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Hardness estimation for pile-up materials by strain gradient plasticity incorporating the geometrically necessary dislocation density †

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Abstract

A plastic strain gradient theory incorporating the geometrically necessary dislocation density based on the low order displacement finite element method is proposed for calculation of the hardness value by Berkovich indentation. The obtained analysis results by this work are found to be in good agreement with the experimental data. Three-dimensional modeling technique of Berkovich indentation is also suggested. An empirical coefficient that includes the strain gradient effect into the yield stress formula is introduced and determined by reviewing area factors and hardness curves generated from the analyses. As pile-up occurs, classical plasticity theory gives a higher area factor and lower hardness value than those from experiment. However the strain gradient plasticity theory used in this work gives corrected area factor and hardness values. Dislocation density plots are generated that can explain the size effect during indentation and the availability of the three-dimensional modeling of Berkovich indentation.

Keywords: Geometrically necessary dislocation; Strain gradient plasticity; Finite element method; Nano indentation; Pile-up phenomenon; Indentation size effect

1. Introduction

Nano and micro indentation experiments have been carried out to determine mechanical properties, such as the elastic modulus and hardness since Oliver and Pharr [1] proposed a relevant method of interpreting test data. Such experiments eliminate the need for destructive testing or electron microscopy observations. Nano indentation experiments are routinely conducted by researchers as well as industry [2-6].

Size effects are often observed for nano and micro depths of indentation. Size effects refer to an increase in the yield stress and hardening when the configuration size of the structure or micro-structure is reduced to the micron and sub-micron (hundreds of nanometers) level [7-9]. Such effects are explained by the presence of geometrically necessary dislocations and can be modeled by strain gradient plasticity in continuum mechanics [7-12]. Hardness values obtained during nano indentation tests may exhibit strong size effects for nano and micro indentation depths [2-4, 9].

Analytic methods that take into account size effects have been proposed by many researchers. Such approaches (e.g., finite element methods) differ in their implementation. Major theories include the higher order formulation of Fleck and Hutchinson [8], the low order formulation of MSG (Mechanism-based Strain Gradient) and TNT (Taylor-based Nonlocal Theory) by Nix and Gao [9], a phenomenological model developed by Abu-Al Rub and Voyiadjis [12], and a crystallographic plasticity model devised by Kysar et al. [13]. With such theories and models, researchers are restricted in their use of finite element methods because limitations are encountered, especially when the most effective and versatile low order displacement-based elements such as linear triangular and hexahedral elements are adopted. Significant limitations stem from the requirements of higher order continuity and/or higher order polynomials of their field variables due to the terms of higher order tensor variables in their strain gradient formulations. Detailed features of limitations resolved are found in Park et al. [14, 15] who recently proposed low order finite elements with strain gradient plasticity using Abaqus.

In this work, a finite element analysis method for estimation of hardness values considering with measurement by a nano indentation machine (Nano Indenter XP, MTS) is proposed. For pile-up materials, classical techniques of finite element with nano indentation measurements may lead to erroneous values of modulus and hardness. We have chosen commercially available aluminum and copper as test materials which are industrially popular engineering metals that are widely used as one of base metals of coated and composite materials.

The hardness measurements are conducted in separate work

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by the authors. Specimens for the indentation were Al 6061 T6 and C 12200, which have shown vivid pile-up phenomena. In this work, relevant theories and experiments are briefly mentioned. Modeling and post-processing aspects for a Berkovich indentation and a cone indentation are explained. An area factor indicating either pile-up or sink-in is introduced and reviewed for a comparison of the cone and Berkovich indenter models. An empirical coefficient that relates the strain gradient effect to the yield stress formula is characterized and determined by fitting analyses data to the experimental results. Finally, the hardness for both materials will be calculated and compared with the experimental data. The calculated plastic strain gradient or density of geometrically necessary dislocations for both materials will be plotted for Berkovich indentation.

2. Theory

2.1 Hardening by the plastic strain gradient

According to the Taylor dislocation model, the flow stress of a material can be expressed as [16]:

$$\sigma = \kappa\, m\, G\, b\sqrt{\rho_{ssd} + \rho_{gnd}} \tag{1}$$

where ρ_{ssd} and ρ_{gnd} are the densities of statistically stored dislocations and geometrically necessary dislocations, respectively. m is the Taylor factor, G is the shear modulus, b is the magnitude of Burgers vector, and κ is an empirical coefficient. Using the uniaxial plastic equation, $\sigma = \sigma_u(\bar{\varepsilon}_p)$, the Taylor model can be expressed as [14]:

$$\sigma = \sqrt{\sigma_u{}^2 + \Omega\bar{\eta}} \tag{2}$$

where, $\Omega = (\kappa m G)^2 \bar{r}\, b$ and $\bar{r}$ is the Nye factor. The following relationships are used in Eq. (2)

$$\rho_{ssd} = (\sigma_u / \kappa m G b)^2 \tag{3a}$$

$$\rho_{gnd} = \bar{r}\,\bar{\eta} / b \tag{3b}$$

where $\bar{\eta}$ is the equivalent plastic strain gradient that must be calculated as described in the next section. The first term in Eq. (2) represents classical plasticity while the second term in Eq. (2) represents strains gradient dependency on the plastic behavior. The hardening due to the strain gradient is proportional to the geometrically necessary dislocation density as in Eq. (3b) which is proportional to the strain gradient and inversely proportional to the magnitude of Burgers vector.

In this work, Eq. (2) is implemented by UHARD in Abaqus with the following derivative according to the material model used.

$$\frac{d\sigma}{d\bar{\varepsilon}_p} = \frac{\sigma_u}{\sigma}\sigma_u' + \frac{1}{2}\frac{\Omega}{\sigma}\bar{\eta}' \tag{4}$$

Here, $(\cdot)' = d(\cdot)/d\bar{\varepsilon}_p$. For more detail steps regarding the implementation and calculation of strain gradient hardening, the reader is referred to the paper by Park et al. [14].

2.2 Calculation of the plastic strain gradient

There exist several definitions of the plastic strain gradient on which formulation and length scales are based. Among these formulations and length scales, the following is used in this work [10].

$$\bar{\eta} = \tfrac{1}{2}\sqrt{\eta_{ijk}\eta_{ijk}} \tag{5}$$

Denoting $\rho_{ijk} = \rho_{jik} = \varepsilon^p_{ij,k}$, the components of the strain gradient are expressed as:

$$\eta_{ijk} = \eta_{jik} \equiv u_{k,ij} = \rho_{kij} + \rho_{kji} - \rho_{ijk}. \tag{6}$$

Calculation of the strain gradient matrix, $[\rho]$, with plastic variables averaged at nodes can be performed using the gradient of shape functions, $[B]$, and the Jacobi matrix, $[J]$, as follows:

$$[\rho] = [J]^{-1}[B][\varepsilon^p] \tag{7}$$

where $[\varepsilon^p]$ is the averaged-at-nodal plastic strain matrix. Eq. (7) is implemented by URDFIL in Abaqus [14].

3. Experiments

In this work, typical non-ferrous engineering metals are selected because they may show pile-up phenomena during indentation. Specifically, the materials under investigation are aluminum Al 6061 T6 and copper C 12200. Tensile tests were carried out with sheet form 13B per KS B 0801 so as to obtain Young's moduli and stress-strain curves as reference to the measurements by nano indentation. The test results are plotted in Fig. 1 and summarized in Table 1.

The plastic part of the stress-strain curves in Fig. 1 exhibits linear hardening in case of the aluminum sample and linear softening in case of the copper specimen. The negative work hardening value of the copper specimen means strain softening in tensile experiment which is in agreement with the published catalogue values. Therefore, the plastic stress-strain relationships are modeled by the Ludwick equation:

$$\sigma_u = \sigma_0 + E_p\bar{\varepsilon}_p \tag{8}$$

where σ_u, σ_0, E_p, and $\bar{\varepsilon}_p$ are the uni-axial yield stress, initial yield stress, work hardening modulus, and plastic strain, respectively.

Indentation tests were also carried out to obtain Young's modulus and hardness values. Specimens were $10\,\text{mm} \times 10\,\text{mm} \times 5\,\text{mm}$ with the same batch samples as the

Table 1. Mechanical properties of the materials, as determined by the tensile tests.

	Al 6061 T6	C 12200
Young's modulus (*GPa*)	75	115
Poisson's ratio[§]	0.33	0.34
Initial yield stress (*MPa*)	249	285
Work hardening modulus (*MPa*)	867	-122
E/σ_0 , E_p/σ_0	300, 3.5	400, -0.43

[§] Poisson's ratios were taken from widely accepted values.

Table 2. Micro-nano material properties required for the analyses.

	Al 6061 T6	C 12200
Taylor factor	3.08	3.08
Burgers vector (*nm*)	0.286	0.255
Nye factor	1.85	1.93

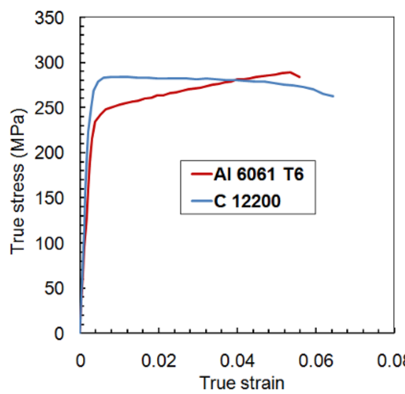

Fig. 1. Stress-strain curves of Al 6061 T6 and C 12200 obtained from the tensile tests.

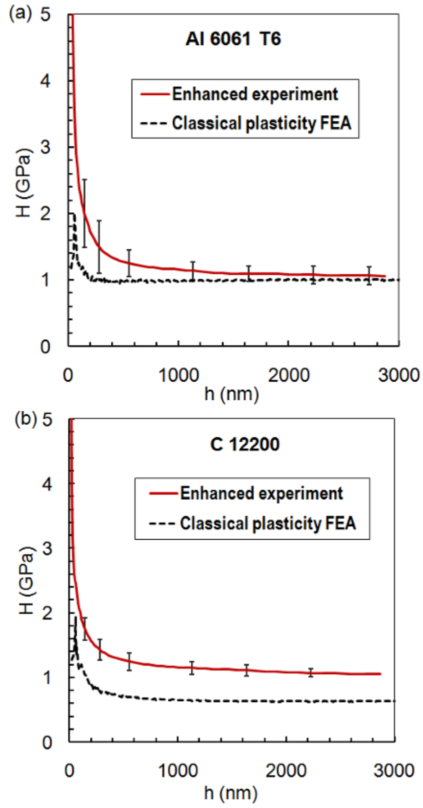

Fig. 2. Hardness from experiment (solid line) and classical finite element analysis (dashed line): (a) Al 6061 T6; (b) C 1220.

tensile test samples. A nano indentation machine (Nano Indenter XP, MTS) was employed and six to nine indentation runs were performed at various locations on the same specimen up to a depth of 3000 nm. While electron microscopy images showed that both the aluminum and copper sample exhibited pile-up phenomena, the copper showed more pile-up.

Hardness results from the nano indentation tests are shown in Fig. 2 where both the experimental and finite element analysis findings are displaced. The experimental values have been corrected by the enhanced technique of measuring the hardness for a pile-up situation. The hardness was measured as 1.1 and 1.0 GPa for the aluminum and copper samples, respectively, while it was 1.0 and 0.6 GPa from the finite element analysis. The finite element analysis used here was based on the classical theory of plasticity. From Fig. 2, it can be seen that the finite element method without consideration of the strain gradient or geometrically necessary dislocations cannot predict the macro hardness as well as the micro hardness. As such, the strain gradient plasticity finite element method which is described in the previous section is needed for more accurate analysis.

4. Analyses

4.1 Finite element modeling

The modeling aspects of the indentation process will now be described. Since the theory presented in Section 2 is applicable to a plane element as well as a solid element, a cone indenter will be used for the axi-symmetric plane element, while a Berkovich indenter will be employed for the three dimensional solid element. The Berkovich indenter is shown in Fig. 3(a) and the cone indenter is displayed in Fig. 3(b); pile-up and sink-in configurations with schematic illustrations of geometrically necessary dislocations are also represented. The conical indenter is an area-equivalent indenter that gives the same depth (h) to area (A_t) function as the Berkovich indenter [1].

$$A_t = 24.56h^2 \tag{9}$$

Axi-symmetric and three dimensional finite element models are presented in Fig. 4. Only a sixth of the full specimen for the solid model can be used when the repetitive symmetry of the Berkovich indentation is accounted. Boundary conditions can be given by such symmetry through the use of a coordi-

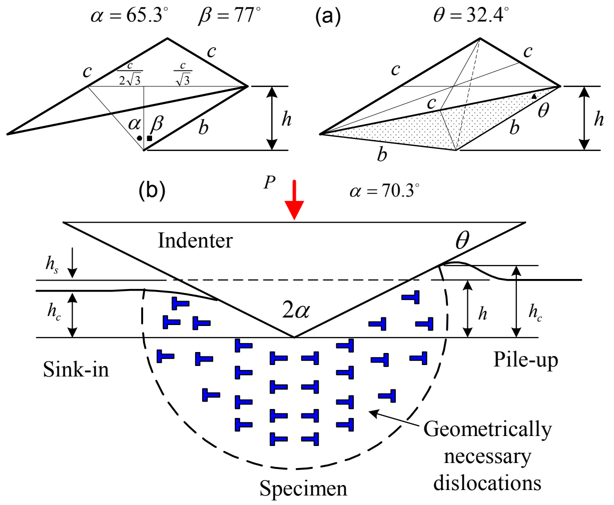

Fig. 3. Indenter geometry with illustrations of the geometrically necessary dislocations, as well as the pile-up and sink-in phenomena: (a) Berkovich indenter; (b) area-equivalent conical indenter.

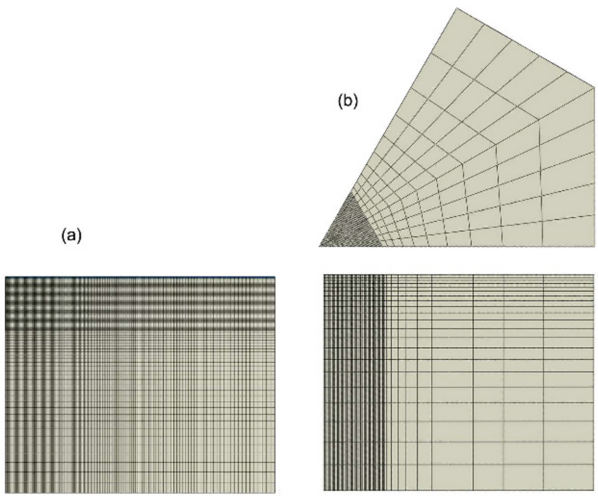

Fig. 4. Finite element meshes for the specimens: (a) axi-symmetric model (2D) for the equivalent conical indentation; (b) solid model (3D) for the Berkovich indentation.

nate transformation of the degrees of freedom. Once results are obtained for part of the geometry, results for the 360° specimen can easily be recovered by mirroring and rotational patterning. For the solid model, 13485 linear hexahedral elements are used, while 12333 linear quadrilateral elements are employed for the axi-symmetric model.

4.2 Contact area and hardness

The finite element analysis software, Abaqus, gives the contact area as an output variable, CAREA. For the Berkovich indentation, this output variable can be equated to the following side wall area (shown in Fig. 3(a)).

$$A_b = (3/4)c^2 \tan\theta = \sqrt{3}\tan\theta \cdot A_t \tag{10}$$

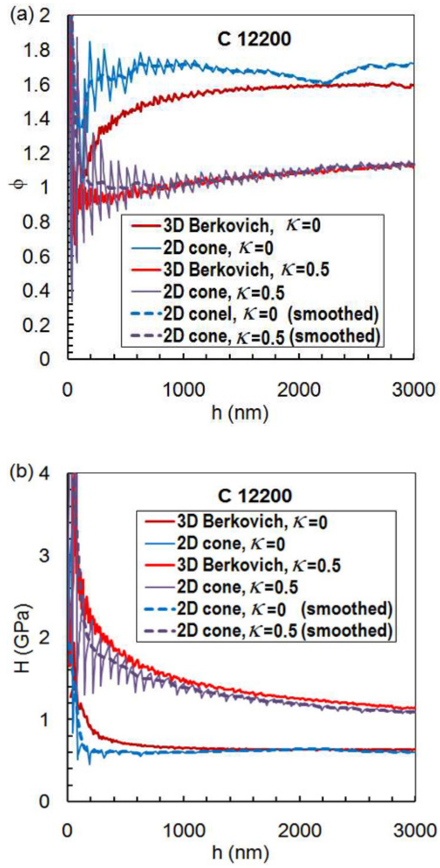

Fig. 5. Berkovich (3D) and cone (2D) indentation analysis by classical plasticity (κ = 0) and strain gradient plasticity (κ = 0.5) theory for C 12200: (a) area factor, ϕ ; (b) hardness, H.

An area factor, ϕ , can be introduced as follows:

$$\phi = A_b^{FEA} / A_b = A_c^{FEA} / A_t . \tag{11}$$

This area factor can be calculated through the relationship, $A_b^{FEA} = CAREA$. In Eq. (11), A_c^{FEA} is the projected contact area by the analysis and ϕ is indicative of whether the result reflects pile-up or sink-in phenomena. A similar way of applying finite element analysis with the conical indenter can be derived in a straightforward manner.

The analysis results for Berkovich and cone indentations are shown in Fig. 5. The area factor ϕ is displayed in Fig. 5(a), while Fig. 5(b) shows the hardness values, H.

$$H = P / A_c^{FEA} \tag{12}$$

The strain gradient plasticity and classical plasticity results are shown in Fig. 5. The 2D conical model exhibits excessive zigzags as shown in Ref. [18] that are not present in the 3D Berkovich model. Such a discrepancy between the models is due to the fact that contact in the 3D model is made more gradually than in the axi-symmetric model. Therefore, the axi-

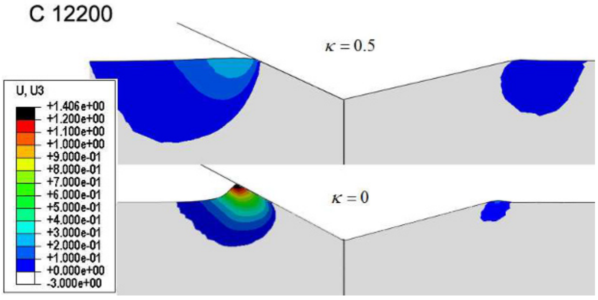

Fig. 6. Pile-up displacement (vertical) for C 12200 at the maximum depth (h = 3000 nm) of the Berkovich indentation analysis by classical plasticity (κ = 0) and strain gradient plasticity theory (κ = 0.5).

symmetric results must be smoothed to obtain the area factor and hardness. Smoothing was performed with a Gaussian kernel [17]; this is represented in Fig. 5 as a dashed line. As expected, the materials are strengthened by the plastic strain gradient during indentation due to geometrically necessary dislocations beneath the indenter tip. In addition, the pile-up phenomenon becomes smaller and the hardness value becomes larger due to the strengthening. A reduction in pile-up due to strain gradient effects is clearly shown in Fig. 6 for the copper specimen. The vertical displacement is reduced from 1.4 to 0.3 μm by the strain gradient effect in Fig. 6. In Fig. 5, the empirical coefficient in Eq. (1) is assumed to be κ = 0.5; this will be more accurately determined in the next section.

4.3 Characterization of the empirical coefficient

In Eqs. (1) and (2), there is an empirical coefficient κ that relates the strain gradient effect to the flow stress formula. If experimental results such as those in Fig. 2 are known, κ can be determined by comparing analysis results with the experimental values.

The analysis results like Fig. 5 for κ = 0, 0.5, and 1.0 are believed to be appropriate to fit with the test results. From the analyses, the hardness curve for a particular value of κ can be fitted with the following equation:

$$H(\kappa,h) = H_0 + a(\kappa)h^{b(\kappa)} \tag{13}$$

where H_0 is the approximated hardness when κ = 0 and a and b are the coefficients of fitting for κ = 0.5 and 1.0, respectively. Based on the careful observation from the analysis behaviors with respect to the values of κ such as Fig. 5, the coefficients themselves can be fitted with the variable κ as follows:

$$a(\kappa) = c\kappa^m \tag{14a}$$
$$b(\kappa) = b_{0.5} + 0.5(b_1 - b_{0.5})\kappa \tag{14b}$$

where c and m are the fitted coefficients with $a(0) = a_0$,

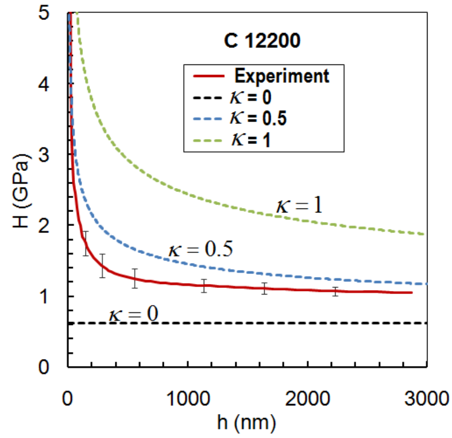

Fig. 7. Determination of the material coefficient κ by fitting finite element analysis data to the experimental results.

$a(0.5) = a_{0.5}$, $a(1) = a_1$, $b(0.5) = b_{0.5}$, and $b(1) = b_1$. Finally, a particular κ that fits the experimental data can be determined by minimizing the following least squares error:

$$\min . \sum_i (H_i - H(\kappa,h_i))^2 . \tag{15}$$

The process described here is illustrated in Fig. 7. The value of κ for the copper sample must be between 0 and 0.5 in the figure. From the fitting, κ = 0.45 for Al 6061 T6 and κ = 0.38 for C 12200. These values will be used to obtain final hardness values by the finite element analysis.

4.4 Results and discussion

Hardness values obtained with the present method are shown in Fig. 8 with the experimental data for comparison. The analysis results for both the macro and micro hardness are in good agreement with the experimental data. With the proposed technique, hardness values for the pile-up materials are estimated with greatly improved accuracy.

The equivalent plastic strain and strain gradient for both materials are shown in Fig. 9. The strains and dislocations present along the edges of the Berkovich tip were larger than those on the faces of the tip. When compared to the aluminum specimen, higher values were observed for the copper sample. Geometrically necessary dislocations in Fig. 9(b) are proved to contribute to the hardening or strengthening of the material by Eq. (1).

If the conical modeling of Fig. 4(a) is used, the strains and strain gradients are axi-symmetric, which is quite different from the scenario in Fig. 9. While the axi-symmetric model can yield reasonable data (as in Fig. 5), the results are not equivalent to those obtained with the three-dimensional Berkovich model. Therefore, the three dimensional model can describe pile-up phenomena better than axi-symmetric model.

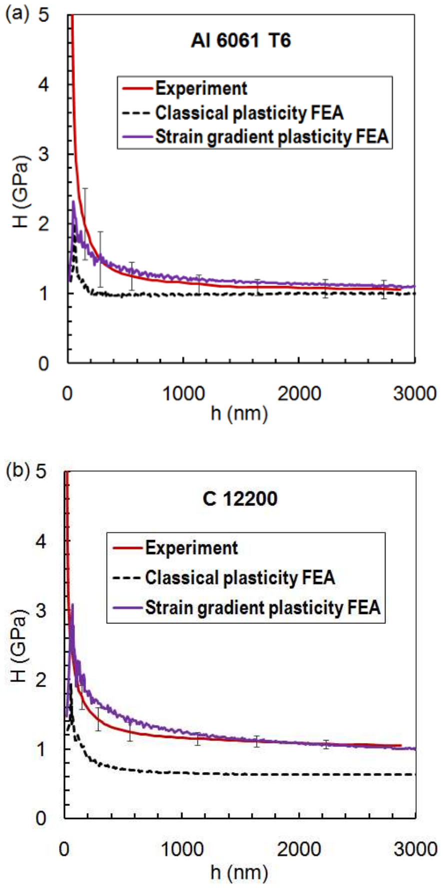

Fig. 8. Comparison of the experimental hardness values and the hardness obtained by the classical finite element analysis and the strain gradient plasticity finite element analysis: (a) Al 6061 T6; (b) C 12200.

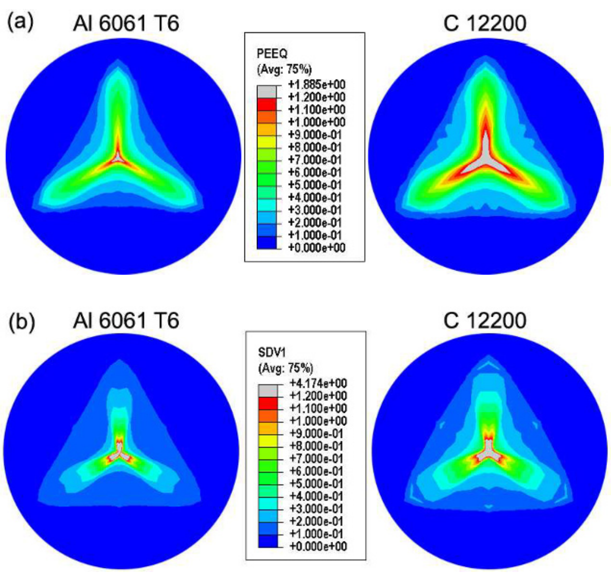

Fig. 9. Finite element analysis results obtained with the present method for Al 6061 T6 and C 12200: (a) equivalent plastic strain, $\varepsilon_p = \text{PEEQ}$; (b) plastic strain gradient; $\bar{\eta} = \text{SDV1}[\mu m^{-1}]$, geometrically necessary dislocation density, $\rho_{Al} = 6470 \cdot \text{SDV1}[\mu m^{-2}]$, $\rho_{Cu} = 7570 \cdot \text{SDV1}[\mu m^{-2}]$ at maximum depth, $h_{\max} = 3000\,nm$.

5. Conclusion

Strengthening or hardening during micro indentation was evaluated by the finite element method based on a theory of strain gradient plasticity that incorporate geometrically necessary dislocations. Hardening by the plastic strain gradient was considered with the Taylor dislocation model for Al 6061 T6 and C 12200, which were modeled, respectively, as linear work hardening and softening materials in terms of their uniaxial tensile behavior. Strain gradients were calculated via an isoparametric interpolation of the averaged-at-nodal plastic strain components. This method is proved to be efficient and versatile through an analysis of axi-symmetric conical indentation and three-dimensional Berkovich indentation. The unknown empirical coefficient of the strain gradient hardening model was determined by fitting several analytic hardness-depth curves to the experimental values obtained by the nano indentation tests.

With the classical plasticity theory, both the micro hardness and macro hardness cannot be calculated accurately. This limitation of the classical plasticity theory tends to increase for soft materials that exhibit more pile-up during indentation. In this study, C 12200 showed more pile-up than Al 6061 T6. The micro and nano hardness for both materials cannot be estimated by classical theory because there is no size effect. Even the macro hardness of the copper sample cannot be estimated by the classical plasticity.

With the proposed technique using strain gradient plasticity theory, both the micro hardness and macro hardness can be calculated more accurately. Results of the analysis with the proposed Berkovich indentation model are in good agreement with the experiments carried out with the nano indentation machine. In addition, the present method can yield geometrically necessary dislocation density plots that can help the interpretation of the mechanisms relevant to the strength of materials.

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