Available online at www.sciencedirect.com

ScienceDirect

Acta Materialia 65 (2014) 326–337

Acta MATERIALIA

www.elsevier.com/locate/actamat

Emergence of localized plasticity and failure through shear banding during microcompression of a nanocrystalline alloy

Amirhossein Khalajhedayati [a], Timothy J. Rupert [a,b,*]

[a] Department of Chemical Engineering and Materials Science, University of California, Irvine, CA 92697, USA
[b] Department of Mechanical and Aerospace Engineering, University of California, Irvine, CA 92697, USA

Received 30 August 2013; received in revised form 23 October 2013; accepted 25 October 2013
Available online 6 December 2013

Abstract

Microcompression testing is used to probe the uniaxial stress–strain response of a nanocrystalline alloy, with an emphasis on exploring how grain size and grain boundary relaxation state impact the complete flow curve and failure behavior. The yield strength, strain hardening, strain-to-failure and failure mode of nanocrystalline Ni–W films with mean grain sizes of 5, 15 and 90 nm are studied using taper-free micropillars that are large enough to avoid extrinsic size effects. Strengthening is observed with grain refinement, but catastrophic failure through strain localization is found as well. Shear banding is found to cause failure, resembling the deformation of metallic glasses. Finally, we study the influence of grain boundary state by employing heat treatments that relax nonequilibrium boundary structure but leave grain size unchanged. A pronounced strengthening effect and increased strain localization are observed after relaxation in the finer grained samples.

Keywords: Nanocrystalline metals; Yield behavior; Compression test; Nickel alloy; Grain boundary relaxation

1. Introduction

Nanocrystalline metals, commonly defined as polycrystals with a mean grain size (d) of less than 100 nm, are promising structural materials [1], mainly due to reports of high strength [2,3], fatigue resistance [4,5] and wear resistance [6–8]. When grain size is reduced below ~100 nm, new physical mechanisms begin to carry plastic deformation. First, there is a shift to plasticity that is controlled by grain boundary sites acting as sources and sinks for dislocation activity. Van Swygenhoven et al. [9] used molecular dynamics simulations to study the deformation physics of nanocrystalline Al and found that dislocation nucleation and propagation were limited by activity at grain boundary sites. Interestingly, they found that these dislocations often became pinned at grain boundary ledges as they moved through the grain, giving the spacing between boundary pinning points as the characteristic length scale of the mechanism. This interpretation has been supported by Huang et al. [10], who showed that experimental data could be well-described by a model that invokes Orowan-type pinning of dislocations with the grain size taken as the distance between obstacles. The behavior of nanocrystalline materials with grain sizes below ~10–20 nm has been attributed to the emergence of grain boundary sliding and rotation as the dominant carriers of plastic deformation. Schiøtz et al. [11,12] were the first to report such a mechanism when they detected local sliding events during molecular dynamics simulations of nanocrystalline Cu.

The common feature of the new deformation physics described above is the increased importance of grain boundaries as facilitators for plastic deformation. Since grain boundaries are more abundant and more important in nanocrystalline systems, increased attention has been

* Corresponding author at: Department of Mechanical and Aerospace Engineering, University of California, Irvine, CA 92697, USA. Tel.: +1 949 824 4937.
E-mail address: trupert@uci.edu (T.J. Rupert).

1359-6454/$36.00
http://dx.doi.org/10.1016/j.actamat.2013.10.074

focused on studying the atomic structure of these interfaces. A number of studies have reported that nanocrystalline metals often contain nonequilibrium grain boundaries, characterized by excess free volume or grain boundary dislocations, in their as-prepared state [13,14]. However, this nonequilibrium structure can be easily relaxed by applying low temperature heat treatments [13,15], with a more ordered and connected grain boundary structure found after relaxation. Perhaps not surprisingly since boundaries are so important for nanocrystalline plasticity, reports have shown that mechanical strength is highly dependent on this grain boundary structural state. Detor and Schuh [16] showed that grain boundary relaxation resulted in a significant increase in hardness, even though grain size was unchanged. Rupert et al. [17] further isolated this effect through systematic nanoindentation at different grain sizes, showing that this hardening occurred quickly and was grain-size-dependent.

The discussion above highlights the fact that novel deformation physics control plasticity in nanocrystalline materials and shows that these mechanisms are sensitive to grain boundary state. However, the vast majority of studies which probe mechanical behavior systematically as a function of grain size or grain boundary state rely on indentation experiments (e.g., Refs. [16–21]). While such techniques allow for large numbers of tests and only require small volumes of material, they also only give a scalar measurement of strength. As a result, the community has very little information about how the novel deformation mechanisms in nanocrystalline materials impact more complex behavior, like strain hardening and failure. In addition, nanoindentation imposes a complex, three-dimensional stress state, which makes it difficult to connect with constitutive theories for yielding and also adds concerns about the effect of a large confining pressure on plasticity. In order to study strain hardening behavior, full plastic flow and the failure of nanocrystalline materials in a straightforward manner, a simple uniaxial tension or compression test is required. However, such testing has to date been problematic as premature failure can occur for two main reasons: (1) improper specimen geometry and (2) processing defects.

Early attempts at uniaxial testing of nanocrystalline materials largely consisted of creating dog-bone specimens from thin sheets of nanocrystalline metals (e.g., Refs. [22–24]). This means that the samples commonly had thicknesses that were orders of magnitude smaller than the in-plane dimensions. Such geometries are problematic, as they introduce a geometric sample size effect, with strain-to-failure decreasing as sample thickness decreases [25]. Brooks et al. [26] explored this effect specifically in nanocrystalline Ni, showing that samples with thicknesses below ~100 μm experienced macroscopically brittle fracture that was not representative of the intrinsic material response. Zhao et al. [27] showed that by making sample geometries defined by ASTM Standard E8 [28], the strain-to-failure becomes independent of thickness, leading to a recommendation that the comparison of strain-to-failure measurements of a nanocrystalline material taken from different tensile test specimen geometries should be done cautiously.

Another complication that precludes the simple application of traditional uniaxial testing techniques to nanocrystalline materials is the effect of processing defects. Research has shown that nanocrystalline materials are commonly plagued by incomplete consolidation of particles, surface flaws, sulfur-induced grain boundary embrittlement and hydrogen pitting, all of which can cause premature failure [23,29,30]. For example, nanocrystalline Cu [31] and Ni–Fe [30] showed increased strain-to-failure with improved processing chemistry that reduced particulate contamination and hydrogen pitting. Brooks et al. [26] also showed that nanocrystalline Ni samples produced by an optimized process experienced twice as much plastic strain before failure, while the samples without this optimization always failed at large void-like defects produced when hydrogen gas was trapped in the deposit. Therefore, without having a proper geometry for mechanical testing and samples that are free of processing defects, conventional testing methods cannot provide us with accurate results and an alternative uniaxial testing technique is needed to adequately probe the plastic flow and failure response of nanocrystalline materials.

Recently, microcompression testing has become a promising and reliable technique that can be used to acquire the mechanical properties from a small volume of material [32,33]. Although this testing method is often used to study the effects of external sample size on mechanical behavior (e.g., Ref. [34]), such micropillars can actually serve as a bulk mechanical testing technique if the characteristic length scale associated with the microstructure of the material is much smaller than the pillar size. For a material with a grain size in the nanometer range, hundreds of thousands to millions of crystallites will be contained inside a pillar with a diameter of at least a few microns. For this reason, we suggest that micropillar compression can be used to measure the intrinsic properties of nanocrystalline materials. With microcompression, one can use sample aspect ratios that are small and within the range of ASTM standards while also minimizing the possibility of processing voids and defects being trapped in the small volume of material that is probed.

In this paper, we use uniaxial microcompression testing to study the full flow curve and failure behavior of a nanocrystalline alloy, with a specific focus on understanding the importance of grain size and grain boundary relaxation state. Nanocrystalline Ni–W was chosen as a model system, since grain size can be easily manipulated during electrodeposition, and this system has been studied extensively with nanoindentation [16,35,36]. To the authors' best knowledge, this is the first study to systematically explore uniaxial flow and failure in specimens with grain sizes from near 100 nm to below 10 nm. By studying this entire range, we are able to probe the effects of the entire gamut of