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A robust procedure of data analysis for micro/nano indentation

Wei Hang[a], Libo Zhou[b,*], Jun Shimizu[b], Julong Yuan[c]

[a] Graduate School of Science and Engineering, Ibaraki University, Nakanarusawa 4-12-1, Hitachi 316-8511, Japan
[b] Department of Intelligent Systems Engineering, Ibaraki University, Nakanarusawa 4-12-1, Hitachi 316-8511, Japan
[c] Key Laboratory of E&M, Zhejiang University of Technology, Chaowang Road-18, Hangzhou 310014, China

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ABSTRACT

This paper reported a robust procedure of data processing in micro/nano indentation for determining the elastic modulus and hardness. As both elastic modulus and indentation hardness strongly depend on the contact stiffness, the contact area and the accuracy with which they are determined, described in the first part of this paper was a comparison of three approaches to estimate the contact stiffness. From the experimental results, it is concluded that the contact stiffness computed from the curve fitting method is most reliable and robust. Subsequently in the second part of this paper, a new procedure for computation of the contact area was proposed by use of multivariable estimation via a least square fitting. In the final part of this paper, indentation tests on silicon and aluminum alloy were conducted to verify that the proposed procedure is not only valid for a variety of materials from hard-brittle to soft-ductile, but also robust and applicable to indenters with imperfect geometry.

1. Introduction

The elastic modulus E and hardness H are two most important parameters used for evaluation of the mechanical properties of materials. The instrument and measurement procedure are very critical for precise estimation of the values of E and H. The most extensively used method is load–displacement sensing indentation, in which no image of indent impression is required. Recently, many efforts have been made in developing micro/nano indentation equipment and techniques for probing the mechanical properties of materials and thin films on the sub-micron and nano-scale [1–4].

When a small indentation is made at sub-mN load and nanometer displacement, however, the measured load–displacement data becomes unstable because it always contains a certain degree of noise. The instability of acquired data will directly affect the calculation results of the elastic modulus and hardness. In addition, due to blunting and roundness at the tip of indenter, the contact area calculated from a perfect geometry is no longer valid especially in case of micro/nano indentation [5]. A proper procedure of data analysis and compensation is thus essential to determine the values of elastic modulus and hardness with high accuracy and precision. Reported in this paper is a robust approach of data analysis for determining the contact stiffness and contact area. Comparisons of results are made on different test materials (silicon vs. aluminum alloy) with different geometries of indenters (brand new vs. blunted), to demonstrate the stability and reliability of the proposed approach for data analysis in micro/nano indentation.

2. Theoretical expression of hardness and elastic modulus at micro/nano indentation

Micro/nano indentation test performs indenting a specimen by a very small load using a high precision instrument, which records both load and displacement simultaneously and continuously. Fig. 1(a) is a schematic representation of load–displacement curve, where F_{max} is the peak load, h_{max} is the indenter displacement at the peak load, h_p is the final depth of the contact impression after unloading. Fig. 1(b) shows a cross-section of the corresponding indentation and identifies the parameters used in the subsequent analysis. h_c is the contact depth of indenter at the peak load and h_s is the displacement of the surface at the perimeter of the contact. Thus, the relationship of $h_{max} = h_c + h_s$ is satisfied. Upon unloading, the elastic displacements are recovered, and final depth of residual indent impression is left as h_p. Fig. 1(c) is the top-view of the residual indent impression by a Berkovich type indenter, where a is the length from the center of indenter to the impression corner.

* Corresponding author. Tel.: +81 294 385217; fax: +81 294 38 5217.
E-mail address: lbzhou@mx.ibaraki.ac.jp (L. Zhou).

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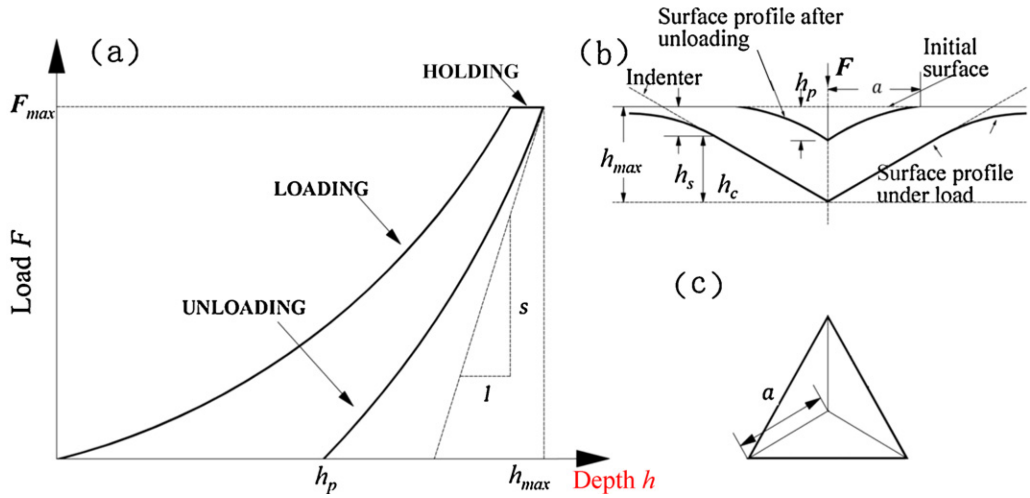

Fig. 1. A schematic representation of an indentation: (a) load–displacement curve, (b) cross-section of indentation process and (c) residual indent impression.

During indentation, the compliance of the specimen and the indenter tip can be combined as springs in series. Therefore,

$$\frac{1}{E_r} = \frac{(1-\nu_s^2)}{E_s} + \frac{(1-\nu_i^2)}{E_i} \quad (1)$$

where E_r is the "reduced modulus", E is the elastic modulus, ν is the Poisson ratio, and suffix i and s refer to the indenter and specimen, respectively.

The reduced modulus was first related to the contact stiffness of indentation by Sneddon [6] who yielded

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \quad (2)$$

where S is the contact stiffness and A is the contact area. Later, Pharr et al. [7] proved that Eq. (2) is a robust equation which applies to indenter with a wide range of geometries.

The indentation hardness is simply defined as

$$H = \frac{F_{\max}}{A} \quad (3)$$

It should be noted that hardness evaluated using Eq. (3) may be different from that of the conventional definition in which the contact area is determined by direct measurement of the size of residual indent impression. In the micro/nano indentation analysis the hardness is calculated utilizing the contact area at peak load whereas in conventional tests the area of the residual indent after unloading is used.

Table 1
Specifications of indentation instrument.

Load range	0.1–1960 mN
Load accuracy	±19.6 μN
Indentation depth range	0–10 μm
Indentation depth resolution	10 nm
Indenter type	Berkovich, Vickers and Knoop

From Eq. (2) and (3), it is easy to understand that both values, elastic modulus as well as indentation hardness, strongly depend on the contact stiffness S, the contact area A and the accuracy with which they are determined. Unfortunately, the contact stiffness S and the contact area A are unable to be directly derived from the acquired load–displacement data. A proper analysis procedure is therefore essential to get reliable contact stiffness and contact area.

3. Micro/nano indentation instrument and experimental procedure

The micro/nano indentation instrument used in this study was Shimadzu DUH-W201, as shown in Fig. 2(a). Its specifications are listed in Table 1. A diamond Berkovich indenter was used for indentation tests. Shown in Fig. 2(b) was a brand new indenter. Its tip angles of each side were 115°01′, 115°05′ and 115°08′, respectively. The departure from a perfect Berkovich indenter was very limited, and its effect on the area function was described in Section 5. Fig. 2(c) was a blunted indenter which was used to investigate

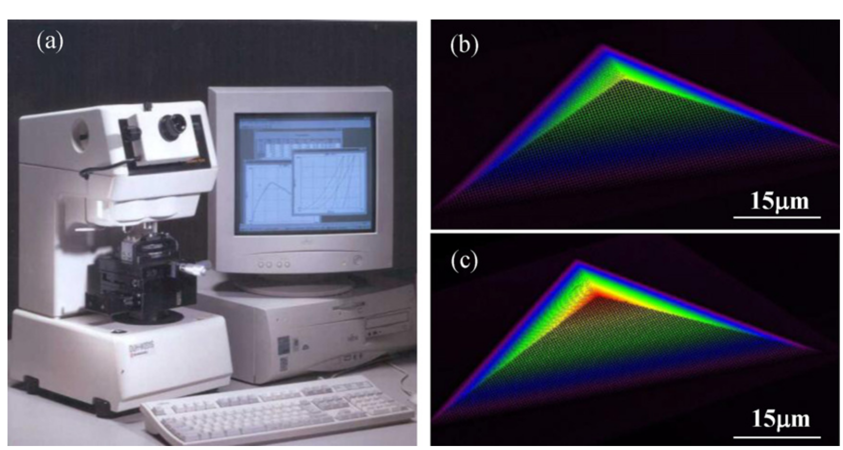

Fig. 2. Instrument and indenters used for experiments: (a) indentation instrument, (b) brand new indenter, (c) blunted indenter.