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SciVerse ScienceDirect
Acta Materialia 60 (2012) 3953–3964
Acta MATERIALIA
www.elsevier.com/locate/actamat

Determination of the mechanical properties of amorphous materials through instrumented nanoindentation

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Received 16 January 2012; received in revised form 9 March 2012; accepted 18 March 2012
Available online 1 May 2012

Abstract

A novel methodology based on instrumented indentation is developed to determine the mechanical properties of amorphous materials which present cohesive-frictional behaviour. The approach is based on the concept of a universal hardness equation, which results from the assumption of a characteristic indentation pressure proportional to the hardness. The actual universal hardness equation is obtained from a detailed finite element analysis of the process of sharp indentation for a very wide range of material properties, and the inverse problem (i.e. how to extract the elastic modulus, the compressive yield strength and the friction angle) from instrumented indentation is solved. The applicability and limitations of the novel approach are highlighted. Finally, the model is validated against experimental data in metallic and ceramic glasses as well as polymers, covering a wide range of amorphous materials in terms of elastic modulus, yield strength and friction angle.

Keywords: Nanoindentation; Microindentation; Amorphous material; Yield phenomena; Micromechanical modelling

1. Introduction

Instrumented indentation, in which the load and depth of indentation are continuously recorded during the indentation process, is a simple testing technique, and a large research effort has been devoted to obtaining the elasto-plastic properties of materials from this test. Most of these studies focused on polycrystalline solids, in which the flow stress follows the von Mises criterion and is equivalent under uniaxial tension and compression [1–3]. This criterion is not representative, however, of the behaviour of amorphous solids such as ceramic glasses, bulk metallic glasses and both thermoset and thermoplastic polymers. They usually display brittle behaviour in tension, while deforming plastically in compression and/or shear, and the flow stress depends on the hydrostatic pressure [4]. As a result, the elasto-plastic properties of these cohesive-frictional materials are difficult to measure by conventional methods such as tensile testing, owing to their inherent brittleness. In contrast, they are readily deformed by indentation, owing to the large hydrostatic compression under the indenter tip, the small volume of deformed material and the constraint of the surrounding material. Therefore, instrumented indentation stands as an ideal technique for characterizing the elasto-plastic constitutive behaviour of cohesive-frictional materials.

Extracting material properties from instrumented indentation is, however, extremely challenging, because there is no one-to-one correspondence between the indentation curve and the material properties [3]. One of the main difficulties in relating both is found in the uncertainty in how the material accommodates the volume displaced by the

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http://dx.doi.org/10.1016/j.actamat.2012.03.027

indenter. On the one hand, purely elastic material accommodates the displaced volume by the elastic deformation of the surrounding material, leading to a sink-in of the material around the indenter and reducing the actual contact area. On the other hand, plastic flow displaces the material upwards around the tip, and the resulting pile-up around the indenter will increase the actual contact area in very ductile materials. Engineering materials normally present intermediate behaviour between these extremes, which has been extensively studied in elasto-plastic solids, following the von Mises yield criterion [5]. However, very little is known about this phenomenon for cohesive-frictional materials. Giannakopoulos and Larsson [6] and Vaidyanathan et al. [7] studied the pyramidal indentation of cohesive-frictional materials using the finite element method and, more recently, Narasimhan [8] and Patnaik et al. [9] analysed the applicability of the expanding cavity model [10,11] to studying the conical and spherical indentation response of elasto-plastic materials in which the flow stress depends on the hydrostatic pressure. However, none of these studies focused on the determination of the constitutive response of the material from the indentation curve, and there is no reliable methodology for estimating the yield stress and pressure sensitivity in amorphous materials from instrumented indentation. In fact, yield stresses of polymers and metallic glasses derived from indentation studies led to unreasonably large values [12].

This work aims to cover this gap by establishing a methodology to determine the constitutive elasto-plastic behaviour of cohesive-frictional materials from instrumented indentation. The methodology is based on a universal hardness equation for cohesive-frictional materials based on the novel concept of the characteristic indentation pressure. The actual universal hardness equation (independent of the ratio of flow stress to elastic modulus and of the pressure sensitivity) was obtained from a systematic numerical simulation of the process of sharp indentation in cohesive-frictional materials. Based on this universal hardness equation, the inverse problem was solved to derive the material properties (elastic modulus, flow stress and pressure sensitivity) from the indentation curve, and the validity and limitations of the new methodology were established. Finally, the method was validated experimentally in various amorphous materials that follow a cohesive-frictional behaviour (metallic and ceramic glasses as well as polymers) and with very different flow stress/elastic modulus ratios.

2. Theoretical considerations

2.1. Constitutive behaviour of cohesive-frictional materials

Amorphous materials deform plastically in compression by the formation of shear bands. Further loading leads to the localization of the deformation within the shear band (or in an array of parallel bands) without any strain hardening. There is a wide body of experimental evidence showing that the yield criterion for shear band formation in metallic [13–16] and ceramic glasses [17,18] as well as in polymers [19–22] depends on the hydrostatic stress and that the yield surface of these materials is adequately represented by either the Mohr–Coulomb or the Drucker–Prager yield criterion. Mathematically, the yield surface ($\Phi = 0$) is given by [23]

$$\Phi = \sqrt{3J_2} - d + \frac{I_1}{3}\tan\beta = 0 \tag{1}$$

where I_1 stands for the first invariant of the Cauchy stress tensor σ_{ij}, and J_2 for the second invariant of the deviatoric part of the Cauchy stress tensor according to

$$I_1 = \sigma_{ii} \quad \sigma'_{ij} = \sigma_{ij} - \frac{I_1}{3} \quad J_2 = \frac{1}{2}\sigma'_{ij}\sigma'_{ji} \tag{2}$$

The elasto-plastic behaviour of the material is thus characterized by the two elastic constants E and ν (assuming isotropic behaviour) and the two parameters which dictate the onset of plastic deformation, namely the cohesion d and the friction angle β, which controls the pressure-sensitivity of the material. Eq. (1) represents a conical surface in principal stress space with the vertex on the hydrostatic stress axis. The trace of the yield surface on the deviatoric plane is circular and leads to the von Mises yield criterion (with yield stress equal to d) in the particular case of $\beta = 0°$.

2.2. Analysis of instrumented indentation test

The output of an instrumented indentation test is the load–displacement curve during loading and unloading of the indenter, as shown in Fig. 1. The main parameters obtained from the test are the maximum indentation load P_{max}, the maximum indentation depth h_{max}, and the elastic stiffness upon unloading S. Another interesting factor is the elastic energy/total energy ratio, $W_e/W_t = W_e/(W_e + W_p)$, where the elastic energy W_e and the energy dissipated by plastic deformation W_p are shown in Fig. 1. It

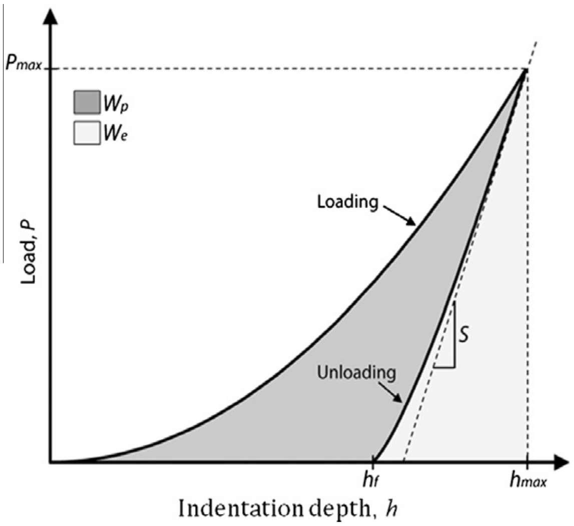

Fig. 1. Schematic of a load–indentation depth curve showing the most relevant experimental parameters that can be obtained from the test.