Mechanical Behavior of Bulk Nanostructured Materials

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1. Introduction

This NIRT program involves a fundamental investigation into the mechanical properties of bulk nanocrystalline (nc) materials, in which grain sizes are in the range 1-200 nm. Understanding mechanical properties is necessary since materials in almost all devices experience mechanical loads that must be supported, and must be predicted to produce reliable designs. Furthermore, understanding the mechanical properties of nc-materials will significantly impact future applications and technological advances in view of several considerations

2. Objectives

The primary objective of the NIRT program is two-fold: (a) to understand the mechanical properties of nc-materials, particularly including their strength, ductility, fatigue, and superplasticity, and (b) to clarify the basic deformation mechanisms that operate in nc-materials. In addition, the program aims at providing graduate and undergraduate students with educational experiences in the area of nanotechnology that will contribute to their training, skills, knowledge, and professional careers.

3. Major research findings

3.1. ***In situ high-resolution TEM studies of indentation***. The objective of this research task is the use of high-resolution studies to reveal and clarify mechanisms of deformation that are important at the nanoscale. This activity is motivated by the fact that in-situ nanoindentation in a TEM makes it possible to explore the role of microstructural length scale in the evolution of plasticity. Previous studies as reported by several investigators showed the dislocation nucleation and evolution during in situ indentation on grains with a grain size of 250nm-400nm.

In this study, the indentation of a film that contained a grain of about 100-150 nm size surrounded by relatively larger grains (250nm-350nm) was performed. Typical experimental results are shown in figure in which indentation was performed on two grains with different sizes. Fig. 1(a)-(c) and Fig. 1(f) were all taken prior to the indentation. Fig 1(a) is the bright-field image showing the two grains before indentation. Fig. 1(b) and 1(c) are the dark-field images of each grain before the indentation. Both images are in a g=[200] diffraction condition. The angle between these two g vectors is 40.9° as showed in Fig. 3(f), indicating a high-angle boundary between the two grains. During the *in situ* experiment, the indenter touched both grains, causing elastic deformation followed by dislocation nucleation and extensive multiplication. After a period of deformation, the grain boundary between the two grains swept quickly across the smaller grain as indentation proceeded. As a result, only one big grain appears in the bright-field image (Fig. 3(d)) and the dark-field image (Fig. 3(e)) taken after the indentation, which confirms that the smaller grain has been eliminated by the growth of the larger grain during indentation.

The above observations reveal stress-induced grain growth during nanoindentation at room temperature. In submicron-grained Al films, it is expected that small grains with grain size less than 150nm would gradually disappear by ordinary coarsening, at a rate that depends exponentially on temperature. When the stress is accelerated by the indentation, however, the originally almost static grain boundaries become so mobile that grain growth via grain migration happens over a very short time. In nanocrystalline Al films, although it is impossible to trace each individual grain as we can do in submicron grains, the sudden contrast changes in dark field modes suggest grain coalescence and/or grain growth mechanisms that are consistent with the results of submicron-grained Al films. For such extremely small grains, grain rotation is also involved in the deformation and the grain coalescence and/or grain growth happen quite frequently even at the very beginning

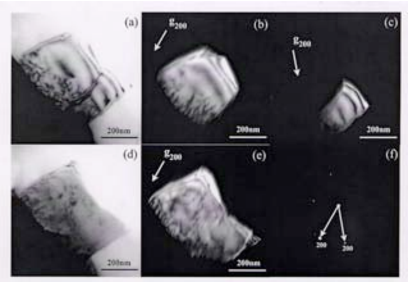


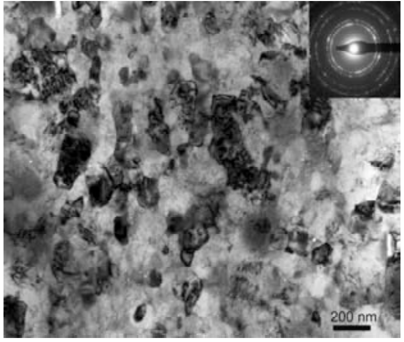
Figure 1

Grain growth due to GB migration and due to grain rotation-coalescence is not expected in low-temperature deformation of coarse-grained materials. It seems to be the reduction of grain size that triggers the GB-related deformation mechanisms. The excess free energy associated with the significant amount of grain boundary area makes the grain growth thermodynamically favored. The application of stress might either liberate the grain boundaries to make it easy for them to move or rotate, or increase the driving forces for GB migration and grain rotation.

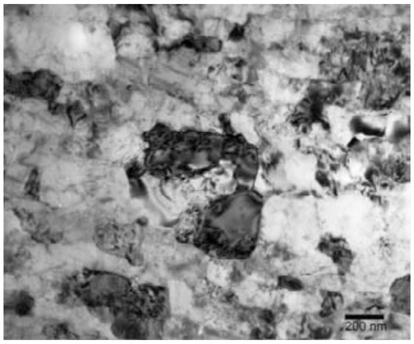
It is not yet clear what specific role grain coarsening plays in the load-displacement response. However, it does seem clear that boundary processes are critical in the deformation of nanocrystalline materials. Since, given Le Chatelier's Principle, all spontaneous processes that happen under load contribute to the relaxation of the load, it is very likely that coalescence is a significant role. We are conducting further *in situ* experiments in both nanocrystalline and ultrafine-grained materials to clarify this issue.

3.2. <u>The Hall-Petch Relation</u>. A primary aspect of the mechanical behavior of nc-material that represents one of the issues being addressed by the present NIRT program is their potentially high strength. As the grain size (d) is refined, the strength increases as $d^{-1/2}$ (the Hall-Petch relation) and can, potentially, reach very high values. However, it is not certainly known whether the strength in the nano-scale range continues to increase or it decreases when the grain size falls below about 20 nm. In order to analytically address this issue, a simple statistical model of dislocation dynamics was incorporated into a simulation framework that allows the investigation of the constant strain rate response of a material. This framework provides a crude model of dislocation pileup formation and relaxation under the conditions of constant strain rate. The simple statistical model of dislocation dynamics was initially applied to micrograined Ni_3Al because this material, like other intermetallic compounds display the so-called yield strength anomaly (the strength increases with increasing temperature). The simulations make clear the origins of the anomalous Hall-Petch exponent. The dislocation dynamics in these compounds lead to exhaustion of dislocation motion (due to core structure transformations). In the pinned state, it can take substantial stresses before a dislocation depins. Consequently, dislocation pileups cannot form completely and the stress multiplication due to pileups no longer scales with the grain size. One expects, therefore, that the yield strength will be independent of grain size. However, there is another source of grain size dependence. The dislocation dynamics are governed by weak-link statistics. Longer dislocations are more likely to have a weak-link than their shorter counterparts. Consequently, materials with a smaller grain size are intrinsically stronger because the dislocations within these materials are intrinsically more difficult to move, and the materials are stronger. For Ni_3Al, this model will need modification for grain sizes below approximately 500 nm, and we are presently working in that direction.

3.3. *Enhancement of Ductility*. A problem with materials that are strengthened by ultra-refinement is the loss of ductility. The source of this problem is not entirely clear, but it is probably due to loss of work hardening as a result of the inability of ultrafine grains to sustain arrays of dislocations. A very recent study in our laboratories, has led to the observation of an enhanced tensile ductility in a nanostructured Al-7.5%Mg alloy with a mean grain size of 90 *nm* processed via consolidation of cryomilled Al- Mg powders. An annealing treatment at a temperature of 773 K for 2.5 hours modified the extruded microstructure slightly without causing significant grain growth, as revealed by TEM and XRD patterns. The annealing treatment significantly improved the ductility with a remarkably small loss in ductility. The reported phenomenon of enhanced tensile ductility was attributed to a mechanism involving dislocation activity in submicron grains during plastic deformation; mechanistic studies are continuing.



(a) After extrusion



(b) After annealing treatment

3.4. *The minimum grain size obtained by ball milling*. The objective of this activity is to account for the origin of the correlations that were reported between the minimum grain size obtained by ball milling and materials parameters such the melting point, the bulk modulus, and hardness. Consideration of available information shows that the two processes of hardening and recovery, which are suggested to control the final grain size obtainable by milling, also occur during creep. An analysis that was based on the treatment of the kinetics of milling process as those associated with a creep process led to the development of a dislocation model. The predictions of the models were found to be in good agreement with experimental results and trends as demonstrated.

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