# Keysight Technologies
# The Revolutionary Impact of the Oliver and Pharr Technique on the Science of Hardness Testing
## Application Brief


## Abstract

In 1992, Warren Oliver and George Pharr published an article in the Journal of Materials Research that revolutionized hardness testing [1]. According to Thomas Reuters Web of Knowledge, this article has been cited more than 8,500 times, making it one of the most cited works in all of material science. The genius of Oliver and Pharr was this: they devised a way to know the size of a hardness indentation without imaging it. This development disrupted Vickers and Knoop microhardness testing which required direct measurement of the lengths of the indentation diagonals [2]. Not having to image the indentation paved the way for fully automated hardness testing. Not only was automated testing independent of human bias, it was also much faster, because multiple tests on multiple samples could be prescribed and executed with no human intervention. Further, the Oliver-Pharr method extended hardness testing to much smaller scales, because one could determine the size of even sub-micron indentations with incredible accuracy. This note explains the theory of the Oliver-Pharr method and shows how it can be used to obtain an equivalent Vickers hardness number (HVc).


## Background on Vickers Hardness

Prior to the advent of instrumented indentation, the Vickers hardness test was the state-of-the-art in microhardness testing. The Vickers hardness test is still used today and is governed by two standards: ASTM E384 and ISO 6507. The test involves pressing a Vickers indenter (an oblique, four-sided pyramid) into a surface to a specific force, P, and holding that force for 10 seconds. Once the indentation process is complete, the lengths of the diagonals of the indentation are measured optically in order to gage the size of the impression. Vickers hardness (HV) is defined as the applied force, P, divided by the surface area of the indentation, As:

$$HV = P/A_S. \qquad \text{Eq. 1a}$$

In terms of the average of the diagonal lengths, this works out to

$$HV = 1854.4 \cdot P/d^2 . \qquad \text{Eq. 1b}$$

where P is the force in units of gf and d is the average of the two diagonal measurements in micrometers. Although it is rarely stated explicitly[1], the units of HV as calculated above are kgf/mm$^2$.

On the lower end, the force-range of the Vickers hardness test is practically limited by one's ability to measure the diagonals of the indentation. ASTM E384-11 recommends that the test not be used unless it produces an indentation having diagonals of 17 µm or greater. If the indented material is actually a coating, this same standard recommends that the indentation depth be less than 10% of the coating thickness. Taken together, these two requirements imply that the Vickers hardness test should not be used at all for coatings which are less than about 60 µm thick.

## The Origins of Instrumented Indentation

In 1983, Warren Oliver and John Pethica founded a company, Nano Instruments, Inc., to commercially produce indentation systems which measured both force and displacement for the entire time that the indenter was in contact with the material. They were motivated by new possibilities in micro-mechanical testing, including novel research which showed that the rebound of the material upon force removal was directly related to the Young's modulus of the material [3]. This rebound could only be sensed if force and displacement were measured continuously throughout the indentation process.

1. This is often a point of confusion, because ASTM E384-11 states that Eq. 1b gives *HV* "in terms of" gf per µm$^2$. But in fact, the units of *HV* are kgf/mm$^2$, because the constant in Eq. 1b includes a conversion in force (from gf to kgf) and a conversion in area (from µm$^2$ to mm$^2$). Expressed with its units, the constant multiplier is actually 1854.4 [(kgf-µm$^2$)/(gf-mm$^2$)].

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In the context of this developing technology, Warren Oliver and George Pharr (both graduates of the doctoral program at Stanford University under William Nix) latched on to another idea: perhaps the size of the indentation could be inferred from the sensed displacement, thus eliminating the need to image the indentation after the fact. The notion that indentation area would be related to the indentation depth was intuitive, but several obstacles had to be overcome. First, one had to know the depth over which the indenter actually made contact with the material, hc. This contact depth could not be taken as identical to the displacement sensed during indentation, h, because during indentation, the material around the contact tended to deflect elastically as shown in Figure 1, making hc less than h. Further, they had to face the possibility that the small flaw at the tip of the indenter (Figure 2), though irrelevant for large indentations, might be significant for the microscopic indentations which they hoped to make. Finally, they wondered whether the final indentation shape would be close enough to the conforming shape under load, or whether the final shape would be smaller due to elastic recovery.

## Oliver-Pharr Method for Determining Contact Area and Hardness

The first problem—that of knowing the contact depth, hc—turned out to be the most challenging. Oliver and Pharr were aware of the work of a Scottish mathematician, Ian Sneddon, who had published a comprehensive theory of elastic contacts. Sneddon had predicted the relationship between contact depth, hc, and total displacement, h, but only for contacts which were completely elastic. Oliver and Pharr knew that Sneddon's analysis couldn't be used to predict deformation under the indenter, due to plasticity, but they suspected that Sneddon's analysis might be used to predict the downward deflection of the surface outside the contact area, thus providing a link between hc and h. The expression for the downward elastic deflection of the surface outside the contact area which Oliver and Pharr derived from Sneddon's analysis was:

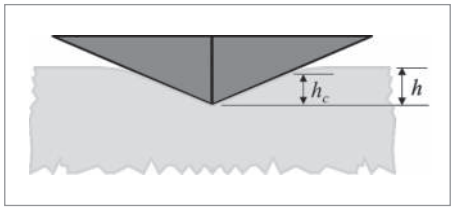


Figure 1. Schematic of Vickers indentation showing downward elastic deflection of surface outside the contact area.

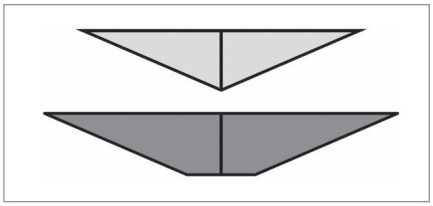

Figure 2. Schematic of an ideal Vickers pyramid (top) and a real Vickers pyramid. The apical flaw causes the indentation area to be larger as a function of indentation depth. ASTM E384 limits the length of the apical flaw to 0.5 µm.

$$h_s = 3P/(4S), \qquad \text{Eq. 2}$$

where P is the applied force, and S is the elastic recovery of the material as the indenter is retracted from the sample. Thus, the contact depth could be calculated as:

$$h_c = h - 3P/(4S) \qquad \text{Eq. 3}$$

Oliver and Pharr intended to use their new method to make very small indentations—so small, in fact, that the flaw at the apex of the diamond pyramid might come into play. Thus, they developed a procedure for "calibrating" the shape of each individual pyramid which accounted for deviations from perfection, especially at the apex. They proposed that the relationship between the projected area, A, and the contact depth, hc should have the general form2

$$A = C_1 h_c^2 + C_2 h_c, \qquad \text{Eq. 4}$$

and they determined the values of the two constants, C1 and C2, by indenting a material of known properties. The first constant, C1, should have a value close to that for an ideal pyramid (24.5 for a Vickers). The second constant, C2, indicates the magnitude of the apical flaw—larger values of C2 indicate a larger apical flaw.

Oliver and Pharr were pleasantly surprised to find that the last issue was not a problem for most materials, so long as the indenter was a pyramid with sharp edges. They compared the contact area under load, which they calculated by Eqs. 3 and 4, with the area of the residual impression, as imaged through a scanning electron microscope. They found that the calculated area was virtually identical to the imaged area for all the conditions and materials which they examined (aluminum, titanium, sapphire, glass, and quartz). They explained that the sharp edges of the pyramid served to permanently mark the corners of the indentation at the peak load, so that in fact, the final indentation was a permanent record of the size of the contact at the peak force.

With this new way of getting contact area, Oliver and Pharr defined hardness as

$$H = P/A, \qquad \text{Eq. 5}$$

where A was the projected indentation area. Although Vickers hardness had always been defined as force divided by surface indentation area (HV = P/As), Oliver and Pharr were informed by contemporary research which showed better agreement among hardness values measured with different indenter shapes, so long as the hardness was defined as the mean indentation stress as in Eq. 5.

## Calculating Vickers Hardness from Instrumented Hardness

There exists a simple calculation for converting between the instrumented hardness, as defined by Eq. 5, and Vickers hardness as defined by Eq. 1. First, the instrumented hardness (H) must be expressed in units of [kgf/mm$^2$] in order to be consistent with the common units of Vickers hardness. Second, the instrumented hardness is multiplied by a constant (0.927) in order to convert from using A to

2. In their 1992 publication, Oliver and Pharr used a more complex form with more terms, but improvements in indenter manufacturing allow this simpler and more intuitive form.

| | | Instrumented indentation | | | | | | ASTM E384 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $P$ | $h$ | $S$ | $h_c$ | $A$ | $H$ | $HV_c$ | $d_1$ | $d_2$ | $d$ | $HV$ | $Diff$ |
| Test | gf | µm | gf/µm | µm | µm² | kgf/mm² | kgf/mm² | µm | µm | µm | kgf/mm² | % |
| 1 | 5.11 | 0.741 | 84.064 | 0.696 | 11.159 | 458.3 | 424.8 | 4.80 | 4.68 | 4.74 | 421.8 | 0.72 |
| 2 | 5.11 | 0.782 | 94.825 | 0.742 | 12.646 | 404.3 | 374.7 | 4.93 | 5.10 | 5.02 | 376.8 | -0.55 |
| 3 | 5.11 | 0.744 | 82.603 | 0.698 | 11.219 | 455.8 | 422.5 | 4.76 | 4.78 | 4.77 | 416.5 | 1.45 |
| 4 | 5.11 | 0.765 | 94.935 | 0.724 | 12.065 | 423.8 | 392.8 | 4.87 | 4.87 | 4.87 | 399.5 | -1.69 |
| 5 | 5.11 | 0.750 | 88.885 | 0.707 | 11.523 | 443.8 | 411.3 | 4.75 | 4.87 | 4.81 | 409.6 | 0.42 |

Table 1. Summary of results.

As. Thus, to obtain Vickers hardness from instrumented hardness, the calculation is

$$HV_c\ [\text{kgf/mm}^2] = 0.927 * H\ [\text{kg/mm}^2], \quad \text{Eq. 6}$$

where the subscript "c" indicates that the value has not been determined using direct measurement of diagonal lengths, but is a "converted" value.

## Experimental Method

In this work, we demonstrate the practical equivalence of direct and converted Vickers hardness by testing a sample of 2205 duplex stainless steel. Duplex stainless steels have a dual-phase microstructure comprising both austenite and ferrite grains. This microstructure provides an advantageous combination of mechanical properties. By volume, the 2205 duplex alloy comprises a roughly 50-50 combination of austenite and ferrite grains [4, 5].

A one-inch (diameter) bar of 2205 duplex stainless steel was purchased from McMaster-Carr (Elmhurst, IL; Part No: 9079K136). The sample was prepared for nanoindentation by Element Materials Technology (Wixom, MI). First, a section was cut from the bar, milled to 0.25 inches, and rough ground with water using silicon carbide starting at 220 grit through 600 grit (US). The samples were polished using 6µm and 1µm diamond on a medium nap cloth with an alcohol-based extender. Final polishing was done using 0.05µm colloidal silica on a low nap cloth, and polished in a vibratory polisher with 0.06µm colloidal silica and a medium nap cloth. After polishing, the sample was mounted for testing using Crystalbond™.

All testing was performed with a Keysight G200 NanoIndenter, configured with the NanoVision option which creates AFM-like surface images. A Vickers indenter was installed in the instrument. This indenter had been calibrated previously to obtain its particular form of Eq. 4:

$$A\ [\mu\text{m}^2] = 21.8945 h_c^2 + 0.8056 h_c,$$

where hc is in units of µm. In accordance with ASTM E384, a test force of 50mN (5.11gf) was applied over 10 seconds and held for 10 seconds. The test force was also removed over 10 seconds. Five different indentations at five different sites were performed in this way.

The resulting indentations had diagonals which were about 5µm in length; thus, they were too small to be measured optically. So in order to accurately measure diagonal lengths, each indentation was subsequently scanned using the NanoVision option. Each scan was positioned to include the indentation, and covered a domain of 10µm x 10µm. Within this domain, 100 scans were performed, with 250 points recorded along each scan.
The slow-scan length divided by the number of scans dictated the image resolution, which was 0.1µm. Each
scan was exported to Gwyddion 2.31 in order to measure diagonal lengths. (Gwyddion is a freely distributed, open-source software package that facilitates analysis of scanning-probe microscopy images.)

Each of the five indentations yielded both a direct and a converted measure of Vickers hardness. To obtain the direct Vickers hardness, HV, the lengths of the indentation diagonals were measured on the scanned image within Gwyddion; the two lengths were averaged and HV was computed by Eq. 1. To obtain the converted Vickers hardness, HVc, an indentation hardness, H, was first computed by the method of Oliver and Pharr (Eqs. 2–5), and then HVc was calculated by Eq. 6.

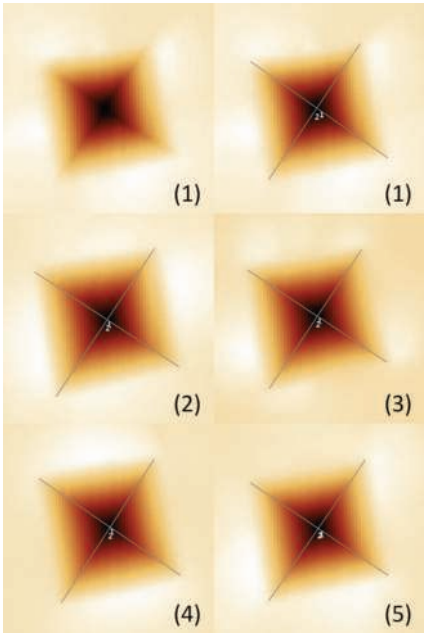


Figure 3. Vickers indentations on 2205 duplex stainless steel (5.11gf). Number on the image identifies the test number. For the first test, indentation is also shown without measured diagonal lengths (top left).

## Results and Discussion

Results for the five indentations are provided in Table 1. The most important finding is that the converted Vickers hardness (HVc) matches the direct Vickers hardness (HV) to within the uncertainty in HV. The relative uncertainty in HV due to the uncertainty in diagonal lengths is

$$\delta HV/HV = 2(\delta d)/d \approx 2(0.1\mu\text{m})/5\mu\text{m} \approx 4\%.$$

Figure 3 shows the good quality of the indentations, even though they are less than one micron deep. The indentations are well

defined and square. For all indentations, the two diagonal lengths matched to within the 5% limit prescribed by ASTM E384.

Strictly speaking, indents at this scale cannot be done in accordance with ASTM E384, because this standard only provides for optical imaging, and these indents were too small to be seen clearly with the best optical magnification. NanoVision scans produce dimensionally accurate images of the indentations, but they are time consuming. In this work, each scan required four minutes, and further analysis to get diagonal lengths took even longer. Thus, even if alternate imaging techniques were allowed, ASTM E384 becomes more and more onerous as indentations become smaller and smaller.

For small indentations, instrumented indentation provides an accurate, fast, and easy way to get Vickers hardness.
The total time required for each indentation is about 2 minutes, and this includes the time for moving the sample from test site to test site. All prescribed tests run unattended and the value of HVc is reported immediately after the completion of each test. In fact, multiple tests can be prescribed on many samples, and testing can run overnight!

There is nothing about the determination of HVc which inherently limits the method to small test forces. However, the advantages of determining HVc with an instrumented indenter become clearer for smaller test forces. Keysight's G200 NanoIndenter can be used to determine HVc accurately for test forces between 1 gf and 50 gf. Smaller forces are not recommended, because the apical flaw of the Vickers pyramid may dominate the geometry of the indentation.

## Conclusions

In 1992, Warren Oliver and George Pharr revolutionized microhardness testing by developing a way to gage the size of the indentation without imaging it. On a well polished sample of 2205 duplex stainless steel, we showed that the Vickers hardness obtained by the method of Oliver and Pharr (HVc) matched the Vickers hardness measured according to ASTM 384 (HV) to within the uncertainty in HV. The practical advantages of determining HVc by means of instrumented indentation are myriad. Instrumented indentation affords increased accuracy for small forces and thus can be used to characterize the Vickers hardness of thin coatings. Further, instrumented indentation is fast, simple, fully automated, and independent of human bias.

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