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Indentation of elastic solids with a rigid Vickers pyramidal indenter

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Abstract

Experimental investigations of the normal loading of a rigid Vickers pyramidal indenter on to several blocks of elastic solids, namely neoprene, rubber, and optically clear polydimethylsiloxane (PDMS) containing 10%, 5%, and 2.5% by volume of the curing agent have been described. An instrumented indentation machine was used and several types of measurement were made. These included (1) indentation load versus indenter penetration behaviour, (2) *in situ* photography of the contact area between the indenter and the substrate, (3) the depth of the points of contact where a plane going through an indenter diagonal and containing the indenter tip intersects the surface of the specimen, and the depth of the contact points lying along a direction at an angle of 45° to the planes containing the diagonals. The measurements were compared with the predictions of the theory of a rigid cone indenting an elastic half space and by assuming that the rigid pyramid could be likened to a cone of a semi-included angle of 70.3° . It is shown that in all cases there were significant discrepancies between the predictions of the theory and the experimental measurements. It is concluded that a rigid pyramidal indenter normally loading on to an elastic solid cannot be likened to a conical indenter for such studies. It is suggested that it is the high friction at the ridges of the indenting pyramid, which gives rise to the discrepancies between the experimental data and the theory for a frictionless indentation with a rigid cone. This conclusion has very significant implications for a commonly used method of nanoindentation data analysis. These implications are also discussed. © 2006 Elsevier Ltd. All rights reserved.

Keywords: Vickers indenter; Elastic solids; Geometric similarity; Nanoindentation; Data analysis; Friction

1. Introduction

During the past quarter of a century instrumented indentation machines have been used increasingly for determining Young's modulus and indentation hardness of bulk solids and thin coatings deposited on solid substrates. The most common indenter used is a three-sided pyramid, which has an equilateral triangular base. Such an indenter is known as a Berkovich indenter. Another commonly used indenter is a Vickers pyramid, which is four-sided and has a square base.

A typical experimental run with an instrumented indentation machine consists of loading normally a Vickers or Berkovich indenter on to the test surface

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Nomenclature

- α semi-included angle of the cone
- β numerical factor
- $A_{\rm c}$ calculated projected contact area of an indentation (see Eq. (3))
- A_{insitu} projected contact area of an indentation, as measured from photographs taken *in situ*
- *E* Young's modulus of the test solid
- $E_{\rm i}$ Young's modulus of a non-rigid indenter
- $E_{\rm r}$ reduced modulus for the case of the nonrigid indenter and is given by $\frac{1}{E_{\rm r}} = \frac{1-v^2}{E} + \frac{1-v_1^2}{E_{\rm i}}$
- v Poisson's ratio of the test solid
- v_i Poisson's ratio of a non-rigid indenter
- $H_{\rm e}$ elastic hardness calculated using Eq. (4)
- H_{insitu} elastic hardness measured *in situ h* penetration depth of the indenter tip below the original surface of the specimen for a given load, *P*, on the indenter
- $h_{\rm c}$ contact depth of indentation, as calculated using Eq. (2); it is also the distance between the indenter tip and the contact edge between the specimen and the conical indenter

and gradually increasing the load at a pre-determined rate to a pre-selected value and then unloading the indenter gradually to zero load. Throughout the indenter loading and unloading the indenter load versus indenter displacement with respect to the original surface of the specimen are recorded. In the case of an elastic-plastic solid, plastic flow will occur around the pointed indenter and when the indenter is unloaded and removed from the indented surface, a permanent impression will be left in the surface of the specimen. To determine the indentation hardness of the test solid, it is necessary to determine the size of the residual indentation. In the case of indentations of size 2-3 µm across and larger, optical microscopy provides an adequately accurate method. However, for indentations of smaller sizes, the use of other techniques, such as scanning electron microscopy or scanning probe microscopy is necessary (Bec et al., 1996; Lim and Chaudhri, 1999; Lim et al., 1999; Miyahara et al., 2002; Randall, 2002). In a commonly used method (Oliver and Pharr, 1992) and in a recently adopted international standard, ISO 14577-1:2002, it is advocated that an accurate

- $h_{\rm er}$ error in the measurement of the point of first contact between the indenter and the test solid
- $h_{in \, situ}$ penetration of the indenter tip below the original surface of the specimen, as measured from the photographic images
- h_{BB} contact depths of the points *BB* on the indenter (see Fig. 2), as measured *in situ*
- $h_{B'B'}$ contact depths of the points B'B' on the indenter (see Fig. 2), as measured *in situ*
- L_D length of the projected contact area diagonal measured *in situ*
- $L_{\rm S}$ length of the side of the projected contact area measured *in situ*
- *P* indenter load
- S stiffness of the elastic loading curve $\left(=\frac{dP}{dh}\right)$

estimate of the size of an elastic/plastic indentation can be made from an analysis of the unloading part of the load-displacement curve, which is generally thought to be a totally elastic process. Although the unloading of the indenter occurs within a permanent indentation formed during the loading cycle, the proposed method (Oliver and Pharr, 1992) likens the situation to the elastic loading/unloading of the indenter on to a flat half space. Moreover, the pyramid is likened to an axisymmetric cone (see also Bhattacharya and Nix, 1988) or a paraboloid of revolution, whose cross-sectional area at a given distance from its tip is exactly equal to the crosssectional area of the indenting pyramid at exactly the same distance from the tip of the pyramid (The cross-sectional area of a cone of a semi-included angle of 70.3° at a given distance from its tip is the same as that of the Vickers pyramid at the same distance from its tip.). Neither of the above two assumptions, that is, a pyramid can be likened to a cone as far the process of elastic normal loading is concerned and that the elastic unloading of the pyramid inside a permanent indentation is like loading of an equivaDownload English Version:

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