ASM Handbook, Volume 8: Mechanical Testing and Evaluation
H. Kuhn, D. Medlin, editors, p221–231
DOI: 10.1361/asmhba0003272

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Microindentation Hardness Testing

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IN MICROINDENTATION HARDNESS TESTING (MHT), a diamond indenter of specific geometry is impressed into the surface of the test specimen using a known applied force (commonly called a "load" or "test load") of 1 to 1000 gf. Historically, the term "microhardness" has been used to describe such tests. This term, taken at face value, suggests that measurements of very low hardness values are being made, rather than measurements of very small indents. Although the term "microhardness" is well established and is generally interpreted properly by test users, it is best to use the more correct term, microindentation hardness testing.

There is some disagreement over the applied force range for MHT. ASTM E 384 states that the range is 1 to 1000 gf, and this is the commonly accepted range in the United States. Europeans tend to call the range of 200 to 3000 gf the "low-load" range. They do this because forces smaller than 200 gf generally produce hardness numbers that are different from those determined from tests conducted with forces ≥200 gf. This problem is discussed later in this article.

The hardness number is based on measurements made of the indent formed in the surface of the test specimen. It is assumed that recovery does not occur upon removal of the test force and indenter, but this is rarely the case. The Knoop test is claimed to eliminate recovery, but again, this is not true for tests of metallic materials. For the Vickers test, both diagonals are measured and the average value is used to compute the Vickers hardness (HV). The hardness number is actually based on the surface area of the indent itself divided by the applied force, giving hardness units of kgf/mm^2. In the Knoop test, only the long diagonal is measured, and the Knoop hardness (HK) is calculated based on the projected area of the indent divided by the applied force, also giving test units of kgf/mm^2. In practice, the test units kgf/mm^2 (or $gf/\mu m^2$) are not reported with the hardness value.

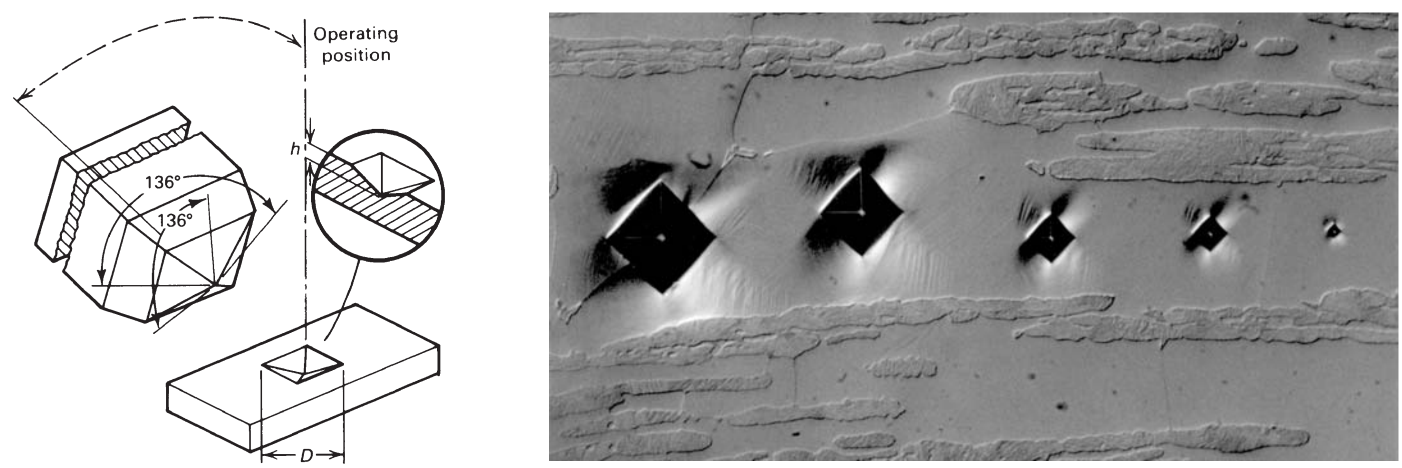
Vickers Hardness Test

In 1925, Smith and Sandland of the United Kingdom developed an indentation test that employs a square-based pyramidal-shaped indenter made from diamond (Fig. 1a). Figure 1(b) shows examples of Vickers indents to illustrate the influence of test force on indent size. The test was developed because the Brinell test, using a spherical hardened steel indenter, could not test hard steels. They chose the pyramidal shape with an angle of 136° between opposite faces in order to obtain hardness numbers that would be as close as possible to Brinell hardness numbers for the same specimens. This made the Vickers test easy to adopt, and it rapidly gained acceptance. Unlike Rockwell tests, the Vickers test has the great advantage of using one hardness scale to test all materials.

In this test, the force is applied smoothly, without impact, and held in contact for 10 to 15 s. The force must be known precisely (refer to ASTM E 384 for tolerances). After the force is removed, both diagonals are measured and the average is used to calculate the HV according to:

$$HV = \frac{2000P \sin(\alpha/2)}{d^2} = \frac{1854.4P}{d^2} \quad \text{(Eq 1)}$$

where d is the mean diagonal in μm, P is the applied load in gf, and α is the face angle (136°).



(a) (b)

Fig. 1 Vickers hardness test. (a) Schematic of the square-based diamond pyramidal indenter used for the Vickers test and an example of the indentation it produces. (b) Vickers indents made in ferrite in a ferritic-martensitic high-carbon version of 430 stainless steel using (left to right) 500, 300, 100, 50, and 10 gf test forces (differential interference contrast illumination, aqueous 60% nitric acid, 1.5 V dc). 250×

The hardness can be computed with the formula and a pocket calculator, or using a spreadsheet program. Most modern MHT units have the calculation capability built in and display the hardness value along with the measured diagonals. A book of tables of HV as a function of d and P also accompanies most testers, and ASTM E 384 includes such tables.

The macro-Vickers test (ASTM E 92) operates over a range of applied forces from 1 to 120 kgf, although many testers cover a range of only 1 to 50 kgf, which is usually adequate. The use of forces below 1 kgf with the Vickers test was first evaluated in 1932 at the National Physical Laboratory in the United Kingdom. Four years later, Lips and Sack constructed the first Vickers tester designed for low applied forces.

Knoop Hardness Test

In 1939, Frederick Knoop and his associates at the former National Bureau of Standards developed an alternate indenter based on a rhombohedral-shaped diamond with the long diagonal approximately seven times as long as the short diagonal (Fig. 2a). Figure 2(b) shows examples of Knoop indents to illustrate the influence of applied load on indent size. The Knoop indenter is used in the same machine as the Vickers indenter, and the test is conducted in exactly the same manner, except that the Knoop hardness (HK) is calculated based on the measurement of the long diagonal only and calculation of the projected area of the indent rather than the surface area of the indent:

$$HK = \frac{1000P}{C_p d^2} = \frac{14229P}{d^2} \quad \text{(Eq 2)}$$

where C_p is the indenter constant, which permits calculation of the projected area of the indent from the long diagonal squared.

The Knoop indenter has a polished rhombohedral shape with an included longitudinal angle of 172° 30′ and an included transverse angle of 130° 0′. The narrowness of the indenter makes it ideal for testing specimens with steep hardness gradients. In such specimens, it may be impossible to get valid Vickers indents as the change in hardness may produce a substantial difference in length of the two halves of the indent parallel to the hardness gradient. With the Knoop test, the long diagonal is set perpendicular to the hardness gradient and the short diagonal is in the direction of the hardness gradient.

For the same test force, Knoop indents can be more closely spaced than Vickers indents, making hardness traverses easier to perform. The Knoop indenter is a better choice for hard brittle materials where indentation cracking would be more extensive using the Vickers indenter at the same load. The Knoop indent is shallower (depth is approximately $\frac{1}{30}$ the long diagonal) than the Vickers indent (depth is approximately $\frac{1}{7}$ the average diagonal). Hence, the Knoop test is better suited for testing thin coatings. On the negative side, the Knoop hardness varies with test load and results are more difficult to convert to other test scales.

Expression of Test Results

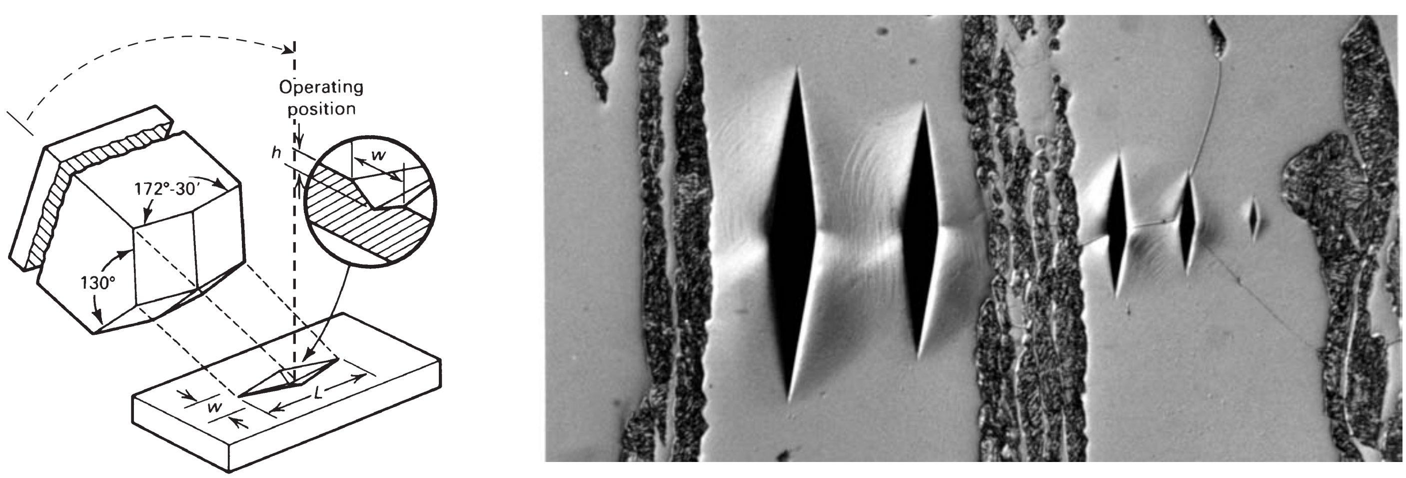
Historically, the official way in which Vickers and Knoop hardness numbers have been presented has varied with time, although many users seem to be unaware of the preferred style. The acronyms VHN and KHN were introduced many years ago and stand for Vickers hardness number and Knoop hardness number. DPN, for diamond-pyramid hardness number, was introduced at approximately the same time. While some have claimed the DPN and VHN are not the same, this is not true. In the early 1960s, ASTM initiated a more modern, systematic approach for all hardness tests and adopted the acronyms HV and HK for the two tests, yet the former acronyms are still widely used (as are many other obsolete acronyms, like BHN and R_C instead of HB and HRC). Style guides for many publications do not seem to track these changes carefully.

For stating the actual hardness results, ASTM advocates the following approach. ASTM E 384 recommends expressing a mean hardness of 425 in the Vickers test using a 100 gf applied force as 425 HV_{100}, while by ISO rules, it would be expressed as 425 $HV_{0.1}$ (because 100 gf would be expressed as 0.1 kgf). ASTM Committee E-4 is currently recommending adoption of a slightly different approach: 425 HV 100 gf. While it has proven difficult to get people to adopt a unified expression style, it is important that the stated results indicate the mean value, the test used, and the test force as a minimum.

Microindentation Hardness-Testing Equipment

A variety of microindentation test machines are produced, ranging from relatively simple, low-priced units (Fig. 3) to semiautomated systems (Fig. 4a) and fully automated systems (Fig. 4b). In most cases, either a Knoop or a Vickers indenter can be used with the same machine, and it is a relatively simple matter to exchange indenters. The force is applied either directly as a dead weight or indirectly by a lever and lighter weights. New testers using a closed-loop load-cell system (Fig. 5) are also available. The magnitude of the weights and force application must be controlled precisely (refer to ASTM E 384).

Most tester systems use an automated test cycle of loading, applying the load for the desired



(a) (b)

Fig. 2 Knoop hardness test. (a) Schematic of the rhombohedral-shaped diamond indenter used for the Knoop test and an example of the indentation it produces. (b) Knoop indents made in ferrite in a ferritic-martensitic high-carbon version of 430 stainless steel using (left to right) 500, 300, 100, 50, and 10 gf test forces (differential interference contrast illumination, aqueous 60% nitric acid, 1.5 V dc). 300×

time, and unloading to ensure reproducibility in the test. Vibrations must be carefully controlled, and this becomes even more important as the applied force decreases. Manual application and removal of the applied force is not recommended due to the difficulty in preventing vibrations that will enlarge the indent size.

The indenter must be perpendicular to the test piece. An error of as little as 2° from perpendicular will distort the indentation shape and introduce errors. A larger tilt angle may cause the specimen to move under the applied force. To aid in controlling this problem, most testers come with a device that can be firmly attached to the stage (Fig. 6). The mounted specimen, or a bulk unmounted specimen of the proper size, can be placed within this device and the plane-of-polish is automatically indexed perpendicularly to the indenter. Historically, it has been a common practice to simply place a specimen on the stage and proceed with indentation, but if the plane-of-polish is not parallel to the back side of the specimen, it will not be perpendicular to the indenter, introducing tilt errors.

Fig. 3 Example of a simple, low-cost manual microindentation hardness-testing unit with a Filar micrometer for measurements but no automation

The stage is an important part of the tester. The stage must be movable and movement is usually controlled in the *x* and *y* directions by micrometers. Once the specimen is placed in the top-indexed holder, the operator must move the stage micrometers to select the desired location for indenting. If a traverse of several hardness readings is desired at inward intervals from a side surface of the specimen (as in case-depth measurements), then the surface of interest should be oriented in the holder so that it is perpendicular to either the *x* or *y* direction of the traverse. If the Knoop indenter is chosen, its long diagonal must also be parallel to the surface of interest. For example, if the Knoop long axis is in the direction going from the front to the back of the tester, then the surface of interest must also be aligned in the same direction. Accordingly, the *x*-axis (left to right) micrometer is used to select the desired indentation positions. The micrometers are ruled in either inches or millimeters and are capable of making very precise movement control.

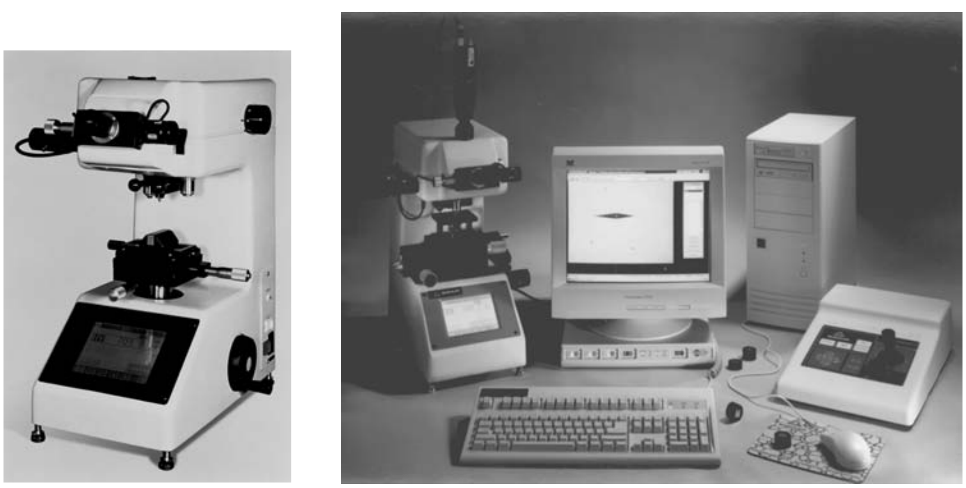
Because the diagonals must be measured after the force has been removed, the tester is equipped with at least two metallurgical objectives (i.e., reflected light), usually 10× and 40×. Some systems may have a third or fourth objective on the turret. For measurement of small indents (<20 μm in diagonal length), a higher-power objective (60×, 80×, or 100×) can be used in place of the 40× objective if the tester has places for only two objectives. The objectives should have a reasonably high numerical aperture for their magnifications to give the best resolution. The 10× objective is usually used as a spotter, that is, simply to find the desired test location. The measuring eyepiece is generally 10×. Naturally, the optical system must be carefully calibrated using a stage micrometer. In general, indents are measured to the nearest 0.1 μm with an accuracy of no more than ±0.5 μm in length. A proper Köhler illumination system is necessary to fully illuminate the specimen. In general, a magnification that makes the diagonal between 25% and less than 75% of the field width is ideal; however, it is not always possible to follow this rule.

Calculation of the hardness is based on the length of the diagonals. The major problem is defining where the indent tips are located. This requires proper illumination, adjustment of the optics for best resolution and contrast, and careful focusing. Every laboratory should have a regular schedule for cleaning the optical components of their MHT apparatuses, as well as for verifying their calibration. A Filar micrometer is used for the diagonal measurement. The micrometer lines have a finite thickness, which requires use of a systematic measurement scheme. Several indent measurement approaches can be used. One popular approach is to bring the two Filar lines just into contact and then zero the micrometer. The interior sides of the Filar lines are then adjusted so that the indent tips just touch the inside of each line.

In recent years, the MHT system has been automated by coupling it to an image analyzer (Fig. 4b). The image-analysis system software is used to control all of the functions regarding indent location, indent spacing, number of indents, indenting, measurement of the indents, calculation of hardness values, and data plotting. For those who perform a substantial number of hardness traverses, this equipment is very useful because the test work is automated, allowing the metallographer to do other tasks.

Hardness Conversions

Sometimes it is desirable to know the equivalent hardness in a scale other than the Vickers



(a) (b)

Fig. 4 Semiautomated and fully automated microindentation hardness testers. (a) Semiautomated tester with a Filar micrometer for measurements, automated readout of the test results with its equivalent hardness in another selected scale. (b) Fully automated tester interfaced to an image analyzer to control indenting, measurement, and data manipulation

Fig. 5 Closed-loop load-cell microindentation hardness tester

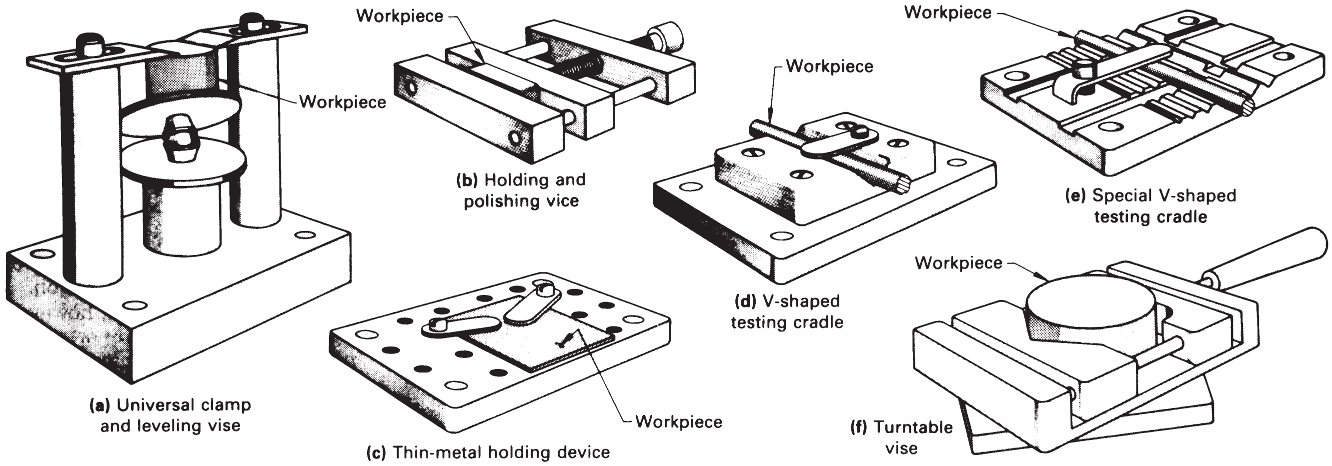


Fig. 6 Examples of fixtures for holding test pieces for microindentation hardness testing

or Knoop. It is not uncommon for product specifications to define the hardness for a case depth in the Rockwell C scale, which, of course, is a bulk test scale unsuitable for case depth determination. Although this seems (and is) illogical, it is widely practiced, probably because designers are not familiar with the Vickers or Knoop scales. Hardness conversions are developed empirically, and there is a degree of error associated with all conversions. The primary source for hardness conversions is ASTM E 140, which lists the conversions in tabular form and also contains equations based on the tabular data. Some MHT units have these tables or the equations built in and will list an equivalent hardness of your choice with each measurement. The most common conversion is from a Vickers or Knoop scale to a Rockwell C scale. In general, these conversions are most commonly available for steels, aluminum alloys, and nickel alloys. Conversions between various scales may be material sensitive.

Conversion of Vickers data to other scales is more straightforward than converting Knoop data to other scales. Basically, the ASTM E 140 conversions between Vickers and other scales can be used for any test force greater than 100 gf. Conversions of Knoop to other scales are problematic because Knoop hardness varies more with load. If the published conversion is for a 500 gf applied load, then this conversion is best for that load and reasonably accurate for loads slightly lower and generally adequate for greater loads, as the Knoop hardness is reasonably constant for loads of 500 gf and above. Aside from the E 140 conversions, two published conversion charts are worth noting. First, Emond (Ref 1) published a correlation chart of Vickers hardness (10 kgf load) to Knoop hardness at loads of 10, 25, 50, 100, 200, and 500 gf (Fig. 7). Second, Batchelder (Ref 2) published conversions from Knoop hardness, with loads of 15, 25, 50, 100, 200, 300, 500, and 1000 gf, to Rockwell C (Fig. 8). Before using these conversions, it is a good practice to test your material with both scales to see how well the conversion chart agrees with your bulk test specimens before utilizing the conversions.

Specimen Preparation

Specimen preparation for microindentation hardness testing is not a trivial matter and becomes more critical as the applied force decreases. Further, if testing is to be done near an edge, then edge preservation (i.e., flatness out to the edge) is also required. For relatively high test forces, for example, 300 to 1000 gf, a perfectly prepared specimen is not required. However, this does not mean that sectioning and grinding damage need not be removed. Rather, the normal preparation procedure could be stopped after grinding and polishing down to a 6, 3, or 1 μm diamond finish. For lower loads, it is advisable to completely prepare the specimen to a damage-free condition. Excessive residual damage from sectioning and grinding will influence test results and produce erroneous hardness values. Depending on the nature of the specimen, preparation damage can cause either an increase or a decrease in the apparent hardness relative to the true hardness. Guidelines for preparing metallographic test specimens are given in ASTM E 3 and in standard textbooks (Ref 3) and handbooks (Ref 4, 5).

Microindentation hardness testing near the edge of a specimen is used frequently to determine the hardness of coatings or to evaluate the extent of the increase in surface hardness due to treatments such as induction hardening, carburizing, or nitriding, or due to the loss in hardness because of decarburization. A variety of

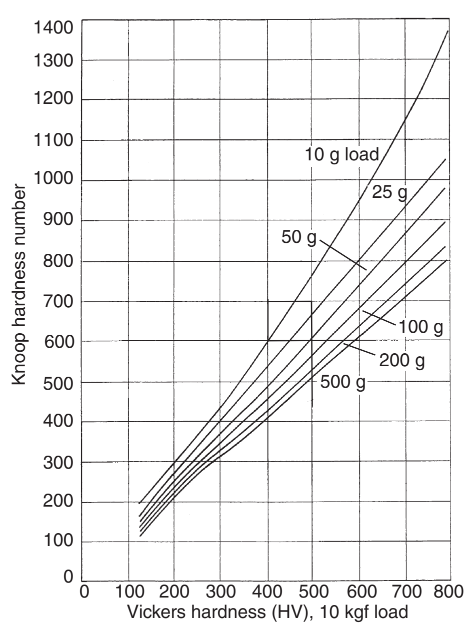


Fig. 7 Correlations between Vickers hardness determined with a 10 kgf load and Knoop hardness determined with loads from 10–500 gf. Source: Ref 1

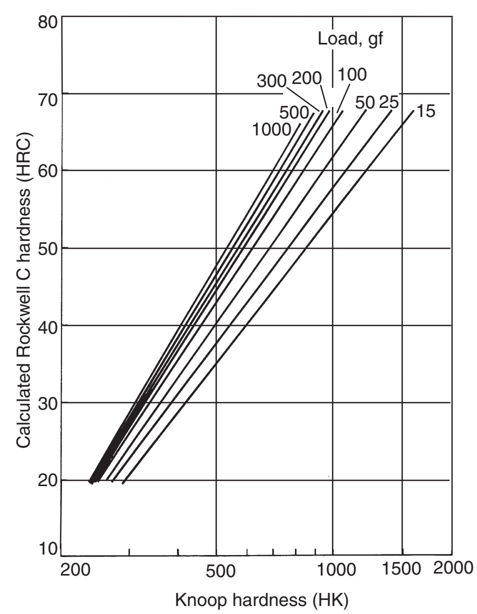


Fig. 8 Correlations between Knoop hardness at loads from 15–1000 gf and Rockwell C hardness. Source: Ref 2

procedures have been developed to provide good edge retention. Today, with a good thermosetting epoxy resin (for best results, cool back to ambient temperature under pressure during mounting), automated preparation equipment, and modern consumable products (use napless cloths for best results), adequate edge retention is readily achievable without requiring protective surface platings (e.g., electroless nickel). It is also possible to prepare unmounted bulk specimens with adequate edge retention using automated equipment and consumables.

Important Test Considerations

All tests require both properly operating equipment and knowledge of how to use it. To obtain precise, unbiased hardness data, a properly prepared specimen must be tested in the correct manner using a properly operating, calibrated tester. ASTM E 384 provides guidance on operating variables developed both theoretically and empirically over a long period of time. Conservative application of these rules is advisable.

Indent Size. In general, the larger the indent is, the better the precision will be. Due to the mathematical approach to defining the Vickers and Knoop hardnesses (Eq 1 and 2, where the denominator is d^2), the curves of diagonal length versus HV or HK get steeper as the test force decreases, as shown in Fig. 9 and 10. Note that as the test force decreases, smaller and smaller variations in diagonal length correlate to larger and larger variations in hardness.

Experience has shown that a single operator typically exhibits a ±0.5 μm variation when measuring the same indent over a period of time, while multiple operators exhibit approximately a ±1.0 μm variation over time. Larger variations have also been observed (Ref 6, 7). A ±0.5 μm variation in the measured diagonal has a greater influence on hardness as the test load decreases, that is, as the diagonal size decreases.

As an example, Fig. 11 shows the change in Vickers hardness when 0.5 μm is either added to, or subtracted from, the diagonal measurement for diagonals ≤40 μm in length. Note that subtracting 0.5 μm has a greater effect on the calculated HV than adding 0.5 μm. This is again due to the d^2 divisor in Eq 1. The graph shows that for a Vickers indent with a 10 μm average diagonal, a ±0.5 μm measurement variation can produce approximately a 10% rise or drop in the hardness. If the hardness is low, this is not too much of a problem, but for high-hardness specimens, a ±10% variation is substantial.

ASTM E 384 recommends that the operator should try to keep indents larger than 20 μm in *d*. Figure 11 demonstrates the reason for this recommendation. A similar graph could be constructed for the Knoop test. In general, determining the location of the tips of the Knoop indent to measure the long diagonal is more difficult than with a Vickers indent because the contrast at the Knoop indent tips is not as strong. The ±0.5 μm measurement variability for the same person as a function of time may be a bit conservative for the Knoop test.

If the operator has a rough idea of the hardness of the test piece, then a good estimate can be made of the appropriate test load to choose. The harder the specimen, the greater the test load needed to keep *d* greater than 20 μm. Figures 9 and 10 can be used as a guide. For example, assume that a hardness traverse is to be made on an induction-hardened specimen that is expected to vary in hardness from approximately 750 HV at the surface to 250 HV in the core. Figure 9 says that an applied force of 200 gf will produce approximately a 22 μm diagonal indent for a 750 HV steel and close to a 40 μm diagonal indent for a 250 HV steel. For a 100 gf applied force, the diagonal for 750 HV is less than 16 μm, so it would be best to use a higher load. A 300 gf applied force produces approximately a 27 μm diagonal for 750 HV and approximately a 47 μm diagonal at 250 HV, and it may be a better choice than a 200 gf or 100 gf load. If the hardened case is rather shallow, it may be necessary to space indents along several different parallel traces at different depths so that the gradient can be assessed satisfactorily without tight indent spacing adversely influencing the test data.

The opposite problem, that of an excessively large (d > 75% of the field width) indent is less common, but may arise depending on test conditions. In general, MHT is performed in an effort to measure spatial variations in hardness or the hardness of small regions. But sometimes it is used as a convenient substitute for a bulk hardness test on a small specimen of homogenous nature at the same time as the structure is examined. In that case, the indent size is not too

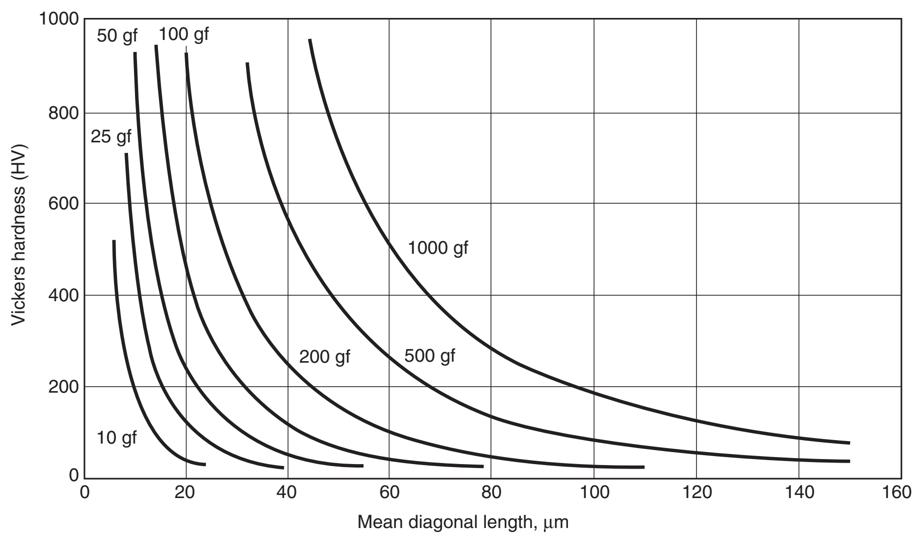


Fig. 9 Relationships between the mean diagonal length and the Vickers hardness for loads of 10–1000 gf

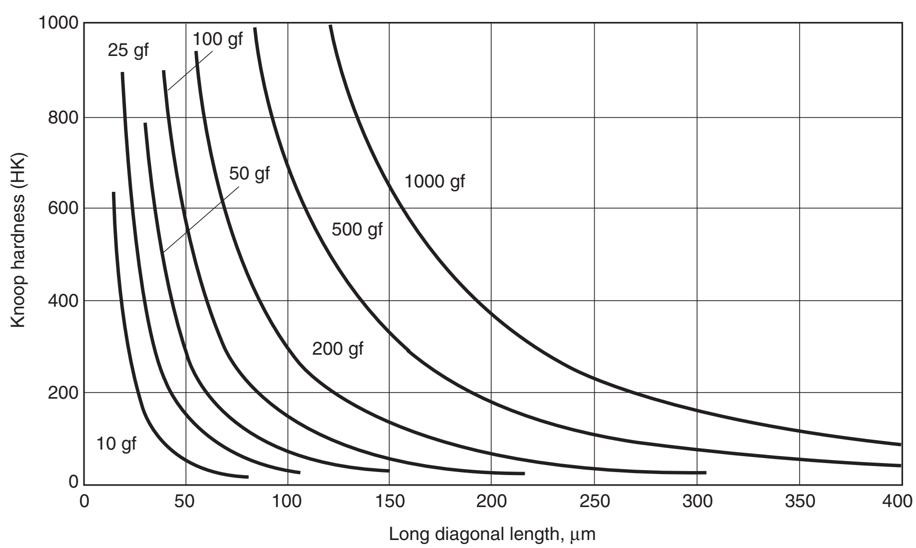


Fig. 10 Relationships between the long diagonal length and the Knoop hardness for loads of 10–1000 gf

critical as long as a ±0.5 μm measurement variation has only a small influence on the calculated HV. With a very soft material, the indent should be small enough that it can be kept entirely in the field of view of the optics.

Indent Spacing. In general, the same guidelines used in bulk hardness tests are used for MHT. Indenting creates both elastic and plastic deformation and a substantial strain field around the indent. If a second indent is made too close to a prior indent, its shape will be distorted on the side toward the first indent. This produces erroneous test results.

In general, the spacing between indents should be at least 2.5 times the d length for the Vickers test and at least twice the length of the short diagonal for the Knoop test. The minimum spacing between the edge of a specimen and the center of an indent should be 2.5d, although values as low as 1.8d have been demonstrated to be acceptable.

Hardness versus Applied Test Force

For the Vickers test, especially in the macro applied force range, it is commonly stated that the hardness is constant as the load is changed. For microindentation tests, the Vickers hardness is not constant over the entire range of test forces. For Vickers tests with an applied force of 100 to 1000 gf, the measured hardnesses are usually equivalent within statistical precision. The Vickers indent produces a geometrically similar indent shape at all loads, and a log-log plot of applied force (load) versus diagonal length should exhibit a constant slope, n, of 2 for the full range of applied force (Kick's Law); however, this usually does not occur at forces under 100 gf.

Reference 6 shows four trends for force (load) and Vickers MHT data:

- *Trend 1:* HV increases as force decreases ($n < 2.0$).
- *Trend 2:* HV decreases as force decreases ($n > 2.0$).
- *Trend 3:* HV essentially constant as force varies ($n = 2.0$).
- *Trend 4:* HV increases, then decreases with decreasing force.

Trends 1, 2, and 4 are more easily detected in hard specimens than on soft specimens where trend 3 is observed. Many publications, particularly those reporting trends 1 and 2, have attributed these trends to material characteristics.

The Knoop indenter does not produce geometrically similar indents, so the hardness should increase with decreasing test force. Due to the poor image contrast at the Knoop indent tips (long diagonal), it is far more likely that d will be undersized, leading to a higher hardness number. Consequently, the Knoop hardness increases with decreasing test force, and the magnitude of the increase rises with increasing hardness. However, a few studies reported a variation in this trend: HK increased with decreasing force and then decreased at the lowest applied force.

It is widely claimed in the literature that the Vickers hardness is constant with test force in the macro force range (≥1 kgf). However, a search in the literature for data to prove this point yielded very little evidence. Reference 3 gives measurements made on five polished HRC test blocks, with hardnesses ranging from 22.9 to 63.2 HRC, using six test forces from 1 to 50 kgf. At each force, six impressions were made, and the mean results are in Fig. 12. The Filar micrometer used a magnification of 100×. Note that the HV is essentially constant for forces of 10 kgf and greater. For each test block, the hardness decreased for test forces less than 10 kgf. The degree of decrease increased with increasing hardness. Thus, for this macro Vickers tester, HV was not constant but exhibited trend 2, the most commonly observed trend for studies of MHT and HV force.

The exact same steel test blocks were also subjected to Vickers microindentation hardness tests using nine different forces from 5 to 500 gf (Ref 3). Again, six impressions were made at each test force, and the mean values are plotted in Fig. 13. These impressions were measured at 500×. Again, the same basic trend is observed. In most cases, HV is essentially constant at forces down to 100 gf, then the hardness decreases. The magnitude of this decrease again increases with increasing specimen hardness.

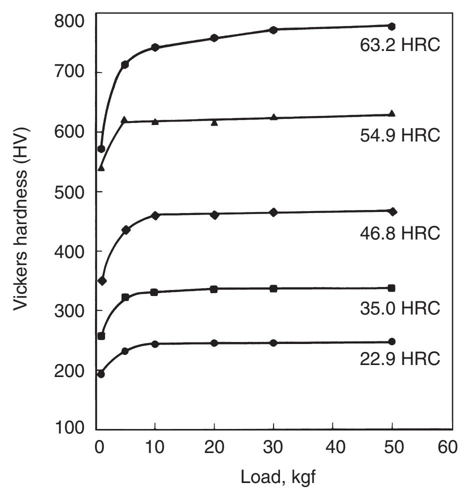


Fig. 12 Measured Vickers macrohardness for five steel test blocks using test forces from 1–50 kgf. Source: Ref 3

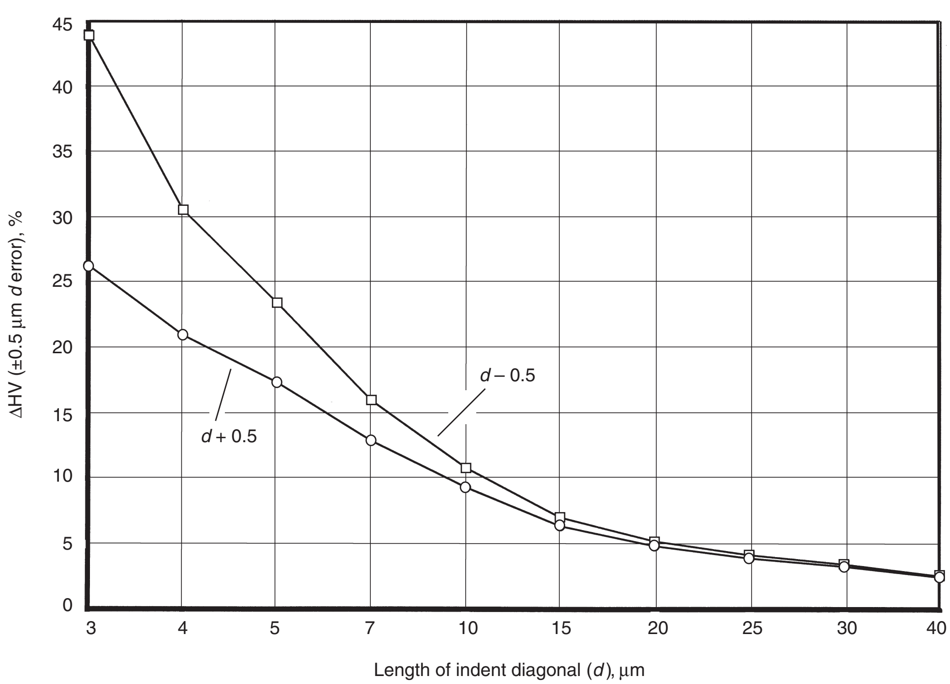


Fig. 11 Influence of a measurement error of ±0.5 μm on the calculated Vickers hardness as a function of diagonal length

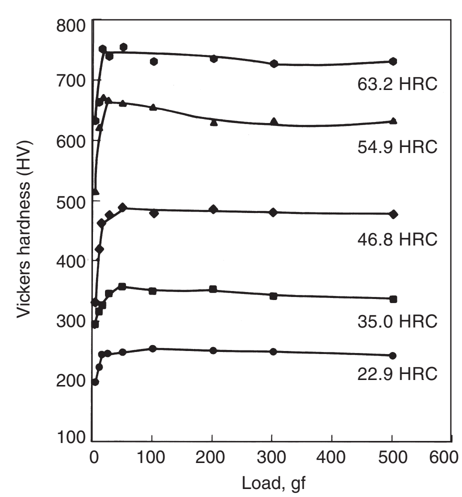


Fig. 13 Measured Vickers microindentation hardness for five steel test blocks using test forces from 5–500 gf. Source: Ref 3

For several of the data, the hardness appears to rise slightly as the force drops below 100 gf, and then it decreases (trend 4). Thus, for the work detailing MHT in HV versus the test forces, both trends 2 and 4 were obtained.

These results, using the same set of five specimens with a wide range of hardnesses and tests with both micro- and macro-Vickers units, revealed basically the same trend. At small indent sizes for both testers, measurements yielded lower hardness (indents being oversized) than they should. This can only be due to visual perception problems in sizing small indents at the tester magnifications employed (100× for the macro system and 500× for the micro system). No material characteristic can possibly explain this problem.

To further demonstrate that the observed trends of HV versus test force (load) are due to measurement difficulties, the results of an ASTM Committee E-4 interlaboratory round-robin test program is cited (Ref 6, 7). In this study, one person indented three ferrous and four nonferrous specimens at test forces of 25, 50, 100, 200, 500, and 1000 gf (five times at each force). Then, twenty-four people measured the indents: thirteen measured all of the Knoop and Vickers indents in the ferrous specimens (fourteen actually measured specimen F1), and eleven measured the Knoop and Vickers indents in the nonferrous specimens. Agreement was best for the low hardness specimens, as would be expected, because they had the largest indents and the effect of small measurement errors is minimal. The Vickers hardness, in most cases, decreased with forces below 100 gf, but all four possible trends reported in the literature can be seen in the measurement data for the same indents.

As an example, Fig. 14 shows the data for nine of the fourteen people who measured the Vickers indents in the hardest ferrous specimen (specimen F1). The overall trend for the data is trend 2. However, examination of the data shows that test lab 8 followed trend number 1, lab 1 followed trend 3, and lab 3 followed trend 4. Statistical analysis of all of the test data suggested that these nine people obtained essentially the same test results while some or all of the data from the other five people represented "outlier" conditions. Figure 15 shows the data for the five outlier labs for the F1 specimen (where lab F was defined as an outlier lab based on results for other specimens—their results for specimen F1 were marginal). The "good max" and "good min" lines in Fig. 15 encompass the range of "good" data shown in Fig. 14. Again, several HV-versus-force trends are observed: labs E, H, and J follow trend 1, and labs F and M follow trend 2. Because exactly the same indents were measured, these variations in test results come only from measurement inconsistencies. This study reveals that the most commonly obtained trend was trend 2, decreasing HV with decreasing test force, and this is the most commonly reported trend in the literature. Thus, it is more likely for an operator to oversize small Vickers indents than to undersize them or to measure their true size.

Measurements of the Knoop indents also reveal substantial variations in the data. In most cases, the HK rose as the test force decreased, with most of the increase occurring at forces less than 200 gf. In general, HK results were statistically identical for each specimen at forces from 200 to 1000 gf. For the nonferrous specimens, one rater consistently obtained the very unusual trend of decreasing HK with forces less than 200 gf. One other rater obtained a similar, but less pronounced, decrease in HK with decreasing test forces; but this was only for the hardest nonferrous specimen (mean hardness, approximately 330 HK).

The visibility of the tips of the long diagonal on the Knoop indent is poorer than for Vickers indents. Thus, for Knoop indents, undersizing the indent is far more likely than oversizing. However, it is clear that one of the eleven people who measured the Knoop indents in this study consistently oversized the Knoop indents. At test forces above 200 gf, this person's results agreed with the mean results in two cases, were below the mean in one case, and were above the mean in another case. A calibration error would produce a consistent bias in all of the data; however, this could not be the case for this person's test results. Interestingly, this person was an experienced metallographer, not a novice.

There are times when the hardness tester can be the source of a variation in the load-hardness relationship. Before using a new MHT unit, it is a good practice to select a specimen with a homogeneous microstructure and a known hardness and then perform a series of tests using the full range of applied test forces available for the unit. To obtain good statistics, make a number of impressions at each load. As an illustration of this problem, two testers were evaluated over their full ranges using a hardened specimen of type 440C martensitic stainless steel. For tester A, six indents were made at each available test load, while for tester B, only three indents were made at each load due to time limitations with the unit. The mean results are plotted in Fig. 16. While tester A produced virtually identical results over the full load range, it is clear that tester B was applying excessively high test forces at all loads under 1000 gf. Clearly this was a machine problem because the same person performed both sets of measurements on the same specimen. Verification of the instrument using properly calibrated test blocks should help identify this type of problem.

Repeatability and Reproducibility

Appendix X2 of ASTM E 384, along with Ref 6 and 7, describes the results of an ASTM interlaboratory round-robin program used to determine the precision of measuring Knoop and Vickers indents and the repeatability and reproducibility of such measurements. Repeatability is a measure of how well an individual operator can replicate results on different days with the same specimen and the same equipment. Reproducibility measures the ability of different operators, in different laboratories, to obtain the same results, within statistical limits. Repeatability and reproducibility were best for low-hardness specimens and got poorer as the hardness increased; that is, as the indent size decreased. Repeatability was always somewhat better than reproducibility, as might be expected.

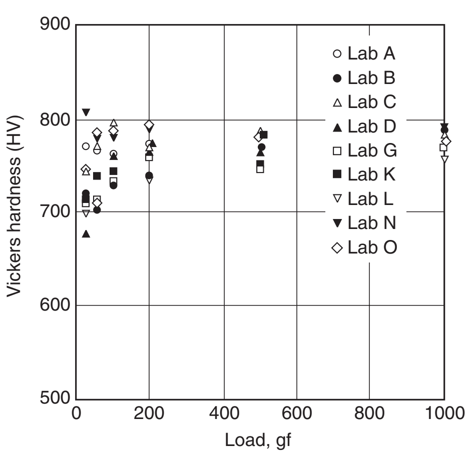


Fig. 14 ASTM E-4 round-robin interlaboratory Vickers microindentation hardness-testing data for the hardest (F1) test specimen and nine people (measuring the same indents) who produced "good" data for test loads from 25–1000 gf. Source: Ref 6, 7

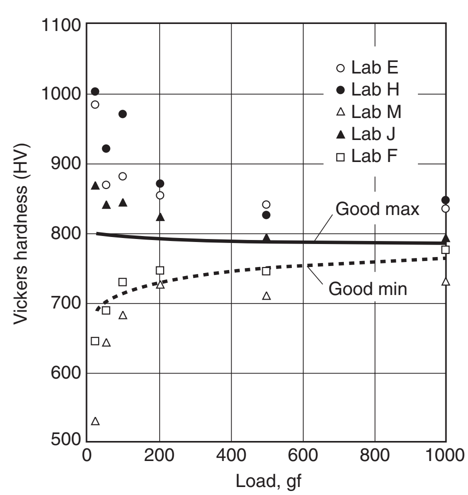


Fig. 15 Data shown in Fig. 14 (all points fall within the two lines) plus the individual data from four "outlier" raters. Source: Ref 6, 7

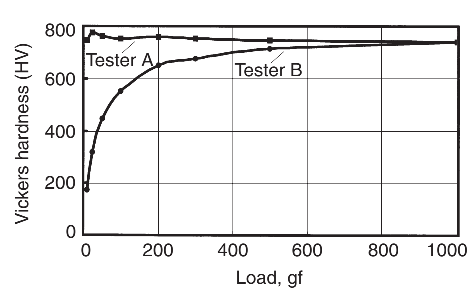


Fig. 16 Curves showing load versus Vickers hardness for two testers (with the same operator) evaluating the hardness of the same type 440C martensitic stainless steel specimen (62.7 HRC)

For a material with a hardness of 900 HV, repeatability for a 25 gf load was approximately ±170 HV, and for a 1000 gf load it was approximately ±25 HV, while reproducibility for a 25 gf load was approximately ±220 HV, and for a 1000 gf load it was approximately ±40 HV. For a material with a hardness of 900 HK, repeatability for a 25 gf load was approximately ±75 HK, and for a 1000 gf load it was approximately ±25 HK, while reproducibility for a 25 gf load was approximately ±105 HK, and for a 1000 gf load it was approximately ±40 HK. This shows that the repeatability and reproducibility values at the highest loads were similar for both types of indents, but as the test load decreased, the longer Knoop indent (at each load) yielded better repeatability and reproducibility than the smaller Vickers indent at the same load. These trends again highlight the importance of trying to use the greatest possible load for any test.

Applications

Because hardness tests are a quick and convenient way to evaluate the quality or characteristics of a material, hardness testing is widely used in quality-control studies of heat treatment, fabrication, and materials processing. It is also a key test used in failure analysis work.

Microindentation hardness testing provides the same benefit as bulk hardness testing, but with a much smaller indent. Because the indents are small, MHT can be used for many parts or material forms that are too small or too thin to test with bulk test procedures. Likewise, MHT allows hardness measurements of microstructural constituents. For example, the determination of hardness of specific types of carbides, nitrides, borides, sulfides, or oxides in metals has been widely performed, particularly in wear and in machinability research.

There is a long list of applications where MHT is indispensable. A few examples are described in this section. The examples are just a few of the many that could be chosen to demonstrate the value of MHT. To a large extent, MHT can be considered as simply an extension of bulk hardness testing, in that it can be used for all the same purposes as bulk hardness tests. However, due to the very small size of the indent, MHT has a host of applications that cannot be performed with bulk tests. It can also be considered as a strength microprobe and, thus, an extension of tensile testing. When properly used, MHT is a great asset in any laboratory.

Hardness Testing of Thin Products

Foil or wire product forms depend on MHT in quality-control programs. In general, the indent depth should be no more than 10% of the thickness or diameter of the products. Figure 17 shows the relationship among the minimum foil thickness that can be tested, the applied force, and the Knoop hardness. As this figure shows, for thicknesses less than 0.010 in. (254 μm), test-force selection becomes more critical as the thickness decreases and the hardness decreases. For example, for a foil 0.002 in. thick (51 μm) with high hardness (e.g., greater than 500 HK), test forces up to 800 gf can be used. However, if the hardness is not known, and a 500 gf load indicates a hardness of approximately 200 HK, then it would be advisable to retest the foil using a force of, at most, 300 gf because the test at 500 gf may not be valid.

Hardness tests of thin materials and thin coatings often require very low applied forces (loads). As already demonstrated, it is quite difficult to measure very small indents. MHT units are readily available for making impressions at forces down to 1 gf, and special testers are available that can indent at even lower forces. (These devices are not discussed in this article, however.) In the case of MHT systems using indenting forces less than 25 gf and indents between 1 and 25 μm, it may be advisable to place the tester on an antivibration platform and to use at least 60× objectives with a high numerical aperture for measurements. Oil-immersion objectives may be required, particularly for materials with poor light reflectivity.

Case Hardness Measurement

Perhaps the classic application of MHT is the assessment of changes in surface hardness: usually increases due to surface treatments, such as carburizing, nitriding, or localized surface-hardening processes, are analyzed, but decreases in hardness due to local chemistry changes (decarburization) or localized heating are also examined. While these changes are usually detectable by eye on a properly prepared metallographic cross-section, hardness traverses define the magnitude and extent of such changes with greater precision and detail. It is not uncommon for quality-control tests to require determination of the depth to a specific hardness for a carburized or nitrided part.

Figure 18 demonstrates the measurement of case depth by a series of indentations that traverse

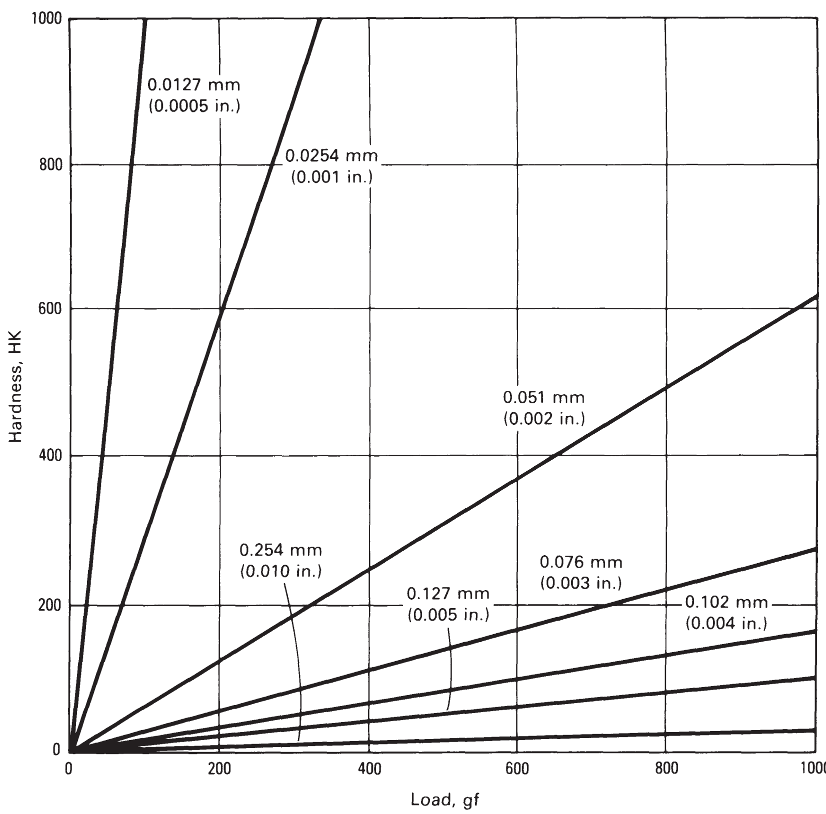


Fig. 17 Minimum thickness of test specimens for the Knoop test as a function of applied force (load) and Knoop hardness

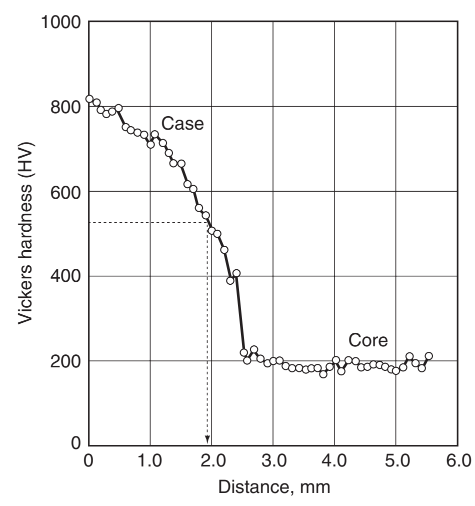


Fig. 18 Vickers traverse showing the hardness profile results from a flame-hardened SAE 8660 gear using a fully automated microindentation hardness-testing system

a cross-section from a flame-hardened SAE 8660 specimen. The hardness traverses used a Vickers tester with the fully automated device (Fig. 4b) and a 300 gf load. The surface hardness is approximately 830 HV, and the hardness drops steadily until, at 2.5 mm depth, the core hardness (~200 HV) is reached. The effective case depth (the depth to 550 HV) occurs at a depth of 1.95 mm.

Figure 19 shows the hardness profile for an induction-hardened SAE 1053 carbon-steel gear using the fully automated system and a 300 gf load. Note that the surface hardness increased slowly from the surface to a depth of 4.1 mm. In this specimen, the microstructure contained at the surface substantial retained austenite, which decreased until it was undetectable at a depth of approximately 3 mm. The prior-austenitic grain size was coarse at the surface and decreased in size through the hardened case. These trends are caused by the temperature profile from induction heating. The hardness drops rapidly in the depth range of 4 to 4.6 mm, and the microstructure changes from predominantly martensite to ferrite and pearlite with a hardness of approximately 230 HV.

When manual MHT systems are used to determine the effective case depth, it is quite common to etch the specimen and find the depth where the microstructure changes from hardened to unhardened. Then, the operator places a few indents in this region and interpolates the depth to the desired hardness, most often 500 or 550 HV, depending on the carbon content. Of course, the very interesting rise in hardness (Fig. 19) from the surface to 4.1 mm would not be detected. This may have an adverse effect on the wear behavior and presents a dilemma for the analyst because the surface hardness is less than the hardness criteria for the effective case depth. Note that the surface does not exceed 550 HV until a depth of approximately 1.5 mm. Then, the hardness raises to approximately 680 HV at approximately 4 mm depth. The hardness falls again to 550 HV at approximately 4.5 mm depth. The detailed variation of hardness with depth can be observed more easily with automated traverse hardness tests.

Figure 20 shows a hardness traverse for a carburized SAE 8620 mold that exhibited substantial retained austenite in the hardened case. Again, the specimen was evaluated with the fully automated system in Fig. 5 with a 300 gf load. Note that the hardness is somewhat erratic in the fully hardened surface layer (surface to approximately 1.8 mm depth). This is due to the presence of retained austenite in this zone, which is substantially lower in hardness than plate martensite. If a lower test force were used, the scatter would be greater. Very low test forces, producing very small indents, might produce a hardness variation of several hundred HV in the case. The effective case depth (depth to 550 HV) is at 2.1 mm, and the core is reached at approximately 2.5 mm (~400 HV). Again, if testing were performed manually and only in the transition zone, the metallographer would not have observed the variability in hardness in the fully hardened zone.

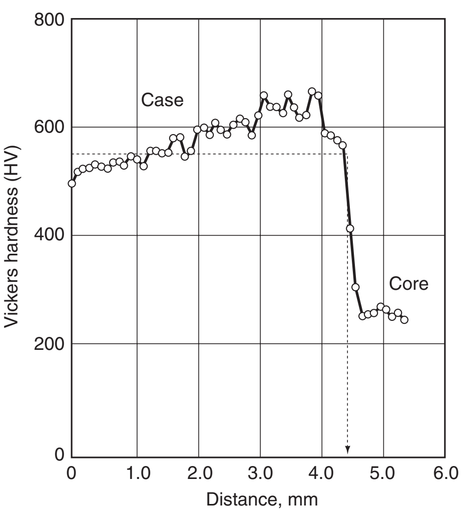


Fig. 19 Vickers traverse showing the hardness profile results from an induction-hardened SAE 1053 gear using a fully automated microindentation hardness-testing system

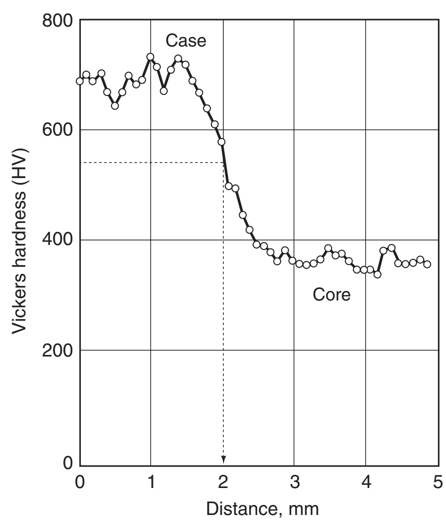


Fig. 20 Vickers traverse showing the hardness profile results from a carburized and hardened SAE 8620 mold using a fully automated microindentation hardness-testing system

Alloy Phase Hardness Measurements

Microindentation hardness testing has been widely used in alloy development research, particularly in multiphase alloy studies. Because hardness can be correlated to strength,

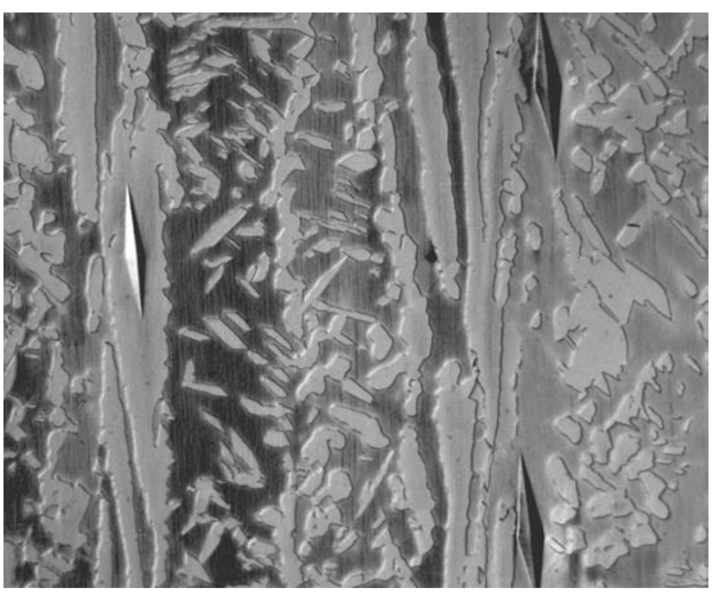


Fig. 21 Knoop indents in ferrite (dark) and austenite (white) grains in a dual-phase stainless steel (differential interference contrast illumination, aqueous 20% nitric acid, 3 V dc). 500×

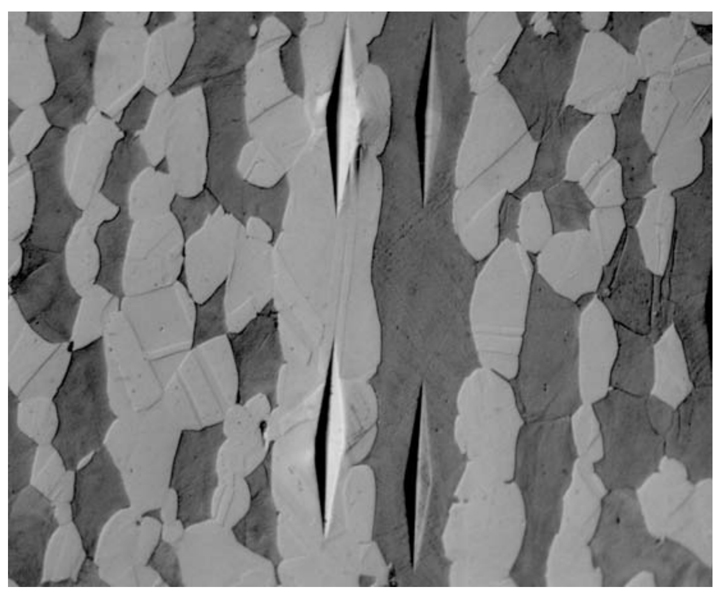


Fig. 22 Knoop indents (50 gf) in alpha (white) and beta (dark) grains in naval brass (C 46400) (differential interference contrast illumination, Klemm's I reagent). 500×

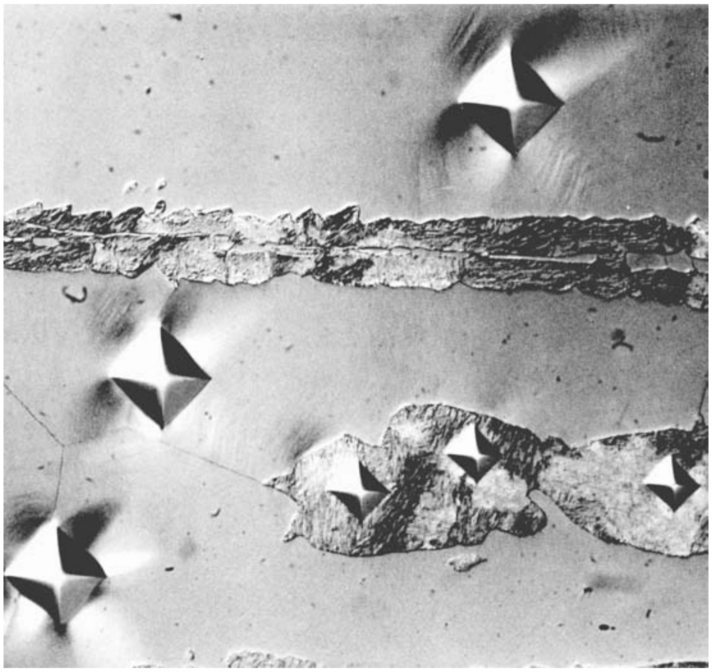


Fig. 23 Vickers indents (100 gf) in alpha (white) and martensite (dark) grains in a high-carbon version of 430 stainless steel (differential interference contrast illumination, aqueous 60% nitric acid, 1.5 V dc). 500×

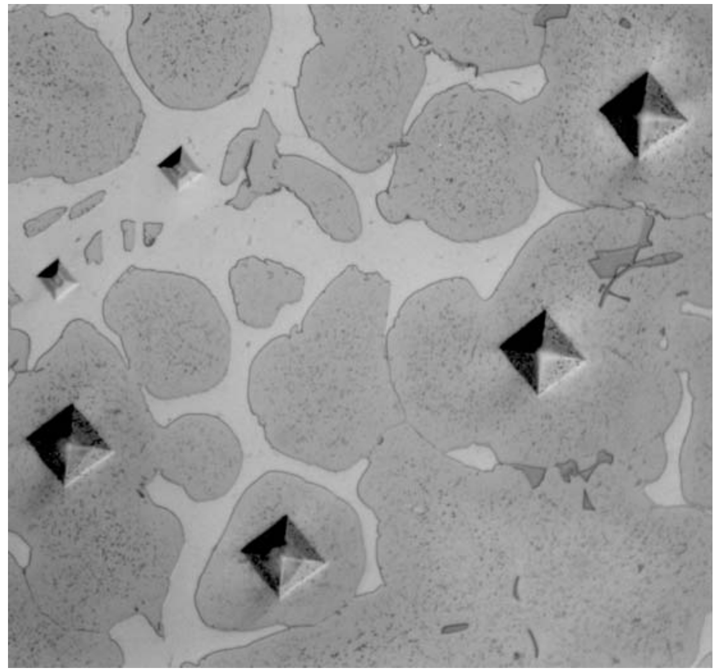


Fig. 24 Vickers indents (50 gf) in the matrix (dark) and in the intergranular beta (white) phase in as-cast beryllium copper (C 82500) that was burnt in solution annealing (differential interference contrast illumination, aqueous 3% ammonium persulfate and 1% ammonium hydroxide). 500×

MHT can be used to determine the properties of phases or constituents. Some such examples are described here.

Example 1: Hardness Measurement on Ferrite and Austenite Grains in Dual Phase Steel. Microindentation testing was performed on the ferrite and austenite grains in a specimen of hot-rolled dual-phase stainless steel. The specimen was prepared so that a plane parallel to the hot-working direction could be observed. Because the phases were elongated rather than equiaxed, the Knoop indenter was used (with a 50 gf load). The specimen was lightly etched electrolytically with 20% nitric acid, which colors the ferrite grains. Indents were made in a number of grains (six or more indents per constituent type, as a rule) to calculate the mean, standard deviation, and the 95% confidence interval. The ferrite had a hardness of 263.5 ± 5 HK_{50} (mean ±95% confidence interval), while the austenite had a hardness of 361.8 ± 18.6 HK_{50}. This difference was significant at the 99.9% confidence level. Figure 21 shows the microstructure of this specimen along with a number of Knoop indents.

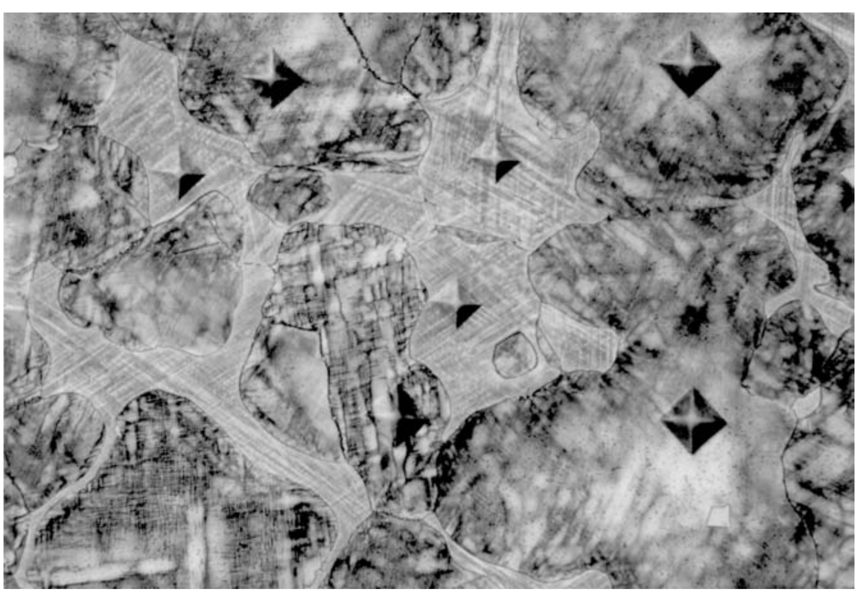


Fig. 25 Vickers indents (100 gf) in the matrix (dark) and in the intergranular beta (white) phase in an age-hardened as-cast beryllium copper (C 82500) that was burnt in solution annealing (differential interference contrast illumination, aqueous 3% ammonium persulfate and 1% ammonium hydroxide). 500×

Example 2: Hardness Measurement on Alpha and Beta Phases in Naval Brass. Microindentation testing with a Knoop indenter was performed on the alpha and beta phases in a specimen of naval brass (C 46400). A longitudinally oriented test plane was evaluated, and the Knoop indentor was used due to the elongated shape of the grains. A test load of 50 gf was used to keep the indents within the grains. The specimen was tint etched with Klemm's I, which colors the beta phase. Again, indents were made on a number of grains of each phase. The alpha phase had a hardness of 178.1 ± 8.8 HK_{50}, while the beta phase had a hardness of 185.4 ± 13.7 HK_{50}. The difference in hardness between alpha and beta phases was not statistically significant. Figure 22 shows the microstructure of this specimen and several of the Knoop indents.

Example 3: Microindentation Hardness of Phases in 430 Stainless Steel. Similar tests were performed on a dual-phase, ferrite and martensite, high-carbon, type 430 stainless-steel specimen. It was possible to test with a 100 gf load using the Vickers indenter. The ferrite had an average hardness of 152.3 ± 5.7 HV_{100} while the martensite had a mean hardness of 473 ± 41.5 HV_{100}. Again, at least six impressions were made in each constituent. The specimen, shown in Fig. 23, was electrolytically etched with aqueous 60% nitric acid at 1.5 V dc. The difference in hardness between the alpha phase and martensite was statistically significant at the 99.9% confidence level.

Example 4: Hardness of Phases in As-Cast Beryllium Copper. MHT can be used to study effects of heat treatment and segregation on the hardness of the phases in as-cast beryllium copper (C 82500) that has been solution treated at 871 °C, hot enough to cause incipient melting. One specimen was age hardened and one was not. Because the phases were essentially equiaxed in shape, the Vickers indenter was used. In the unaged specimen, a 50 gf test force was used, while in the harder, aged specimen, 100 gf could be used. Again, a number of indents, at least six, were made in each phase. For the unaged specimen, shown in Fig. 24, the alpha matrix had a hardness of 107.6 ± 4.8 HV_{50}, while the intergranular beta had a hardness of 401.0 ± 63.0 HV_{50}. For the aged specimen, shown in Fig. 25, the alpha matrix exhibited light and dark crosshatched etched areas suggesting chemical segregation. The light etching alpha had a hardness of 316.1 ± 38.3 HV_{100}, while the dark etching alpha had a hardness of 416.6 ± 8.6 HV_{100}. This difference in hardness was statistically significant at the 99.9% confidence level. The intergranular beta phase also exhibited a crosshatched etched appearance and had a hardness of 521.6 ± 31.9 HV_{100}. The difference in hardness of the intergranular beta phase in the aged versus unaged condition was statistically significant at the 99.9% confidence level. The specimens were etched with aqueous 3% ammonium persulfate-1% ammonium hydroxide. It is best to use the same applied force for each phase or constituent when doing such comparisons, rather than the highest possible applied force in each phase or constituent.

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