Evaluation of macro-hardness from nanoindentation tests

DEJUN MA
Surface Engineering Research Institute, Chinese Mechanical Engineering Society, Beijing 100072, People's Republic of China

CHUNG WO ONG*, SING FAI WONG
Department of Applied Physics and Materials Research Center, The Hong Kong Polytechnic University, Hung Hom, Kowloon, Hong Kong, People's Republic of China
E-mail: apacwong@inet.polyu.edu.hk

Anomaly in the measured hardness of a material increases with decrease in the indentation depth below one or two microns has been observed by many researchers. This phenomenon is named as indentation size effect (ISE) in literature. Obviously, the existence of ISE would give rise to a problem that hardness values measured at shallow depths are incomparable with the macro-hardness, namely the asymptotic value at deep indentation depths. Although a variety of mechanisms including a dependence of flow strength on gradients of plastic strain, surface effects, and the influence of harder surface layers, etc. have been proposed to interpret this phenomenon [1–7], little work has been done to seek practical method for evaluating macro-hardness based on indentation data at depths in submicron range (nanoindentation). The establishment of a method of this type is particularly important when the area of interest is so small that the evaluation of hardness can only reply on nanoindentation results, such as for a thin film sample or a modified surface region.

In this study, we illustrated experimentally the feasibility of a practical way to extract macro-hardness values of a material from nanoindentation tests. The effectiveness of the method replied on the revelation of a relationship between the ratio of nanoindentation hardness with macro-hardness, and some indentation parameters.

The depth dependence of the nanoindentation hardness of five materials, including S45C carbon steel, 6061 aluminum alloy, tungsten single crystal, aluminum single crystal and fused silica were measured. The surfaces of the samples were carefully polished to mirror finish. A commercial Nano indenter® IIs (Nano Instruments, Inc.) equipped with a diamond Berkovich indenter was used to perform the experiments. The area function of the indenter was carefully measured according to the standard procedures proposed by Oliver and Pharr [8]. Each experiment was repeated 10 times at different positions on a sample surface to ensure statistical reliability. The measured hardness values at different depths derived from Oliver and Pharr method [8] are shown in Fig. 1a–e.

Although Nix and Gao [3] proposed a formula $H = H_0\sqrt{1 + h^*/h}$ to describe the indentation depth (h) dependence of measured hardness (H), where H_0 is the hardness in the limit of infinite depth, i.e., the macro-hardness and h^* is a coefficient representing the length scale for the depth dependence, unfortunately, it seems not to fit to our data shown in Fig. 1a–e. Therefore, we employed another formula:

$$H = H_0 \exp[a/(h + b)] \tag{1}$$

with a and b being two fitting parameters. This formula is much more satisfactory in reproducing the depth dependence of H for all the five materials under investigations.

It is known that the results of ISE would also be influenced by the bluntness of the indenter [5]. In order to incorporate the contribution of bluntness effect into the analysis, we introduce a quantity called absolute bluntness, Δh, of the indenter, which is defined as the distance between the apex of the ideal pyramidal shape and the top of the real indenter. Through calibrating the area function of the indenter, Δh is determined to be 40.4 nm.

To simplify the analysis, we specifically took the data of indentation tests with the maximum indentation depth h_m equal to five times of Δh, i.e., 202 nm. Three groups of data for all the five materials under investigations were obtained. The first is the ratio of H to H_0, with H being derived according to Oliver and Pharr method [8]. The second is the power-law exponent $X|_{\mathrm{hm}=5\Delta\mathrm{h}}$ extracted from fitting the nanoindentation loading curves as shown in Fig. 2 according to a power-law function:

$$P = Ch^X \tag{2}$$

where P is the indentation load and C is a fitting coefficient. Each $X|_{\mathrm{hm}=5\Delta\mathrm{h}}$ value is an average of the results of 10 repetitive measurements. The third group of data is the reduced Young's modulus, E_r, defined as $1/E_r = (1 - \nu^2)/E + (1 - \nu_i^2)/E_i$, where E and ν, and E_i and ν_i are the Young's modulus and Poisson's ratio of the indented material and the indenter, respectively. These three groups of data are tabulated in Table I.

*Author to whom all correspondence should be addressed.

0022–2461

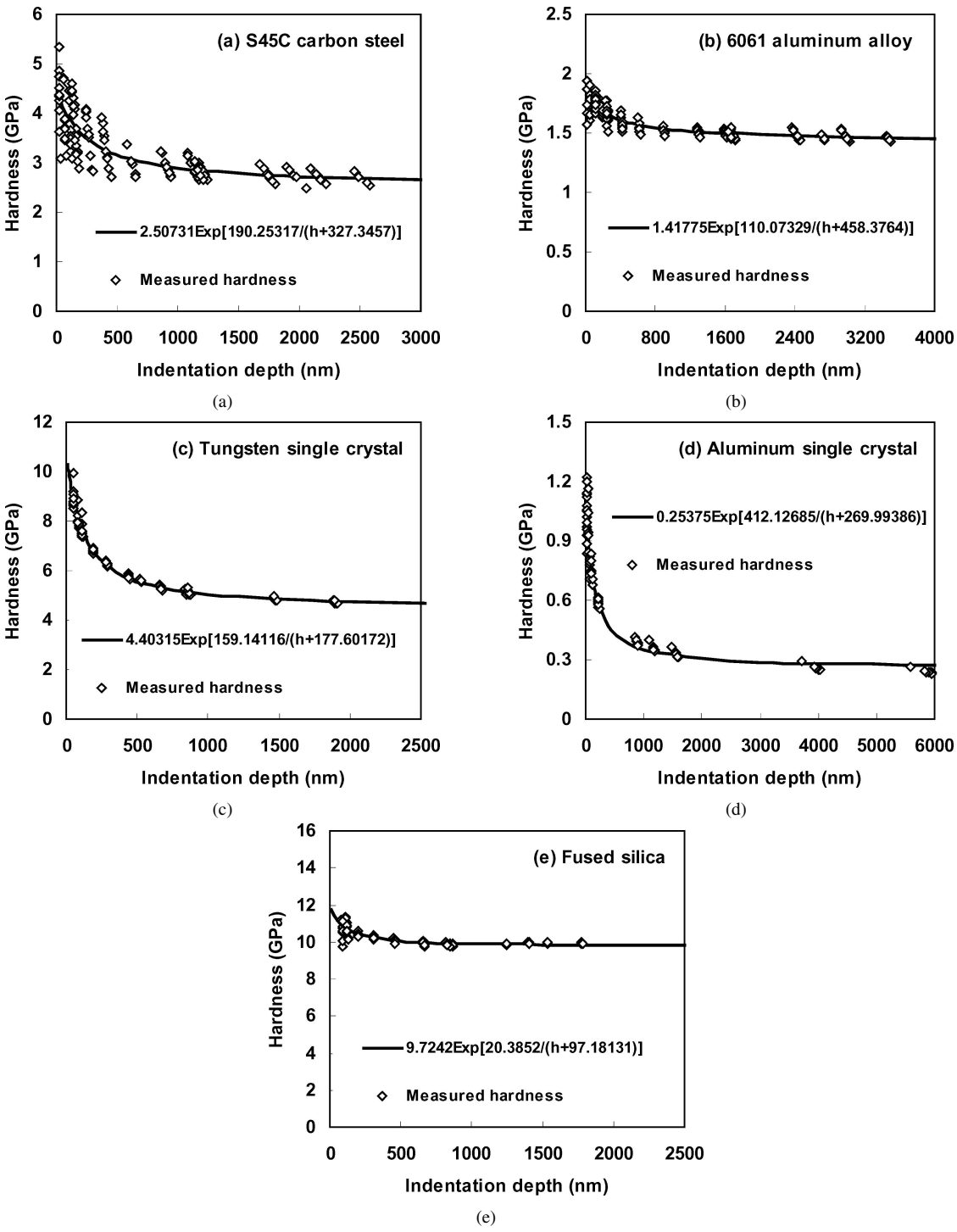

Figure 1 Depth dependence of the measured hardness of: (a) S45C carbon steel, (b) 6061 aluminum alloy, (c) tungsten single crystal, (d) aluminum single crystal and (e) fused silica.

It was found that the aforementioned three groups of data, though obtained from five different materials, fall in a single linear relationship in the form:

$$H/H_0|_{\mathrm{hm}=5\Delta\mathrm{h}} = k_1 - k_2(X|_{\mathrm{hm}=5\Delta\mathrm{h}} + k_3 E_\mathrm{r}) \quad (3)$$

where k_1, k_2 and k_3 are fitting coefficients equal to 6.7986, 3.8303 and 3.7162×10^{-4} GPa^{-1} respectively according to regression analysis. Fig. 3 shows how excellent the data are fitted to Equation 3. It should be emphasized that the values of k_1, k_2 and k_3 may depend on the indenter bluntness (Δh) and the design of indentation tests ($h_\mathrm{m}/\Delta h$ ratio).

The revelation of the earlier relationship between $H/H_0|_{\mathrm{hm}=5\Delta\mathrm{h}}$, $X|_{\mathrm{hm}=5\Delta\mathrm{h}}$ and E_r provides a practical approach to determine the macrohardness from nanoindentation tests. The procedure of such a method consists of the following steps:

(i) Measuring the nanoindentation load–unload curves with the maximum indentation depth $h_\mathrm{m} = 5\Delta h$ by employing a depth-sensing indentation instrument;

(ii) Determining the nanoindentation hardness $H|_{\mathrm{hm}=5\Delta\mathrm{h}}$ and the reduced Young's modulus, E_r by using Oliver and Pharr method [8];

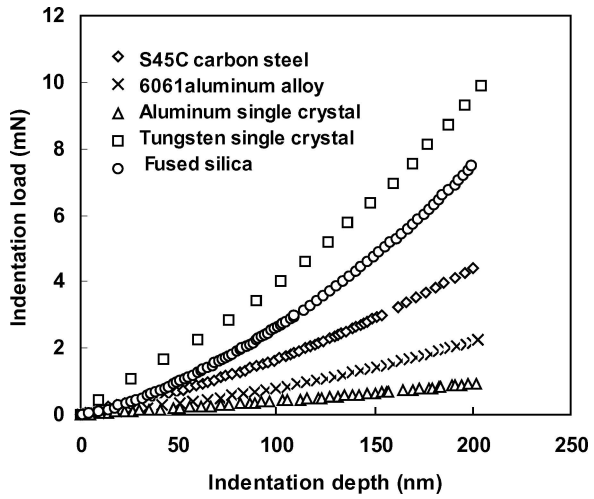

Figure 2 Nanoindentation loading curves with $h_m = 5\Delta h = 202$ nm for five materials.

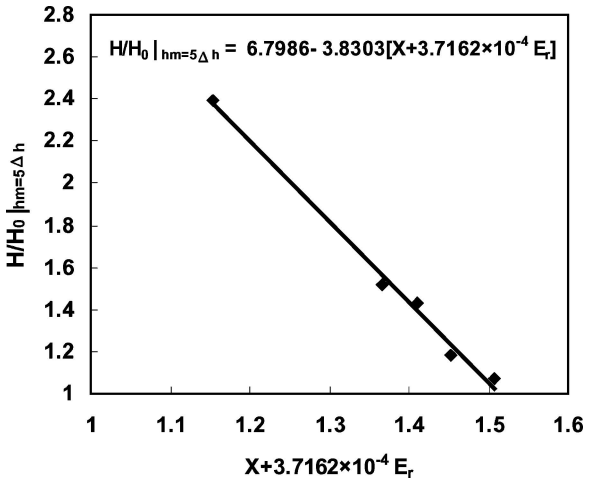

Figure 3 A linear relationship between $H/H_0|_{\mathrm{hm}=5\Delta\mathrm{h}}$, $X|_{\mathrm{hm}=5\Delta\mathrm{h}}$ and E_r.

(iii) Determining the power-law exponent $X|_{\mathrm{hm}=5\Delta\mathrm{h}}$ by fitting the nanoindentation loading curve;

(iv) Calculating the macro-hardness H_0 by using the expression:

$$H_0 = (H|_{\mathrm{hm}=5\Delta\mathrm{h}})/(H/H_0|_{\mathrm{hm}=5\Delta\mathrm{h}}) = (H|_{\mathrm{hm}=5\Delta\mathrm{h}})/[k_1 - k_2(X|_{\mathrm{hm}=5\Delta\mathrm{h}} + k_3 E_\mathrm{r})]$$

It should be pointed out that the method was valid for the indenter with a specific bluntness $\Delta h = 40.4$ nm. For a more general situation, the absolute bluntness Δh should have different values for different indenters, and so should the constants k_1, k_2 and k_3. Once the constants k_1, k_2 and k_3 are determined for several different indenters by following the same line of thought, a universal method for evaluating the macro-hardness from nanoindentation tests can be established. Obviously, more work should be done to fulfill this aim.

In conclusion, the present study sheds light on the possibility of evaluating the macro-hardness of a material from nanoindentation tests. A practice method can be established on basis of the revelation of a relationship between the hardness ratio $H/H_0|_{\mathrm{hm}=5\Delta\mathrm{h}}$, the power-law exponent $X|_{\mathrm{hm}=5\Delta\mathrm{h}}$ of a nanoindentation loading curve and the reduced Young's modulus E_r of the indented material.

TABLE I The values of $H/H_0|_{\mathrm{hm}=5\Delta\mathrm{h}}$, $X|_{\mathrm{hm}=5\Delta\mathrm{h}}$ and E_r of five materials

Materials	$H/H_0\|_{\mathrm{hm}=5\Delta\mathrm{h}}$	$X\|_{\mathrm{hm}=5\Delta\mathrm{h}}$	E_r (GPa)
S45C carbon steel	1.4325	1.3419	184
6061 aluminum alloy	1.1814	1.4249	74
Tungsten single crystal	1.5208	1.2483	320
Aluminum single crystal	2.3945	1.1262	74
Fused silica	1.0705	1.4819	68

Acknowledgments

The work described in this paper was substantially supported by a grant from the Center for Smart Materials (account Code: 1.A.310), an internal grant (account Code: A-PE07) and another internal grant of The Hong Kong Polytechnic University.

References

1. N. A. FLECK, G. M. MULLER, M. F. ASHBY and J. W. HUTCHINSON, *Acta Metall. Mater.* **42** (1994) 475.
2. W. J. POOLE, M. F. ASHBY and N. A. FLECK, *Scripta Mater.* **34** (1996) 559.
3. W. D. NIX and H. GAO, *J. Mech. Phys. Solids* **46** (1998) 411.
4. N. I. TYMIAK, D. E. KRAMER, D. F. BAHR, T. J. WYROBEK and W. W. GERBERICH, *Acta Mater.* **49** (2001) 1021.
5. J. G. SWADENER, E. P. GEORGE and G. M. PHARR, *J. Mech. Phys. Solids* **50** (2002) 681.
6. W. W. GERBERICH, N. I. TYMIAK, J. C. GRUNLAN, M. F. HORSTEMEYER and M. I. BASKES, *ASME J. Appl. Mech.* **69** (2002) 433.
7. T. Y. ZHANG and W. H. XU, *J. Mare. Res.* **17** (2002) 1715.
8. W. C. OLIVER and G. M. PHARR, *ibid.* **7** (1992) 1564.

Received 28 June
and accepted 31 August 2004