MICROINDENTATION HARDNESS TESTING

Microindentation hardness testing, more commonly (but incorrectly) called microhardness testing, is widely used to study fine scale changes in hardness, either intentional or accidental. Heat treaters have utilized the technique for many years to evaluate the success of surface hardening treatments or to detect and assess decarburization. Metallographers and failure analysts use the method for a host of purposes including evaluation of homogeneity, characterization of weldments, as an aid to phase identification, or simply to determine the hardness of specimens too small for traditional bulk indentation tests.

Metallurgists and metallographers tend to develop their own jargon, often as a matter of linguistic simplicity, which is not always as rigorously correct as it could be. Although the term "microhardness" is generally understood by its users, the word implies that the hardness is extremely low, which is not the case. The applied load and the resulting indent size are small relative to bulk tests, but the same hardness number is obtained. Consequently, ASTM Committee E-4 on Metallography recommends use of the term "microindentation hardness testing" which could be given the acronym MHT. ASTM Standard E 384 fully describes the two most common microindentation tests - the Vickers and the Knoop tests.

The Vickers Test

In 1925, Smith and Sandland of the UK developed a new indentation test for metals that were too hard to evaluate using the Brinell test. The hardened steel ball of the Brinell test limited the test to steels with hardnesses below ~450 HBS (~48 HRC). (The harder tungsten carbide ball was not available in 1925. The WC indenter extends the Brinell test to metals up to 615 HBW (~58 HRC). The WC ball has now replaced the steel ball for the Brinell test.) In designing the new indenter, a square-based diamond pyramid (see Figure 47), they chose a geometry that would produce hardness numbers nearly identical to Brinell numbers in the range where both tests could be used. This was a very wise decision as it made the Vickers test very easy to adopt. The ideal d/D ratio (d = impression diameter, D = ball diameter) for a spherical indenter is 0.375. If tangents are drawn to the ball at the impression edges for d/D = 0.375, they meet below the center of the impression at an angle of 136°, the angle chosen for the Vickers indenter.

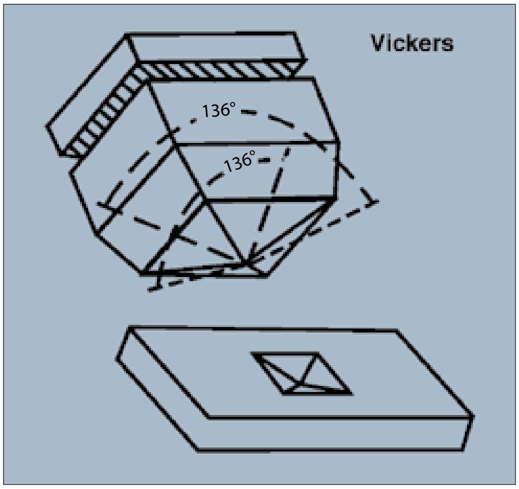

Figure 47. Schematic of the Vickers indenter and the shape of an impression.

Use of diamond allowed the Vickers test to be used to evaluate any material (except diamond) and, furthermore, had the very important advantage of placing the hardness of all materials on one continuous scale. This is a major disadvantage of Rockwell type tests where different scales (15 standard and 15 superficial) were developed to evaluate materials. Not one of these scales can cover the full hardness range. The HRA scale covers the broadest hardness range, but it is not commonly used.

In the Vickers test, the load is applied smoothly, without impact, forcing the indenter into the test piece. The indenter is held in place for 10 or 15 seconds. The physical quality of the indenter and the accuracy of the applied load (defined in E 384) must be controlled in order to get the correct results. After the load is removed, the two impression diagonals are measured, usually to the nearest 0.1-µm with a filar micrometer, and averaged. The Vickers hardness (HV) is calculated using:

$$HV = \frac{1854.4L}{d^2}$$

where the load L is in gf and the average diagonal d is in µm (this produces hardness number units of gf/μm^2 although the equivalent units kgf/mm^2 are preferred; in practice the numbers are reported without indication of the units). The original Vickers testers were developed for test loads of 1 to 120kgf that produce rather large indents. Recognizing the need for lower test loads, the National Physical Laboratory (UK) reported on use of lower test loads in 1932. Lips and Sack developed the first low-load Vickers tester in 1936.

Because the shape of the Vickers indentation is geometrically similar at all test loads, the HV value is constant, within statistical precision, over a very wide test load range as long as the test specimen is reasonably homogeneous. Numerous studies of microindentation hardness test results conducted over a wide range of test loads have shown that test results are not constant at very low loads. This problem, called the "indentation size effect", or ISE, has been attributed to fundamental characteristics of the material. However, the same effect is observed at the low load test range (1-10kgf) of bulk Vickers testers [2] and an ASTM interlaboratory "round robin" of indents made by one laboratory but measured by twelve different people, reported all three possible ISE responses [24,25] for the same indents!

Since the 1960s, the standard symbol for Vickers hardness per ASTM E 92 and E 384, has been HV. This should be used in preference to the older, obsolete symbols DPN or VPN. The hardness is expressed in a standard format. For example, if a 300 gf load is used and the test reveals a hardness of 375 HV, the hardness is expressed as 375 HV_{300}. Rigorous application of the SI system results in hardness units expressed not in the standard, understandable kgf/mm^2 values but in GPa units that are meaningless to most engineers and technicians. ASTM recommends a 'soft" metric approach in this case.

In the Vickers test, it is assumed that elastic recovery does not occur once the load is removed. However, elastic recovery does occur, and sometimes its influence is quite pronounced. Generally, the impression (Figure 48) appears to be square, and the two diagonals have similar lengths. As with the Brinell test, the Vickers hardness number is calculated based on the surface area of the indent rather than the projected area. If the impression shape is distorted due to elastic recovery (very common in anisotropic materials), Figure 49, should the hardness be based on the average of the two diagonals? It is possible to calculate the Vickers hardness based on the projected area of the impression, which can be measured by image analysis. While rigorous studies of this problem are scant in the literature, the diagonal measurement is the preferred approach even for distorted indents, at this time.

The Knoop Test

As an alternative to the Vickers test, particularly to test very thin layers, Frederick Knoop and his associates at the former National Bureau of Standards developed a low-load test using a rhombohedral-shaped diamond indenter, Figure 50.

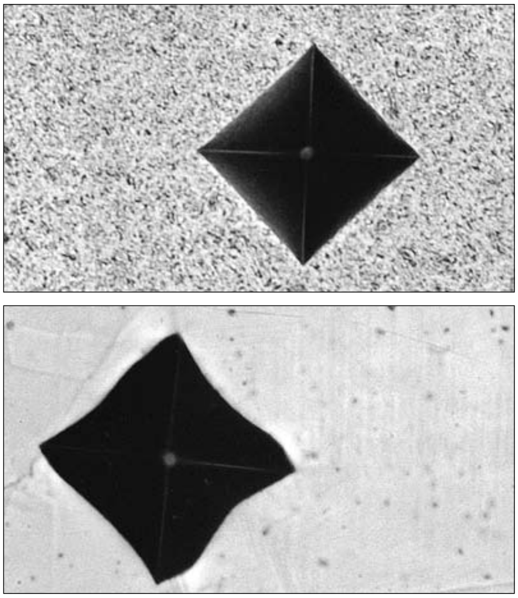

Figure 48. (top) Example of a well-formed Vickers indentation (400X). Figure 49. (bottom) Example of a distorted Vickers indentation (400X).

The long diagonal is seven times (7.114 actually) as long as the short diagonal. With this indenter shape, elastic recovery can be held to a minimum. Some investigators claim there is no elastic recovery with the Knoop indent, but this cannot be true as measurements of the ratio of long to short diagonal often reveal results substantially different than the ideal 7.114 value.

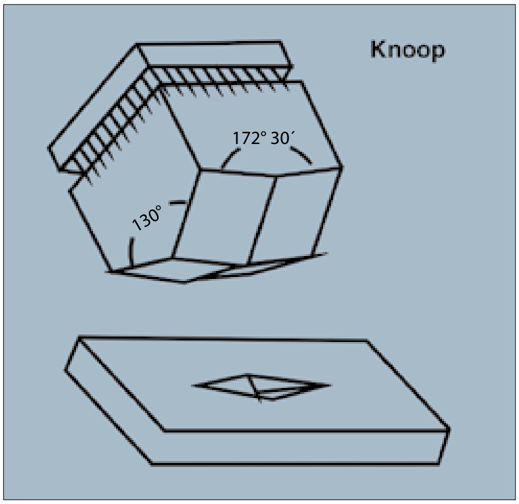

Figure 50. Schematic of the Knoop indenter and the shape of an impression

The Knoop test is conducted in the same manner, and with the same tester, as the Vickers test. However, only the long diagonal is measured. This, of course, saves some time. The Knoop hardness is calculated from

$$HK = \frac{14229L}{d^2}$$

where the load L is in gf and the long diagonal d is in µm. Again, the symbol HK was adopted in the early 1960's while other terms; e.g., HKN or KHN, are obsolete and should not be used. The Knoop hardness is expressed in the same manner as the Vickers hardness; i.e., 375 HK_{300} means that a 300 gf load produced a Knoop hardness of 375. (The kgf/mm^2 unit information is no longer reported).

Aside from a minor savings of time, one chief merit of the Knoop test is the ability to test thin layers more easily. For surfaces with varying hardness, such as case hardened parts, Knoop indents can be spaced closer together than Vickers indents. Thus, a single Knoop traverse can define a hardness gradient more simply than a series of two or three parallel Vickers traverses where each indent is made at different depths. Furthermore, if the hardness varies strongly with the depth, the Vickers indent will be distorted by this change; that is, the diagonal parallel to the hardness change will be affected by the hardness gradient (i.e., there is a substantial difference in the lengths of the two halves of the diagonal), while the diagonal perpendicular to the hardness gradient will be unaffected (both halves of this diagonal of the same approximate length).

The down side of the Knoop indent is that the three dimensional indent shape will change with test load and, consequently, HK varies with load. At high loads, this variation is not substantial. Conversion of HK values to other test scales can only be done reliably for HK values performed at the standard load, generally 500gf, used to develop the correlations. All hardness scale conversions are based on empirical data. Conversions are not precise but are estimates.

Factors Affecting Accuracy, Precision and Bias

Many factors (see Table 44) can influence the quality of microindentation test results [26]. In the early days of low-load (< 100gf) hardness testing, it was quickly recognized that improper specimen preparation may influence hardness test results. Most text books state that improper preparation will yield higher test results because the surface contains excessive preparation induced deformation. While this is certainly true, there are other cases where improper preparation can create excessive heat that will lower the hardness and strength of many metals and alloys. So either problem may be encountered due to faulty preparation. For many years, it was considered necessary to electrolytically polish specimens so that the preparation-induced damage could be removed thus permitting bias-free low-load testing. However, the science behind mechanical specimen preparation, chiefly due to the work of Len Samuels [3], has led to development of excellent mechanical specimen preparation procedures, and electropolishing is no longer required.

There are several operational factors that must be controlled in order to obtain optimum test results. First, it is a good practice to inspect the indenter periodically for damage, for example, cracking or chipping of the diamond. If you have metrology equipment, you can measure the face angles and the sharpness of the tip. Specifications for Vickers and Knoop indenter geometries are given in E 384.

Table 44. Factors Affecting Precision and Bias in Microindentation Hardness Testing

Instrument Factors	Measurement Factors	Material Factors
Accuracy of the applied load	Calibration of the measurement system	Heterogenity of the specimen
Inertia effects, speed of loading	Numerical aperture of the objective	Strength of crystallographic texture, if present
Lateral movement of the indenter or specimen	Magnification	Quality of specimen preparation
Indentation time	Inadequate image quality	Low reflectivity or transparency
Indenter shap deviations	Uniformity of illumination	Creep during indentation
Damage to the indenter	Distortion in optics	Fracture during indentation
Inadequate spacing between indents or form edges	Operator's visual acuity	Oil, grease or dirt on indenter indenter or specimen
Angle of indentation	Focusing of the image	

A prime source of error in the tests is the alignment of the specimen surface relative to the indenter. The indenter itself must be properly aligned perpendicular (±1°) to the stage plate. Next, the specimen surface must be perpendicular to the indenter. Most testers provide holders that align the polished face perpendicular to the indenter (parallel to the stage). If a specimen is simply placed on the stage surface, its back surface must be parallel to its polished surface. Tilting the surface more than 1° from perpendicular results in nonsymmetrical impressions and can produce lateral movement between specimen and indenter. The occurrence of non- symmetrical indents is generally easily detected during measurement.

In most cases, errors in indenting with modern testers are not the major source of error, although this can occur [26]. It is important to check the performance of your tester regularly using a certified test block. It is safest to use a test block manufactured for microindentation testing and certified for the test (Vickers or Knoop) and the load that you intend to use. Strictly speaking, a block certified for Vickers testing at 300 or 500 gf (commonly chosen loads) should yield essentially the same hardness with loads from about 50 to 1000 gf. That is, if you take the average of about five indents and compare the average at your load to the average at the calibrated load (knowing the standard deviation of the test results), statistical tests can tell you (at any desired confidence level) if the difference between the mean values of the tests at the two loads is statistically significant or not.

Because of the method of defining HV and HK (equations given above) where we divide by d^2, measurement errors become more critical as d get smaller; that is, as L decreases and the material's hardness increases (discussed later). So departure from a constant hardness for the Vickers or Knoop tests as a function of load will be a greater problem as the hardness increases. For the Knoop test, HK increases as L decreases because the indent geometry changes with indent depth and width. The degree of the change in HK also varies with test load being greater as L decreases.

The greatest source of error is in measuring the indent as has been documented in an ASTM interlaboratory test [24,25]. Place the indent in the center of the measuring field, if it is not already there, as lens image quality is best in the center. The light source should provide adequate, even illumination to provide maximum contrast and resolution. The accuracy of the filar micrometer, or other measuring device, should be verified using a stage micrometer.

Specimen preparation quality becomes more important as the load decreases, and it must be at an acceptable level. Specimen thickness must be at least 2.5 times the Vickers diagonal length. Because the Knoop indent is shallower than the Vickers at the same load, somewhat thinner specimens can be tested. Spacing of indents is important because indenting produces plastic deformation and a strain field around the indent. If the spacing is too small, the new indent will be affected by the strain field around the last indent. ASTM recommends a minimum spacing (center to edge of adjacent indent) of 2.5 times the Vickers diagonal. For the Knoop test where the long diagonals are parallel, the spacing is 2.5 times the short diagonal. The minimum recommended spacing between the edge of the specimen and the center of the indent should be 2.5 times. Again, Knoop indents can be placed closer to the surface than Vickers indents.

Several studies have stated that the resolution of the optical system is the major factor limiting measurement precision. They state that this causes indents to be undersized by a constant amount. However, as undersizing would increase the measured hardness, not decrease it, resolution limits *per se* cannot explain this problem. For the Knoop indent, the chief problem is image contrast at the indent tips that results in undersized indents. This problem, plus the variable indent shape, both result in increasing HK with decreasing test load. For the Vickers test, one would assume that undersizing or oversizing would be equally likely; but, experience suggests that oversizing is much more commonly encountered for low loads and small indents.

Automation

Microindentation hardness testing is tedious, so anything that can be done to simplify testing is valuable, especially for laboratories that do substantial testing. Many adjuncts to indent measurement have been tried and a variety of such systems are available. There has been considerable interest in applying image analyzers to the indent measurement task. Further, with stage automation, it is possible to automate the indenting process itself, and with the same equipment. Figure 51 shows the Buehler OmniMet MHT automated microindentation hardness testing system. This system can be used in the fully automatic, semiautomatic or manual mode, depending upon the nature of the testing.

Figure 51. OmniMet MHT

An automated system can be programmed to make any number of indents in either a defined pattern (x number of equally spaced indents, or x number between two chosen points) or a random pattern (locations selected at random with a mouse). Curved patterns may be used, not just straight-line patterns. The system then makes the indents at the requested load and locations, measures each indent, calculates the hardness (and desired conversions to other scales), and prints/plots the results. Statistical analysis can also be preformed.

In general, Vickers indents are easier to measure by image analysis than Knoop indents due to the lower contrast at the tips of the Knoop indents (leads to undersizing and higher HK values). Metal flow (plastic deformation) around the indent edges can interfere with measurement precision. Vickers indents, like the ones shown in Figure 48, exhibit excellent contrast and shape and are easily measured. If the magnification is too high (and this may be influenced by the numerical aperture of the objective) for a given indent size, image contrast suffers and correct detection of the indent will be very difficult. On the other hand, if the indent is very small on the screen, it will be hard for the system to detect it automatically. In this case, use a higher magnification, or use the semiautomatic measurement mode if this is not possible. The operator fits a box around the indent with a mouse to measure the indent.

Microindentation hardness testing is a very valuable tool for the materials engineer but it must be used with care and a full understanding of the potential problems that can occur. Always try to use the highest possible load depending upon the indent spacing or closeness to edges. Try to keep indents greater than 20-μm in diameter, as recommended in ASTM standard E 384. If you have since you read it (all ASTM standards must be reviewed and revised, if necessary, every five years), get the latest copy and go over it. The Precision and Bias section (and appendix X1 of E 384) contains a wealth of practical advice. Automation of indentation and measurement is possible and, for laboratories that do substantial testing, greatly reduces operator fatigue while reducing testing times.

Helpful Hints For Microindentation Hardness Testing

If you suspect that the test results obtained on a specimen are questionable, verify the tester using a certified test block. When testing thin coatings, use the Knoop indenter with the long axis parallel to the surface. If you have a specimen where the hardness changes rapidly, Vickers indents may be distorted badly in the direction of the hardness gradient. Switch to a Knoop indenter and place the long axis perpendicular to the hardness gradient.