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Correcting power-law viscoelastic effects in elastic modulus measurement using depth-sensing indentation

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Abstract

The standard Oliver–Pharr method for measuring the elastic modulus by depth-sensing indentation makes use of the unloading response of the material as it is assumed that the unloading behaviour is purely elastic. However, under certain conditions, the unloading behaviour can be viscoelastic, and if the viscosity effects are not corrected, the calculated modulus can be seriously erroneous. Feng and Ngan have proposed a correction formula which can eliminate the creep effects. However, this formula has been proven to be correct for the case of linear viscoelasticity only; the general case of power-law viscoelasticity has not been proven. In this paper, this formula is proved for the general power-law viscoelastic situation using a Maxwell material model. Finite-element calculations are also performed to illustrate the result. The correction formula is applied to experimental data on amorphous selenium at ambient and elevated temperatures and is found to be effective in correcting for creep effects which are very prominent in this material.

Keywords: Mechanical properties; Nanoindentation; Elasticity; Creep; Hardness testing

1. Introduction

Depth-sensing indentation has become a standard technique for the measurement of the elastic modulus of small samples. In the well-known Oliver–Pharr method for modulus measurement (Oliver and Pharr, 1992), the reduced modulus E_r, defined as

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$$\frac{1}{E_r} = \left(\frac{1-\nu^2}{E}\right)_{\text{sample}} + \left(\frac{1-\nu^2}{E}\right)_{\text{indenter}} \tag{1}$$

is calculated from the contact stiffness S using the following formula which is derived from Sneddon's solution (Sneddon, 1965) to the elastic contact problem between a half space and an axi-symmetric punch:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}}. \tag{2}$$

Here ν and E are Poisson's ratio and Young's modulus, and A_c is the tip–sample contact area at full load $P_{\max}$. The contact area A_c is calculated from the contact depth h_c through a pre-calibrated shape function $A_c = f(h_c)$ of the indenter, and h_c is given by

$$h_c = h_{\max} - \varepsilon\frac{P_{\max}}{S}, \tag{3}$$

where $h_{\max}$ is the maximum indenter displacement, and ε is a constant depending on the indenter geometry ($\varepsilon = 0.72$ for the Berkovich tip).

Eq. (2) was derived based on the assumption that the material behaviour is purely elastic. However, many experiments have indicated that creep effects usually occur during nanoindentation (Mayo and Nix, 1988; LaFontaine et al., 1990; Baker et al., 1992; Ramman and Berriche, 1992; O'Connor and Cleveland, 1993; Syed and Pethica, 1997; Lucas and Oliver, 1999; Feng and Ngan, 2001a,b; Ngan and Tang, 2002; Li and Ngan, 2004). In low-melting metals, indentation creep can occur before general yield (Feng and Ngan, 2001a,b) but for high-melting metals, crystal plasticity is a pre-requisite condition for indentation creep (Syed and Pethica, 1997; Feng and Ngan, 2001a,b). In extreme creeping situations, the unloading curve can exhibit a "nose", meaning that the indenter can continue to sink into the specimen even though the load is decreasing. The conditions for the occurrence of unloading "nose" have been investigated by Ngan and Tang (2002). Recently, it has also been demonstrated that the stress exponent of nanoindentation creep exhibits a strong dependence on the indent size (Li and Ngan, 2004), indicating a transition of creep mechanism as indent size approaches the incipient plasticity situation.

On the specific question of how indentation creep affects the calculated modulus, a number of reports have indicated that the measured modulus can be seriously affected by creep (Chudoba and Richter, 2001; Feng and Ngan, 2001a,b; Feng and Ngan, 2002; Tang and Ngan, 2003). Summarising these findings, indentation creep will exert significant effects on the measured modulus when (i) the material itself is low-melting or soft, (ii) the load hold before unloading is too brief, (iii) the unloading rate is too slow, and (iv) the full load is too large. For reasonably hard materials like metals or ceramics at testing temperatures low compared to their melting points, the effects of creep can be eliminated by using an extended load hold or a rapid unloading rate, as is perhaps well-known. However, for soft materials like polymers or biological tissues, or if the aim of the experiment is to deliberately measure the elastic modulus at a high temperature compared to the melting point, the load hold or the unloading rate required to eliminate creep effects may be difficult to achieve in practice. Ngan and co-workers (Feng and Ngan, 2001a,b, 2002; Tang and Ngan, 2003) have developed correction formulas to eliminate creep effects in the post-experiment, data-processing stage. It was proposed that, in a viscoelastic situation, the correct elastic stiffness S_e can be calculated as

$$\frac{1}{S_e} = \frac{1}{S} - \frac{\dot{h}_h}{\dot{P}_u}, \tag{4}$$

where S is the apparent contact stiffness $\mathrm{d}P/\mathrm{d}h$ at the onset of unloading (P = load, h = indenter displacement), $\dot{h}_h$ is the tip displacement rate at the end of the load hold just prior to unloading, and $\dot{P}_u$ is the