Available online at www.sciencedirect.com
ScienceDirect
Acta Materialia 56 (2008) 511–517
Acta MATERIALIA
www.elsevier.com/locate/actamat

Sample-size effects in the yield behavior of nanocrystalline nickel

A. Rinaldi [a,b], P. Peralta [a], C. Friesen [a], K. Sieradzki [a,*]

[a] Fulton School of Engineering, Arizona State University, Tempe, AZ 85287, USA
[b] University of Rome "Tor Vergata", Via della Ricerca Scientifica, Roma 00133, Italy

Received 5 June 2007; received in revised form 28 September 2007; accepted 28 September 2007
Available online 3 December 2007

Abstract

The compressive plastic strength of nanometer-scale single-crystal metallic pillars is larger than that found in conventionally sized samples. This behavior is generally associated with a change in the length scale that determines plastic behavior and the consequent inability of nanoscale samples to store dislocations. Here, we show in the case of nanocrystalline nickel pillars, for which there is a fixed microstructural length scale set by the grain size, that smaller is still stronger and find that this behavior derives from statistical expectations that have long been used to understand the size-dependent strength of brittle solids such as glass.

Keywords: Yield strength; Crystal plasticity; Extreme value statistics

1. Introduction

According to conventional solid mechanics, the plastic properties of a metal are independent of size [1]. However, this view is based on being able to select a representative volume element (RVE) of the solid that incorporates all of the microstructural characteristics of a correspondingly larger volume element. Since the plastic properties of a metal are governed by its dislocation content, the grain size, size and spacing of second phase particles, etc., as long as the sample size is significantly larger than the largest of these length scales (here notionally defined as a RVE), one observes sample-size-independent plastic behavior.

Consider the plastic behavior of a perfect, defect free, single crystal of finite size that has been "cut" or somehow machined from a larger sample. If such a sample is deformed, plasticity will initiate by the heterogeneous nucleation of dislocations at the free surface of the sample [2–4]. Surface defects, either in the form of steps (i.e. a vicinal surface) or "scratches" (that may result from machining), will act as stress concentrators and the initial nucleation event will occur at the most severe of these. Imagine now that we investigate the response of smaller samples. Similar behavior would be observed except that the stress level required for nucleation would, on average, increase because the likelihood of having a similarly sized defect or stress concentrator would be reduced owing to the reduction in the sample area. In general, then, this would result in sample-size-dependent plastic behavior at all length scales and in a well-defined statistical distribution for sample strength at fixed sample size.

Now consider the behavior of a real single crystal that contains some fixed density, ρ, of dislocations. The microstructural length scale is set by the mean spacing, $\rho^{-1/2}$, of dislocations. When the sample dimension, L, is large enough such that many RVEs are contained within the sample, conventional plastic behavior is observed. In this case, bulk dislocation sources apparently operate at stress levels below that associated with the operation of surface sources unless there is a large surface-stress concentrator. However, when L is of order $\rho^{-1/2}$, a number of different scenarios can evolve. Here, for example, bulk dislocation sources may operate as in the case of conventionally sized samples, or if there is no suitable bulk source, nucleation may occur at the sample surface as in the case of the perfect

* Corresponding author.
E-mail address: karl.sieradzki@asu.edu (K. Sieradzki).

1359-6454/$30.00 © 2007 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.
doi:10.1016/j.actamat.2007.09.044

crystal. In either case, dislocation interactions would not evolve as they do in samples containing many RVEs, since mobile dislocations would have a high probability to exit the sample prior to interacting with other dislocations. As L decreases, the likelihood that these smaller volumes contain any dislocations is further reduced and eventually dislocation production must cross-over from volume to surface sources.

A well-annealed metal crystal contains about 10^6 dislocations per square centimeter of surface, with a resultant dislocation spacing of the order of 10 μm. When the sample dimensions approach this microstructural length scale, one should begin to observe qualitative changes in the plastic behavior of these materials. This has now been observed in compression testing of pillars machined by focused ion beam (FIB) [5–9]. Specifically, the yield strength, σ_y, has been examined as a function of pillar diameter, D, and power-law scaling of the form $\sigma_y \propto D^{-\beta}$ has been reported. Various mechanisms and explanations have been proposed to explain this behavior, many of which predict this scaling [5,8,10–12].

In order to differentiate between these mechanisms, a fundamental question is the following. During plastic deformation of these nanoscale structures, do the dislocations originate from surface or volume sources? One may be tempted to address this question by simply deforming these structures while viewing the outcome using an appropriate microscope; however, this is like looking for a needle in a haystack. Even if such an experiment were feasible, we require an answer that "averages over" the results of many events rather than one based on the results of a few observations. A different approach to this that we have pursued is to examine the strength fluctuations at a fixed sample size and to use these results to ascertain the location of the operative dislocation source.

In nanoscale pillars which contain few, if any, dislocations the likelihood of activating a dislocation source at a particular level of stress is determined by the statistical distribution in source strengths. The source requiring the lowest stress for activation will be responsible for the first microscopic yield event. The statistics of this "weakest-link" concept are classically described by the Weibull distribution, which has been used for many years to understand the size-dependent fracture strength of brittle materials [13,14]. The difference between application of this idea to plastic yielding and brittle fracture is that in the latter case these defects result in a fracture event which is always observable. In conventionally sized structures, the initial yield event is virtually imperceptible; observable yield occurs by the gradual build-up of plastic strain owing to the operation of many dislocation sources. This has led to different engineering definitions of yielding as applied to the behavior of nanoscale pillars [5–10]. In nanoscale samples such as those discussed here, the number of potential dislocation sources is significantly reduced so that initial yield events evolving from a single source are observable. If the first dislocation that is nucleated from a weakest-link source moves out of the crystal or into a grain boundary and the geometric configuration of the source remains unaltered, we can expect continued nucleation from this source. As long as this situation prevails and no other sources are activated, the weakest-link scenario should be a good statistical model of yield behavior.

The Weibull distribution has the form

$$F(\sigma; L^d) = 1 - \exp[-L^d(\sigma/\sigma_0)^m]$$

where $F(\sigma; L^d)$ is the failure probability, L is a characteristic sample dimension (e.g. pillar diameter), $d = 2$ or 3, σ_0 is the so-called shape factor and m is the modulus [13,14]. When comparing two samples, L^d and a reference, L^d_{REF}, at constant probability of failure, $F(\sigma; L^d) = F(\sigma_{\mathrm{REF}}; L^d_{\mathrm{REF}})$, $\sigma/\sigma_{\mathrm{REF}} = (L^d_{\mathrm{REF}}/L^d)^{1/m}$ or $\sigma \propto L^{-d/m}$. Since experiments find $\sigma_{\mathrm{YIELD}} \propto D^{-\beta}$, we can identify L with D and $\beta = d/m$. Therefore, statistical evaluation of m should allow for the determination of whether surface ($d = 2$) or volume ($d = 3$) defects control the observed yield behavior of nanopillars.

2. Plastic behavior of nanocrystalline Ni

In order to test these ideas, we have chosen to examine the behavior of electrodeposited nanocrystalline Ni (nc Ni) obtained from Integran with a mean grain size of ∼30 nm. There is a log-normal grain size distribution in this material, with the largest grains being ∼50 nm [15]. While the plastic properties of nc Ni have received considerable attention, there is not yet a complete understanding of the mechanisms of inelastic behavior. There seems to be general consensus on the following [16–18]. For grain sizes above ∼20 nm, dislocation-mediated plasticity is operative. Below ∼10 nm in grain size, a majority of the plastic flow occurs by grain boundary sliding and/or Coble creep. This transition in the operative plastic flow mechanism depends on the elastic work required for the nucleation of the trailing partial dislocation, which is nominally described by the unstable stacking energy, γ_{us}, and the stacking fault energy, γ_{sf}, which defines the separation between the leading and trailing partial dislocation [19]. In nc materials, when the ratio γ_{sf}/γ_{us} is small in comparison to unity, other plasticity mechanisms such as grain boundary sliding or Coble creep become operative. While there have been reports of dislocation-mediated plasticity at grain sizes of 30 nm, there is no evidence of dislocation pile-up or storage in nc Ni [16]. Thus, during deformation of 30 nm grain size nc Ni, there is no mechanism for microstructural evolution of dislocation structures that can result in work hardening or eventual flow at reduced levels of stress owing to the formation of dislocation pile-ups.

3. Results of compression tests of nc Ni pillar arrays

The results of preparation of the FIB-machined nc Ni pillar arrays, together with a montage of stress–strain curves under compression loading are shown in Fig. 1