



Comparison of nano-indentation hardness to microhardness

Linmao Qian^a, Ming Li^b, Zhongrong Zhou^{a,*}, Hui Yang^a, Xinyu Shi^a

^aTribology Research Institute, National Traction Power Laboratory, Southwest Jiaotong University, Chengdu 610031, Sichuan Province, PR China

^bState Key Laboratory of Tribology, Tsinghua University, Beijing 100084, PR China

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Abstract

With a nano-indenter and a microhardness testing machine, nano-indentation hardness and microhardness are measured in a wide load range (0.1–19600 mN) for five materials. Even fused silica and silicon almost have constant hardness during the load range, the nano-indentation hardness of copper, stainless steel and nickel titanium alloy shows obvious indentation size effect, namely that the hardness decreases with the increase of depth. For the measured materials, the nano-indentation hardness is about 10–30% in magnitude larger than the microhardness. The main reasons can be explained as the analysis of the nano-indentation hardness using the projected contact area at peak load A_c instead of the residual projected area A_r , as well as the purely elastic contact assumption describing the elastic/plastic indentation process. The analysis based on a simple model indicates that A_c is always smaller than A_r , and the more heavily the indent piles up (or sinks in), the larger the difference between the nano-indentation hardness and microhardness.

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1. Introduction

As an indicator of the material's ability against deformation, hardness has been studied for more than 100 years [1]. Usually, a prescribed load is applied to an indenter in contact with a specimen and the area of the residual impression A_r is measured often by optical imaging after the load is removed, the microhardness is then obtained through dividing the load by A_r [1]. During the last 20 years, researchers have become more and more interested in the mechanical properties of ever smaller volumes, such as thin films and microelectromechanical systems (MEMS) [2–5]. The reason is not only attributed to that the reliability of MEMS is determined by their mechanical property, but also that the mechanical properties at micro-scale may differ from the bulk properties due to the size effect and surface effect [6–8]. As a guide of the material's ability against micro-deformation and of the thin film's ability against wear, the nano-indentation hardness has been widely studied.

For the indents at nano-scale, the remaining area is difficult to be measured with the traditional optical microscopy because of a too low resolution. Consequently, different from the microhardness, the determination of the nano-indentation hardness usually uses the depth-sensing indentation technique which obviates the need for imaging the residual indent. In a depth-sensing indentation hardness test, a diamond tip is pressed into specimen till a given maximum depth or load and then removed, synchronously the load on, as well as the displacement of, the indenter are recorded. From such a load–depth curve, hardness and Young's modulus of the specimen can be analyzed using the Oliver and Pharr method [9]. The method starts from the fit of the unloading curve to the power-law relation:

$$P = C(h - h_f)^m \quad (1)$$

where P is the indentation load, C and m are the fitting parameters, h is the depth and h_f is the residual depth. The unloading stiffness S is then obtained by

$$S = mC(h_{\max} - h_f)^{m-1} \quad (2)$$

* Corresponding author. Fax: +86 28 87600971.

E-mail address: zzzhou@home.swjtu.edu.cn (Z. Zhou).

The contact depth h_c between the indenter and the specimen can be estimated using:

$$h_c = h_{\max} - \varepsilon \frac{P_{\max}}{S} \quad (3)$$

Here, $P_{\max}$ is the peak load and ε is a constant related to the indenter. Thus, the projected contact area A_c is established by evaluating an empirically determined indenter shape function at the contact depth. Finally, the nano-indentation hardness H_{O-P} and effective elastic modulus E_{eff} are calculated by

$$H_{O-P} = \frac{P_{\max}}{A_c} \quad (4)$$

$$E_{\text{eff}} = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} \quad (5)$$

E_{eff} has the following relationship with the specimen modulus E

$$\frac{1}{E_{\text{eff}}} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \quad (6)$$

where ν is Poisson's ratio for the specimen, E_i and ν_i are the elastic modulus and Poisson's ratio for the indenter, respectively.

Obviously, the advantage of this method is that it is not needed to image the residual indent for the evaluation of hardness, which facilitates property measurement from very small indentations. However, the measurement accuracy of H_{O-P} and E is mainly depended on how well the projected contact area A_c replaces the indent residual projected area A_r , as well as how well Eqs. (2)–(5) describe the indentation deformation behavior. The distinct definitions of A_c and A_r may induce their different values. Since Eqs. (2)–(5) were derived from a purely elastic contact solution developed by Sneddon [10], how well they work for elastic/plastic indentation is not entirely clear. Using the finite element (FE) method, Bolshakov and Pharr [11] found when pile-up is significant the area deduced from the Oliver–Pharr analysis could underestimate the true contact area by as much as 60%, which through Eqs. (4) and (5) leads to large errors in the hardness and modulus. More recently, with dimensional and FE analysis, Cheng and Cheng [12,13] reported that the Oliver–Pharr procedure should be used with caution for materials with a wide range of the ratio of initial yield strength Y to Young's modulus E (Y/E : 10^{-4} – 10^{-2}).

However, except the FE analysis [11–13], few studies were found on the experimental comparison of nano-indentation hardness and microhardness. Using a nano-indenter and a microhardness testing machine, we performed the indentation hardness measurements in a wide load range (0.1–19600 mN) for five materials. The

limitation of the nano-indentation hardness was studied and discussed by the comparison to the microhardness.

2. Materials and methods

The materials were chosen to span a wide range of hardness and modulus, including single crystal copper, stainless steel 304, single crystal silicon (110), fused silica, and superelastic nickel titanium (NiTi) shape memory alloy (Shape Memory Applications, San Jose, CA, USA). Single crystal silicon and fused silica are optically flat and measured as received. The nominal NiTi alloy composition is Ni-51.33 at.% Ti-48.67 at.%. With a differential scanning calorimeter (DSC 92, SETARAM, France), the characteristic transformation temperatures indicate that the material is in austenite phase at room temperature ($\sim 22^\circ\text{C}$) and exhibits superelasticity under stress. To prepare the samples for indentation tests, copper, stainless steel and NiTi were polished with a series of aluminum oxide sand papers (Nominal grain diameter: 14, 8, 5, 1, 0.3 and 0.05 μm) to optical flatness.

Nano-indentation hardness was measured on a nano-indenter (CSEM, Instruments, Switzerland) by a Berkovich type diamond tip with a face angle of 142.3° . Before the experiments, the indenter shape was calibrated by a standard indentation procedure in fused silica between 0.1 and 100 mN. Thirty-two different loads were chosen between 0.1 and 200 mN and 20 measurements were repeated at each load. At each test, the loading speed was adjusted to keep 30-s loading time, 2 s delay at peak load and 30 s unloading time. The microhardness was obtained through dividing the load by the area of the residual indents, where the remaining indents were imaged by a laser confocal scanning microscopy (Olympus-OLS-1100, Japan). For the values of load below 200 mN, the microhardness was measured from the Berkovich nano-indents obtained by nano-indenter. At high loads of 200–19600 mN, the microhardness was measured from the Vickers indents obtained by a microhardness test machine (MVK-H, Akashi, Japan).

3. Results and discussion

3.1. Nano-indentation hardness

The typical load–depth curves of five materials are plotted in Fig. 1. The large variations of the residual indentation depths show their distinctions in the material's ability against deformation or the hardness. On the load–depth curves of single crystal copper and stainless steel, the high initial unloading slope and small recovery depth suggest their high contact stiffness and elastic modulus. The stress-induced reversible phase transition leads to the large recovery depth of NiTi [14], as well as the pop-in jump in the unloading part of the load–depth curve of Si(100) [15,16].

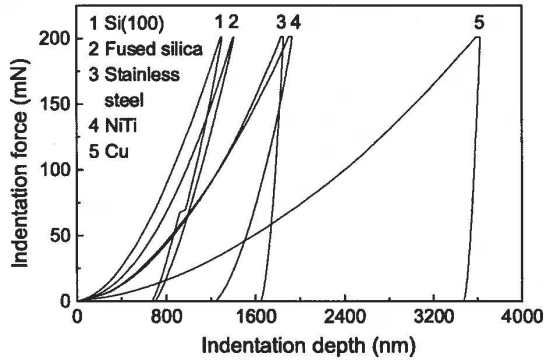


Fig. 1. Typical indentation force versus indentation depth curves for various samples. The nano-indentation hardness and elastic modulus of specimen can be analyzed by the curves with the Oliver–Pharr method. The stress induced reversible phase transition leads to the large recovery depth of NiTi, as well as the pop-in jump in the unloading part of the load–depth curve of Si(100).

As shown as the open circle plots in Fig. 2, the load dependence of nano-indentation hardness H_{O-P} for various samples is different. Clearly, the H_{O-P} of copper appears the strongest load effect, namely from 2.5 GPa at 1 mN to 0.8 GPa at 200 mN, which coincides with McElhaney et al.'s [17] measurements. The H_{O-P} of the stainless steel and NiTi exhibit a relatively weak load effect, namely from 5.5 to 3.8 and 5 to 4.5 GPa, respectively. Nevertheless, fused silica and silicon almost have constant H_{O-P} . To understand the load effect of H_{O-P} , the H_{O-P}/H_{max} (the maximum hardness of the sample in the load range) versus contact depth curves were plotted together in Fig. 3. Similar to Fig. 2, except fused silica with a constant H_{O-P} , the H_{O-P} of three metal samples decrease with the increase of depth. This kind of depth effect is usually called the indentation size effect [9]. Since the hardness of copper measured by keeping the constant indentation strain ($1/P \, dP/dt$) shows the similar variation with load, the loading rate effect cannot explain

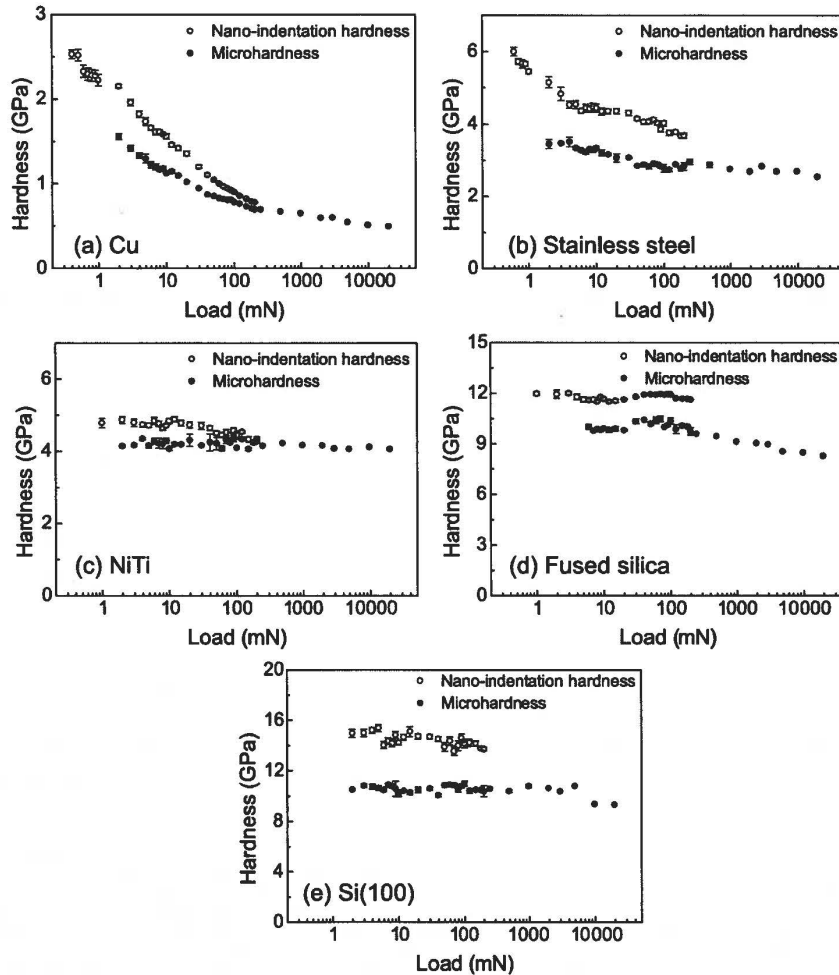


Fig. 2. Comparison of the nano-indentation hardness to microhardness. For all the measured materials, the microhardness are always below than their nano-indentation hardness, even the two kinds of hardness exhibit similar load effect between 2 and 200 mN owing to the indentation size effect.

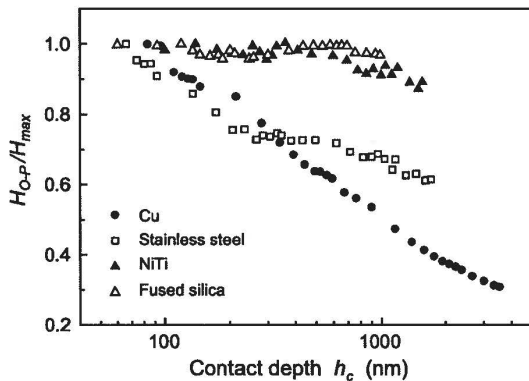


Fig. 3. H_{O-P}/H_{max} versus contact depth curves. Even fused silica shows constant hardness during the load range, the nano-indentation hardness of metals appears strong indentation size effect.

the results in Fig. 3. Strain-hardening effect was usually used to explain the increase of hardness with strain [18,19]. However, for sharp Berkovich indenter (with a curvature radius of ~ 150 nm) and large indentation depth, the representative strain is only determined by the face angle of indenter and independent of the indentation load [1]. It should then induce a constant hardness at various depths. Nevertheless, people also give some possible explanations on the indentation size effect [20,21]. By applying the strain gradient plasticity theory in indentation, Nix and Gao [20] pointed out that the geometrically necessary dislocations could increase the effective yield stress of material and the dislocation density increases with the decrease of indentation depth, resulting in the indentation size effect. From the energy viewpoint, Froehlich et al. [21] indicated that hardness is in fact the energy per volume plastic deformation, including volume deformation energy and free surface energy. It is that the free surface area of indentation grows up with depth to cause the indentation size effect. Whereas, due to the multifariousness of the materials and the complication of the micro elastic/plastic deformation during indentation process, the indentation size effect is still far from entire understanding.

Different from the other materials, the nano-indentation hardness of NiTi and Si(100) shows unique load effects due to the stress-induced phase transition. Even with similar hardness as stainless steel, NiTi appears superelastic property under stress and its apparent elastic modulus is only 1/3 magnitude larger than that of stainless steel (Fig. 4). As plotted in Fig. 1, during loading process, the load–depth curves of NiTi and stainless steel are repeated at low loads, implying no phase transition occurs in NiTi. While at high loads, the indentation depth of NiTi becomes larger than that of stainless steel, suggesting that the phase transition happens in NiTi and the phase transition yield increases the deformation volume. During unloading process, the indentation depth of NiTi falls down quickly owing to the recovery of the phase transition deformation. There-

fore, even the maximum indentation depth of NiTi is larger than that of stainless steel, the residual depth of NiTi is much smaller due to its unique stress induced phase transition (Fig. 1). As shown in Fig. 2(e), because no phase transition is generated at low loads below 5 mN, the nano-indentation hardness of Si(100) keeps constant between 2 and 5 mN. While the load increases above 5 mN, the maximum contact pressure attends to its phase transition stress and phase transition will occur during indentation process, which therefore results in the pop-in jump in the unloading part of the load–depth curve and the variation of the nano-indentation hardness [15,16].

The elastic moduli of five materials at various loads are plotted in Fig. 4. The slight decrease of elastic modulus of metals with the increase of load is consistent with the results of Rodriguez and Gutierrez [22], who reported that the elastic modulus of pearlite steel decreases about 30% with the increase of indentation depth from 100 nm–1000 nm. The large decrease of the elastic modulus of Si(100) may be due to the generation of cracks.

3.2. The microhardness

To obtain the microhardness, the micro topographies of indents are characterized by a laser confocal scanning microscopy. Fig. 5 shows the typical images of the Berkovich nano-indents obtained by nano-indenter. Fig. 6 shows the typical images of the Vickers indents obtained by microhardness test machine. Clearly, except the indents of copper and stainless steel exhibiting little pile-up, the indents of the other materials appear obvious sink-in. These experimental phenomena are consistent with the calculation of Bolshakov and Pharr [11], who found that pile-up occurred for $h_f/h_{max} > 0.7$ and sink-in for $h_f/h_{max} < 0.7$. As plotted in Fig. 7, it is clear that the indents of copper and stainless steel pile up with $h_f/h_{max} > 0.7$, while the indents of the other materials sink in with $h_f/h_{max} < 0.7$.

Some cracks generated around the indent of Si(100) along the indenter edges (Figs. 5 and 6). It can be attributed to the elastic recovery of material which induces tensile stresses in the vicinity of the plastic zone [23]. Since the reversible phase transition during the indentation process of silicon may result in an abrupt volume variation, namely during loading from diamond cube phase to β -tin phase with 22% volume contraction and during unloading the inverse phase transition with 22% volume expansion [15,16], the elastic recovery induced by the phase transition may accelerate the generation of the micro-cracks.

The microhardness H of five materials is plotted as the solid circles in Fig. 2. The values between 2 and 200 mN are measured from the Berkovich nano-indents, while the others from the Vickers indents. As the same as the nano-indentation hardness H_{O-P} , the H of copper exhibits the strongest load effect, and the H of the stainless steel has a relatively weak load effect. No obvious load effect on the H of the other materials is observed.

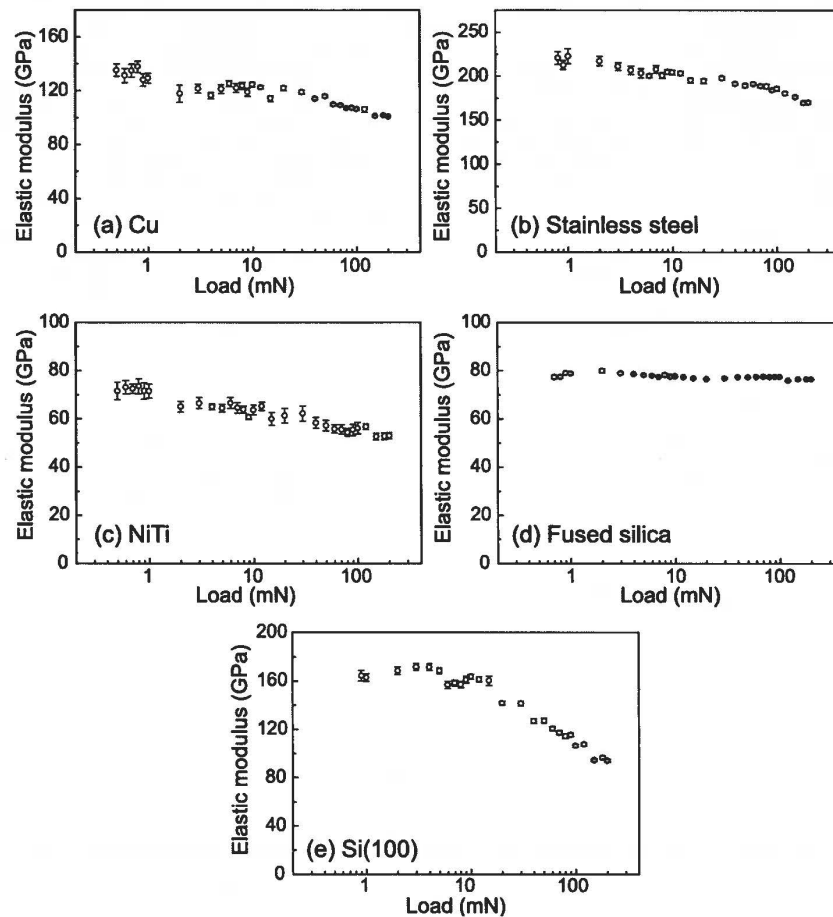


Fig. 4. Load effect on the elastic modulus for various samples.

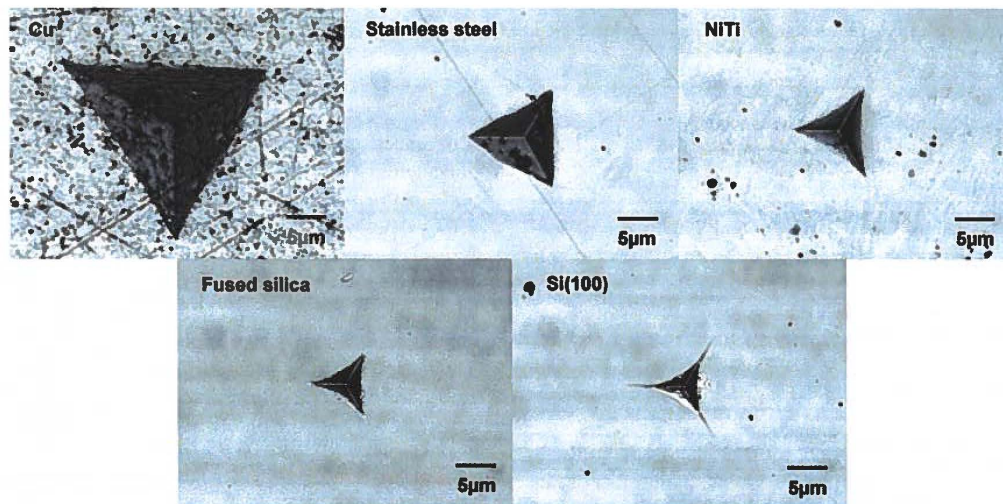


Fig. 5. Topography of indents. Except the indents of copper and stainless steel exhibiting little pile-up, the indents of the other materials appear obvious sink-in.

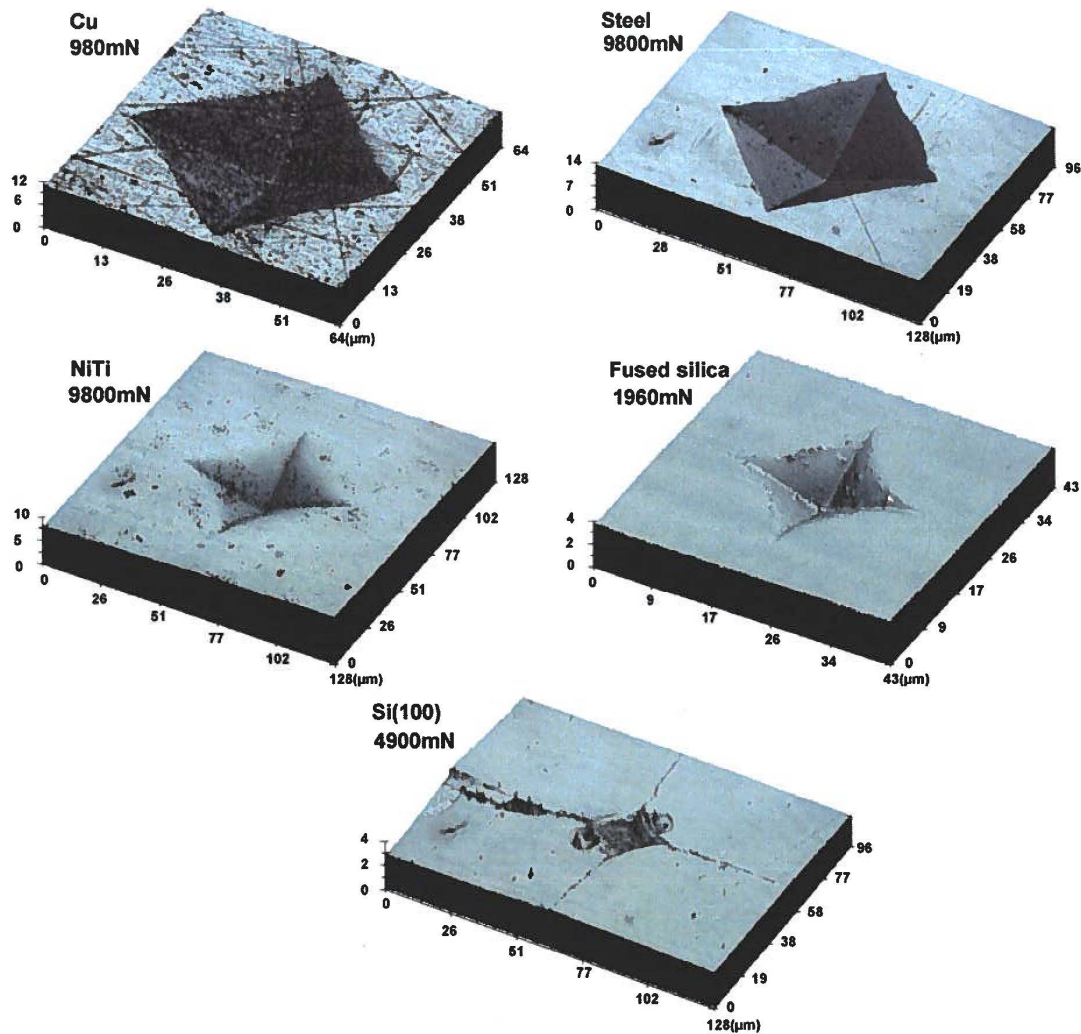


Fig. 6. Three-dimensional images of indents. The generation of the cracks on Si(100) is attributed to elastic recovery of material which induces tensile stresses in the vicinity of the plastic zone.

3.3. Comparison of nano-indentation hardness to microhardness

As discussed in introduction, the nano-indentation hardness H_{O-P} is obtained from the analysis on the load–depth curve with the Oliver–Pharr method. Since the Oliver–Pharr method applies the purely elastic solution to describe the elastic/plastic process, how well the H_{O-P} reflects the material's ability against deformation still need to be understood. The results in Fig. 2 provide an intuitionistic answer for the question.

In Fig. 2, for all the measured materials, the microhardness H are always below than their nano-indentation hardness H_{O-P} even the two kinds of hardness exhibit similar load effect between 2 and 200 mN. To show the

differences quantitatively, the ratios of H to H_{O-P} are collected in Table 1. Clearly, either the pile-up materials Cu and steel or the sink-in material Si(100) have the values of H/H_{O-P} below 0.8. Recently, Li and Cheng [24] calculated H/H_{O-P} versus Y/E curves with the FE method. In Table 1, E are the measured values; Y of the metals are taken to be 1/3 of their microhardness [1], Y of Si(100) and fused silica are taken to be 4.41 and 3.90 GPa, respectively [25,13]. Thus, the FE calculated H/H_{O-P}^* are obtained from the H/H_{O-P} versus Y/E curves in reference [24] with the experimental Y/E . Obviously, the experimental H/H_{O-P} consist well with the calculated H/H_{O-P}^* .

In Table 1, both the experiments and the FE calculation show that the nano-indentation hardness is 10–30% higher than the microhardness. With the FE method, Bolshakov

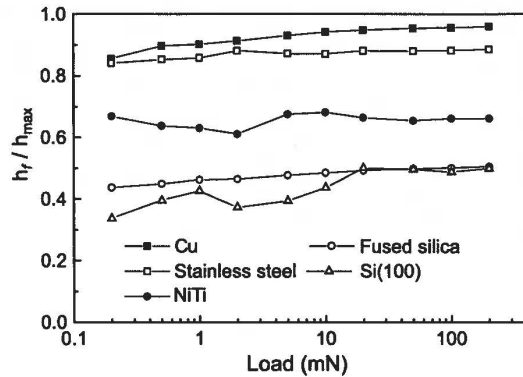


Fig. 7. Load dependence of the depth ratio for various samples. $h_r/h_{\max} > 0.7$ for copper and stainless steel suggests their piling-up indents, while $h_r/h_{\max} < 0.7$ for the other materials implies their sinking-in indents.

and Pharr [11] found that the pile-up of indent made the real contact area larger than A_c from the Oliver–Pharr method, which in turn induced the high nano-indentation hardness. However, pile-up only occurs for the materials with small Y/E , and the materials with large Y/E usually sink in [24]. Therefore, the main reasons may be attributed to the analysis of the nano-indentation hardness using the projected contact area A_c instead of the residual projected area A_r , as well as the purely elastic contact assumption describing the elastic/plastic indentation process.

The distinct definitions of A_c and A_r may cause great differences on their values. Since the indentation hardness is usually determined as the normal force per residual deformation area, the H calculated with A_r can precisely reflect the material's indentation hardness. In the following, a simple physical model is proposed to explore the difference between A_c and A_r . In Fig. 8, the thick solid lines and thick dashed lines separately plot the half profile of indent at peak load and the half profile of the residual indent. a_c and a_r are the radii of A_c and A_r , h_e and h_a are the elastic recovery depths at the contact center and radius a_c , respectively. If the unloading process of indentation is perfectly elastic, thus $h_a = (\pi - 2)h_e/\pi$ [10]. From Fig. 7, it is found that $h_a/h_{\max}$ is 0.04–0.07 for pile-up, as well as 0.11–0.25 for sink-in. Consequently, as shown in Fig. 8(a), if the edge of the pile-up indent is set as the top line of the

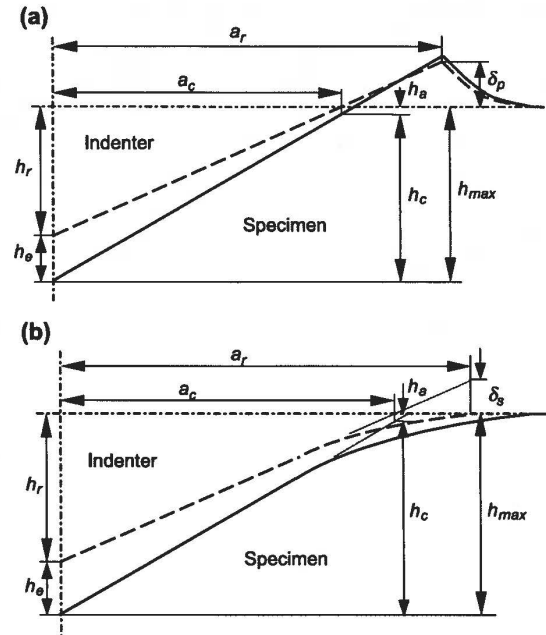


Fig. 8. Comparison of A_c and A_r . (a) Pile-up. (b) Sink-in. Either pile up or sink in, a_c (or A_c) is smaller than a_r (or A_r), and $H_{O-P}/H = (1 + \delta_p/h_r)^2$ or $H_{O-P}/H = (1 + \delta_s/h_r)^2$ is always larger than 1. The more heavily the indent piles up (or sinks in), the larger the δ_p/h_r (or δ_s/h_r), and the larger the error of H_{O-P} .

residual indent, as well as δ_p is defined as the depth difference between the edge and the original specimen surface, a_c (or A_c) is smaller than a_r (or A_r), and $H_{O-P}/H = (1 + \delta_p/h_r)^2$ is always larger than 1. Similarly, in Fig. 8(b), if the edge of the sink-in indent is defined as the cross line of the residual indent and the original specimen surface, as well as δ_s is defined as the depth difference between the original specimen surface and the cross point of the extended straight-line of the residual indent profile and the vertical line at the edge point, a_c (or A_c) is smaller than a_r (or A_r), $H_{O-P}/H = (1 + \delta_s/h_r)^2$ is also always larger than 1. The more heavily the indent piles up (or sinks in), the larger the δ_p/h_r (or δ_s/h_r), and the larger the error of H_{O-P} . Theoretically, while no pile-up or sink-in occurs, H_{O-P} is equal to H .

According to the elastic assumption, the fitted power law exponent m on the unloading part of the load–depth curve for the Berkovich indenter should be 2, while the experimental results in Fig. 9 show that the m of the materials are mainly distributed in 1.2–1.6, which consists with the results of Refs. [9,11] and suggests that the indentation unloading is a complicatedly elastic/plastic deformation process. Recently, Pharr and Bolshakov [26] found that the value of exponent is associated with the value of E/H of the material and a higher value of E/H will results in a larger value of m . Owing to the simplification of the elastic/plastic deformation to a purely elastic deformation process, the contact area A_c based on the Oliver–Pharr

Table 1

Comparison of the nano-indentation hardness H_{O-P} to the microhardness H

Materials	E (GPa)	Y (GPa)	Y/E	H/H_{O-P}	H/H_{O-P}^*
Single crystal copper	110	0.2	0.002	0.80	0.88
Stainless steel	180	1.0	0.006	0.73	0.89
NiTi	55	1.6	0.03	0.91	0.95
Fused silica	72	3.9	0.05	0.86	0.94
Si(100)	172	4.4	0.03	0.76	0.95

H/H_{O-P} are the experimental values, H/H_{O-P}^* are obtained from the H/H_{O-P} versus Y/E curves in Ref. [24] with the experimental Y/E .

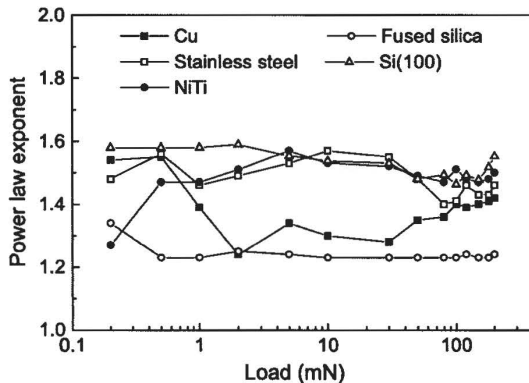


Fig. 9. Load effect on the power law exponent for various samples. The m of the materials are mainly distributed in 1.2–1.6, which suggests that the indentation unloading is a complicate elastic/plastic deformation process.

analysis cannot precisely describe the real contact area at peak load. Therefore, even for the indent without pile-up and sink-in, H_{0-P} may be still different from H .

Whereas, the experimental results in Fig. 2 show that nano-indentation hardness and microhardness have similar load effect and their differences are between 10% and 30%, indicating that the nano-indentation hardness can qualitatively characterize the material's ability against deformation. Especially for the hardness analysis of the small volume parts, where the residual projected area of nano-indent is difficult to be measured precisely, the nano-indentation hardness based on the Oliver–Pharr method will be a good choice. However, while using the nano-indentation hardness, we should fully estimate the error from the measurement method. One suggestion is to calibrate the nano-indentation hardness with the microhardness at the same load.

4. Conclusion

Different samples exhibit various indentation size effect of nano-indentation hardness. Even fused silica and silicon show constant hardness during the load range, the nano-indentation hardness of metals appears strong indentation size effect.

The nano-indentation hardness is about 10–30% magnitudes larger than the microhardness for the measured materials. The main reasons can be attributed to the analysis

of the nano-indentation hardness using the projected contact area at peak load A_c instead of the residual projected area A_r , as well as the purely elastic contact assumption describing the elastic/plastic indentation process. The analysis indicates that A_c is always smaller than A_r for indent either pile-up or sink-in. Therefore, the nano-indentation hardness can approximately imply the material's ability against deformation.

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References

- [1] D. Tabor, *The Hardness of Metals*, Clarendon Press, Oxford, 1951.
- [2] L.M. Qian, X.D. Xiao, Q.P. Sun, T.X. Yu, *Appl. Phys. Lett.* 84 (2004) 1076.
- [3] L.M. Qian, F. Tian, X.D. Xiao, *Tribol. Lett.* 15 (2003) 169.
- [4] L.M. Qian, G. Luango, E. Perez, *Europhys. Lett.* 61 (2003) 268.
- [5] L.M. Qian, G. Luango, D. Douillet, et al., *Rev. Sci. Instrum.* 72 (2001) 4171.
- [6] L.M. Qian, X.D. Xiao, S.Z. Wen, *Langmuir* 16 (2000) 662.
- [7] X.D. Xiao, L.M. Qian, *Langmuir* 16 (2000) 8153.
- [8] J.B. Luo, L.M. Qian, S.Z. Wen, et al., *Tribol. Trans.* 42 (1999) 912.
- [9] W.C. Oliver, G.M. Pharr, *J. Mater. Res.* 7 (1992) 1564.
- [10] I.N. Sneddon, *Int. J. Eng. Sci.* 3 (1965) 47.
- [11] A. Bolshakov, G.M. Pharr, *J. Mater. Res.* 13 (1998) 1049.
- [12] Y.-T. Cheng, C.-M. Cheng, *Philos. Mag. Lett.* 78 (1998) 115.
- [13] Y.-T. Cheng, C.-M. Cheng, *Surf. Coat. Technol.* 133–134 (2000) 417.
- [14] T. Duerig, K.N. Melton, D. Stockel, et al., *Engineering Aspect of Shape Memory Alloys*, Butterworth-Heinemann Press, Boston, 1990.
- [15] E.R. Weppelmann, J.S. Field, M.V. Swain, *J. Mater. Res.* 8 (1993) 830.
- [16] X. Li, B. Bhushan, *Thin Solid Films* 340 (1999) 210–217.
- [17] K.W. McElhane, J.J. Vlassak, W.D. Nix, *J. Mater. Res.* 13 (1998) 1300.
- [18] Y.Y. Le, M.M. Chaudhri, *Phys. Status Solidi, A Appl. Res.* 194 (2002) 19.
- [19] M.M. Chaudhri, *Acta Mater.* 46 (1998) 3047.
- [20] W.D. Nix, H. Gao, *J. Mech. Phys. Solids* 46 (1998) 411.
- [21] F. Froehlich, P. Grau, W. Wrellmann, *Phys. Status Solidi* 42 (1977) 79.
- [22] R. Rodriguez, I. Gutierrez, *Mater. Sci. Eng., A* 361 (2003) 377.
- [23] D.B. Marshall, B.R. Lawn, A.G. Evans, *J. Am. Ceram. Soc.* 65 (1982) 561.
- [24] Z.-Y. Li, Y.-T. Cheng, et al., *Surf. Coat. Technol.* 154 (2002) 124.
- [25] T.A. Laursen, J.C. Simo, *J. Mater. Res.* 7 (1992) 618.
- [26] G.M. Pharr, A. Bolshakov, *J. Mater. Res.* 17 (2002) 2660.