Low-Load Vickers Microindentation

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Different trends observed for low-load Vickers hardness are due to visual perception problems of operators of where indent tips are, not a material problem.

In 1925 in the UK, Smith and Sandland developed an indentation test that used a square-based pyramidal-shaped indenter made of diamond. The test was developed because the Brinell test (introduced in 1900), which (until recently) used a round hardened steel ball indenter, could not test steels harder than ~450 HB (~48 HRC). They chose this shape with an angle of 136° between opposite faces to obtain hardness numbers that would be as close as possible to Brinell hardness numbers for the same specimens over the usable Brinell range. This made the Vickers test easy to adopt, and it rapidly gained acceptance. The Vickers test has the great advantage of one hardness scale being used to test all materials, unlike the 30 different Rockwell test scales, each yielding numbers of hardness numbers for the same specimens over the usable Brinell range.

In the Vickers test, the force is applied smoothly, without impact, and held in contact for 10 to 15 seconds. The force must be controlled precisely. After removing the force, both diagonals are measured and the average is used to calculate the HV according to (for loads ≤1 kgf):

\[ HV = 2000L \sin \left( \frac{a}{2} \right) / d^2 = 1854.4L / d^2 \]  

(1)

Where \( d \) is the mean diagonal length in \( \mu \)m, \( L \) is the load in gf, and \( a \) is the face angle (136°).

The original Vickers test operated over a range of applied forces from 1 to 120 kgf; many testers today cover a range of 1 to 50 kgf, which is adequate. The use of forces below 1 kgf with the Vickers test was first evaluated in 1932 at the National Physical Laboratory in the UK. Four years later, Lips and Sack constructed the first Vickers tester designed for applied forces ≤1 kgf.

Why macro- and micro-Vickers tests?

Historically, Vickers hardness testing was separated into two categories, macro and micro. This distinction was made because the nature of the testing and the goals are different. The Brinell test has been widely used since 1900 to evaluate the hardness of as-cast, as-rolled, and annealed metals. In these conditions, the alloys are not homogeneous and the large indent size made by the 10-mm diameter ball averaged out the inhomogeneities to obtain a bulk hardness value. The macro Vickers test creates a smaller impression, so it is not as good at averaging inhomogeneities in metals as the Brinell test. However, macro Vickers tests on such specimens reveal more consistent hardness numbers as the test load is increased and the indent becomes larger. Macro Vickers is very good for testing the hardness of cold-rolled metals and heat treated steels. Micro Vickers tests, by comparison, are used at low loads to make small indents so hardness variations, intentional or unintentional, can be assessed. Micro Vickers tests are very good to characterize segregation and banding, to identify constituents, and to characterize surface hardness/microstructure gradients. Thus, macro tests yield a gross product average, while micro tests indicate variations in hardness. Another difference is that micro Vickers testing is generally performed in the laboratory, while macro Vickers tests (as with Brinell and Rockwell tests) are usually performed on the shop floor to obtain bulk average hardness—usually by different people.

Precision of Vickers hardness

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. However, numerous studies of microindentation hardness test results conducted over a wide range of test loads show that test results usually differ at loads ≤100 gf. 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 of macro Vickers testers, and an ASTM interlaboratory “round robin” of indents on seven test specimens made by one person, where the indents in each specimen were measured by 12 or more different people, reported three or four different ISE trends for the same indents in each specimen.

Top-referenced mount holder used to ensure that the plane-of-polish is perpendicular to the indenter for hardness testing.

Many factors can influence the quality of
Hardness Testing

Microindentation test results. In the early days of low-load (<100 gf) hardness testing, it was quickly recognized that improper specimen preparation influences hardness test results. Assuming the specimen is properly prepared, the greatest source of error is in measuring the indent as documented in the ASTM interlaboratory test. Place the indent in the center of the measuring field (if not already there), as lens image quality is best in the center. The light source should provide adequate even illumination for maximum contrast and resolution. The accuracy of the filar micrometer or other measuring device should be verified using a stage micrometer.

Specimen preparation for microindentation hardness testing is not a trivial matter, and becomes more critical as the applied force decreases. Specimen preparation for macro Vickers testing is less critical as the load increases. Furthermore, 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 (e.g., 300-1000 gf), a perfectly prepared specimen is not required. However, sectioning and grinding damage still must be removed. The normal preparation procedure could be stopped after grinding and polishing down to a 6-, 3-, or 1-µm diamond finish, depending upon the load used. For lower loads, it is advisable to prepare the specimen to remove all damage. Excessive residual damage from sectioning and grinding influences test results, producing 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 correct hardness. Guidelines for preparing metallographic test specimens are given in ASTM Standard E3, Volume 9 of the ASM Handbook, and in standard text books.

Most systems use an automated test cycle of loading, application for the desired 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, which will enlarge the indent size.

The indent 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. 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 perpendicular to the indenter. Historically, it has been 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.

Because the diagonals must be measured after the force is removed, the tester is equipped with two (or more) metallographic objectives (i.e., reflected light), usually 10× and 40×. Some systems may have a third or fourth objective on the turret. A higher power objective (60×, 80×, or 100×) should be used to measure small indents (<20 µm in diagonal length). Objectives should have a reasonably high numerical aperture for its magnification to give best resolution. Brown and Ineson showed that measurement varied more from the true value as the NA of the objective decreases. A 10× objective is usually used as a spotter; i.e., simply to find the desired test location, except for macro Vickers testers. The measuring eyepiece is usually 10×. The optical system must be carefully calibrated using a stage micrometer. In general, indents can be measured to a precision of ±0.5 µm in length. A proper Köhler illumination system is necessary to fully illuminate the specimen. Unfortunately, the optical systems on many microhardness testers are not as good as on a high-quality metallograph.

Calculation of 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. The micrometer lines have a finite thickness. Several approaches can be used to zero the system before measurement. A good 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 the indent tips just touch each line.

Bückle suggested that the metal flow at the edge of the indentation is a major source of measurement errors, particularly at low loads. He noted that ridge height can be nearly 1% of the diagonal width. This produces problems with focusing the image, which leads to errors in measurement of the indent size, either positive or negative. The magnitude of the error increases with decreasing diagonal length, and is large enough to account for hardness deviations at low loads. A positive d error lowers the hardness, while a negative d error raises the hardness.

In general, the larger the indent, the better is precision. Due to the mathematical approach to defining the Vickers hardness (Eq. 1), where the denominator is d², the curves of diagonal length versus HV get steeper as test force decreases (Fig. 1). Note that as the test force decreases, the
same small variation in diagonal length correlates to a larger variation in hardness.

Experience shows that an operator typically exhibits a ±0.5 μm variation in measuring the same indent over a period of time, while multiple operators exhibit about a ±1.0 μm variation over time. Larger variations have also been observed[2]. A ±0.5 μm variation in the measured diagonal has a greater influence on hardness as the test load decreases; i.e., as the diagonal size decreases. As an example, Fig. 2 shows the change in Vickers hardness when 0.5 μm is either added to, or subtracted from, the diagonal measurement for diagonals ≤40 μm. Subtracting 0.5 μm has a greater effect on the calculated HV than adding 0.5 μm, 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 about a 10% rise or drop in hardness. If hardness is low, this is not too much of a problem, but for high hardness specimens, a ±10% variation is substantial. ASTM E384 recommends that, when possible, the operator should try to keep indents larger than 20 μm in $d$.

**Load-hardness relationships**

For the Vickers test, especially in the macro applied force range, it is commonly believed that the hardness is constant as the load is changed. However, for microindentation tests, many publications show that Vickers hardness is not constant over the entire test force range. For Vickers tests with an applied force of 100 to 1000 gf, measured hardness values are usually equivalent within statistical precision. The Vickers indent produces a geometrically similar indent shape at all loads, and a log-log plot of load versus diagonal length should exhibit a constant slope, $n$, of 2 for the full range of applied force (Kick’s Law). But, this law appears to be in disagreement for forces under 100 gf.

The literature (more than 60 publications) shows four trends for load-hardness Vickers data at low loads:

- HV increases as gf decreases ($n<2.0$)
- HV decreases as gf decreases ($n>2.0$)
- HV is essentially constant as gf varies ($n = 2.0$)
- HV increases, then decreases with decreasing gf

Trends 1, 2, and 4 are more easily detected in hard specimens than soft specimens, where trend 3 is observed. Publications reporting trends 1, 2, and 4 attribute the trends to material characteristics. In a 1984 ASTM symposium, Len Samuels stated that these problems were not due to the materials, but to difficulties in visual perception of indent tips as indents became smaller[7]. His conclusion was borne out by the ASTM round robin[3,4].

It has been widely claimed in the literature that Vickers hardness is constant with test forces in the macro force range (≥1 kgf). However, a literature search for data to prove this point yielded no evidence to support this claim. Consequently, macro Vickers tests were performed with measurements made on five polished HRC test blocks (hardness ranging from 22.9 to 63.2 HRC) using six test forces from 1 to 50 kgf[2]. At each force, six impressions were made with the mean results plotted in Fig. 3. The filar micrometer used a magnification of 100×. HV is essentially constant for forces of 10 kgf and greater. For each test block, hardness decreased for test forces ≤10 kgf. The degree of decrease increased with increasing hardness. Thus, for this macro Vickers tester, HV was not constant, but exhibited trend No. 2 above, the most commonly observed trend for HV-micro low-range force studies.

The same steel test blocks were subjected to Vickers tests using nine different forces from 5 to 500 gf, with a microhardness tester. Six impressions were made at each test force, and the mean values are plotted in Fig. 4. Impressions were measured at 500×, and basic trend No. 2 was observed. In most cases, HV is essentially constant at forces down to 100 gf, after which hardness decreases. The magnitude of the decrease increased with increasing specimen hardness. For several curves, hardness appears to rise slightly as the force drops below 100 gf, and then decreased (trend No. 4). Trends 2 and 4 were obtained for the micro HV versus test force work.

The above results using the same set of five specimens with a wide range of hardness 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). This is due to visual perception problems in sizing small indents at
the tester magnifications used (100× for the macro and 500× for the micro system). Data in Figs. 3 and 4 were developed for the same set of specimens by the same metallographer using two different testers, with different measuring systems (100× versus 500×). Tests covered a load range of 5 gf to 50 kgf. This poses the question: “If indentation size effect is a material characteristic, why is it observed at the low-load range of both testers?” These results indicate that the ISE is due to the difficulty of measuring small indents relative to the magnification of the viewing system of the tester.

ASTM Committee E-4 on Metallography conducted an interlaboratory round robin where one person made five indents at each test load from 25 to 1000 gf using both Vickers and Knoop indenters, on three ferrous and four nonferrous specimens5,4. Then, fifteen people measured the indents on the ferrous specimens, while another similar size group measured the indents on nonferrous specimens; two people measured both sets. As the ferrous specimens covered a higher hardness range, these test results are of greater interest to this problem.

For the ferrous specimens, most of the raters’ measurements followed the most common trend; i.e., relatively constant HV values at loads above 50-100 gf with decreasing HV for the lower loads. The drop-off in hardness increased with increasing specimen hardness. Results for the hardest test specimen are shown in Figs. 5 and 6. Examination of the data in Fig. 5 reveals that most of the “good” labs (identified by the statistical approach in ASTM E 691) had trends identical to those observed in Figs. 3 and 4. However, Lab A had almost constant data over the load range, while Lab N obtained higher values at the lowest loads, and Lab C had an increase in hardness followed by a decrease at the lowest loads. Thus, all four load-hardness trends were observed for the same indents. However, several other raters obtained different trends for the same indents (Fig. 6); the blue line is the average for the 9 good Labs shown individually in Fig. 5. For these “outlier” labs (as defined by E 691), Lab M had a consistent bias at all test loads with significantly lower hardness at all loads, especially low loads. Labs E and H also had a consistent bias, obtaining higher hardness values at all loads. But, at low loads, Labs E and H showed substantially higher hardness values with decreasing loads. Lab J had reasonably good results at 500 and 1000 gf loads, but hardness increased slightly at lower loads.

These results and others demonstrate that the indentation size effect is not a characteristic of the material, but is due to visual perception problems associated with sizing small indents. In addition, it is exaggerated because small measurement variations for small d values (Fig. 1) creates a major influence on the calculated hardness5,4.

**Can the load-hardness problem be eliminated?**

Because this is a visual perception problem related to the ability to correctly measure indents, the authors performed micro Vickers measurements on high hardness steel test blocks using the DuraScan 70 microindentation hardness tester (Fig. 7). An optional six-position nosepiece...
turret is available that can accommodate two indenters and four objectives (Fig. 8). Rockwell C certified test blocks were used for the study after polishing them to remove any surface damage from preparation. Complete removal of preparation-induced damage is necessary when performing tests at loads under 100 gf. Data are shown in Figs. 9 to 12 for test blocks with hardness values of 64.2, 61.3, 44.7, and 32.5 HRC. Each data point is the average of six impressions at that load. For the three hardest blocks, Vickers values are statistically identical down to 25 gf, but slightly lower values were obtained at a 10 gf load. Results for the hardest block (64.2 HRC) are actually excellent. For the 32.5 HRC test block, all HV values including 10 gf values are statistically identical.

Experiments measuring the smallest (10 gf) indents using 100× (brightfield and darkfield) and 150× objectives (with high NA values of 0.90 and 0.95) reveal that indents can be focused, and they may remain in focus as fine focus control is moved slightly, but the indent size appears to change. If the smallest appearing indent size is arbitrarily selected, the 10 gf data would look very good and would be consistent with the values at higher loads. But, the selection is biased by knowing the correct answer, which is unacceptable. The values plotted represent indents in focus at what appears to be their largest size. These indents are in the 4–5 μm size range for the 64.2 and 61.3 HRC test blocks. Tests of a 60.7 HRC test block yielded results similar to the 61.3 HRC block in this regard. Figure 2 shows how a minor variation in diagonal size (±0.5 μm) for 4 or 5-μm diagonal indents (the two indent values at the left side of the graph are for 4 and 5 μm diagonal size Vickers indents) impart a huge variation in the measured hardness. This is a fundamental restriction on Vickers hardness precision for small indents using light optical microscope (LOM) measurements. A different measurement technology is required for small indents to obtain greater precision in measuring them and reduce the possible calculated hardness variation. The scanning electron microscope offers the potential for measurements with greater precision and at higher magnifications than possible with the LOM. However, obtaining such precision with the SEM is not simple because the specimen cannot be tilted in the SEM chamber to enhance image contrast. Magnification must be known to a very high precision, and this must be maintained as the working distance is changed.

Conclusions
The ASTM interlaboratory round robin proves that different trends observed for Vickers hardness as a function of load (particularly for ≤100 gf loads) are due to visual perception problems of operators deciding where the indent tips are. The problem is not a material problem as widely claimed in the literature for many years. Tests using a DuraScan 70 show that when the proper magnification using high-quality objectives with high numerical aper-
tures and a good illumination system is selected, HV values are statistically identical down to a 25 gf load for the highest hardness steels, while they are consistent to a 10 gf load for steel at 32.5 HRC. For very small diagonal Vickers indents of \( \leq 5 \mu m \), the LOM is inadequate for such measurements as the repeatability for most operators in measuring diagonals is \( \pm 0.5 \mu m \). Due to the nature of the mathematical definition of Vickers hardness (where the divisor is \( d^2 \)), the effect of a \( \pm 0.5 \mu m \) variation in \( d \) (Fig. 2) on the calculated HV value is excessive. For these small Vickers indents, an alternate observation/measurement technology is required with greater precision to reduce the potential data scatter.

**References**


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![Fig. 12 — Test results for the 32.5 HRC test block are statistically constant from 10 gf to 10 kgf.](image-url)