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Modeling to evaluate the contact areas of hard materials during the nano-indentation tests

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ABSTRACT

A new mechanical model is developed in the present study for hard materials to investigate the behavior arising during the loading/unloading process of an indentation test. Two governing differential equations are derived for the depth solution of the indenter tip and the depth solution formed at the separation point expressed in a power form. The exponent value in either the loading process or unloading process is considered to be a variable as a function of the indentation depth in the governing differential equation. All coefficients shown in these governing differential equations associated with the spring and damping behavior are determined by the real-coded genetic algorithm. Quartz and silicon were used as the examples of hard materials, and the contact projected area predicted by the present model is quite close to the solution predicted by the area function of Oliver and Pharr [W.C. Oliver, G.M. Pharr, An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, J. Mater. Res. 7 (4) (1992) 1564–1583]. The phase lagging behavior demonstrated in the indentation test at two different loading/unloading rates was investigated, and it is enhanced by increasing the loading/unloading rate. No restriction for hard materials in the loading rate is needed in the indentation test if the present model is employed. The contact area is decreased by increasing the loading rate.

1. Introduction

Depth-sensing indentation techniques, which record the data of load, P, or displacement, h, during an indenter contact with a specimen, are used to obtain mechanical properties without measuring the indentation. In particular, a variety of methods were applied to analyze P–h curves and obtain the values of elastic modulus, E and hardness, H [1–5]. These methods have been predominantly used or verified on a variety of materials. For thin film and surface-graded systems, the depth-sensing indentation test, or nano-indentation, is a powerful tool by taking advantage of its evaluating mechanical properties under different penetrations [6]. The continuous stiffness method (CSM), which measures the contact stiffness continuously through tests under an oscillating load, allows the collection of continuous data of mechanical properties using one indentation [7].

However, ambiguity in interpreting P–h curves has been observed in soft materials because of the time-dependent response of indentation depth due to different loading/unloading rates [8]. In order to observe the time-dependent behavior of the loading process, an experimental approach allowing a sufficient time at peak load was used to detect creep [9]. Viscoelastic materials have been observed to not follow Kick's law due to creep, and the power value of h in the P–h relationship was dependent upon the loading rate [9]. Through dimensional analysis, the viscoelastic constitutive model concerning the load as a function of depth and depth rate and a description for the P–h relationship in the loading process has been developed by Ma et al. [10].

For polymer materials, Oyen and Cook [11] modified the Maxwell model by three parameters in order to determine the indentation displacement as a function of load and load history. The experimental P–h response under a proportional-loading-rate condition has shown a quadratic relationship [11]. In the study of Lucas et al. [12], the results of the proportional-loading-rate tests appeared to have a good correlation with the uniaxial data. At room temperature, the creep motions of hard materials are negligible compared with soft materials; however, they become significant in the case of a very high loading rate. Creep motions due to the collision of a Vickers indenter at a very high velocity have been observed by Nagn and Tang [13]. Indentation experiments had been performed on hard materials such as glass and quartz, and creep motions showed an asymptotic behavior that could be described

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by a Voigt system. The experimental creep data of glass has been described by the creep solution of the Voigt model in a study by Feng and Nagn [14].

The CSM is an indentation test that has a specified frequency by adding a small harmonic force on the indenter and measuring the harmonic response of the indenter at the excitation frequency [7]. Apart from the advantages of CSM in its investigation of the initial contact and excellent accuracy, the contact stiffness can be determined directly and the mechanical property data can be obtained at multiple depths from a single test. For viscoelastic materials, the measurement of storage modulus and loss modulus through CSM provides the elastic and viscous properties of viscoelastic materials.

The method to obtain precise contact area arising in the indentation of a specimen is of importance to evaluate the hardness and Young's modulus of a material correctly. A mechanical model is developed in the present study to investigate the contact behavior of the indenter and the specimen arising in the loading/unloading process of an indentation test. Quartz and silicon are adopted as the two examples of brittle materials which can produce the sink-in in the indentation process. Two governing differential equations are established for the depth at the indenter tip (h_i^*) and the depth formed at the separation point of the indenter and the specimen (h_s^*). The independent variables in these two governing equations are first expressed as a power of h_i^* and h_s^* with their individual exponent value. The exponent value of h_i^* or h_s^* in either the loading or unloading process is considered to be a variable as a function of the indentation depth, irrespective of the loading or unloading process. Based on the linear relationship satisfied between h_i^* and h_s^* [1], the exponent of h_s^* is proved to have the same value as that of h_i^*, irrespective of the exponent at large or small values. Before obtaining the h_i^* and h_s^* solutions, all coefficients shown in these two governing differential equations associated with the "elastic" and "viscous" behavior of the specimen are determined by the real-coded genetic algorithm (RGA). With the aid of the experimental results of h_i^* shown at large and small indentation depths, the h_i^* and h_s^* solutions are achieved when good agreements between the results predicted by the present model and the experimental results are obtained. The solutions of h_i^* and h_s^* valid at various indentation depths can be obtained by the asymptotic method. Then, the real contact area at any indentation depth can be calculated if the h_s^* solutions are available. All contact parameters are evaluated at two loading/unloading rates in this study in order to investigate the lagging behavior existed at different load/unloading rates. Two contact projected areas obtained from two kinds of area functions including the one associated with the present model are presented to compare the differences among them. The causes of these differences are also discussed.

2. Theoretical modeling

The viscoelastic and viscoplastic deformations describing the indentation behavior of hard (brittle) materials demonstrated during the loading/unloading process are developed in this section for two indentation depth parameters: the indentation depth created by the indenter tip (h_i) and the depth in which the indenter starts to deviate from the deformed surface of specimen in the loading/unloading process (h_s).

2.1. Oscillation-in-air tests

The indentation-in-air mechanical model is usually developed for the indenter clear of the specimen. This model is established by placing a spring in parallel with a damper between the indenter and its support (the capacitive depth gauge). The dynamic model for the oscillation-in-air tests is established according to the schematic diagram shown in Fig. 1(a). The spring constant and damping coefficient under this non-contact condition are defined as k_M and c_M, respectively. In order to determine the contact force formed between the indenter and the deformed specimen under a given indentation load, the k_M and c_M coefficients in the governing equation of the indentation depth must be solved beforehand. However, these two coefficients can be obtained from oscillation-in-air test. The oscillation-in-air tests with different frequencies were operating under the condition that the indenter is oscillating with a load

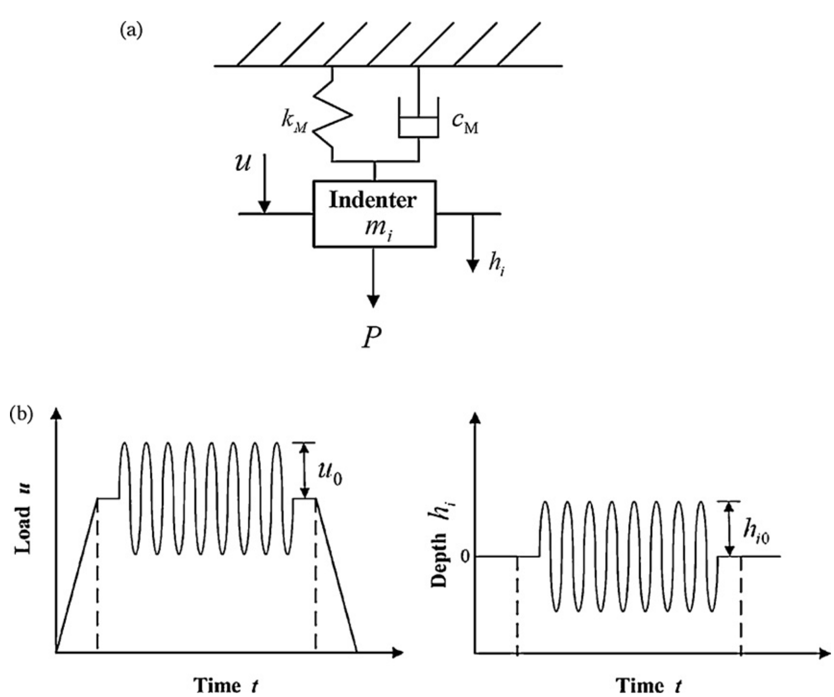

Fig. 1. Oscillation-in-air tests. (a) Indentation mechanical model in the air; (b) an oscillating load with an amplitude of u_0 and the resulting indentation depth amplitude h_{i0}. A frequency of ω is applied as the indentation load condition.