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Correlation between the flow stress and the nominal indentation hardness of soft metals

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For the sharp indentation of soft metals, we first reveal a simple relation between the nominal hardness, H_n, and the flow stress, σ_r, i.e. $H_n = 4.4\sigma_r$. Further, using the relation proposed herein instead of the well-known Tabor's relation and the analysis performed by Nix/Gao on the geometrically necessary dislocations, a simple indentation nominal hardness–depth relation at the microscale is obtained. The model agrees well with the experiment data in the literature and the simulation based on a strain gradient theory.

Keywords: Nominal hardness; Microindentation; Size effects

Size effects were frequently observed in microindentation tests [1–4]. A number of factors, e.g. tip defects [5], creep [6], plastically graded surface [7,8] and geometrically necessary dislocations (GNDs), may be responsible for this phenomenon. In the present paper, we focus on the size effects induced by GNDs, which has undergone much research during the past decade [9–17]. Nix and Gao [9] proposed a model to interpret the depth-dependent hardness observed in microindentation tests by including the effect of GNDs. The model has been widely used recently (e.g. [18–21]), for instance to evaluate the characteristic length of the materials. In the model of Nix and Gao [9], the hardness is defined as the indentation load dividing by the projected contact area. However, in many circumstances the determination of the projected contact area at the microscale is not easy due to the presence of piling-up [22,23]. For example, the standard method by Oliver and Pharr [24] used to measure hardness can significantly underestimate the contact area if the indented materials exhibit piling-up. In this case, if the Young's modulus of the material is known, the method suggested by Joslin and Oliver [25] may be used to evaluate the projected contact area; however, the initial unloading slope is involved, and the errors in this might be large in practice. If the Young's modulus of the material is unknown, additional experimental facilities, such as atomic force microscopy (AFM), may be applied to determine the extent of the piling-up. However, it should be noted that AFM can only be used to scan the residual impression after unloading; direct measurement of the contact area at the maximum indentation depth appears to be impossible. Thus, in order to investigate the role played by GNDs in microindentation tests, it is necessary and important to develop a model to interpret the effect of GNDs on directly measurable quantities. Bucaille et al. [26] and Cao and Lu [27] attempted to address the effect of GNDs on the indentation loading curve. However, these studies were based on the representative strain and the fitting functions presented by Dao et al. [28], which may exhibit significant errors when applied to some important highly plastic metals [29] (e.g. copper, gold and silver, for which the ratios of the Young's modulus to the yield strength are large). Based on this premise, the present study attempted to address the correlation between the material properties and directly measurable quantities in the sharp indentation of soft metals at the microscale.

According to the systematic finite element analysis performed in our recent research [29–31], we first reveal a simple relationship between the nominal hardness H_n and the flow stress σ_r corresponding to the specific representative strain ε_r, i.e.

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$$H_{\rm n} = \kappa_0 \sigma_{\rm r} \tag{1}$$

for conical indentation into soft metals (with a large ratio of Young's modulus to yield strength; Fig. 1 gives a schematic plot of the conical indentation). Here κ_0 is a constant and is equal to 4.4 for a given range of the half-apex angle θ of the indenter (i.e. θ varies from 60 to 80°) according to our recent studies [29–31]. $\varepsilon_{\rm r}$ depends on θ only (see Fig. 2), and the relation between $\sigma_{\rm r}$ and $\varepsilon_{\rm r}$ is given by the following power function:

$$\sigma_{\rm r} = K\varepsilon_{\rm r}^n \tag{2}$$

where K is the strength coefficient and the strain-hardening exponent n lies in the range of 0–0.5. $H_{\rm n}$ is a directly measurable quantity and is defined as

$$H_{\rm n} = \frac{P}{\pi(\tan(\theta)h)^2} \tag{3}$$

where P is the indentation load and h is the indentation depth (see Fig. 1). It should be emphasized here that the nominal hardness defined by Eq. (3) only depends on the half-apex angle θ and the directly measurable quantities, i.e. the indentation load P and the depth h. The hardness used in the model of Nix and Gao [9] depends on the contact depth $h_{\rm c}$ (see Fig. 1) aw well as the load P and the half-apex angle θ; thus the effects of piling-up or sinking-in will come into play.

Eq. (1) is quite similar to the well-known Tabor's relation between the hardness and the flow stress, i.e.

$$H = \kappa\sigma_{\rm r,Tabor} \tag{4}$$

where the hardness $H = P/\pi(\tan(\theta)h_{\rm c})^2$ (see Fig. 1 for $h_{\rm c}$) and κ is a constant; according to the analysis of Cheng and Cheng [32], this constant is around 2.75 for highly plastically materials. It has been noted that friction may affect the contact area at the given indentation load [33], and will thus influence the value of κ. The representative stress $\sigma_{\rm r,Tabor}$ depends on the representative strain defined by Tabor [33], and their relation is given by Eq. (2). The Tabor's representative strain is also a function of the half-apex angle θ; for instance, for $\theta = 70.3°$, it is around 8%.

At the macroscale, the relation given by Eq. (1) permits the determination of the flow stresses corresponding to the representative strains of the materials from the nominal hardness. At the microscale, Nix and Gao [9] showed that the density of the GNDs under the indenter might be expressed as

$$\rho_{\rm GND} = \frac{3}{2\tan^2(\theta)bh} \tag{5}$$

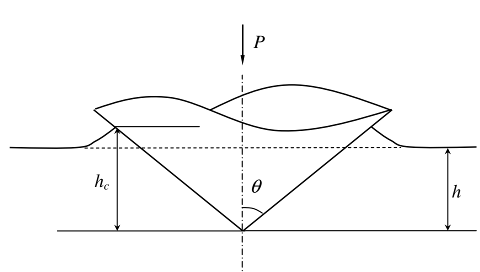

Figure 1. A plot of the conical indenter.

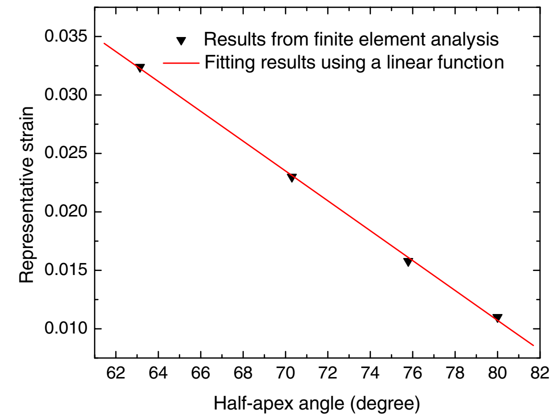

Figure 2. Dependence of the representative strain on the indenter half-apex angle.

where b is the magnitude of the Burgers vector. Durst et al. [34] modified Eq. (5) by considering the size of the plastic zone and gave

$$\rho_{\rm GND,\,m} = \frac{3}{2\tan^2(\theta)bh}\frac{1}{f^3} \tag{6}$$

where the parameter f reflects the effect of the size of the plastic zone. When it is taken as 1, the density of the GNDs is that given in the model of Nix and Gao [9]. In the work of Durst et al. [34], it is taken as 1.9. According to Nix and Gao [9], the flow stresses in the absence of the GNDs and in the presence of GNDs, respectively, are given by

$$\sigma_{\rm r,0} = \sqrt{3}G\alpha b\sqrt{\rho_{\rm s}} \tag{7a}$$

and

$$\sigma_{\rm r} = \sqrt{3}G\alpha b\sqrt{\rho_{\rm s} + \rho_{\rm GND,m}} \tag{7b}$$

where $\rho_{\rm s}$ is the density of the statistically stored dislocations (SSDs) and G represents the shear modulus. α is a constant. Using the relation between the flow stress and the nominal hardness as given by Eq. (1) instead of Tabor's relation (Eq. (4)) as applied in the model of Nix and Gao [9], we obtain from Eq. (7)

$$\frac{H_{\rm n}}{H_{\rm n}^0} = \sqrt{1 + \frac{\rho_{\rm GND,m}}{\rho_{\rm s}}} \tag{8}$$

where $H_{\rm n}^0$ is the nominal hardness at the macroscale. Further, using Eqs. (6) and (7a), the following indentation nominal hardness–depth relation at the microscale can be obtained from Eq. (8)

$$\frac{H_{\rm n}}{H_{\rm n}^0} = \sqrt{1 + \frac{h^*}{h}} \tag{9}$$

where h^* is the material characteristic length and is given by

$$h^* = 87\frac{b\alpha^2}{\tan^2(\theta)}\frac{1}{f^3}\left(\frac{G}{H_{\rm n}^0}\right)^2 \tag{10}$$

Besides the procedure above, the nominal hardness–depth relation may also be derived from the work of Bucaille et al. [26]. In their work, it is assumed that the ratio of the contact depth to the indentation depth is a constant, and the following relation is obtained by using the model of Nix and Gao [9]:

$$P = C_0 h^2 \sqrt{1 + \frac{h^*_{NG}}{h}} \tag{11}$$

where C_0 is the indentation loading curvature at the macroscale and C_0h^2 is the corresponding indentation load. h^*_{NG} is the material characteristic length defined in the model of Nix and Gao [9], i.e.

$$h^*_{NG} = \frac{81}{2}\frac{b\alpha^2}{\tan^2(\theta)}\left(\frac{G}{H^0}\right)^2 \tag{12}$$

Unlike the definition given by Eq. (10), h^*_{NG} defined in the work of Nix and Gao [9] depends on the hardness at the macroscale H^0. Dividing Eq. (12) with $\pi(\tan(\theta)h)^2$ and according to the definition of the nominal hardness given by Eq. (3), we have

$$H_n = H_n^0 \sqrt{1 + \frac{h^*_{NG}}{h}} \tag{13}$$

which has the same form as Eq. (9) and the difference lies in the definition of the material characteristic length as given by Eqs. (10) and (12), respectively.

Eq. (9) has the same form as the model of Nix and Gao [9], with only the hardness in their model replaced by the nominal hardness defined by Eq. (3). However, the accurate measurement of the projected contact area in the use of the model of Nix and Gao [9] may be difficult. Therefore, Eqs. (1) and (9) may provide an easier way to estimate the material characteristic length and the representative stress in the absence of the effect of GNDs. Eq. (1) is supported by the systematic finite element analysis [29–31]; in the present paper, verification of Eq. (9) using the mechanism-based strain gradient (MSG) theory [11] and the experiments performed by Durst et al. [35] has been carried out. Using the MSG theory, Huang et al. [13] have simulated the microindentation test performed by McElhaney et al. [3]. The detail procedure for the simulations can be found in Ref. [13]. Using the indentation loading curve from the simulations in Ref. [13] for the polycrystalline copper, the depth dependence nominal hardness is obtained and plotted in Figure 3. Fitting the computational data using Eq. (9) gives the nominal hardness at the macroscale $H_n^0 = 837$ MPa and $h^* = 0.503$ μm. Further, taking shear modulus $G = 40$ GPa, Burgers vector $b = 0.255$ nm, half-apex angle $\theta = 72°$ and $f = 1.0$, the Taylor's constant α determined using Eq. (10) is 0.3. It is noted that the computational data in Figure 3 from the simulations of Huang et al. [13] corresponds to $\alpha = 0.7$. The difference between the Taylor's empirical constant α obtained from the present model and that from the work of Huang et al. [13] may be explained by considering the effect of the plastic zone size (PZS). As mentioned above, Durst et al. [34] has addressed the effect of the PZS and suggested a parameter f larger than 1.0. In the study of Huang et al. [13], the effect of PZS has been included in the simulation. If α is taken as 0.7, fitting the simulation results in Figure 3 using Eq. (9) gives the parameter $f = 1.74$, which is near to

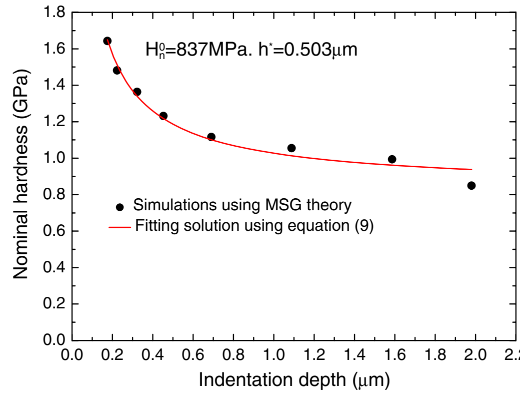

Figure 3. Depth-dependent nominal hardness from the MSG theory [13] and the model given by Eq. (9).

the value of 1.9 as used in the work of Durst et al. [34]. In other words, if, in the model given by Eq. (9), the effect of PZS is included by taking $f = 1.74$, $\alpha = 0.7$ will lead to a good agreement with the simulation result of Huang et al. [13]. In this sense, the predicted result using the model given by Eq. (9) is indeed consistent with that from the simulation based on the MSG plasticity [13]. However, it should be noted that $\alpha = 0.7$ is beyond the general range of 0.1–0.5; this might be due to the effects of mechanical polishing on the experiments of McElhaney et al. [3] as argued by Durst et al. [34]. Bearing this point in mind, we further verify the model by using the experimental data in the work of Durst et al. [35] for Ni SX(100), NiFe28 and tungsten. Using the indentation loading curves in their experiments, the nominal hardness was determined at different indentation depths as given by the points in Figure 4. The solid lines in Figure 4 give the fitting results using Eq. (9). The results show that indeed the model matches the experiments remarkably well and the material characteristic lengths (as shown in the figure) are reasonable when compared with the results reported in Ref. [35].

We now discuss the limitations of the results given by Eqs. (1) and (9). First, the model is limited to the microindentation and the cases in which the indenter can be assumed to be ideally sharp. Second, according to the results in Refs. [29,30], Eq. (1) requires a large ratio of the Young's modulus to the representative stress; for example, it should be larger than 500 for $\theta = 70.3°$. The study of Cao and Lu [27] shows that Eq. (9) may be invalid if the ratio of the Young's modulus to the representative stress is smaller than a certain value.

Thus, it may be concluded that Eqs. (1) and (9) as reported in the present work may only be applicable to highly plastic metals. This conclusion could apply to Eq. (13), which is obtained from the model of Bucaille et al. [26], because the assumption in their study, i.e., the ratio of the contact depth to the indentation depth is constant, is only valid for the highly plastic metals. Although the present results are limited to highly plastic materials, such a property range still includes many important engineering metals, such as some steels, copper, silver and gold [36]. Moreover, the highly plastic metals have larger ratios of the shear modulus to the hardness or nominal hardness; according to the analysis

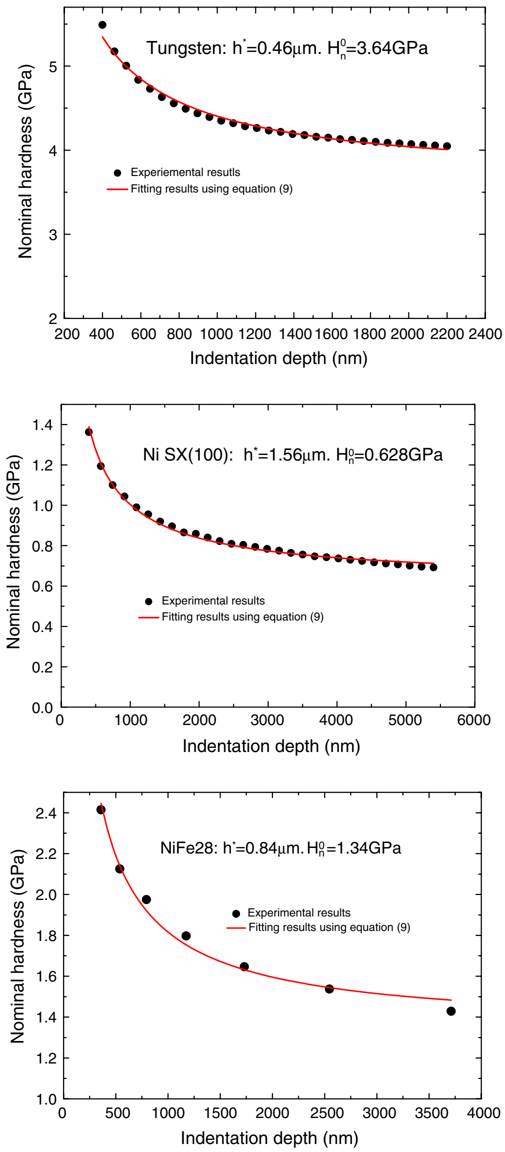

Figure 4. Depth-dependent nominal hardness from the experiment of Durst et al. [35] and the model given by Eq. (9).

of the Nix and Gao [9] and also Eq. (10), the size effects due to GNDs will be more significant in these circumstances. Thus the development of a model to interpret the effect of GNDs on the indentation response of soft metals is particularly necessary and important.

We reveal a simple relation between the nominal hardness and the flow stress corresponding to the representative strain given by Figure 2 in the present work. Using this relation instead of the Tabor's relation and the relation between the flow stresses and the density of dislocations as applied in the work of Nix and Gao [9], a model to predict the depth-dependent nominal hardness is proposed. The model agrees remarkably well with the experiment in the literature and the finite element analysis based on the mechanism-based strain gradient plasticity [13]. The results reported herein may help us to understand the correlation between the directly measurable quantities in the sharp indentation into soft metals and the material properties (e.g. the flow stress) at the macro or microscale.

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