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Representative indentation yield stress evaluated by behavior of nanoindentations made with a point sharp indenter

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1. Introduction

Nanoindentation evaluates the local mechanical properties without having to observe the residual imprint on the indented surface through the analysis of the continuous relationship between the indentation load P and penetration depth h (P-h curve, hereafter) upon loading as well as unloading. Thus, nanoindentation techniques can be used on specimens that are too small for conventional macroscopic mechanical tests, e.g. thin films on a substrate, materials utilized for MEMS devices.

The indentation hardness and reduced modulus can be evaluated with the conventional *P*-*h* curve analysis (Oliver and Pharr, 1992). Regarding the deformation mechanism, plastic and elastic deformation simultaneously occur under an indentation during loading, and the elastically deformed volume recovers when the indentation is unloaded. The elastic recovery during unloading can be used to evaluate the

ABSTRACT

In order to evaluate local plasticity, a numerical analysis is carried out on conical nanoindentations simulated with a finite element method. A representative indentation yield stress *Y*^{*} that characterizes the nanoindentation is derived in order to represent the local plasticity properly in terms of the yield stress *Y*, plastic strain hardening modulus *E*_p, Poisson's ratio ν and the inclined face angle of the indenter, β . *Y*^{*} can be empirically evaluated as a function of the representative indentation elastic modulus *E*^{*}, the relative residual depth ξ and β , where ξ is defined as $\xi \equiv h_r/h_{max}$ with the maximum penetration depth h_{max} and the residual depth h_r . Nanoindentation experiments confirmed the validity of the empirical equation for evaluating the yield stress.

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elastic modulus (Akatsu et al., 2015). There is a possibility of deriving the plastic deformation resistance from the loading process of the nanoindentation by subtracting the elastic deformation from the total deformation during the loading process.

Recent studies have revealed interesting nanoindentation phenomena such as the indentation size effect (Akatsu et al., 2005; Nix and Gao, 1998, Pharr et al., 2010), inverse Hall-Petch rule for fine grained materials (Carlton and Ferreira, 2007; Mohammadabadi and Dehghani, 2008), hardening of film on substrates (Wang et al., 2007; Xu et al., 2011; Idrissi et al., 2011), wherein the indentation hardness is usually used as a measure of plastic deformation resistance. In order to describe these plastic phenomena correctly, the proper plastic deformation resistance of these materials should be evaluated and investigated. Sakai has proposed a measure, called the true hardness, that can be used instead of the indentation hardness to represents pure plastic deformation resistance (Sakai, 1993; Sakai et al., 1999; Sakai, 1999). The true hardness is determined on the basis of the Maxwell combination of an elastic spring and a plastic slider. There is still some





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controversy as to the validity of the simple Maxwell combination model for describing real elastoplastic deformations caused by nanoindentations.

In this study, a simulation of a conical nanoindentation was carried out with a finite element method (FEM), and the simulated *P-h* curve was numerically analyzed in order to determine the plastic deformation resistance that properly represents local plasticity. Moreover, a means of evaluating the representative plastic deformation resistance was devised for experimental nanoindentations. Finally, nanoindentation experiments were conducted to assess the validity of the protocol for deriving the representative plastic deformation resistance from the *P-h* curve.

2. FEM simulation and the experiment of nanoindentation

A nanoindentation made with a conical indenter was simulated with FEM in the same way as reported in the literature (Akatsu et al., 2015). A conical indentation with an inclined face angle β on a cylindrical elastoplastic solid was modeled in order to avoid the difficulty of modeling a real pyramidal indenter. The FEM simulation exploited the large strain elastoplastic capability of ABAQUS code (Version 5.8.1). The FEM simulation used elastoplastic linear strain hardening rules, i.e., $\sigma = E\varepsilon$ for $\sigma < Y$, and $\sigma = Y + E_p\varepsilon_p$ for $\sigma \ge Y$. Here, $E (\equiv d\sigma/d\epsilon_e)$ is Young's modulus, Y yield stress, and $E_{\rm p}$ ($\equiv d\sigma/d\varepsilon_{\rm p}$) plastic strain hardening modulus, where $d\sigma$, $d\varepsilon$, $d\varepsilon_{\rm e}$, and $d\varepsilon_{\rm p}$ are, respectively, the incremental values of stress, total, elastic, and plastic strains. Indentations were simulated for E, ν , Y and E_p ranges of 50–1000 GPa, 0–0.499, 0.1-60 GPa, and 0-200 GPa, respectively. The von Mises criterion was used to determine the onset of the yielding flow.

To confirm the validity of the numerical analysis, nanoindentation experiments with a Berkovich indenter were carried out on a series of commercially available metals with well-known properties, including brass (copper alloy C2680 in the Japanese Industrial Standard (JIS) H3100), duralumin (aluminum alloy Al2024 in JIS H4000), and beryllium copper alloy (C1720 in JIS H3130). The preparation of samples for the nanoindentation experiment and the way of making the nanoindentation are described in detail in the literature (Akatsu et al., 2015).

3. Results and discussions

3.1. Representative indentation yield stress Y* as an index of plastic deformation resistance for indentation

According to the previous study (Sakai, 1999) and as described in Appendix B (see Fig. (B1)), the indentation hardness *H* defined by Eq. (A1) is not a measure of plasticity. Moreover, as described in Appendix B (see Fig. (B2)), the true hardness *H*_t (Sakai, 1993; Sakai et al., 1999; Sakai, 1999) with n = 3 only partially represents plastic deformation resurce. When the constraint factor *C* is defined as $C \equiv \frac{H_t}{Y_R}$, where Y_R is the representative yield stress for a conical indentation with an inclined face angle β on an elastoplastic solid with the yield stress *Y* and the plastic strain hardening modulus E_p (Sakai et al., 2003):

$$Y_{\rm R} \equiv Y + 0.22E_{\rm p} \tan\beta,\tag{1}$$

1.5 X = 0.5 0.5 0.2 0.4 0.50.5

Fig. 1. Indentation elastic–plastic index as a function of relative residual depth ξ .

an indentation elastic-plastic index $\frac{Y_R}{E' \tan \beta}$ (Sakai et al., 2003) can be derived from rewriting Eq. (B1) under the assumption that $k_e \approx k_2$ as follows:

$$\frac{Y_{\rm R}}{E'\tan\beta} = \frac{1}{2C} \left[\left\{ \frac{\gamma_{\rm e}}{\gamma(1-\xi)} \right\}^n - 1 \right]^{-\frac{2}{n}}$$
(2)

where k_2 is the indentation unloading parameter defined as $k_2 = \frac{P_{\text{max}}}{(h_{\text{max}} - h_r)^2}$ with the maximum indentation load P_{max} , maximum penetration depth h_{max} and residual depth h_r , and ξ is the relative residual depth defined as $\xi \equiv \frac{h_r}{h_{\text{max}}}$. Here, for a linearly elastic solid, k_e is the indentation elastic parameter defined as $k_e \equiv P_{\text{max}}/h_{\text{max}}^2$ and γ_e is the a surface deformation parameter defined as $\gamma_e \equiv \frac{h_{\text{max}}}{h_c}$ with the contact depth h_c at P_{max} (see Appendix A and B). The index $\frac{Y_R}{E' \tan \beta}$ infinitely diverges for perfect elasticity with $\xi = 0$ and is equal to 0 for perfect plasticity with $\xi = 1$. Fig. 1 plots the value of $\frac{Y_R}{E' \tan \beta}$ input to the FEM simulation versus ξ determined from the simulated *P*-*h* curve. $\frac{Y_R}{E' \tan \beta}$ is, strictly speaking, not an indentation elastic-plastic index because it does not seem to be determined uniquely as a function of ξ , as shown in Fig. 1. Now let us propose a modification to the indentation elasticplastic index, i.e., $\frac{Y^*}{E^* \tan^{1.2} \beta}$, where Y^* and E^* are the representative values of yield stress and elastic modulus for indentation, respectively, in order to express the index as a unique function of ξ . The representative indentation yield stress Y^* is defined as,

$$Y^* \equiv \frac{Y + E_p \varepsilon^*}{1 - (\nu - b)} \tag{3}$$

where

$$b = 0.225 \tan^{1.05} \beta$$
 (4)

and ε^* is representative strain for conical indentation.

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