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Spherical indentation method to evaluate material properties of high-strength materials

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ABSTRACT

The spherical indentation method of Lee et al. (2005, 2010) [8,10] is extended for property evaluation of high-strength materials. By considering the finite deformation of elastic indenter due to high-strength of the indented material, regression functions are generated to map the indentation load–depth curve into the stress–strain curve. A property evaluation program is then written to produce material properties by using the indentation load–depth data from the loading/unloading process. Finally, the nano-indention tests with a spherical indenter are carried on Germanium Ge (100) and Silicon Si (100) to verify the proposed method using the experimental load–depth curves.

1. Introduction

High-strength materials have wide applications to cutting tools, surface coatings, components for high temperature, light weight automotive structures. In the applications, it is essential to understand the deformation behavior of material to applied load, i.e. stress–strain relationship. Young's modulus E, yield strength σ_o, strain hardening coefficient n are often used to describe the stress–strain relation.

For general metallic and brittle materials, numerous studies and techniques are available in the literature to evaluate these material properties from experimental test data [1]. Tensile/compression tests are traditionally used to determine the properties of metallic materials; however, standard specimens including elaborate machining jobs are needed to accomplish these tests [1]. On the other hand, a non-destructive and localized indentation method can be used with micro-size and non-standard specimens to evaluate constitutive properties, hardness, fracture toughness, residual stress and creep properties. In contrast to its rather simple testing process, extracting properties from the indentation test is far from being easy due to the non-uniform deformation beneath the indenter.

Numerous studies are thus carried out to reveal the characteristics of indention tests [2–10]. In indentation test, required properties are reversely extracted from the indentation load–depth curves (and imprint size) generally by using mapping functions. Sharp indentation produces phase transformation and micro-cracking even at relatively small loads. The indenter sharpness has significant effect on the mechanisms and it is hard to achieve the exact tip-sharpness during the manufacturing process. All these factors make the problem quite complex to be expressed analytically. In spherical indentations, phase transformation and micro-cracking can be avoided.

Tabor [11] proposed an idea to relate the mechanical properties with indentation load and depth. Afterwards, spherical indentation techniques are mainly studied to extract mechanical properties from experimental and numerical data [12–21]. Via extensive finite element (FE) analyses, Lee et al. [8,10] generated the functions mapping the indentation load–depth curve into the true stress–strain curve. They then contrived numerical algorithms which give the material properties of metallic materials from the load–depth data of spherical indentation test. Their reverse approaches provide the properties of general metallic material with high accuracy. However, the used material property ranges $\sigma_o < 1$ GPa keep the method from applying to high-strength materials.

For high-strength materials, relatively higher load is necessary to indent the specimen up to the same indentation depth. Consequently, the elastic indenter experiences finite deformation, the consideration of which is essential in the indentation of high-strength materials. The above studies [8–21] are mostly based on rigid or elastic indenter with negligible indenter deformation; therefore, one cannot directly apply these spherical indentation

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studies for property evaluation of high-strength materials. However, Albayrak et al. [22] evaluated the E and σ_o of transparent Yttria (high-strength material) based on the spherical-tip assumption, while they considered the indenter deformation via effective modulus. Based on spherical-tip approximation, the solution with an irregular indenter tip shape can rather give reasonable elastic load-displacement relationship, but significantly arbitrary stress distribution and contact area beneath the indenter.

A reliable indentation method for evaluation of material properties of high-strength materials can be especially useful, when the cracking of specimen is hard to control during specimen preparation and testing due to the brittleness of material. For example in the tensile test, brittle materials are broken even at very small strain. Despite this compelling usefulness of indentation test, studies on the indentation methods for property evaluation of high-strength materials are limited in the literature.

In this study, by extending the method of Lee et al. [10], a technique is developed to evaluate the properties of high-strength materials from spherical indentation. Considering finite deformation of elastic indenter, we generate enhanced mapping functions. The range of yield strain is divided into three sections based on the regression characteristics; an independent program is generated for each section, and then 3 independent programs are integrated into a single property evaluation program. By applying the program to the spherical indentation test, material properties are obtained from a single indentation load–depth curve. Finally, the nano-indention tests with spherical indenters are carried on Germanium Ge (100) and Silicon Si (100) to verify the proposed method by using the experimental load–depth curves.

2. Finite element model for spherical indentation

The axisymmetric FE model (Fig. 1) for the spherical indentation simulation is formed, and the commercial FE software Abaqus/standard (ver. 6.12) [23] is used for the FE analysis. Considering axisymmetry of both geometry and loading, 4-node axisymmetric elements (CAX4) are used to model the indenter and specimen. To capture the large deformation and steep stress gradient, the sub-indenter region is refined with relatively small elements. To merge the small elements with large elements, the trapezoidal elements are used near the contact region, where the constrained mid-nodes in multi-point constrain (MPC) [23] tends to give discrete stress and strain values [8]. Therefore, MPC is used only in the region far from the contact region. The axisymmetric FE model consists about 16,700 nodes and 16,000 elements. For the spherical indenter with diameter $D=1\,\mu m$, the diamond material properties (Young's modulus $E_I=1000$ GPa, Poisson's ratio $\nu_I=0.07$) are assigned, while the friction coefficient $f=0.1$ is considered in the contact between indenter and the specimen [11,24].

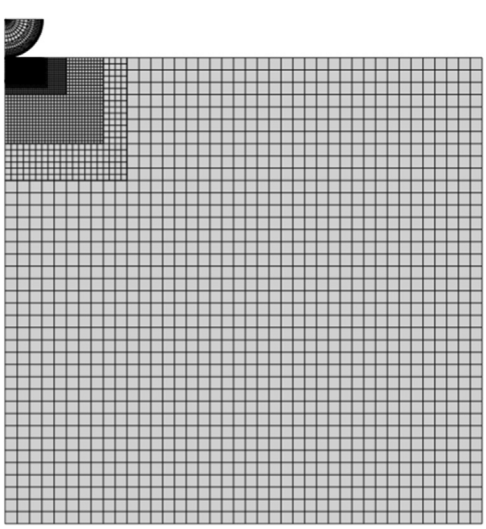

Fig. 1. FE model for $h_{max}/D=20\%$ indentation analyses.

A point at $l/D=0.3$, $2r/d=0.8$, where the strain gradient is relatively small [10], is selected as a data acquisition point to avoid the frictional effect on the effective stress σ and plastic strain ε_p measurements. Here l and r denote the vertical and radial distance, respectively, from the center of the contact, and d denotes the *actual* contact diameter. Note that the use of data acquisition point is only practical in FE simulations and cannot be applied in the experiments. The non-linear geometry change (NLGEOM) FE analyses are performed for the isotropic elasto-plastic materials, which obey J_2 incremental plastic theory.

In a shallow spherical indentation, the indenter deformation is insignificant due to relatively small indentation load P at shallow indentation depth; there is no finite indenter deformation. When the maximum indentation depth is relatively small, materials with different properties can yield almost identical load–depth curves, which result in significant errors of evaluated material properties [8,10]. It can be solved by increasing the indentation depth. In this study, 20% of indenter diameter is thus selected as a maximum indentation depth ($h_{max}/D=20\%$) as that of Lee et al. [10].

2.1. Material model for FE analysis

Eq. (1) is used here to express the stress–strain relationship with Hollomon's piecewise power law hardening material model [25].

$$\frac{\epsilon_t}{\epsilon_o}=\begin{cases}\frac{\sigma}{\sigma_o} & \text{for } \sigma\le\sigma_o\\ \left(\frac{\sigma}{\sigma_o}\right)^n & \text{for } \sigma\ge\sigma_o\ ;\ 1<n\le\infty\end{cases} \tag{1}$$

Here σ_o, ε_o $(=\sigma_o/E)$, ε_t are the yield strength, yield strain and total strain, respectively. ε_t $(=\varepsilon_e+\varepsilon_p)$ is equal to the sum of elastic and plastic strains and n is the strain-hardening exponent. The material is assumed as perfectly elastic for $n=1$ and elastic-perfectly plastic for $n=\infty$. For $1<n<\infty$, the material shows the hardening behavior. Differently from the Ramberg–Osgood material model in other studies, the power-law functions of Eq. (1) has clear distinction between elastic and plastic regions at ε_o.

2.2. Enhanced evaluation of indentation contact diameter

Fig. 2 compares the diamond indenter deformation with rigid indenter, when $E=300$ GPa, $\varepsilon_o=0.1$, $n=3$ are used as the specimen material properties. The diamond indenter deviates from the spherical shape due to large deformation. In FE study, d is calculated from the nodal coordinates of the last nodes in contact,

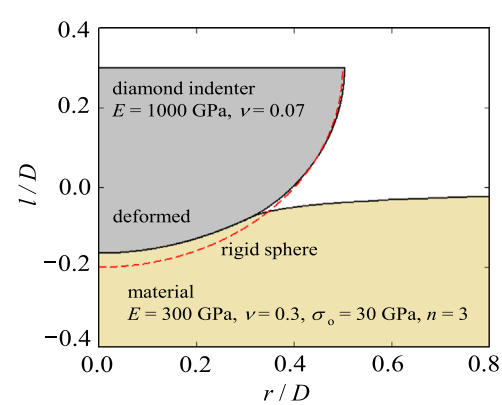

Fig. 2. Deformed shape of diamond indenter at loaded state.