

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 $\text{gf}/\mu\text{m}^2$) 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°).

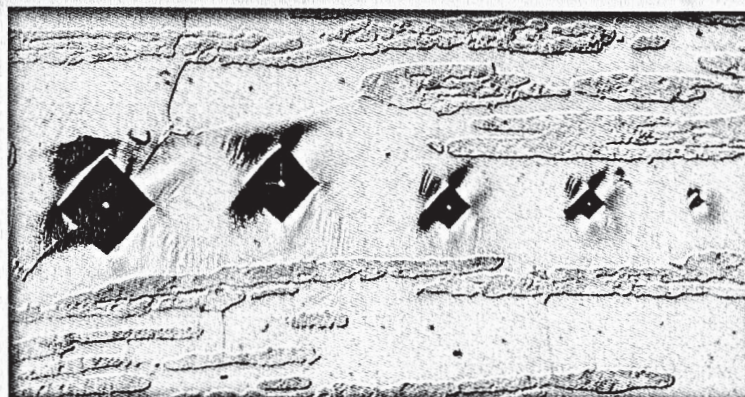
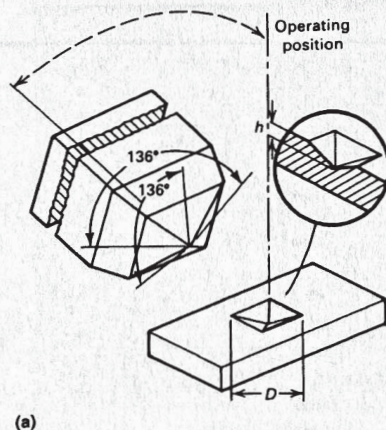


Fig. 1 Vickers hardness test. (a) Schematic of the square-based diamond pyramidal indenter used for the Vickers test and an example of the indentation it produces. (b) Vickers indents made in ferrite in a ferritic-martensitic high-carbon version of 430 stainless steel using (left to right) 500, 300,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

critical as long as a $\pm 0.5 \mu\text{m}$ 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 ($\geq 1 \text{ kgf}$). 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 $100\times$. 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 $500\times$. 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.

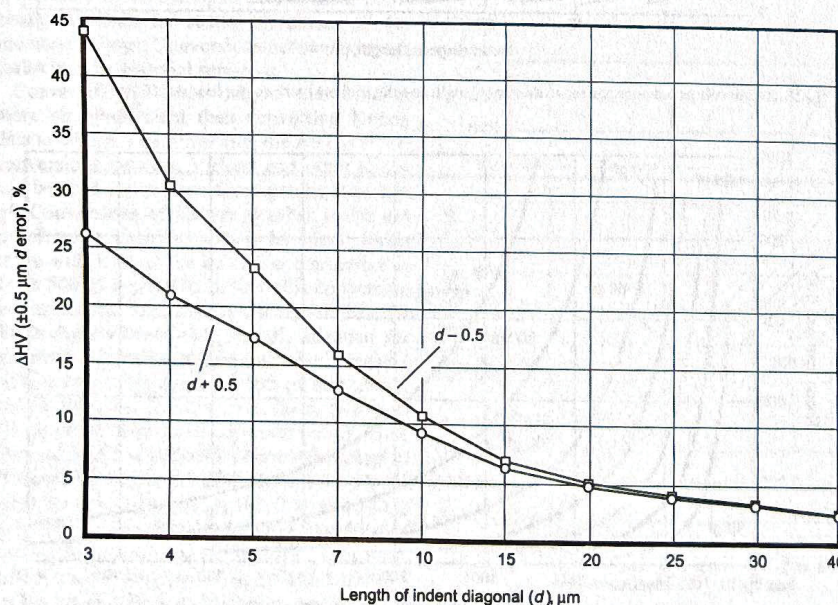


Fig. 11 Influence of a measurement error of $\pm 0.5 \mu\text{m}$ on the calculated Vickers hardness as a function of diagonal length

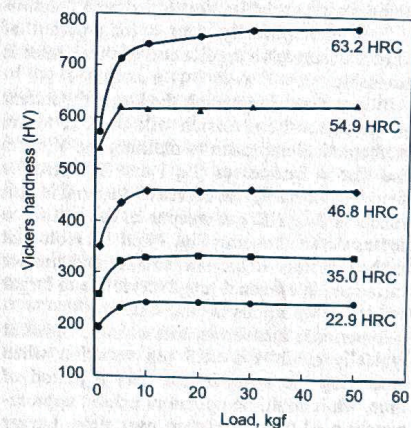


Fig. 12 Measured Vickers macrohardness for five steel test blocks using test forces from 1–50 kgf. Source: Ref 3

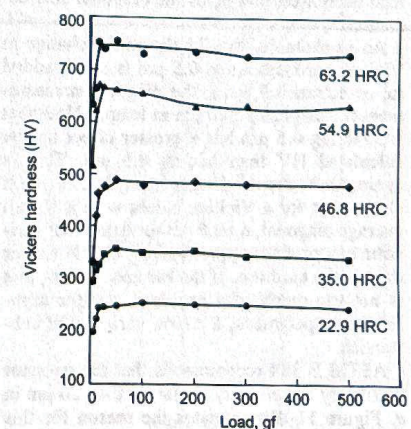


Fig. 13 Measured Vickers microindentation hardness for five steel test blocks using test forces from 5–500 gf. Source: Ref 3

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 under-size 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.

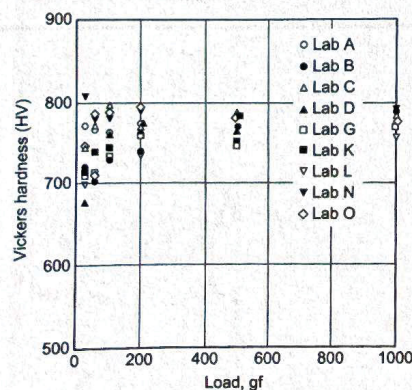


Fig. 14 ASTM E-4 round-robin interlaboratory Vickers microindentation hardness-testing data for the hardest (F1) test specimen and nine people (measuring the same indents) who produced "good" data for test loads from 25–1000 gf. Source: Ref 6, 7

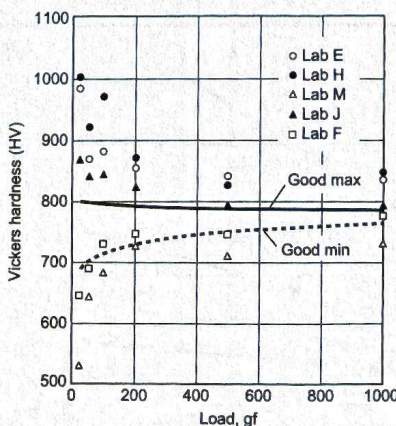


Fig. 15 Data shown in Fig. 14 (all points fall within the two lines) plus the individual data from four "outlier" raters. Source: Ref 6, 7

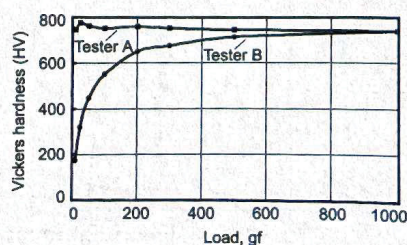


Fig. 16 Curves showing load versus Vickers hardness for two testers (with the same operator) evaluating the hardness of the same type 440C martensitic stainless steel specimen (62.7 HRC)