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Size-dependent sharp indentation—I: a closed-form expression of the indentation loading curve

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Abstract

In this paper, a closed-form expression of the size-dependent sharp indentation loading curve has been proposed based on dimensional analysis and the finite deformation Taylor-based nonlocal theory (TNT) of plasticity (Int. J. Plasticity 20 (2004) 831). The key issue is to link the results of FEM based on TNT plasticity with those obtained using conventional FEM by taking as the effective strain gradient, η, that presented in the work of Nix and Gao (J. Mech. Phys. Solids 46 (1998) 411), thus avoiding large-scale finite element computations using strain gradient plasticity theories. Two experiments carried out on 316 stainless-steel and pure titanium have been used to verify the effectiveness of the present analytical model; the results demonstrate that the present analytical expression of the size-dependent indentation loading curve corresponds very well to the experimental indentation loading curve. The empirical constant, α, in the Taylor model estimated from the experimental data has the correct order of magnitude. Also, the results presented in this part can be further applied to establish an analytical framework to extract the plastic properties of metallic materials with sharp indentation on a small scale where the size effect caused by geometrically necessary dislocations is significant. This will be discussed in detail in the second part of the paper.

Keywords: Taylor-based nonlocal theory of plasticity; Dimensional analysis; Closed-form expression; Size-dependent indentation loading curve; Plastic properties

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1. Introduction

Many experiments have demonstrated that metallic materials have significant size effects when the characteristic length associated with nonuniform plastic deformation is at micron scale. Here we mainly refer to the size effects observed in nano or micro-indentation tests (Fleck et al., 1994; Stelmashenko et al., 1993; De Guzman et al., 1993; Ma and Clarke, 1995; Nix and Gao, 1998; Elmustafa and Stone, 2003; Zhao et al., 2003), based on the consideration that much effort has been devoted recently to the development of systematic methods to extract the mechanical properties of materials with depth-sensing instrumented indentation. Without considering size effects, much work has been presented to extract material properties by means of micro-indentation tests. For single indenter algorithms, Giannakopoulos and Suresh (1999) have proposed a systematic framework to obtain elastic–plastic properties within the context of small-strain finite element analysis. Since then, more comprehensive work has been carried out by Dao et al. (2001) based on dimensional analysis and large deformation FEM. Although in the case of many engineering metals, a single set of plastic properties can be extracted from single indenter algorithms, the results were found to be sensitive to small experimental errors. More recently, this problem has been thoroughly explored by Capehart and Cheng (2003). Their results show that 1% noise levels preclude the accuracy of the plastic properties identified, such as the strain hardening exponent. Based on the fact that the plastic properties extracted from a single P–h curve are sensitive to small experimental errors, two comprehensive studies have been carried out recently (Bucaille et al., 2003; Chollacoop et al., 2003). Dual sharp indenter algorithms were devised to improve the accuracy of the identified results. The authors (Cao and Lu, 2004) have studied the stability of the dual indenter algorithms by introducing the concept of the condition number. In that article, ill-conditioned cases in the inverse problem are examined and corresponding regularization schemes proposed. Since the selection of tip apex angles can change the properties of the sensitivity matrix in the inverse problem, guidelines have been developed for optimal combinations of tip apex angles and verified by means of numerical examples.

It should be emphasized that when using dual sharp indenter algorithms to extract the plastic properties of materials, all that are needed are the Young's modulus and the indentation loading curves which are the basis of reverse algorithms. During the loading procedure, the p–h response of a homogeneous elastic–plastic material to sharp indentation generally obeys the following relationship which was found to be a natural outcome of dimensional analysis (Cheng and Cheng, 1998a, b).

$$p = Ch^2, \tag{1}$$

where C is the loading curvature, constant for given material properties and independent of the indentation depth.

However, Eq. (1) will not be true if the size effects are significant. Here, we will mainly discuss the size effect induced by geometrically necessary dislocations (GND) (Stelmashenko et al., 1993; De Guzman et al., 1993; Ma and Clarke, 1995; Nix and Gao, 1998). Dao et al. (2001) have suggested a large indentation depth to exclude this effect. However, in practice, the indentation depth is limited by many factors such as the sample size and the device itself and cannot be arbitrarily large; on the other hand, in order to exclude the size effect, the indentation depth should be much larger than the material length scale which reflects the effect of GND, but it is difficult to present a guideline for determining the proper indentation depth since the material length scale is material dependent, and generally varies from hundreds of nanometers to several micrometers (Nix and Gao, 1998). Bucaille et al. (2004) adopted second-order polynomials to approximate the indentation loading curve in order to take into account the effect of GND, but the relationship between the GND and the polynomial coefficients was not clear. Further research was therefore needed to interpret the size-dependent indentation loading curve.

Based on the above premise, in this paper, a closed-form expression of the size-dependent indentation loading curve has been proposed in which a strain-independent material length scale was included to express the effect of geometrically necessary dislocations. The present paper is organized as follows. In Section 2, by using dimensional analysis and the finite deformation Taylor-based nonlocal theory of plasticity (Hwang et al., 2004) which is an extension of the work of Gao and Huang (2001), we have proposed an analytical model for predicting the size-dependent indentation loading curve by linking the results of FEM based on the nonlocal theory of plasticity with those obtained using conventional FEM. To achieve this, the effective strain gradient, η, suggested in the work of Nix and Gao (1998) has been applied here. In Section 3, a comparison of the present analytical model with other work has been carried out to prove the effectiveness of the present work. Section 4 contains two experiments carried out on 316 stainless-steel and pure titanium, respectively, to further verify the present model. In Section 5, the main contributions made by the present work have been summarized.

2. Model

Dimensional analysis is a useful tool which has been successfully used to analyze the indentation response. The most representative work was published by Cheng and Cheng (1998a, b): based on dimensional analysis and FEM, they have presented several scaling relationships which provide new insight into the shape of indentation curves. They are also helpful as a guide to the FE analysis of conical indentations. Since then, single indenter algorithms (Dao et al., 2001) and dual indenter algorithms (Bucaille et al., 2003; Chollacoop et al., 2003) to extract the mechanical properties of materials have also been based on dimensional analysis and large deformation FEM. Our discussion also begins with dimensional analysis. Unlike in the previous work (Cheng and Cheng, 1998a,b; Dao et al., 2001), the material length scale (Nix and Gao, 1998) has been included in the relationship to reflect the size effect observed in micro or nano-indentation.

For conical indentation in a power-law material, in loading procedure, the indentation load must be a function of the following independent parameters: the Young's modulus, E, and Poisson's ratio, ν, of the elastic–plastic solid, the Young's modulus, E_i, and Poisson's ratio, v_i, of the elastic indenter, the yield strength $\sigma_{y,0}$ in the absence of a strain gradient, the strain hardening exponent n, the indentation depth h, the material length scale l and the tip apex angle of the indenter θ.

$$p = f(E, v, E_i, v_i, \sigma_{y,0}, n, \theta, h, l). \tag{2}$$

Using the reduced Young's modulus (Johnson, 1985). Eq. (2) can be reduced to

$$p = f(E^*, \sigma_{y,0}, n, h, l, \theta), \tag{3}$$

where the reduced Young's modulus is

$$\frac{1}{E^*} = \frac{1 - v^2}{E} + \frac{1 - v_i^2}{E_i}. \tag{4}$$

Alternatively, Eq. (3) can be given by

$$p = f(E^*, \sigma_{r,0}, n, h, l, \theta), \tag{5}$$

where the representative stress $\sigma_{r,0}$ in the absence of strain gradient (Dao et al., 2001) is

$$\sigma_{r,0} = \sigma_{y,0}\left(1 + \frac{E}{\sigma_{y,0}}\,\varepsilon_r\right)^n, \tag{6}$$

where ε_r is the total effective strain accumulated beyond the yield strain, see the work of Dao et al. (2001) for its definition in detail.

The Π theorem (Barenblatt, 1996) is a key theorem in dimensional analysis, which describes how every physically meaningful equation involving k variables can be equivalently rewritten as an equation of k–m dimensionless parameters, where m is the number of fundamental units used. Furthermore, and most importantly, it provides a method for computing these dimensionless parameters from the given variables, even if the form of the equation is still unknown. Here, by applying the Π theorem, Eq. (5) can be expressed as

$$p = \sigma_{r,0} h^2 \Pi_1\left(\frac{E^*}{\sigma_{r,0}}, n, \frac{l}{h}, \theta\right). \tag{7}$$

From Eq. (7), it can be found that the parameter, l/h, should be contained in the expression of the indentation loading curve which reflects the effect of GND.

In general, to determine the closed-form expression of Eq. (7), FEM based on the higher order theories (Begley and Hutchinson, 1998; Gao et al., 1999; Huang et al., 2000) or other size-dependent plasticity models (Acharya and Bassani, 2000; Gao and Huang, 2001; Evers et al., 2002) should be used. A lack of general purpose FEM software for such theories makes it difficult to carry out corresponding large-scale numerical simulations. Here we will propose a new method to evaluate Eq. (7), aimed at avoiding large-scale finite element computations based on strain gradient plasticity.

Gao and Huang (2001) have proposed a Taylor-based nonlocal theory of plasticity to account for the size dependence of plastic deformation at micron and submicron length scales. Their further analysis showed that for indentation problems, there are only negligible differences between the TNT and MSG (Gao et al., 1999; Huang et al., 2000) theories for the prediction of experimentally measurable quantities. However, the constitutive equations of TNT plasticity are similar to the classical plasticity theories (Hill, 1950), which are significantly simpler than high-order theories such as the MSG theory. As a result, in this article, in order to determine the closed-form of Eq. (7), we invoke the Taylor-based nonlocal theory of plasticity. For simulation of the indentation problems, large deformation FEM based on the flow theory of TNT plasticity should be used. Unfortunately, to the authors' knowledge, the use of this type of method has not yet been explored. For simplicity's sake, the deformation theory of strain gradient plasticity has been widely applied to the analysis of indentation problems (see Shu and Fleck (1998) and Begley and Hutchinson (1998) using the infinitesimal strain assumption and Hwang et al. (2002) using the finite deformation MSG theory). Also, based on numerical results, Guo et al. (2001) have found that, in the case of micro-indentation with monotonic loading, the difference between the results obtained using the TNT deformation theory and those given by the flow theory of TNT plasticity is rather small. Similar conclusions have also been drawn from results based on the MSG theory (Qiu et al., 2003). Consequently, the deformation theory of TNT plasticity has been applied here to evaluate the explicit expression of Eq. (7).

The deformation theory of TNT plasticity retains the same structure as the classical plasticity theory. The constitutive equation based on the infinitesimal strain assumption has been presented by Gao and Huang (2001). For indentation problems, it is anticipated that strains near the indenter tip will be relatively large. The effect of finite deformation should therefore be considered. Recently, Hwang et al. (2004) have presented the finite deformation theory of TNT plasticity. It can be found from their work that, except for the yield function given as follows which is specific to TNT plasticity, all the other governing equations and boundary conditions are identical to those of the classical plasticity theory.

$$\tau_{\mathrm{eq}} = \sigma(\varepsilon, \eta), \tag{8}$$

where τ_{eq} is the effective stress in the current configuration (see Hwang et al., 2004, for a more detailed definition) and the flow stress is given by

$$\sigma = \sigma_{\mathrm{ref}}\sqrt{f^2(\varepsilon) + l\eta}, \tag{9}$$

where σ_{ref} is the reference stress, η is the effective strain gradient whose detailed definition can be found in the work of Gao and Huang (2001) or Hwang et al. (2004), and the material length scale l is expressed according to the shear modulus μ and Burgers vector b by

$$l = 18\alpha^2(\mu/\sigma_{\mathrm{ref}})^2 b \tag{10}$$

α in Eq. (10) is an empirical material constant.

Unlike in the classical plasticity theory, in TNT plasticity, a representative cell needs to be applied to evaluate the effective strain gradient. When using FEM based on TNT plasticity to calculate the effective strain gradient, Gaussian integration should be carried out at the mesoscale cell level, which is specific to TNT plasticity (Guo et al., 2001) and leads to general purpose software such as ABAQUS becoming ineffective and requiring the development of a special FEM program. In this paper, in order to avoid large-scale finite element computations using strain gradient plasticity theories, we have proposed a method of directly linking the results obtained using FEM based on TNT plasticity to those obtained using conventional FEM, by taking as the effective strain gradient, η, that presented in the work of Nix and Gao (1998), i.e.

$$\eta = \frac{1}{h\tan^2(\theta)}. \tag{11}$$

At a given indentation depth and a given tip apex angle, Eq. (11) shows that the effective strain gradient is constant.

Remark. It should be pointed out that, for conical indentation problems, the strain gradient under the indenter might not be constant in general. The assumption of the constant effective strain gradient given by Eq. (11) is in an average sense. Taking into consideration that, same as the hardness discussed in previous work (Nix and Gao, 1998), the total force acting on the indenter and the displacement of the indenter involved in the present work are not localized variables, e.g. stresses or strains which are heavily dependent on the local distribution of the strain gradient, therefore, it is possible to obtain a reasonable analytical model by using the effective strain gradient shown as Eq. (11). Moreover, the results in the work of Nix and Gao (1998) show that the depth dependence of hardness predicted by their model is in excellent agreement with indentation tests. Later, many other experiments (Abu Al-Rub and Voyiadjis, 2004; Elmustafa and Stone, 2002, 2003; Liu and Ngan, 2001; Lou et al., 2003; Mirshams and Parakala, 2004; Rodriguez and Gutierrez, 2003; Swadener et al., 2002; Yuan and Chen, 2001) also prove that Nix and Gao's model (1998) is effective in the analysis of size-dependent indentation problems for a wide range of metal materials at a wide range of indentation depth. On the other hand, at present, to the best of the authors' knowledge, no rigorous proof in theory has been proposed for the actual distribution of the effective strain gradient under the indenter; it is a very complicated problem. Recently, Zhao et al. (2003) have presented a new strain gradient plasticity model to characterize depth dependence of hardness; in their work, the strain gradient is assumed to be nonconstant and explicitly dependent on the position under the indenter. Experimental results have showed the effectiveness of their model. As mentioned in their analysis (Zhao et al., 2003), the effective strain gradient applied in their work makes some approximation to the real situation and may be appropriate to very small deformation levels. However, we emphasize here that the effective strain gradient used in their work (Zhao et al., 2003) is also an assumption; its effectiveness is verified by experiments. Based on the consideration above, in our present work, also for the size-dependent conical indentation problems, we have applied the effective strain gradient (constant at a given indentation depth) suggested by Nix and Gao (1998) to propose a new analytical model to interpret size-dependent conical indentation. A comparison with other work and corresponding experiments will be carried out in the sequel to prove the effectiveness of the application of the effective strain gradient in such a manner.

If the shear modulus, the constant parameter, α, in the Taylor relationship and the Burgers vector are known, using Eq. (11) and for given indentation depth and tip apex angle the yield criterion expressed by Eq. (8) can be rewritten as

$$\tau_{\mathrm{eq}} = \sigma_{\mathrm{ref}} \sqrt{f^2(\varepsilon) + \frac{l}{h \tan^2(\theta)}} = g(\sigma_{\mathrm{ref}}, \varepsilon). \tag{12}$$

By replacing Eq. (8) in the work of Hwang et al. (2004) with Eq. (12), it can be found that the constitutive law of the classical deformation plasticity theory has been respected once again. When using the finite deformation theory of plasticity, the yield function of which is given by Eq. (12) to evaluate the indentation loading curve given by Eq. (7), the computational procedure is, for a given indentation depth (or contact radius), to find a solution which satisfies all the relevant equations and boundary conditions; the result is assumed to be only dependent on the final status, and independent of the loading procedure. FEM can be used to evaluate the above solution. Here, we have considered power-law materials for which the function, f, in Eq. (12) is given by

$$f(\varepsilon) = (\varepsilon_y + \varepsilon_{\mathrm{r}})^n, \tag{13}$$

where ε_y is the strain corresponding to the initial yield stress in the stress–strain curve and n is the strain hardening exponent. Here it varies from 0 to 0.5.

Inserting Eq. (13) into (12), we obtain

$$\tau_{\mathrm{eq}} = \sigma_{\mathrm{ref}} \sqrt{(\varepsilon_y + \varepsilon_{\mathrm{r}})^{2n} + \frac{l}{h \tan^2(\theta)}}. \tag{14}$$

Fitting Eq. (14) with the power function leads to

$$\tau_{\mathrm{eq}} = \sigma_{\mathrm{ref}} \sqrt{(\varepsilon_y + \varepsilon_{\mathrm{r}})^{2n} + \frac{l}{h \tan^2(\theta)}} \approx \sigma'_{\mathrm{ref}} (\varepsilon'_y + \varepsilon'_{\mathrm{r}})^{n'}. \tag{15}$$

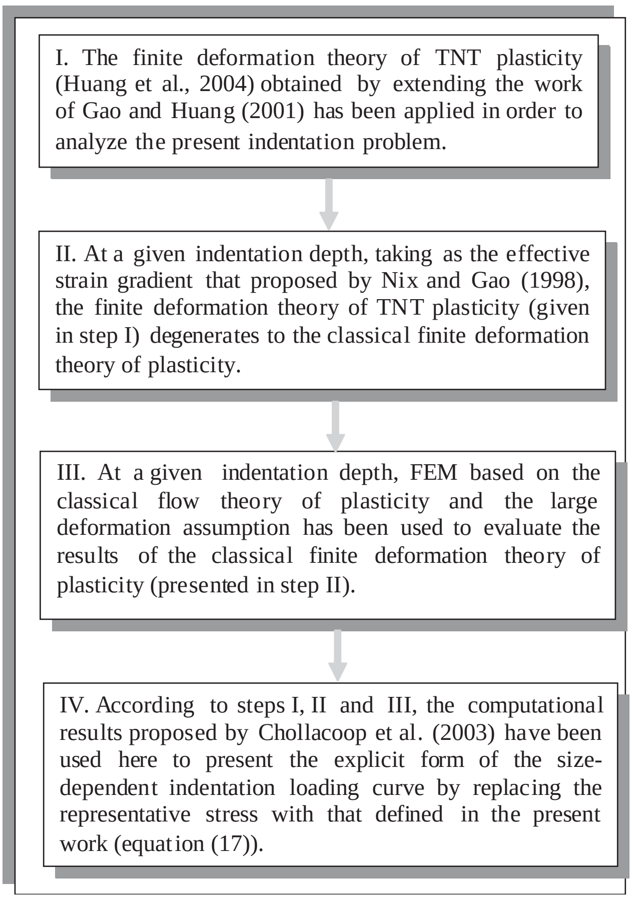
At a given indentation depth, conventional FEM for power law materials can be used to evaluate the results produced by the finite deformation plasticity theory, with the yield function being that given in Eq. (15) above and all the other relevant equations being the same as those in the classical plasticity theory. Here we have invoked the flow theory, based on the large deformation assumption included in the general purpose commercial software, ABAQUS, to evaluate the results of the finite deformation plasticity theory discussed above. The advantage of using conventional FEM to analyze the problem is that the results established in the previous work can be directly used here. In the present work, the results of FEM computations, based on the large deformation assumption made by Chollacoop et al. (2003) for 76 different combinations of elastic–plastic properties representing common engineering metals for each tip geometry, has been applied to produce the closed-form of

Eq. (7), which is given by

$$p = \sigma_r h^2 \Pi_1 = \sigma_r h^2 \left(\phi_3(\theta) \ln^3\left(\frac{E^*}{\sigma_r}\right) + \phi_2(\theta) \ln^2\left(\frac{E^*}{\sigma_r}\right) + \phi_1(\theta) \ln\left(\frac{E^*}{\sigma_r}\right) + \phi_0(\theta) \right). \tag{16}$$

Further details on $\phi_0, \phi_1, \phi_2, \phi_3$ are to be found in the work of Chollacoop et al. (2003). It can be seen that, in the present work, the parameter, l/h, in Eq. (7) which expresses the effect of GND, has been included in the representative stress, σ_r, in Eq. (16), given by

$$\sigma_r = \sigma'_{ref}(\varepsilon'_y + \varepsilon'_r)^{n'} \approx \sigma_{ref}\sqrt{(\varepsilon_y + \varepsilon_r)^{2n} + \frac{l}{h\tan^2(\theta)}}, \tag{17}$$



Flowchart 1. Key steps in the present analytical model.

here for simplicity's sake, we take $\varepsilon_y' = \varepsilon_y$. And the representative strain ε_r is a function of the tip apex angle (Chollacoop et al., 2003), which can be used to normalize the indentation loading curvature independently of the material hardening exponent for a wide range of material properties. For a Berkovich indenter with an equivalent conical tip apex angle of $\theta = 70.3^\circ$, the representative strain is $\varepsilon_r = 0.033$ (see Dao et al. (2001) for further details).

Remark. A closed-form expression of the indentation loading curve with a size effect induced by GND has been proposed in this section. The key notion is that the representative stress defined in the work of Chollacoop et al. (2003) has been replaced with that given in Eq. (17) in which the material length scale has been included. Below is a flowchart (Flowchart 1) to explain the key idea behind the present method. From the flowchart, it can be seen that the advantages of the present method are twofold; first, finite element computations using strain gradient plasticity theories have been avoided and second, the previous analytical results (Dao et al., 2001; Chollacoop et al., 2003) can be directly used to present the closed-form expression of the size-dependent indentation loading curve.

3. Comparison with other work

Taking into consideration that our work has been heavily based on the model of Nix and Gao (1998) and the finite deformation TNT plasticity (Hwang et al., 2004), it will be very useful to compare the results of the present model with that of their work. In order to verify the effectiveness of the finite element formulations based on finite deformation TNT plasticity, Hwang et al. (2004) have presented a numerical example, simulating a micro-indentation hardness experiment. Their results showed that the square of the relative indentation hardness (H/H_0) is linearly dependent on the inverse of the indentation depth, $1/h$, fitting the experimental result very well, and the empirical constant, α, in the Taylor model has the correct order of magnitude. At this point, the performance of their method is consistent with that of Nix and Gao's model (1998). Therefore, in our work we directly compared the present model with the model of Nix and Gao (1998) in a general sense.

Dao et al. (2001) have argued that for most engineering metals the hardness based on large deformation FEM using a standard Berkovich indenter has the following relationship with the representative stress, $\sigma_{0.082}$ (evaluated by means of the representative strain, $\varepsilon_r = 0.082$):

$$H = 2.70\sigma_{0.082}. \tag{18}$$

Therefore, using Eq. (18) and the representative stress defined in Eq. (17), the hardness predicted with the present model (Flowchart 1, i.e. at a given depth, conventional FEM is used to replace that based on TNT plasticity by taking Eq. (15) to be the yield criterion) will also be depth-dependent and consistent with that predicted using the model of Nix and Gao (1998), which is a natural outcome since the effective strain gradient described in their work has been used here.

4. Experimental verification

The present computational model is based on the finite deformation plasticity theory, while for the analysis of indentation problems, FEM based on the flow theory of plasticity and the large deformation assumption is the most appropriate selection. Although the hardness predicted using the present model is expected to be consistent with that produced by the model of Nix and Gao (1998), further indentation experiments need to be carried out to verify the effectiveness of the application of the effective strain gradient given by Eq. (11) and the explicit form of the indentation loading curve given in Eq. (16). First, tension tests using a UTS machine were performed on 316 stainless-steel and pure titanium to obtain the stress–strain relationships of the two materials. The diagram of the specimen is given by Fig. 1. For each material, experiments were carried out on five samples. The true stress–logarithmic strain curves based on the average tension curves for the two materials have been plotted in Figs. 2 and 3. By approximating the stress–strain relationship with a power law description, the reference stresses and strain hardening exponents for the two materials can be obtained. Nanoindentation tests were then carried out on an MTS XP nano-indenter. A standard diamond Berkovich indenter with an equivalent tip apex angel of $\theta = 70.3^{\circ}$ was used. A loading rate such that $\dot{\varepsilon} \sim \dot{p}/p = 0.05\,\mathrm{s}^{-1}$ was selected to approximate a constant strain rate. The indentation depth was large enough compared with the tip defect for the geometrical sensitivity to be eliminated. Bucaille et al. (2003) have demonstrated that, for a Berkovich indenter with an equivalent tip apex angle of $\theta = 70.3^{\circ}$, the effect of friction on the indentation loading curve can be ignored. This conclusion can also be drawn from Table 1 in the work of Mata and Alcala (2004). The surface was ground with SiC paper until the grain size was about 15 μm. Then the samples were polished with 6 and 3 μm diamond suspension for 5 min and 0.05 μm alumina suspension for 20 s. Although the effects of a deformation layer due to mechanical polishing still exist, compared with results based on further chemically polished sample, the effects of surface layer are limited to the indentation depth lower than 200 nm for the two materials studied here. Therefore, in the present work, the indentation data at the indentation depths lower than 200 nm have been eliminated. Based on the above considerations, we have attributed the indentation size-effect to the presence of the GND. For each material, six measurements were made. Figs. 4 and 5 give the indentation loading curves for 316 stainless-steel and pure titanium, respectively. The values of σ_{ref} and ε_y and n in Eq. (17) can be determined from Figs. 2 and 3; they

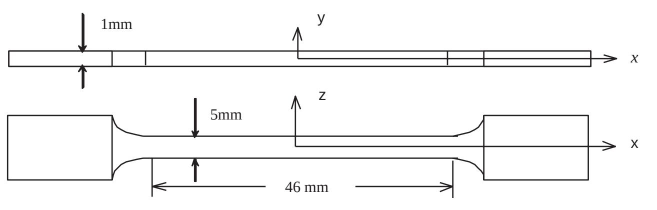


Fig. 1. Schematic of the tension sample.

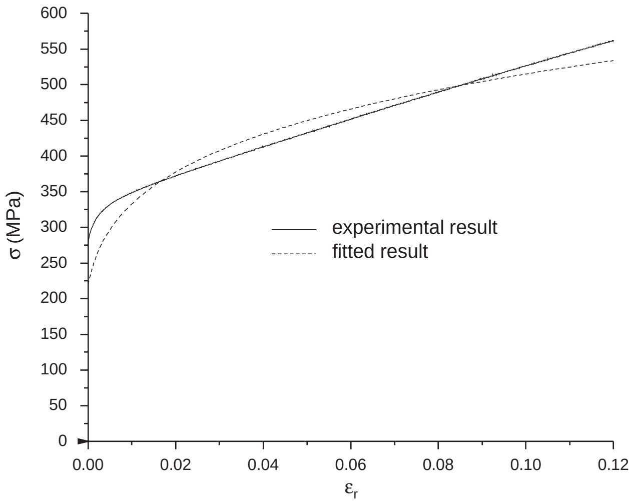


Fig. 2. Plot of the stress–strain relationship of 316 stainless-steel.

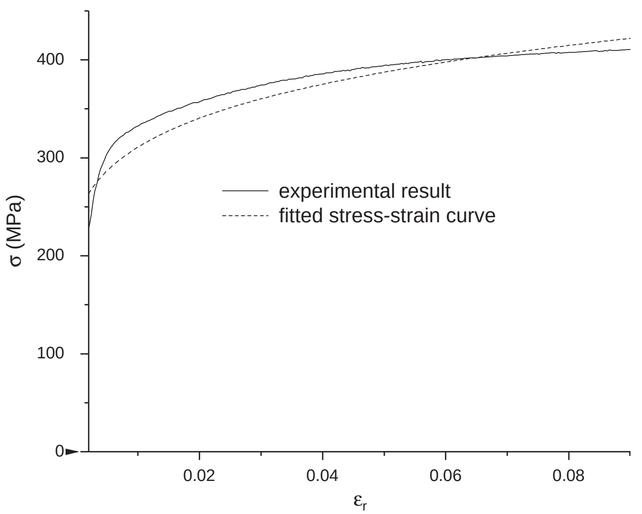


Fig. 3. Plot of the stress–strain relationship of pure titanium.

are $\sigma_{\mathrm{ref}}^{\mathrm{steel}} = 813.9\,\mathrm{MPa}$, $\varepsilon_{y}^{\mathrm{steel}} = 0.00147$, $n_{\mathrm{steel}} = 0.2$ and $\sigma_{\mathrm{ref}}^{\mathrm{Ti}} = 603.5\,\mathrm{MPa}$, $\varepsilon_{y}^{\mathrm{Ti}} = 0.002$, $n_{\mathrm{Ti}} = 0.15$. The Young's modulus was determined from the tension experiments as being $E_{\mathrm{steel}} = 200\,\mathrm{GPa}$ and $E_{\mathrm{Ti}} = 120\,\mathrm{GPa}$, respectively. The empirical constant, α, in the Taylor model was determined for 316 stainless-steel and pure titanium, respectively (see Figs. 4 and 5) by fitting the experimental indentation loading curves with Eq. (16) using the least-square method and taking a Burgers vector of

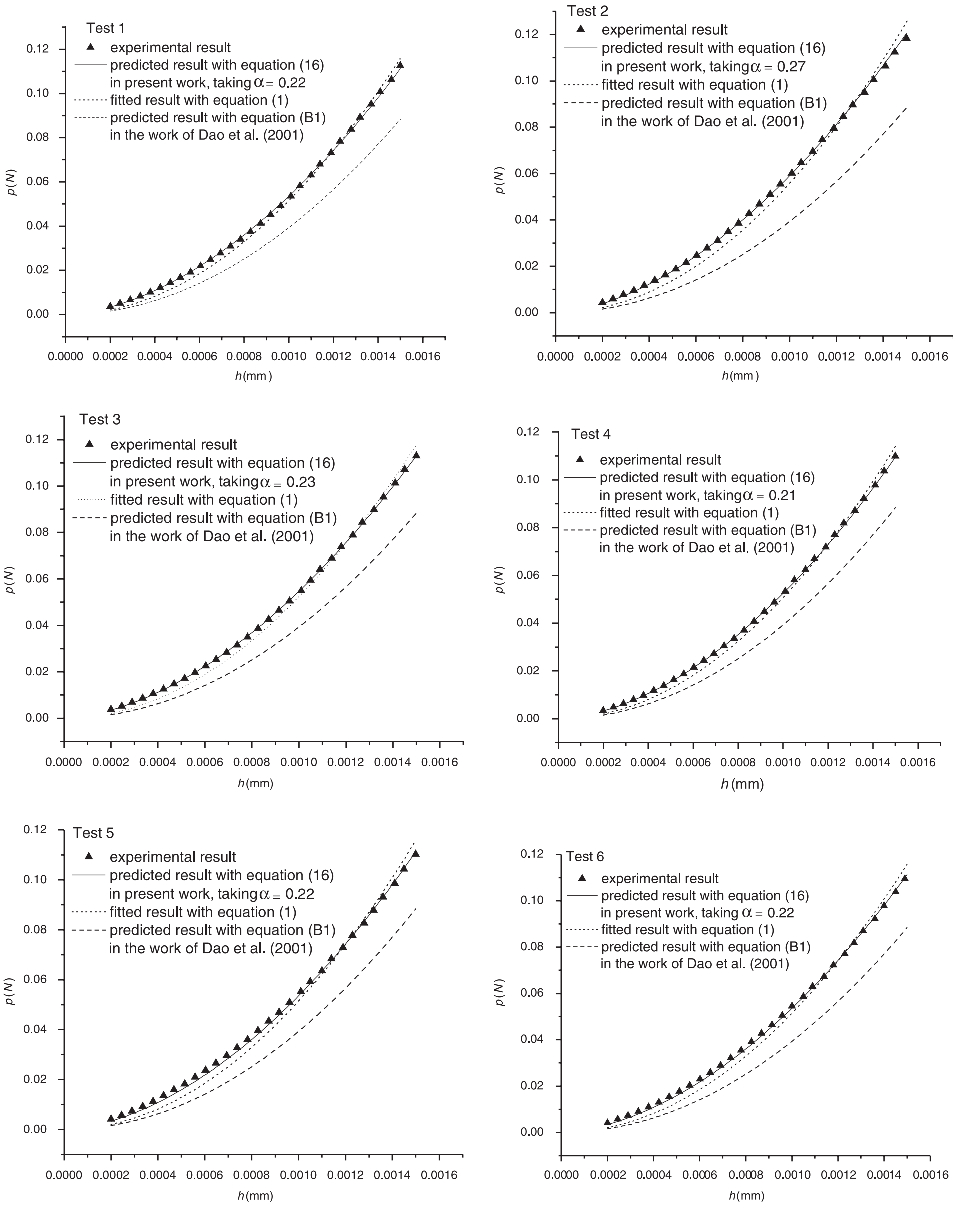


Fig. 4. Plot of the experimental p–h response and corresponding fitted results with the present closed-form expression of indentation loading curves and other equations (316 stainless-steel).

$b = 0.25\,\mathrm{nm}$. The average values of parameter α for the two types of materials studied here are $\alpha_{\mathrm{steel}} = 0.228$ and $\alpha_{\mathrm{Ti}} = 0.363$, which have the correct order of magnitude (between 0.1 and 0.5). Moreover, Figs. 4 and 5 also show that the

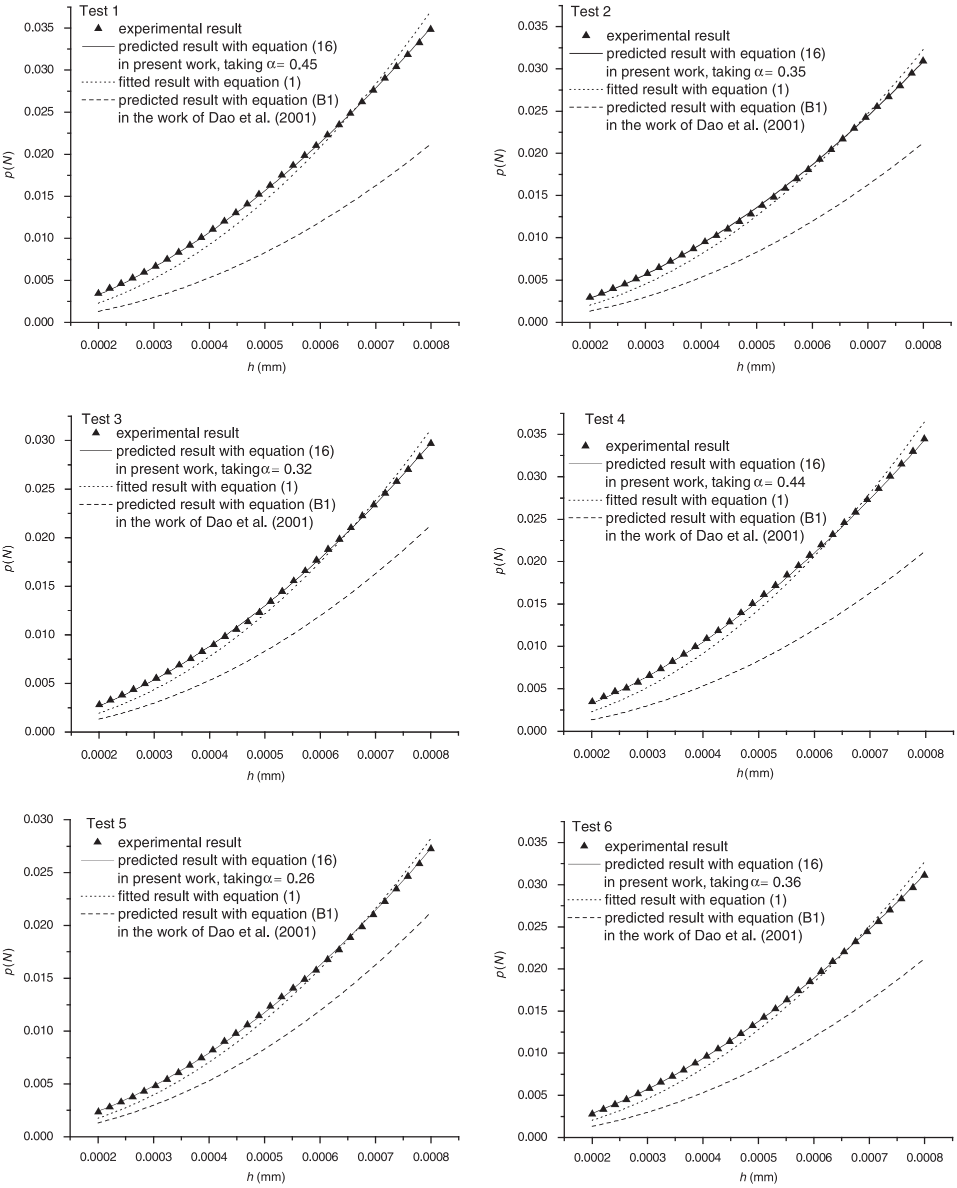


Fig. 5. Plot of the experimental p–h response and corresponding fitted results with the present closed-form expression of indentation loading curves and other equations (pure titanium).

proposed closed-form expression of the indentation loading curves correspond very well to the experimental curves. The results obtained by fitting the experimental indentation curve with a function in the form of Eq. (1) and the results predicted using Eq. (B1) in the work of Dao et al. (2001) are also given in Figs. 4 and 5. It can be seen that the deviation of the size-dependent indentation loading curve from the assumption made in Eq. (1) is significant. At the same time, the results predicted using the work of Dao et al. (2001) show that taking the size effect into consideration is both necessary and important for the maximum indentation depths discussed here.

5. Conclusions

Recently, there has been a mounting interest in the measurement of the mechanical properties of materials on small scales with depth-sensing instrumented indentation. In this work, we have proposed a new method to interpret the conical indentation loading curve including size effects induced by GND, which can be further used to establish an analytical framework to extract the plastic properties of materials from size-dependent indentation loading curves. Below is a summary of the contributions made in this part of the article:

(1) A closed-form expression of the size-dependent indentation loading curve has been proposed based on dimensional analysis and the finite deformation theory of strain gradient plasticity presented by Hwang et al. (2004) which is an extension of the work of Gao and Huang (2001). The key idea of the present work is to link the computational results of FEM based on TNT plasticity with that established using conventional FEM by taking as the effective strain gradient, η, that presented in the work of Nix and Gao (1998). Large-scale finite element computations using strain gradient plasticity theories have been avoided. Based on the present scheme, it is possible to work out another model by using the effective strain gradient presented in the work of Zhao et al. (2003), which will be discussed in our further research.

(2) A comparison with other work has been carried out. Further analysis reveals that hardness evaluated by the present computational model is consistent with the model of Nix and Gao (1998).

(3) Experiments carried out on 316 stainless-steel and pure titanium have been used to verify the effectiveness of the proposed analytical result. The results show that the present analytical expression of the size-dependent indentation loading curve corresponds very well to the experimental indentation loading curve. The empirical constant, α, in the Taylor model estimated from the experimental data has the correct order of magnitude.

(4) As a direct follow-up to the present work, an analytical framework can be established to extract the plastic properties of engineering metals from size-dependent indentation loading curves, which will be presented in Part II of this paper.

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References

Abu Al-Rub, R.K., Voyiadjis, G.Z., 2004. Analytical and experimental determination of the material intrinsic length scale of strain gradient plasticity theory from micro- and nano-indentation experiments. Int. J. Plasticity 20, 1139–1182.

Acharya, A., Bassani, J.L., 2000. Lattice incompatibility and a gradient theory of crystal plasticity. J. Mech. Phys. Solids 48, 1565–1595.

Barenblatt, G.I., 1996. Scaling, Self-Similarity, and Intermediate Asymptotics. Cambridge University Press, Cambridge, U.K.

Begley, M.R., Hutchinson, J.W., 1998. The mechanics of size-dependent indentation. J. Mech. Phys. Solids 46, 2049–2068.

Bucaille, J.L., Stauss, S., Felder, E., Michler, J., 2003. Determination of plastic properties of metals by instrumented indentation using different sharp indenters. Acta Mater. 51, 1663–1678.

Bucaille, J.L., Stauss, S., Schwaller, P., Micher, J., 2004. A new technique to determine the elastoplastic properties of thin metallic films using sharp indenters. Thin Solid Films 447–448, 239–245.

Cao, Y.P., Lu, J., 2004. Depth-sensing instrumented indentation with dual sharp indenters: stability analysis and corresponding regularization schemes. Acta Mater. 52, 1143–1153.

Capehart, T.W., Cheng, Y.T., 2003. Determining constitutive models from conical indentation: sensitivity analysis. J. Mater. Res. 18, 827–832.

Cheng, Y.T., Cheng, C.M., 1998b. Scaling approach to conical indentation in elastic–plastic solids with work hardening. J. Appl. Phys 84, 1284–1291.

Cheng, Y.T., Cheng, C.M., 1998b. Relationships between hardness, elastic modulus, and the work of indentation. Appl. Phys. Lett 73, 614–616.

Chollacoop, N., Dao, M., Suresh, S., 2003. Depth-sensing instrumented indentation with dual sharp indenters. Acta Mater. 51, 3713–3729.

Dao, M., Chollacoop, N., Van Vliet, K.J., Venkatesh, T.A., Suresh, S., 2001. Computational modelling of the forward and reverse problems in instrumented sharp indentation. Acta Mater. 49, 3899–3918.

De Guzman, M.S., Neubauer, G., Flinn, P., Nix, W.D., 1993. The role of indentation depth on the measured hardness of materials. Mater. Res. Symp. Proc. 308, 613–618.

Elmustafa, A.A., Stone, D.S., 2002. Indentation size effect in polycrystalline F.C.C. metals. Acta Mater. 50, 3641–3650.

Elmustafa, A.A., Stone, D.S., 2003. Nanoindentation and the indentation size effect: kinetics of deformation and strain gradient plasticity. J. Mech. Phys. Solids 51, 357–381.

Evers, L.P., Parks, D.M., Brekelmans, W.A.M., Geers, M.G.D., 2002. Crystal plasticity model with enhanced hardening by geometrically necessary dislocation accumulation. J. Mech. Phys. Solids 50, 2403–2424.

Fleck, N.A., Muller, G.M., Ashby, M.F., Hutchinson, J.W., 1994. Strain gradient plasticity: theory and experiment. Acta. Metall. Mater. 42, 475–487.

Gao, H., Huang, Y., 2001. Taylor-based nonlocal theory of plasticity. Int. J. Solids Struct. 38, 2615–2637.

Gao, H., Huang, Y., Nix, W.D., Hutchinson, J.W., 1999. Mechanism-based strain gradient plasticity—I. Theory. J. Mech. Phys. Solids 47, 1239–1263.

Giannakopoulos, A.E., Suresh, S., 1999. Determination of elastoplastic properties by instrumented sharp indentation. Scripta Mater. 40, 1191–1198.

Guo, Y., Huang, Y., Gao, H., Zhuang, Z., Hwang, K.C., 2001. Int. J. Solids Struct. 38, 7447–7460.

Hill, R., 1950. Mathematical Theory of Plasticity. Oxford University Press, Oxford, England.

Huang, Y., Gao, H., Nix, W.D., Hutchinson, J.W., 2000. Mechanism-based strain gradient plasticity—II. Analysis. J. Mech. Phys. Solids 48, 99–128.

Hwang, K.C., Jiang, H., Huang, Y., Gao, H., Hu, N., 2002. A finite deformation theory of strain gradient plasticity. J. Mech. Phys. Solids 50, 81–99.

Hwang, K.C., Guo, Y., Jiang, H., Huang, Y., Zhuang, Z., 2004. The finite deformation theory of Taylor-based nonlocal plasticity. Int. J. Plasticity 20, 831–839.

Johnson, K.L., 1985. Contact Mechanics. Cambridge University Press, London.

Liu, Y., Ngan, A.H.W., 2001. Depth dependence of hardness in copper single crystals measured by nanoindentation. Scripta Mater. 44, 237–241.

Lou, J., Shrotriya, P., Buchheit, T., Yang, D., Soboyejo, W.O., 2003. Nanoindentation study of plasticity length scale effects in lithographie, galvanoformung, abformung Ni microelectromechanical structures. J. Mater. Res. 18, 719–728.

Ma, Q., Clarke, D.R., 1995. Size dependent hardness in silver single crystals. J. Mater. Res. 10, 853–863.

Mata, M., Alcala, J., 2004. The role of friction on sharp indentation. J. Mech. Phys. Solids 52, 145–165.

Mirshams, R., Parakala, P., 2004. Nanoindentation of nanocrystalline Ni with geometrically different indenters. Materials Science and Engineering A 372, 252–260.

Nix, W.D., Gao, H., 1998. Indentation size effects in crystalline materials: a law for strain gradient plasticity. J. Mech. Phys. Solids 46, 411–425.

Qiu, X., Huang, Y., Wei, Y., Gao, H., Hwang, K.C., 2003. The flow theory of mechanism-based strain gradient plasticity. Mech. Mater. 35, 245–258.

Rodriguez, R., Gutierrez, I., 2003. Correlation between nanoindentation and tensile properties: influence of the indentation size effect. Mater. Sci. Eng. A 361, 377–384.

Shu, J.Y., Fleck, N.A., 1998. The prediction of a size effect in microindentation. Int. J. Solids Struct. 35, 1363–1383.

Stelmashenko, N.A., Walls, M.G., Brown, L.M., Milman, Y.V., 1993. Micro-indentation on W and Mo oriented single crystals: an STM study. Acta. Metall. Mater. 41, 2855–2865.

Swadener, J.G., George, E.P., Pharr, G.M., 2002. The correlation of the indentation size effect measured with indenters of various shapes. J. Mech. Phys. Solids 50, 681–694.

Yuan, H., Chen, J., 2001. Identification of the intrinsic material length in gradient plasticity theory from micro-indentation tests. Int. J. Solids Struct. 38, 8171–8187.

Zhao, M.H., Slaughter, W.S., Li, M., Mao, S.X., 2003. Material-length-scale-controlled nanoindentation size effects due to strain-gradient plasticity. Acta Mater. 51, 4461–4469.