J Mater Sci (2011) 46:1083–1086
DOI 10.1007/s10853-010-4876-7

Measurement of mechanical properties of 1045 steel with significant pile-up by sharp indentation

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Received: 10 March 2010 / Accepted: 30 August 2010 / Published online: 11 September 2010

Abstract The Oliver–Pharr method has extensively been adopted for measuring hardness and elastic modulus by indentation techniques. However, the method assumes that the contact periphery sinks in, which limits the applicability to the materials pile up. This study proposed an improved methodology to calculate the real contact area of 1045 steel with significant pile-up. The contact boundary between indenter and specimen was assumed to overlap with the top points on the residual surface profile, and the real contact depth was defined by a sum of the indentation depth at maximum load, $h_{\max}$, and average pile-up height, $h_{\text{pile-up}}^{\text{ave}}$, measured from the analysis on the residual indent morphology with atomic force microscope (AFM). The mechanical properties calculated by the newly proposed method were compared with those by the Oliver–Pharr method.

Introduction

At present, nanoindentation technique is widely used for measuring the hardness and elastic modulus of both bulk solids and thin films [1–3]. In 1992, the method was introduced by Oliver and Pharr [4] based on an earlier work of Doerner and Nix [5]. Its attractiveness stems largely from the fact that mechanical properties can be directly obtained from indentation load–displacement data. According to the unloading curve, the hardness H and reduced modulus E_r can be determined by Eqs. 1–4 [6].

$$H = \frac{P_{\max}}{A_c}, \tag{1}$$

$$E_r = \frac{S\sqrt{\pi}}{2\sqrt{A_c}}, \tag{2}$$

$$A_c = \sum_{n=0}^{8} C_n (h_c)^{2-n} = C_0 h^2 + C_1 h + \cdots + C_8 h^{1/128}, \tag{3}$$

$$h_c = h_{\max} - 0.72\frac{P_{\max}}{S}, \tag{4}$$

where $P_{\max}$ is the maximum load, h_c is the real contact depth, $h_{\max}$ is the maximum displacement, $S = dP/dh$ defined as the slope of the upper portion of the unloading curve during the initial stages of unloading, and $C_0,\ldots,C_8$ are constants determined by curve-fitting procedures.

Once the contact area is determined, the hardness and reduced modulus can be estimated from Eqs. 1 and 2. Therefore, the hardness and reduced modulus rely heavily on an accurate measurement of the contact area. However, when pile-up occurs, the real contact area will be underestimated by as much as 60%, leading to similar errors in calculated material properties [7]. Joslin and Oliver [8] extracted the real contact area implying pile-up deformation, but the method requests one property of hardness or elastic modulus, which limits its wide applications.

Thus, a newly improved method has been proposed by redefining the real contact depth for pile-up materials. The mechanical properties were calculated by the proposed method and were compared with those measured by the Oliver and Pharr method.

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Experimental method

The material used in this study was a 1045 steel rectangular flat-plate with the dimension of 25 mm × 15 mm × 8 mm. The chemical composition of the 1045 steel was shown in Table 1. The specimen was water quenched at 840 °C, then it was mechanically ground and polished. Roughness of the sample surface was 6.1 ± 2.0 nm.

Nanoindentation tests were performed using TriboIndenter system (Hysitron, Inc) with a Berkovich diamond pyramid indenter. The load and depth resolutions of the system are 0.1 nm and 0.1 μN, respectively. Prior to indentation, the geometry of the Berkovich indenter was calibrated on standard fused quartz sample. Indentation tests were performed under the constant temperature of 20 °C. Six different maximum loads were chosen as 3, 5, 6, 7, 8, and 9 mN, respectively. 5 × 5 matrix indents (as shown in Fig. 1) spaced 15 μm were made at each load.

Results and discussion

Figure 2 shows the three-dimensional images of nano-indents at 5 mN, and the nano-indents at other peak loads

Table 1 Chemical composition of the 1045 steel

Element	C	Si	Mn	S	P	Cr	Ni	Cu	Fe
wt%	0.45	0.25	0.62	0.015	0.018	0.23	0.008	0.009	Balance

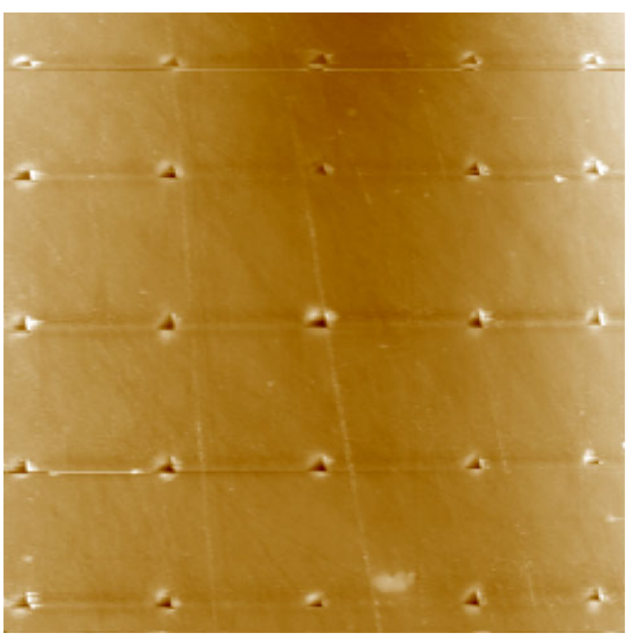

Fig. 1 5 × 5 Matrix indents

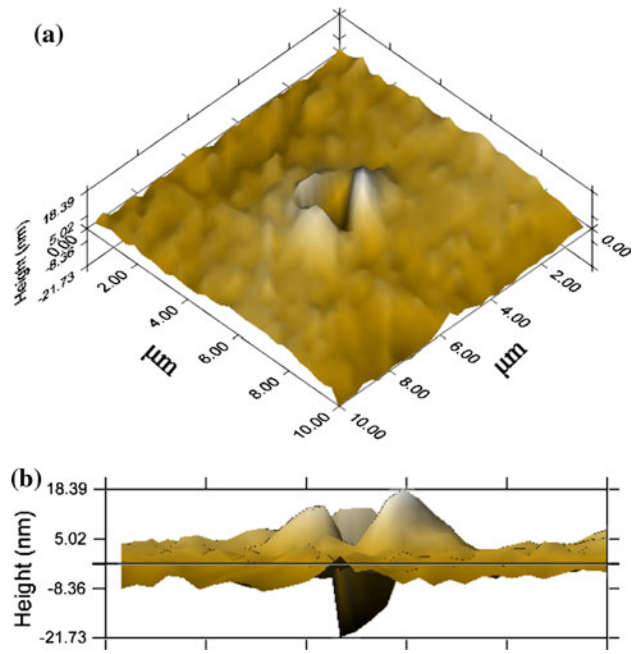

Fig. 2 Typical three-dimensional images of nano-indents at 5 mN

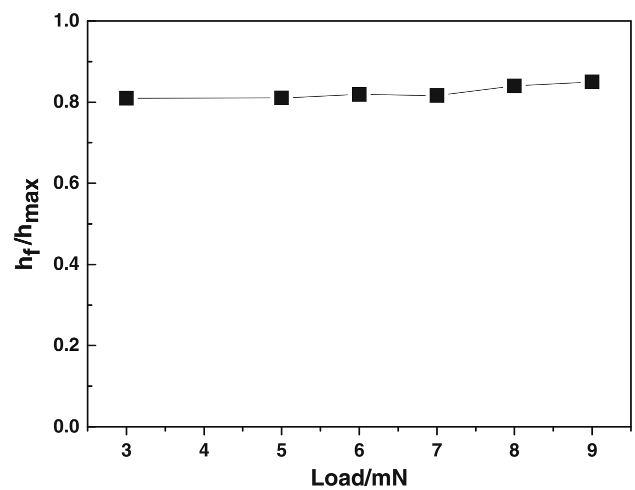

Fig. 3 Depth ratio h_f/h_{max} for different maximum loads

are similar in image to that at 5 mN. It is seen that the nano-indenters exhibit obvious pile-up. This phenomenon is consistent with the finite element studies by Bolshakov and Pharr [7]. The amount of pile-up is related to the ratio of final indentation depth, h_f, to the indentation depth at maximum load, h_{max}. The ratio of h_f/h_{max} can be obtained easily from the unloading curve in a nanoindentation experiment. The pile-up occurs when $h_f/h_{max} > 0.7$ [9]. As shown in Figs. 2 and 3, it is obvious that the nano-indents of the 1045 steel pile up with $h_f/h_{max} > 0.7$. This observation is particularly important when considered in relation to the contact areas. When pile-up occurs, the real contact area is greater than that predicted by the

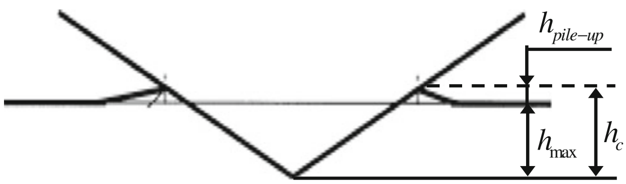

Fig. 4 Definition of real contact depth for materials that pile up

Oliver–Pharr method, and both the hardness calculated from Eq. 1 and the reduced modulus from Eq. 2 are overestimated.

From Eq. 4, h_c defined in the Oliver–Pharr method is always smaller than h_{max}. However, the contact boundary of materials pile-up rises above the original sample surface, and the real contact depth cannot be consistent with h_c defined in the Oliver–Pharr method anymore. Therefore, the real contact depth for pile-up materials should be redefined. In the present study, we have developed an improved method to extract the contact area of the material that pile-up. According to the previous study [10], the pile-up morphology can be assumed to be invariant regardless of the loaded or unloaded states. Therefore, the real contact depth h_c can be defined by $h_{max} + h_{pile\text{-}up}$, as shown in Fig. 4. The pile-up height $h_{pile\text{-}up}$ was measured by AFM cross-section profile, as shown in Fig. 5. Three pile-up height values were averaged to derive a representative value $h^{ave}_{pile-up}$. $h_c = h_{max} + h^{ave}_{pile-up}$ was substituted in Eq. 3 to calculate the real contact area A_c. The values of A_c, H, and E_r calculated by the Oliver–Pharr method and improved method are listed in Table 2.

Figure 6 shows the hardness and reduced modulus calculated by the Oliver–Pharr method and improved method at different maximum loads. It is clear that both the hardness and reduced modulus exhibit a strong load effect. In general, the hardness and reduced modulus tend to decline with the increasing load, i.e., indentation size effect (ISE). ISE phenomenon exists in many materials, which is probably caused by the following reasons: (1) The commonly used empirical equation for describing the ISE is the Meyer's law, which correlates the load and the resultant indentation size using a simple power law [11]. (2) From the strain gradient plasticity theory in indentation, the geometrically necessary dislocations could increase the effective yield stress of material and the dislocation density increases with the decrease of indentation depth, leading to the ISE [9]. (3) The ISE is the result of a high surface area to volume ratio of the indentation at low applied test forces [12].

As the Oliver–Pharr method neglects the pile-up deformation, the real contact area is underestimated, leading to

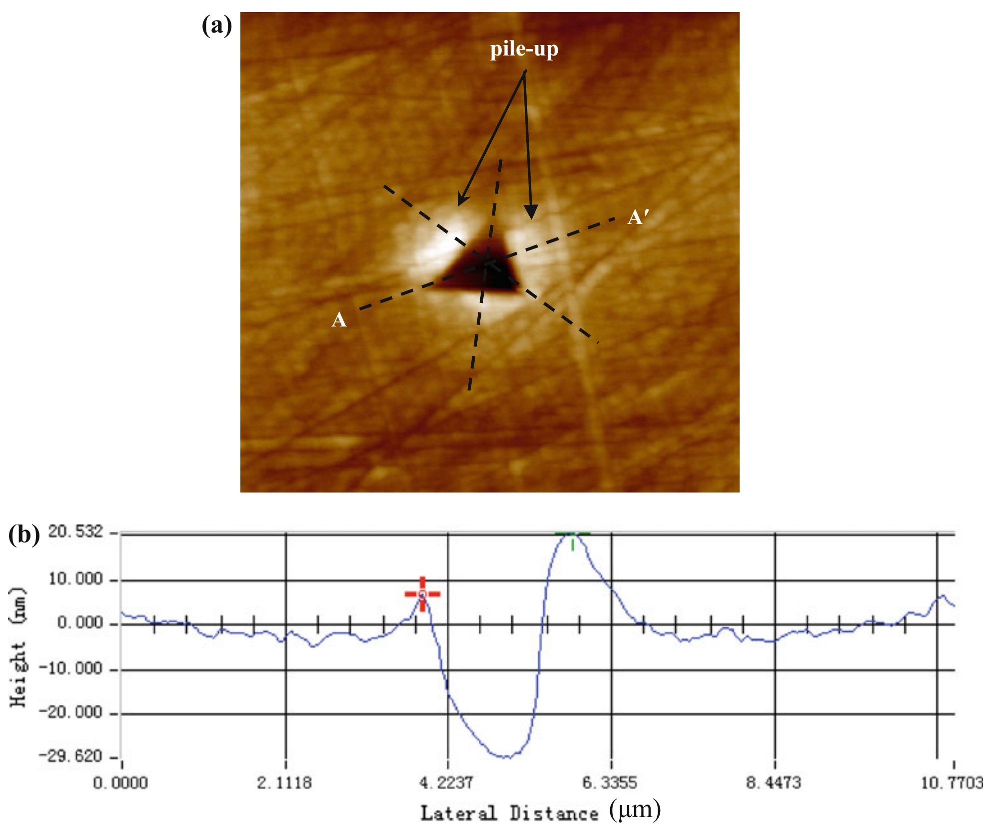

Fig. 5 Significant pile-up (**a**) and its cross-section profile (**b**)

Table 2 A_c, H and E_r calculated by the Oliver–Pharr method and improved method

Load (mN)	Oliver–Pharr method			Improved method		
	A_c (nm^2)	H (GPa)	E_r (GPa)	A_c (nm^2)	H (GPa)	E_r (GPa)
3	441590	6.79	248.30	753210	3.98	190.17
5	926927	5.40	201.71	1401897	3.57	179.86
6	1015290	5.91	238.64	1677017	3.58	185.70
7	1194252	5.84	234.60	1904373	3.68	184.32
8	1602344	4.99	227.32	2640957	3.03	179.97
9	1844498	4.86	233.25	3155871	2.85	183.73

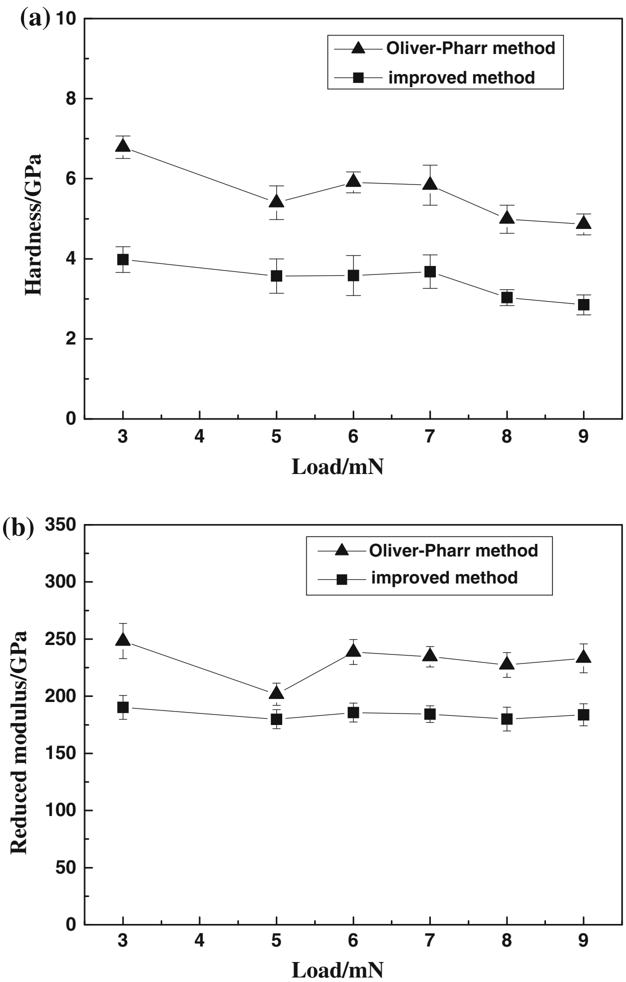

Fig. 6 Comparison between the Oliver–Pharr method and improved method

the overestimates of hardness and reduced modulus. The differences of the hardnesses obtained by the two methods are between 34 and 44%, and the differences of the moduli are between 10 and 23%, which agrees with the finite element studies by Bolshakov and Pharr [7]. For this newly proposed method, the top points on the residual surface profile are taken as the contact boundary, but further study should be made in order to confirm its validity. As the pile-up deformation around nano-indents is complicated, it is important to develop a much better understanding of the contact mechanics on which the proposed method is based. If the contact boundary is confirmed to be the peaks of pile-up region, the analysis on the residual nano-indent morphology will become a promising tool to make the most accurate mechanical property measurement for materials that pile up.

Conclusion

In this article, we proposed an improved method to calculate the hardness and elastic modulus of the materials pile-up. This method redefined the real contact depth as a sum of the indentation depth at maximum load, h_{max}, and average pile-up height, $h^{ave}_{pile-up}$, measured from the direct observation of the residual indent morphology. The measurement of the mechanical properties for the specimen 1045 steel by the improved method was more accurate than that by the commonly used Oliver–Pharr method. However, further study should be made to confirm the validity of the newly proposed method.

Acknowledgements This paper was financially supported by Advanced Maintenance Research Project (9140A270304090C8501), NSFC (50875053, 50735006), Equipment Research Project and Fundamental Research Funds for the Central Universities (2009PY08).

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