many cases, Vickers or Knoop values are converted to other scales, such as Rockwell. For example, a 700HV500 reading is considered to be approximately equal to a 60 HRC reading. However, a 700HV500 indent is only 36.5µm in diameter whereas the indent resulting from a Rockwell C test is about 500µm in diameter. The same amount of area is not being sampled. A Rockwell value will be more representative of the bulk properties whereas the Vickers value will be more dependent on the local properties because the depth of penetration is considerably less. Both ISO documents 4545 and 6507 and ASTM E 384 caution users about conversions. "There is no general process of accurately converting Vickers or Knoop hardness values into other scales of hardness. Such conversions therefore should be avoided, unless a reliable basis for conversion can be obtained by comparison tests. A strict comparison of hardness values is only possible at identical test forces."

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