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Size effect in the initiation of plasticity for ceramics in nanoindentation

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Abstract

The indentation size effect has been observed for many years and is usually associated with increasing hardness as the depth of indentation is reduced for pyramidal indenters. The indentation size effect for spherical indenters has recently been associated with an increase in the yield stress of metals proportional to the inverse cube root of indenter radius [Spary, I.J., Bushby, A.J., Jennett, N.M., 2006. On the indentation size effect in spherical indentation. Philos. Mag. 86 (33), 5581–5593]. Here we investigate ceramic materials where the yield point is high enough to be easily distinguished in nanoindentation tests. A robust method for determining the yield point from a nanoindentation test with spherical indenters is presented. The results for a range of ceramics confirm that the increase in yield pressure is directly proportional to the inverse cube root of indenter radius. Furthermore, the yield pressure is also shown to be proportional to the inverse square root of the contact radius. Revisiting data in the literature shows that this inverse square root relationship is also true for pyramidal indenters. This implies that the indentation size effect is driven by the contact area rather than by the depth of indentation or by the indenter radius.

Keywords: Contact mechanics; Crystal plasticity; Ceramic material; Indentation and hardness; Indentation size effect

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1. Introduction

Small-scale mechanical behavior is at the cutting edge of research in materials science and applied mechanics. Nanoindentation testing is one of the most popular means for testing the mechanical properties of materials at small scales, because the size of the specimen can be extremely small and the procedure is usually non-destructive. It has been shown repeatedly that the indentation hardness of materials displays a strong size effect with pointed indenters. The measured hardness is seen to decrease as the depth of indentation increases, typically for metals by a factor two or three (Oliver and Pharr, 1992; Ma and Clarke, 1995; Nix and Gao, 1998). For spherical indenters, Lim and Chaudhri (1999) first showed that the entire flow curve appears at

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higher contact pressures for smaller radius indenters in oxygen-free copper. Similar results have been obtained in iridium by Swadener et al. (2002).

For the sharp-pointed indenters, there is significant plastic flow from an early stage in the indentation cycle. Consequently, few papers report a size effect at the initiation of plasticity. Spherical indenters induce a larger area of contact at small depths, and this enables the entire stress–strain curve to be measured, including the transition from the elastic regime to plastic deformation. However, there are still difficulties in characterizing initial yield behavior and defining a reproducible yield point. The yield strength of the ductile metals is so low that the elastic regime is below the resolution of the measuring system. In contrast, for hard ceramics, the main difficulty is the discontinuous yield phenomenon known as ‘pop-in’.

The hardness size effect has been explained in terms of strain gradient plasticity (Nix and Gao, 1998), in which arrays of geometrically necessary dislocations are required to accommodate the plastic strain gradient around the indentation, which is steeper for small contact depths. An increased rate of hardening is expected due to the presence of these dislocations interacting with existing dislocations in metals. Similar arguments have been proposed to explain the spherical indenter size effect (Durst et al., 2006; Swadener et al., 2002), in which the size effect appears to vary with indenter radius rather than contact depth. On the other hand, Lim and Chaudhri (1999) argued that strain gradient plasticity did not explain their spherical indentation data for copper. They attributed the size effect to homogenous nucleation of dislocations. Gerberich et al. (2002) reported an interesting interpretation of the indentation size effect, based on the surface to volume ratio. They proposed that the work done by an applied indentation load contains both bulk and surface terms. They found that the hardness of a wide range of metals increases with the inverse cube root of the indenter radius. Recently, Spary et al. (2006) demonstrated that the spherical indenter size effect could be simulated in a finite element model by simply increasing the initial yield strength in the constitutive equation for the material. They also found that the initial yield strength of a wide range of metals increased linearly with the inverse cube root of indenter radius. Gerberich et al. (2002) found the same dependence for hardness. An increase in the initial yield stress cannot be explained by strain gradient plasticity according to the model where the effect is caused by geometrically necessary dislocations, since at the yield point there is no plastic strain gradient. In this paper, we show that the size effect in the initial yield strength, implied for metals by finite element modeling (Spary et al., 2006), is clearly observed in ceramics. We demonstrate rigorous methods to determine the onset of plasticity in ceramics using spherical nanoindentation, even when pop-in occurs. This enabled us to measure well-defined and reproducible yield pressures of ceramics with a high degree of accuracy and over a large range of indenter radii (hundreds of nanometers to several tens of micrometers). Consequently, clear relationships among yield strength, indenter radius and contact radius have been observed accurately. The key result is an inverse square-root dependence of yield strength on the indent contact radius.

2. Experimental details

In nanoindentation tests the material response is detected throughout the loading cycle by continuously recording the force and penetration depth. Using a partial unloading (quasi-static) or continuous stiffness (dynamic) method of monitoring the elastic recovery of the specimen, the transition from elastic to plastic behavior can be determined. In our experiments, we used spherical indenters with the multiple partial unloading technique (Field and Swain, 1993). The indentation loading proceeds incrementally. Following each load increment to a force F_i, the load is reduced (partial unloading) by a small amount (in our experiments, to $0.75F_i$) before proceeding to the next higher load increment, F_{i+1}. Force and penetration are recorded at each load and partial unload step. Assuming that the unloading is elastic, Hertzian mechanics (Hertz, 1882) can be applied to calculate the radius, a, of the circle of contact made between the indenter and the specimen surface for each load increment (see Appendix A, Eq. (A.2), (A.3), (A.4), (A.6). Knowing a as a function of load indentation stress is expressed as the mean pressure acting over the projected contact area, $P_{\mathrm{m}} = F/\pi a^2$. Indentation strain is expressed as a/R, where R is the spherical indenter tip radius. The indentation strain is a normalized contact dimension, which expresses the geometry of the contact regardless of the spherical tip radius. (Bushby, 2001). Then the plot of P_{m} against a/R is the so-called indentation stress–strain curve.

Nanoindentation tests were conducted on a UMIS 2000 instrument (CSIRO, Lindfield NSW, Australia). The ceramics investigated were: (i) a sapphire, α-Al_2O_3 $(1\,1\,\bar{2}\,0)$-oriented single crystal; (ii) a 2.5-μm-thick layer