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Application of a modified slip-distance theory to the indentation of single-crystal and polycrystalline copper to model the interactions between indentation size and structure size effects

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Abstract

Plasticity size effects offer both measurement challenges and opportunities for material engineering. We have used nano-indentation to study the relationship between different size effects. Hardness varies significantly with indent size in single crystals, and also in polycrystals, whenever indent sizes and structure sizes are within an order of magnitude of each other. We exploit the geometric self-similarity of a Berkovich indenter and apply slip distance theory to indents of different sizes at a constant indentation strain. We show that indent size, grain size and pinning defects combine in a single, length-scale-dependent deformation mechanism, to determine the yield strength (hardness) of a material. This provides an excellent foundation for: improved grain size determination by indentation, design rules for combining different methods of yield stress enhancement and using indentation to probe local stress–strain properties of a material, or for mapping residual stress.

Keywords: Size effects; Nanoindentation; Grain size; Crystal plasticity; Dislocation density

1. Introduction

It is becoming increasingly important to understand and develop fully validated models for plasticity size effects. Size effects cause both problems and opportunities. Most finite element simulations use continuum mechanics as the basis for the constitutive properties of a material having an elastic modulus, a Poisson ratio, a yield stress (deviation from the elastic proportional limit) and a plastic work-hardening relationship (where the yield stress of a strained material increases with plastic strain). Input data to FE models are often in the form of a true stress–strain curve obtained by uniaxial testing of a bulk material, which is assumed to be in the same work-hardened state as the material to be modelled. Continuum mechanics assumes that there is no length scale dependence in material properties (of particular importance here – the yield stress), although the yield stress is allowed to increase with work-hardening. However, enhanced yield (or flow) stress of materials linked to an indentation size effect (ISE) or to a structure size effect (SSE), is known to occur when some critical dimension such as contact size or grain size is reduced [1,2]. In most cases, strength enhancement is proportional to an inverse square root of dimension; the most well-known being (arguably) the Hall–Petch (H–P) effect [3,4]:

$$\sigma(\varepsilon) = \sigma_0(\varepsilon) + \frac{k_{HP}(\varepsilon)}{\sqrt{d}} \tag{1}$$

where σ is the maximum elastic stress sustained by a material (also called the flow stress) at the particular strain ε, k_{HP} is the H–P coefficient, d is the mean grain size, and σ_0 is that of an infinite single crystal work-hardened to the particular strain ε. Whilst a variation in yield stress (hardness) due to a material microstructure can be treated as if it is a constant (length scale independent) material parameter,

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variation in hardness as a function of both material grain size and indentation contact size renders FE models unable to model compression of small sized samples or small mechanical contacts using the usual bulk property input data. "Smaller is harder" i.e., smaller contact size results in a genuine increase in the yield stress (hardness) of that contact. This means that attempts to calibrate instrumented indentation test (IIT) systems using hardness blocks becomes extremely problematic as blocks are generally certified for hardness only at a single indentation size. In conventional hardness scales, indentation size effects suggest that the practice of correcting hardness tests for systematic errors using an estimated offset derived from a hardness block is invalid if the indent sizes differ significantly. Size effects are so significant a contribution to indentation response that high resolution quantitative constitutive property mapping or residual stress mapping by using two-dimensional arrays of small indentations is not possible without an ability to evaluate and correct for the indentation size effect. Conversely, the ability to use length-scale engineering to enhance material properties and performance is of great technological interest. Higher performance components are often achieved by surface engineering, but in structural components the entire material must be improved. An increase in the yield stress and toughness of components can increase their lifetime and/or enable a reduction in size (mass), which for vehicle components, translates directly into fuel efficiency. Fatigue lifetime is also closely related to the yield stress of materials. An ability to length-scale engineer enhanced strength is directly applicable to a wide range of industrial sectors (from automotive and aerospace through to the electronics and communications sectors). There is, therefore, a requirement to validate size effect theories and measurement methods to enable length scale engineering of materials; the goal being structural materials with an increased yield stress that retain or increase their toughness (typically ductility) and the ability of components to fail "gracefully".

Early theories developed to explain the H–P phenomenon often invoked a restriction of dislocation movement that resulted in dislocation "pile-up" at grain boundaries causing a back stress [4]. However, more recently, Dunstan et al. have shown that a theory based on dislocation slip-distance plasticity size effect mechanisms [5], originally suggested by Conrad et al. [6,7], is able to explain not only the H–P effect, but also the fact that the uniaxial, microcompression of single crystal pillars also follows an H–P like enhancement of yield strength (proportional limit) with pillar diameter – yet without any constraint at the free surfaces. Indeed, work by Jennett et al. has shown that tungsten single crystal monolithic structures exhibit a "thinness effect" where walls (thin in one lateral dimension) exhibit the same yield stress as pillars of the same width in both lateral dimensions, and which exhibit the same inverse square root of size strengthening as H–P grains [8]. This result is very strong evidence for a purely dimension-related mechanism, since pillars and walls of the same "thinness" have the same yield stress but have very different volume, surface area and surface-to-volume ratio.

The scaling of the ISE has been shown by Spary et al. [9], when indenting metal single crystals, to be material-independent and dimension-related. Bushby et al. demonstrated exactly the same effect for ceramics and metals; both sets of results follow the same function of inverse square root of indent dimension when normalized for yield strain [10]. Hou et al. indented Cu polycrystals to show that the SSE and ISE combine; the hardness of a single crystal depends on indent size and a further H–P like hardening is seen, as grain size is reduced, but only when grain size is less than six times the contact radius [2].

In this paper, we investigate further the combination of indentation size effect and H–P effect in a range of Cu polycrystal grain sizes, using spherical and Berkovich indentation, the latter providing a variable contact dimension at constant indentation strain. We show that our results are entirely consistent with the modified slip distance theory of Dunstan et al. [5] and that ISE and SSE do appear to be equivalent components of the same effect, at least for face-centred cubic (fcc) Cu. We demonstrate that the flow stress of a material can be modelled as a function of the average spacing of obstacles to dislocation movement, whether they be free surfaces, grain boundaries, plastic zone boundaries or other pinning points such as sessile dislocations. We also show that differences in the work-hardened state as a result of indenting to different indentation strains can be identified.

2. Model derived from slip distance theory and spherical indentation

Hou et al. [2] demonstrated that indentations of different sizes (at the same strain) made into different grain size Cu polycrystals could be plotted using a single H–P relationship by combining the individual length scales for indentation size and grain size into a single length scale, D, as given in:

$$P_{\mathrm{m}} = P_0 + \frac{k_{\mathrm{HP}}}{\sqrt{D}}, \quad \text{where} \frac{k_{\mathrm{HP}}^2}{D} = \frac{k_1}{a} + \frac{k_2}{d} \tag{2}$$

where (at a particular and constant strain) P_{m} is the mean indentation pressure (hardness); P_0 is the hardness of an infinite single crystal; k_{HP} is an H–P-like constant; a is the contact radius; and k_1 and k_2 are scaling parameters.

Originally, this relationship was empirically determined, although the mathematical form was driven by expectations based upon critical thickness theory [11]. It has subsequently been shown that this is exactly the type of relationship that would be predicted by slip distance theory [5,7]. In slip distance theory, plastic strain, $\varepsilon_{\mathrm{pl}}$, is provided by mobile dislocations moving an average mean free path, $\bar{x}$:

$$\varepsilon_{\mathrm{pl}} = b\rho_{\mathrm{m}}\bar{x} \tag{3}$$

where ρ_{m} is the density of mobile dislocations and is a fraction λ of the total dislocation density ρ_{Total}; b is the Burgers vector in the direction of slip. Thus: