



# Effect of grain size on the indentation hardness for polycrystalline materials by the modified strain gradient theory<sup>☆</sup>



Bong-bu Jung<sup>a</sup>, Hun-kee Lee<sup>b</sup>, Hyun-chul Park<sup>a,\*</sup>

<sup>a</sup> Department of Mechanical Engineering, POSTECH, Pohang, Gyeongbuk 790-784, Republic of Korea

<sup>b</sup> Department of Materials Science and Engineering, MIT, 77 Massachusetts Avenue, Cambridge, MA 02139-4307, USA

## ARTICLE INFO

### Article history:

Received 30 January 2013

Received in revised form 24 April 2013

Available online 15 May 2013

### Keywords:

Indentation test

GNDs

Indentation size effect

Grain boundary effect

## ABSTRACT

Numerous indentation tests in the micrometer and nanometer scale have shown that the measured hardness decreases significantly with increasing indentation depth, and this is known as the indentation size effect (ISE). However, several other nanoindentation results for polycrystalline materials show that the indentation hardness increases with increasing indentation depth because of the grain boundary (GB) effect. In this work, we propose a new model for the indentation test using the modified strain gradient plasticity theory. The GB effect is considered by evaluating the density of GNDs on the GB. Using the proposed model, the indentation hardness of polycrystalline materials in micrometer-scale structures is investigated, and compared with experimental results from other researchers.

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## 1. Introduction

Indentation tests are commonly used to measure the mechanical properties of thin films and small volumes because of their fast, convenient and nondestructive characteristics (Oliver and Pharr, 1992). However, indentation experiments exhibit the indentation size effect (ISE) for the micrometer and nanometer scale. Numerous indentation tests in the micrometer and nanometer scale have shown that the measured indentation hardness increases significantly with decreasing indentation depth (Abu Al-Rub and Voyiadis, 2004; Cao et al., 2006). The classical plasticity cannot predict the size effect because the constitutive equation of classical mechanics does not involve an intrinsic materials length scale as a parameter for the deformation.

Nix and Gao model (1998) based on the geometrically necessary dislocations (GNDs) with the Taylor dislocation model (Taylor, 1938), together with indentation results for various materials, have led to the development of the mechanism-based strain gradient (MSG) plasticity theory. In this model, the ISE model was suggested as the linear dependence of the square of the indentation hardness to the reciprocal of the indentation depth by considering the GNDs, and agrees with many experimental results. However, several other nanoindentation results show that the Nix and Gao model

cannot predict the indentation hardness with a small indentation depth, in particular because of the tip rounding, grain boundary (GB) effect and change in the storage volume for GNDs (Elmustafa and Stone, 2002; Feng and Nix, 2004; Xue et al., 2002; Yang and Vehoff, 2007).

Yang and Vehoff (2007) performed the nanoindentation test in the center of individual grains to study the effect of indentation size and grain size on the nanohardness. In this test, for larger grains the hardness always decreases with the increase in indentation depth, the classical ISE. However, for smaller grains the hardness exhibited a behavior opposite to that of coarse grains: it increases with increase in the indentation depth, because of the GB effect. In the Nix and Gao model, the GB effect is not considered because the constitutive equation does not include the constituent grain size as a parameter for deformation.

In this paper, a model for the grain size and the ISE is proposed and compared with experimental results for polycrystalline materials using the modified MSG plasticity. This model can explain both the hardening and softening effects experienced in the polycrystalline materials by considering the GB effect.

## 2. Gradient plasticity theories

### 2.1. MSG plasticity

Taylor's dislocation model (Taylor, 1938) gives the shear flow stress in terms of a dislocation density as:

$$\tau = \frac{\alpha \mu b}{L} = \alpha \mu b \sqrt{\rho}, \quad (1)$$

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\* Corresponding author. Tel.: +82 54 279 2167; fax: +82 54 279 5899.

E-mail address: [hcpark@postech.ac.kr](mailto:hcpark@postech.ac.kr) (H.-c. Park).

where  $\alpha$  is an empirical constant usually ranging from 0.2 to 0.5,  $\mu$  the shear modulus,  $b$  the magnitude of the Burgers vector and  $\rho$  the dislocation density.

The dislocation density is composed of the density of the statistically stored dislocations (SSDs), which accumulate by trapping each other in a random way, and the density of the GNDs, which is required for the compatible deformation of various parts of the specimen.

Nix and Gao (1998) started from Taylor's hardening law between the shear strength and the dislocation density in a material:

$$\tau = \alpha \mu b \sqrt{\rho_T} = \alpha \mu b \sqrt{\rho_S + \rho_G}, \quad (2)$$

where  $\rho_T$  is the total dislocation density, and  $\rho_S$  and  $\rho_G$  are the densities of the SSDs and GNDs, respectively. If the Taylor factor,  $\bar{m}$ , is used, the uniaxial flow stress of the material can be described as:

$$\sigma = \bar{m} \tau = \bar{m} \alpha \mu b \sqrt{\rho_S + \rho_G}. \quad (3)$$

The Taylor factor  $\bar{m}$  acts as an isotropic interpretation of the crystalline anisotropy at the continuum level:  $\bar{m} = \sqrt{3}$  for an isotropic solid and  $\bar{m} = 3.08$  for the face-centered cubic (fcc) polycrystalline material.

In the absence of  $\rho_G$ , the flow stress can be derived using the power-law hardening rule (Bishop and Hill, 1951a,b):

$$\sigma = \sigma_{ref} \varepsilon^N = \bar{m} \alpha \mu b \sqrt{\rho_S}, \quad (4)$$

where  $\sigma_{ref}$  is the reference stress for the uniaxial tension,  $\varepsilon$  the effective strain and  $N$  the work hardening exponent ( $0 \leq N < 1$ ). The effective strain in the deformation theory of the MSG plasticity can be defined as:

$$\varepsilon_e = \sqrt{\frac{2}{3} \varepsilon_{ij} \varepsilon_{ij}}. \quad (5)$$

The gradient in the strain field is accommodated by the GNDs, so the effective strain gradient is described by the deformation shape:

$$\eta = \sqrt{\frac{1}{4} u_{k,ij} u_{k,ij}} = \sqrt{\frac{1}{4} \eta_{ijk} \eta_{ijk}}. \quad (6)$$

From Eqs. (4)–(6), the flow stress for the MSG plasticity is obtained as (Nix and Gao, 1998):

$$\sigma = \sigma_{ref} \sqrt{\varepsilon^{2N} + l \eta}, \quad (7)$$

where  $l$  is the material characteristic length described as:

$$l = \bar{m}^2 \alpha^2 \left( \frac{\mu}{\sigma_{ref}} \right)^2 b. \quad (8)$$

Generally, the flow theory of MSG plasticity theory is needed for complex indentation test (Qiu et al., 2003). However, in this study, deformation theory of MSG plasticity was used for simplicity because the difference between deformation and flow theory of MSG plasticity is vanishingly small.

## 2.2. Modified strain gradient theory

The modified strain gradient theory proposed by Lee et al. (2009) is based on the assumption that the metallic polycrystalline materials are plastically nonhomogeneous. When a material deforms, each grain in the material deforms by different amounts depending on its orientation. Thus, overlaps and voids tend to occur on the GBs, and are corrected by shear displacement. This shear displacement is interpreted as local shear by the GNDs (Ashby, 1970). Therefore, two types of GNDs are considered during deformation in this theory: one type occurs in the slip system because of the strain gradient and the other on the GB because of incompat-

ibilities associated with potential overlaps and voids. To calculate the density of the GNDs on the GB, the following assumptions are used.

1. The grains are approximately cubic and arranged randomly.
2. The density of the GNDs on the GB is proportional to the small strain.

For deformation of the polycrystalline materials, the two types of the GNDs are generated (Lee et al., 2009). Then the total density of the GNDs is:

$$\rho_G = \rho_{GS} + \rho_{GG}, \quad (9)$$

where  $\rho_{GS}$  and  $\rho_{GG}$  are the densities of the GNDs on the slip planes and the GB, respectively. Each density of the GNDs is related to the effective strain gradient as:

$$\rho_{GS} = \frac{\bar{r} \eta_S}{b}, \quad (10)$$

$$\rho_{GG} = \frac{\eta_G}{b}, \quad (11)$$

where  $\eta_S$  and  $\eta_G$  are the strain gradients caused by the slip plane and the GB, respectively, and  $\bar{r}$  is the Nye factor, which depends on the deformation shape and the slip plane. The Nye factor is employed to relate the macroscopic strain gradient to scalar measures of the GNDs density in polycrystalline materials. In general, the factor has a value of 1 for single crystals and 1.85 for the bending deformation and 1.93 for the torsion deformation of FCC polycrystalline materials (Arsenlis and Park, 1999). A value of 1.9 is assumed in this paper in order to determine the characteristic length for the polycrystalline materials in nanoindentation test.

The effective strain gradient caused by the density of the GNDs on the slip plane,  $\eta_S$ , is generally calculated from Eq. (6).

From Eqs. (4)–(6) and Eqs. (9)–(11), we propose the flow stress relationship for the polycrystalline materials as:

$$\sigma_{MSG} = \sigma_{ref} \sqrt{\varepsilon^{2N} + l(\eta_S + \eta_G)}. \quad (12)$$

Then, the corresponding indentation hardness can be obtained by choosing the Tabor's factor of 3:

$$\begin{aligned} H_{MSG} &= 3\sigma_{MSG} = 3\sigma_{ref} \varepsilon_p^N \sqrt{1 + l(\eta_S + \eta_G)/\varepsilon_p^{2N}} \\ &= H_A \sqrt{1 + \left( \frac{9}{H_A^2} \right) \cdot (\bar{m}^2 \alpha^2 \mu^2 b)(\eta_S + \eta_G)}, \end{aligned} \quad (13)$$

where the classical indentation hardness value,  $H_A$ , is given by:

$$H_A = 3\sigma_{ref} \varepsilon_p^N. \quad (14)$$

## 3. Indentation model for polycrystalline materials

For the nanoindentation model, the indenter tip was assumed as a rigid circular cone. Fig. 1 shows the simplified axisymmetric indentation model, where  $a$  is the radius of the contact area and  $h$  the indentation depth. The angle between the tip and the surface of the specimen was  $\theta$ . In this study, the indenter tip angle was converted to  $19.68^\circ$ , which is equivalent to the Berkovich tip. The relationships between  $a$ ,  $h$  and  $\theta$  are given as:

$$A = 24.56h^2 = \pi a^2, \quad \tan \theta = h/a. \quad (15)$$

Nix and Gao developed the equation for the ISE in single crystals by considering the density of the GNDs around a conical indenter. In this model, to calculate the density of the GNDs they assumed that the indentation is accommodated by circular loops

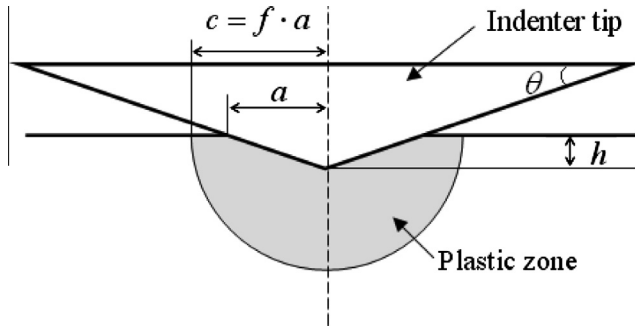


Fig. 1. Simplified axisymmetric indentation model.

of the GNDs with the Burgers vectors normal to the surface plane. As the indenter penetrates into the surface, the GNDs on the slip system are required to account for the permanent shape change at the surface. Then, the total length of injected dislocation loops to form the shape of conical indenter is given as:

$$\lambda = \frac{\pi h a}{b} = \frac{\pi a^2 \tan \theta}{b}, \quad (16)$$

where  $b$  is the Burgers vector.

It is assumed that the evolution of GNDs during the indentation is primarily governed by a large hemispherical volume with the radius of contact area  $a$ . However, the GNDs will spread beyond the hemispherical, because of the strong repulsive force between GNDs for very small indentation depth. To modify the plastic zone radius, Durst et al. (2006) proposed that the radius of the plastic zone is assumed to be  $f \cdot a$ , where  $f \geq 1$  can be interpreted as the ratio of the radii of the plastic zone and the contact. It is assumed that all injected GNDs loops remain within a hemisphere with radius  $c$ . Then, the volume of the plastic zone is:

$$V_p = \frac{2}{3} \pi c^3 = \frac{2}{3} \pi f^3 a^3. \quad (17)$$

Therefore, the density of the GNDs on the slip systems is determined as:

$$\rho_{GS} = \frac{\lambda}{V_p} = \frac{3}{2f^3 b h} \tan^2 \theta. \quad (18)$$

From Eqs. (10) and (18), the strain gradient caused by the slip plane is given as:

$$\eta_s = \frac{3}{2f^3 h} \cdot \frac{1}{\bar{r}} \cdot \tan^2 \theta. \quad (19)$$

For a conical indenter, it is assumed that the displacement is proportional to the indentation depth  $h$  and the strain field should depend only on the normalized indentation depth,  $h/a$  based on the assumption of a self-similar deformation field (Biwa and Storakers, 1995; Storakers et al., 1997). Then, the average plastic strain is given by:

$$\varepsilon_p = k(h/a) = k \tan \theta, \quad (20)$$

where  $k$  is the constant usually ranging from 0.2 to 0.4. A value of 0.2 is assumed in this paper. The average strain is independent of the indentation depth.

It is assumed that the GNDs on the GB are considered in the plastic region only to calculate the density of the GNDs on GB. The radius of the plastic zone varies directly with the indentation depth. Therefore, the density of the GNDs on the GB changes with

the range of indentation depth as shown in Fig. 2. For a radius of plastic zone,  $c$ , is smaller than half of the grain size,  $d/2$ , as the GB is not included within the plastic zone as shown in Fig. 2(a), the GNDs are not generated on the interface. Then, density of the GNDs on the GB is defined as:

$$\rho_{GG} = 0. \quad (21)$$

For this case, one can assume that there is no GB effect, and because the grain can be treated as single crystal in this range, the Nye factor,  $\bar{r}$ , and Taylor factor,  $\bar{m}$ , have values for single crystals:

$$\bar{r} = 1, \quad \bar{m} = \sqrt{3}. \quad (22)$$

From Eqs. (13), (19), (21), and (22), the indentation hardness can be obtained by:

$$H_{MSG} = H_A \sqrt{1 + \left( \frac{27}{H_A^2} \right) \cdot \left( \frac{3\alpha^2 \mu^2 b \tan^2 \theta}{2f^3} \right) \cdot \frac{1}{h}}. \quad (23)$$

For  $d/2 \leq c < \sqrt{2}d/2$ , parts of the GB are contained within the plastic zone, as seen in Fig. 2(b) and increase with increasing indentation depth. Assuming that the normal strain in the direction of indentation is the dominant component, we consider that the GNDs are generated on the GB, which is perpendicular to the indentation direction. Because the number of the GNDs on the polycrystalline GB is given as  $\varepsilon_p d/4b$  based on the Ashby relationship (1970), the total length of the GNDs on the GB between  $r$  and  $r + dr$  can be calculated as:

$$dl_G = 2\pi r \frac{\varepsilon_p}{4b} dr. \quad (24)$$

Integration of Eq. (24) from 0 to  $\sqrt{c^2 - d^2/4}$  yields the total length using the probability of the GBs in the plastic zone,  $N' = \frac{c}{d/2}$ , as:

$$l_G = 2 \int_0^{\sqrt{c^2 - d^2/4}} 2\pi r \frac{\varepsilon_p}{4b} dr \times \left( \frac{c}{d/2} \right) = \frac{\pi \varepsilon_p}{b} \left( \frac{c^3}{d} - \frac{cd}{4} \right), \quad (25)$$

The density of the GNDs can be obtained as:

$$\rho_{GG} = \frac{l_G}{V_p} = \frac{3\varepsilon_p}{bf^3 a^3} \left( \frac{c^3}{d} - \frac{cd}{4} \right). \quad (26)$$

As the Nye factor,  $\bar{r}$ , and Taylor factor,  $\bar{m}$ , have values for the polycrystalline material, the indentation hardness can be obtained from Eqs. (13), (19), and (26):

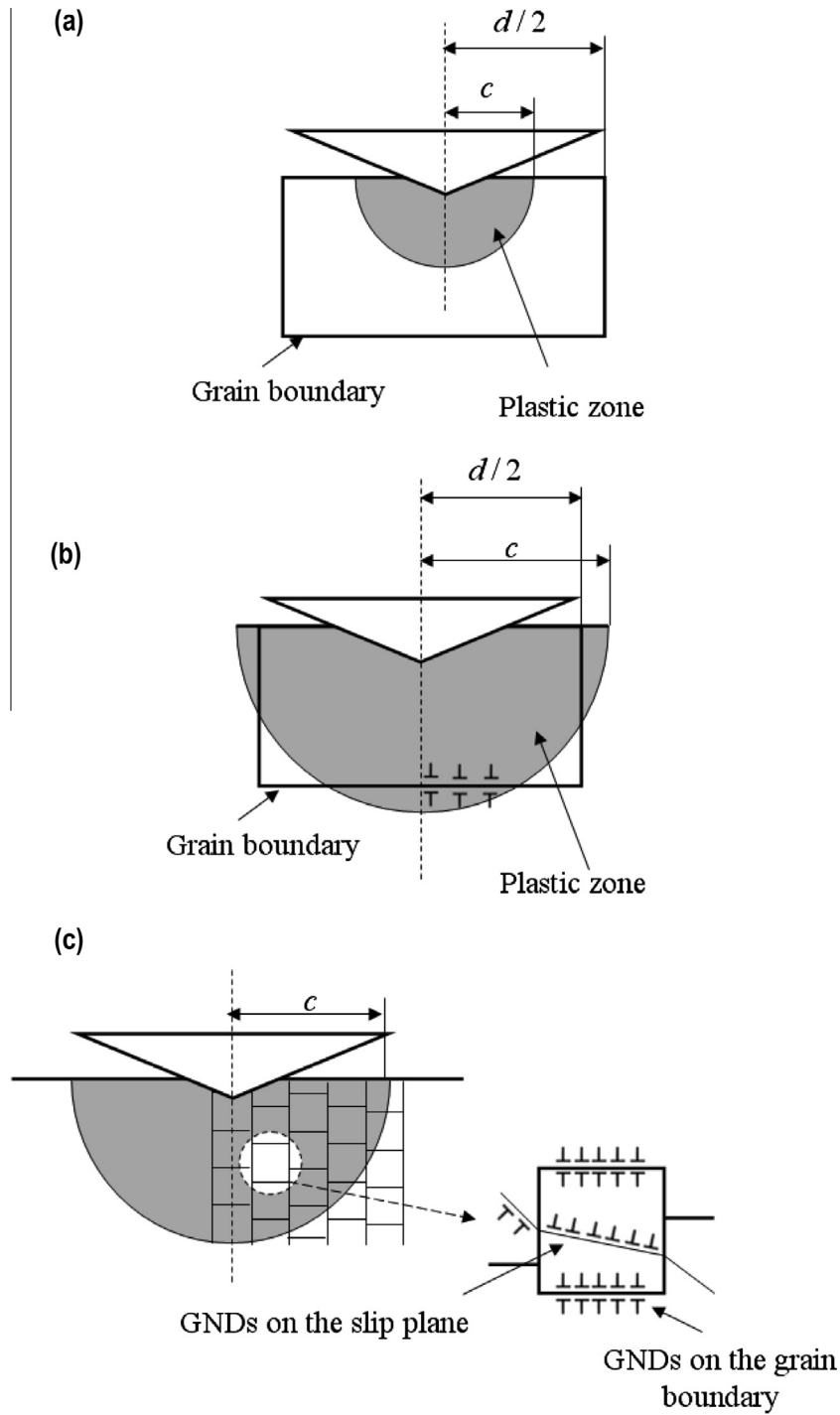
$$H_{MSG} = H_A \sqrt{1 + \left( \frac{81^2 \alpha^2 \mu^2 b}{H_A^2} \right) \cdot \left( \frac{3}{3.8f^3 h} \tan^2 \theta + \frac{3\varepsilon_p}{bf^3 a^3} \left( \frac{c^3}{d} - \frac{cd}{4} \right) \right)}. \quad (27)$$

As seen in Eq. (27), the indentation hardness increases with the increase in the indentation depth because of the GB effect in this depth range.

For  $c \geq \sqrt{2}d/2$  as shown in Fig. 2(c), the GB probability in the plastic zone is  $N' = 1 + \frac{(c-d/2)}{d/2}$ . Therefore, using the procedure proposed by Lee et al. (2009), the length and density of the GNDs on the GBs are given by:

$$l_G = 2 \int_0^c 2\pi r \frac{\varepsilon_p}{4b} dr \times (N') \times \frac{\pi}{4} = \frac{\pi^2 \varepsilon_p}{8} \cdot \frac{c^2}{b} \cdot N', \quad (28)$$

$$\rho_{GG} = \frac{l_G}{V_p} = \frac{3\pi \varepsilon_p}{16bf^3 a^3} N'. \quad (29)$$



**Fig. 2.** The occurrence of GNDs on the grain boundary according to relation between the radius of plastic zone and the grain size. (a) For  $c < d/2$ , (b) For  $d/2 \leq c < \sqrt{2}d/2$ , (c) For  $c > \sqrt{2}d/2$ .

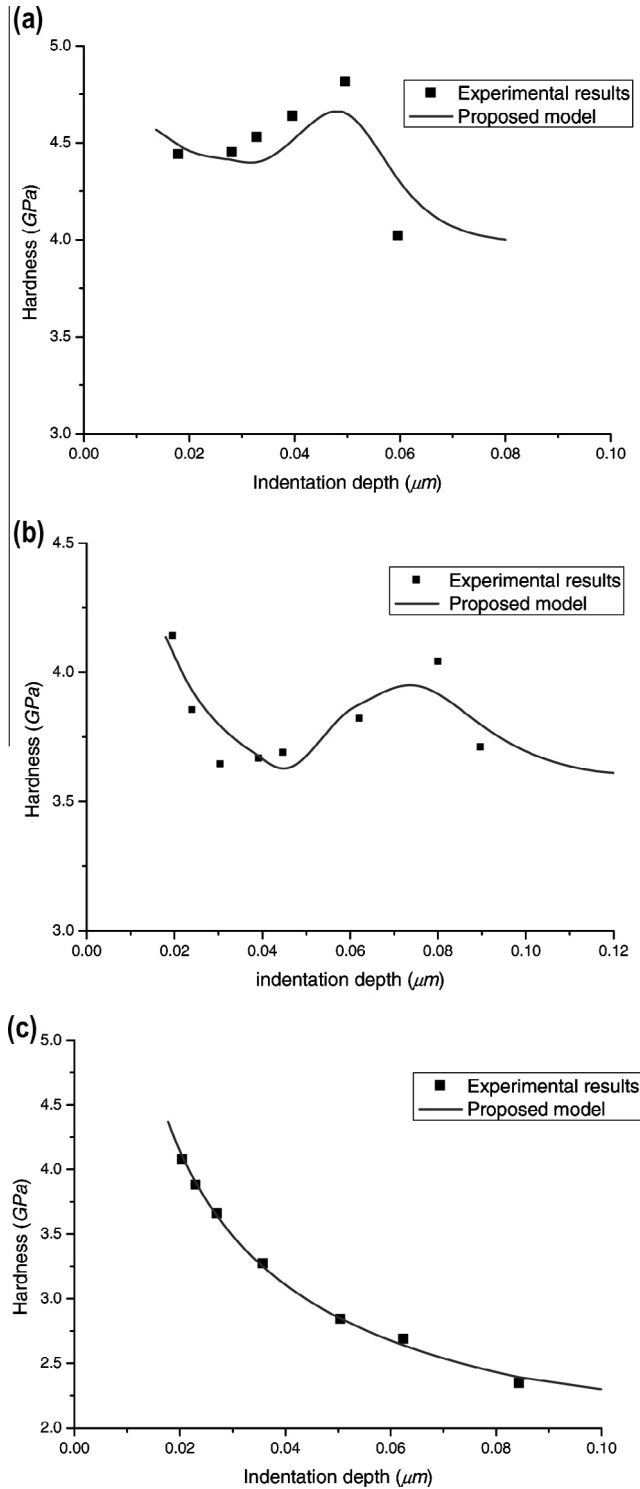
The indentation hardness can be obtained from Eqs. (13), (19), and (29):

$$H_{MSG} = H_A \times \sqrt{1 + \left( \frac{81^2 \alpha^2 \mu^2 b}{H_A^2} \right) \cdot \left( \frac{3}{3.8 f^3 h} \tan^2 \theta + \frac{3 \pi \epsilon_p}{16 b f^3 a^3} N' \right)}. \quad (30)$$

As seen in Eqs. (23), (27), and (30), the proposed model considers the GNDs on both the slip plane and Gb.

#### 4. Results and discussion

Fig. 3 compares the hardness predictions by the proposed model with the experiment result of Yang for the Ni plate specimen (Yang and Vehoff, 2007). In this test, the dependence of the hardness on indentation size and on grain size was studied by performing a nanoindentation test at the center of the individual grains and by varying the grain size and indentation depth. As seen in Fig. 3, the hardness decreases monotonically with increasing indentation depth for a grain size of 80  $\mu\text{m}$  which is considered a



**Fig. 3.** Comparison of the experimental data and the predicted indentation hardness by proposed bending model for (a) 0.85  $\mu\text{m}$  grain size, (b) 1.35  $\mu\text{m}$  grain size, and (c) 80  $\mu\text{m}$  grain size.

large grain. However, for smaller grain sizes, hardness increases with increasing indentation depth at a specific range. Nix and Gao model cannot predict this result because it does not consider the GB effect. Generally, for small indentation depth as shown in Fig. 2(a), the flow stress is governed by the continued nucleation and activation of new sources because the grain is considered as single crystals in this case. Then the flow stress in this range is soft-

**Table 1**

Mechanical parameters derived by fitting of experimental data (by Yang and Vehoff).

$d$ ( $\mu\text{m}$ )	$\alpha$	$H_A$ (GPa)	$\mu$ (GPa)	$f$
80	0.48	2.15	83	3.2
1.35	0.44	3.52	83	4.3
0.85	0.43	4.21	79	4.2

ened by the presence of dislocations as in the case of the Nix and Gao model. Plasticity is initiated by dislocation nucleation at theoretical shear stress. As plastic deformed volume is contacted with GBs as shown in Fig. 2(b), the hardening is preceded due to the GB. The GBs are regarded as obstacles to dislocation motion or a source of dislocations. Therefore the dislocation pileup or dislocation forest is generated in regions close to the GBs. Then higher stress is required to move dislocations through these pileup or forest. This effect caused by the GB is considered by calculating the density of the GNDs on the GB in this model. The proposed model agrees with experiments, as shown in Fig. 3.

In calculating the hardness, the material parameters were determined by curve fitting and the results are shown in Table 1. The relationship between  $H_A$  and the grain size in Table 1 follows the Hall–Petch relation (Hall, 1951; Petch, 1953). Generally, for indentation test, value of the empirical constant  $\alpha = 0.5$  is chosen due to the complex stress field dislocation substructures (Durst et al., 2007). Therefore, fitted empirical constant  $\alpha$  has from 0.43 to 0.48 in our study, which is reasonable. The average calculated shear modulus is about 81.67 GPa, which agrees well with that in Meyers and Chawla (1999), and the range of the parameter  $f$  is from 3.2 to 4.3, which agrees well with the reference value. (Ty-miak et al., 2001)

To estimate the size effect using the proposed model, the indentation hardness in Eq. (30) was normalized by the classical indentation hardness,  $H_A$ . The normalized indentation hardness is then given as:

$$\frac{H_{MSG}}{H_A} = \sqrt{1 + l' \left[ \frac{A}{d} + \left( \frac{A}{2f} + \frac{3 \tan \theta}{4f^3} \right) \frac{1}{h} \right]} \quad \text{for } c \geq \sqrt{2}d/2 \quad (31)$$

$$\therefore l' = \frac{81\alpha^2 \mu^2 b}{H_A^2}, \quad A = \frac{3\pi\epsilon_p}{16}.$$

As shown in Eq. (31), the normalized hardness is related to the grain size and the indentation depth. This relation shows a linear relationship between  $H_{MSG}$  and  $1/h$ , which corresponds to the Nix and Gao model, and agrees with the indentation data for the polycrystalline material with fine grains by Cao et al. (2006). This relation also predicts that the specimen with smaller grains has higher hardness at the same indentation depth, which agrees with the Hall–Petch relation.

## 5. Conclusions

We have investigated the indentation hardness for polycrystalline materials in micrometer and nanometer scale structures. The following conclusions are drawn.

1. We propose a new model for predicting indentation hardness of the polycrystalline materials using the modified MSG theory. The density of the GNDs is calculated based on the nonhomogeneity of the polycrystalline materials and the slip plane.
2. The total density of the GNDs changes with indentation depth. For small indentation depth ( $c < d/2$ ), the grain can be considered as single crystals because the GNDs are not generated on the GBs. However, for middle depth range ( $d/2 \leq c < \sqrt{2}d/2$ ), the GB effect is considered by the density of the GNDs on GBs,



which act as obstacles to dislocation movement. Then, the hardness increases with the increase in indentation depth in this range. For a larger depth ( $c > \sqrt{2}d/2$ ), the GNDs density decreases with increasing indentation depth and increases with the decrease in the grain size. The predicted hardness by the proposed model agrees fairly well with the experimental results conducted by Yang and Vehoff (2007).

3. We have demonstrated that the proposed model can explain the indentation hardness in polycrystalline materials. The proposed model shows that the grain size is an important factor in estimating the ISE for the polycrystalline materials in the micrometer and nanometer scale.

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