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A methodology to investigate size scale effects in crystalline plasticity using uniaxial compression testing

Michael D. Uchic *, Dennis M. Dimiduk

Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817, USA

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Abstract

A methodology for performing uniaxial compression tests on samples having micron-size dimensions is presented. Sample fabrication is accomplished using focused ion beam milling to create cylindrical samples of uniform cross-section that remain attached to the bulk substrate at one end. Once fabricated, samples are tested in uniaxial compression using a nanoindentation device outfitted with a flat tip, and a stress–strain curve is obtained. The methodology can be used to examine the plastic response of samples of different sizes that are from the same bulk material. In this manner, dimensional size effects at the micron scale can be explored for single crystals, using a readily interpretable test that minimizes imposed stretch and bending gradients. The methodology was applied to a single-crystal Ni superalloy and a transition from bulk-like to size-affected behavior was observed for samples 5 μm in diameter and smaller.

Keywords: Size scale effects; Micro-compression testing; Focused ion beam milling; Nickel superalloys

1. Introduction

Materials scientists have known for many decades that size scales in crystal plasticity – length scales associated with dislocation nucleation, motion, and substructure evolution – greatly influence flow stresses and strain-hardening rates during plastic deformation. Size scale effects due to internal microstructural features are commonly observed, and are often utilized to improve bulk properties (for example, Hall-Petch hardening) [1,2]. In stark contrast, size scale effects due to the physical geometry of a sample have been generally overlooked, especially for sample dimensions smaller than 100 μm. In particular, one would expect that as the sample geometry begins to shrink to a few microns, the fundamental response and organization of dislocations should be affected by this change. For example, it is well known that the characteristic size of the dislocation cell substructure formed during plastic deformation of fcc crystals in stage II flow is a few microns [3]. One might ask what happens to the flow behavior of fcc crystals when the sample size systematically approaches this length scale? Similar questions could be asked for all classes of materials that deform by dislocation motion, yet the scientific community has only recently begun to systematically examine the effect of sample size on mechanical properties in the micron to sub-millimeter range.

Within the past 15 years there has been renewed attention paid to the dimensional size effects at the micron-size scale that are related to plastic deformation in the presence of strong strain gradients, such as those imposed during nanoindentation testing [4,5], tension–torsion testing of wires [6], and bending of thin foils [7]. Dimensional effects are generally observed for these types of tests, which are interpreted to be a direct result of a higher dislocation density due to the generation of geometrically necessary dislocations (GND). By comparison, testing for size effects at the micron-size scale using test methods that minimize the effect of strain gradients has primarily focused on the testing of materials that can be grown to have dimension(s) that are on the order of microns, such as whiskers and thin-films. Many test methodologies were successfully developed to measure the flow properties of these small structures, such as wafer curvature [8], bulge

* Corresponding author.
E-mail address: Michael.Uchic@wpafb.af.mil (M.D. Uchic).

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testing [9], nanoindentation [10], micro-beam bending [11], and uniaxial tension testing [12–16]. It is obvious that there is one significant barrier that prevents the application of these methodologies to an arbitrary material of interest, which is that each has evolved around a specialized product form that is the result of carefully controlled fabrication processes. As an example, many of the existing tension-testing methods that operate at the micron-size scale have evolved around wafer processing of films on Si substrates, which produce specimens in the form of a freestanding ligament that is partially supported on a thick Si substrate. This type of fabrication route limits the broad applicability of these methods, as synthesizing a given material in a thin-film or whisker form such that it contains the same composition and internal microstructural features as a bulk-processed material, is difficult or often impossible. Based on the proliferation of testing devices that operate on micron-size volumes, it is clear that the difficulties associated with sample fabrication at the micron scale has primarily limited the exploration of dimensional size effects.

This research was motivated by an engineering challenge which may be simply stated as the following question: is it possible to measure the single-crystal mechanical properties from a fully processed, polycrystalline engineering alloy when the grain size of these materials is often smaller than 50 μm [17]? Here, it is envisaged that one could obtain single-crystal properties by fabricating and extracting a single-crystal specimen from the interior of a grain. The methodology described in this paper addresses one possible solution for obtaining this type of mechanical property data using commercially available instrumentation.

The recent development of new nanoscale fabrication, manipulation, and actuation technologies allows for the creation of new testing methodologies. As the scale of mechanical and microelectronic devices grows smaller, there has been a continuing push to develop mechanical test devices to probe the properties of materials having decreasing dimensional scales. Drawing from this technology base, we developed a methodology to fabricate miniature compression samples that have micron-size dimensions from almost any inorganic material. Once fabricated, the samples are tested under a simple loading condition (uniaxial compression). Perhaps of equal scientific interest is that this methodology can also be used to explore dimensional size effects in a simple and straightforward manner. For these tests, the total volume of the micro-compression samples typically ranges from less than 1 μm^3 to over 100,000 μm^3. Much of this size range is directly accessible for modern dislocation-based plasticity simulations. Therefore, in the future it may be possible to use the direct output from the micro-compression tests (or other types of similar-size experiments) to help validate this class of simulations.

This paper is structured as follows. The primary focus is to provide a detailed account of the methods that have been used to prepare and test micron-sized compression testing specimens. The paper gives particular emphasis to the use of focused ion beam-based (FIB) fabrication methods, in part because these methods have not been traditionally used for preparation of mechanical test structures, save for a limited number of studies [18,19]. It is the authors' opinion that FIB instrumentation will play a critical role in future test methodologies that extend beyond the simple test method discussed in this work. Lastly, the methodology is demonstrated through tests of a single-crystal Ni-base superalloy, in order to explore dimensional size effects in a precipitation-strengthened material.

2. Experimental procedure

2.1. Micro-sample fabrication using Focused Ion Beam milling

The micro-sample fabrication methodology is based on FIB milling – highly localized ion sputtering using energetic ions – to micro-machine small compression samples into the surface of a bulk crystal. Focused Ion Beam systems are uniquely suited in their ability to fabricate 3D structures while maintaining sub-micron precision in a variety of metallic and non-metallic systems [20]. A FEI Company Strata Dual Beam 235 was used for this study, and the FIB column on this instrument supplies 30 kV Ga^+ ions at beam currents ranging from 0.001 to 20 nA.

The micro-sample fabrication process begins with the preparation of the bulk material of interest. A small section of a bulk material is prepared using standard metallographic methods, so that the section can be placed on a standard scanning electron microscope (SEM) stub. Typically the bulk section is mechanically polished using an automated lapping machine (Allied High-Tech Multi-Prep System) to produce a parallel-sided sample, which is subsequently electropolished to remove any residual damage from the mechanical polishing. The sample is then mounted to a SEM stub using silver-based conductive paste or epoxy.

The micro-compression sample preparation process consists of two steps. First, the surface of the bulk section is oriented normal to the FIB column, and an area of interest is located using FIB imaging. In that area a series of concentric annular milling patterns are used to mill a cavity or crater that creates each sample blank in relief, as shown in Fig. 1. The large cavity serves the following purposes: (i) allows one to image the sidewalls of the micro-sample both before and after deformation using the SEM or FIB (which is necessary for subsequent fabrication steps), (ii) ensures that the flat indenter tip does not contact any other surfaces than the micro-sample, (iii) lessens the probability that sputtered material will redeposit back onto the freshly milled micro-sample during subsequent fabrication steps, and (iv) facilitates locating the micro-sample using the optical microscope of the nanoindenter test system. Note that this step is usually performed using high-beam currents (20–5 nA), since the goal is to quickly remove material from around the sam-