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Grain growth in nanocrystalline nickel films at low temperature and stress

B. Wang, M.T. Alam and M.A. Haque*

Department of Mechanical and Nuclear Engineering, The Pennsylvania State University, University Park, PA 16802, USA

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The influence of temperature and stress on grain growth was investigated in nanocrystalline nickel thin films in situ inside a transmission electron microscope. Independently, temperature and stress did not show any appreciable effect. However, concurrent loading at only 20% of melting temperature and 20% of yield stress resulted in significant grain boundary mobility and grain growth. We propose that grain growth in nanocrystals is a stress-heterogeneity-relieving mechanism that may not necessarily be associated with plasticity as proposed in the literature.
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Grain size is undoubtedly the most influential microstructural parameter governing the properties of polycrystalline materials. Mechanical properties have been studied for grain sizes from a few nanometers to hundreds of microns [1,2]. By directly controlling defect density and grain boundary volume fraction, grain size is known to govern both elastic [3,4] and plastic [5–7] behavior. It is generally accepted that as grain size becomes smaller, materials exhibit higher yield strength and lower ductility, primarily because of the confining of dislocations [8]. The trend reverses below a critical size due to large fraction of grain boundaries at the nanoscale [9]. Grain size effects go beyond mechanical behavior, since defects and grain boundaries are also responsible for scattering electrons and phonons. At the nanoscale, grain size can significantly impact electrical and thermal conductivity [10], primarily because grain boundary scattering is stronger than that by dislocations and vacancies [11]. The literature contains similar evidence for optical, magnetic, etc., properties [12]. The wide-ranging impact of grain size has encouraged vigorous multidisciplinary research.

In this study, we focus on grain size growth in thin metal films, which are used in a wide variety of devices, including among many others microelectronics, sensors and energy conversion [13]. Classical ways of increasing grain size are thermal annealing, precipitate/impurities and irradiation [14]. For nanocrystalline materials, thermal annealing works primarily through surface diffusion, a reason why grain growth stagnates at temperatures far below the melting point ($0.2–0.3T_m$) [15,16]. Here, the excess free volume associated with the grain boundaries is transformed into vacancies as the grains grow. The atomic rearrangement in the grain boundaries decreases the grain boundary mobility to effectively halt the grain growth. Increasing temperature decreases the activation energy barrier only temporarily since any atomic rearrangement lowers the grain boundary energy and slows down its mobility [16,17]. A more recent study suggests that in addition to grain boundary energy reduction, a complex interaction between the anisotropy of grain boundary energy, grain boundary grooving and solute/triple junction drag may contribute to stagnation in grain growth [18]. Stagnation is therefore a key impediment to grain growth in nanocrystalline films.

Externally applied stress can also act as a stimulus for grain growth in nanocrystalline materials that are both pure and [19,20] and alloyed [21,22]. Typically, growth is viewed as a grain-boundary-mediated plastic deformation mode [9,23,24]. The phenomenon has been consistently observed at or near room temperature [19,21,25–27], which is very intriguing because grain boundary mobility requires higher temperatures. Moreover diffusional flow does not necessarily imply grain

* Corresponding author. Tel.: +1 (814) 865 4248; fax: +1 (814) 865 9693; e-mail: mah37@psu.edu

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growth. It is suggested that room temperature grain growth is associated with shear-stress-driven grain boundary migration [25]. The concept is borrowed from recent studies showing that shear stress can initiate normal grain boundary migration [28] at high-angle boundaries and is energetically more favorable than the pure migration of a grain boundary [29]. When such coupled migration occurs over numerous grains, the global effect is an increased average grain size.

In this paper, we present experimental evidence of grain growth due to the simultaneous action of low stress and low temperature. This is in stark contrast with the grain growth literature, which involves either high stresses and low temperatures or low stress and high temperatures [19,30]. Since both temperature and stress play a role in grain growth, then intuitively, external stress applied in nanocrystalline materials could catalyze the temperature-driven grain growth and vice versa. However, there has been no concerted effort in the literature to investigate such synergy, if any. This has motivated us to develop an experimental setup in which independently controlled uniform stress and temperature can be applied to a freestanding nanocrystalline thin film in situ inside a transmission electron microscope (TEM). The advantages of merging the high resolution of the TEM with quantitative in situ characterization have been demonstrated previously in studies of the dynamics of grain boundary motion [21,22].

Figure 1a shows a custom designed and fabricated microelectromechanical system (MEMS) device with an electrothermal actuator and heater to apply uniaxial tensile stress and heat, respectively, to the specimen. The 100 nm thick, 99.99% pure Ni films used in this study were first evaporated on a silicon-on-insulator wafer. Standard photolithography and deep reactive ion etching was performed on the wafer so that the actuator and heater structures are co-fabricated with the specimen. Such co-fabrication ensures perfect specimen alignment and gripping [3]. The electrothermal actuator and heater were calibrated for various values of input current using an infrared microscope with 0.1 K resolution. After calibration, the 3 mm × 5 mm MEMS devices are mounted on an electrical biasing TEM specimen holder so that the experiments could be performed in situ in the TEM.

In situ TEM tensile tests on the 100 nm thick freestanding Ni thin films gave Young's modulus and yield stress values of about 120 ± 5 GPa and 1.8 ± 0.1 GPa, respectively, which compares very well with the literature [31]. The as-deposited grain size was approximately 10 nm with a few grains as large as 50 nm. Figure 1b and c shows bright-field TEM and selected-area diffraction images, respectively. Figure 1d shows secondary grain growth to a bimodal distribution (first mode 25 nm and second mode 100 nm) at room temperature without any applied stress for 30–60 days. Grain growth is also evident from the distinct changes in the electron diffraction pattern (Fig. 1e). Such growth has previously been reported on nanocrystalline Pd and Cu, but not on Ni [16,20]. The grain growth stagnated at this point. In a set of in situ TEM heating (no applied stress) experiments, the specimen temperature was raised to 373 K (about $0.2T_m$) for several hours. This is shown in Figure 2a and b. No appreciable grain growth or grain boundary mobility was observed, which is expected because Ni is thermally stable below 473 K. In a separate set of experiments, room temperature mechanical loading was performed to investigate any stress-assisted grain growth. The results around the ultimate stress range (1.7 GPa) are shown in Figure 2c and d, where discernible grain growth is observed in a few hours.

The third set of experiments involved applying stresses to heated (373 K or $0.2T_m$ for Ni) specimens. The experimental results are given in Figure 3, where clearly discernible grain growth started at about 325 MPa stress, which is only about 20% of the yield stress (σ_Y). The growth rate at this low-temperature/low-stress loading configuration was rapid enough to be visualized in the quasi-statically (of the order of minutes) captured images, which suggests that further growth might have occurred had longer times been allowed. Interestingly, most of the growth was in normal (or uniform) mode, where the smaller grains grew appreciably with the larger ones. Figure 3b shows further grain growth at $0.5\sigma_Y$, resulting in equiaxed grain structure with many grains larger than the thickness. This uniform growth pattern is opposite to the non-uniform growth seen with individual temperature and stress loading and as predicted in the literature [32]. Interestingly, the relative magnitude of grain growth decreased with increased stress in the plastic deformation zone, where the in situ TEM observations clearly show other modes (primarily grain rotation and dislocation) of plastic

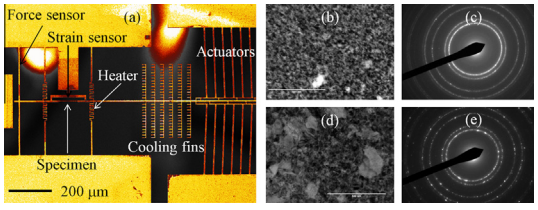

Figure 1. (a) Scanning electron micrograph showing various components of a MEMS device for quantitative in situ TEM testing of thin films (false colored); (b and c) bright-field and selected-area electron diffraction pattern of the as-deposited nanocrystalline Ni specimen; (d and e) room temperature grain growth over a span of a few days. Scale bar represents 500 nm.

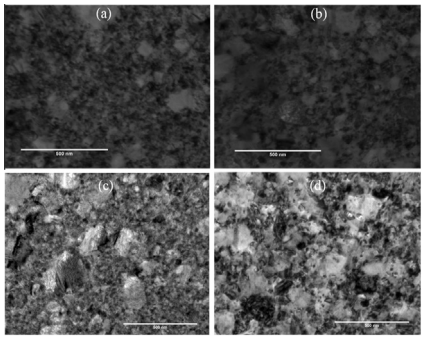

Figure 2. TEM images of the specimens at (a) room temperature, (b) $0.2T_m$ with no applied stress, (c) no applied stress and (b) 1.7 GPa stress at room temperature. Scale bar is 500 nm.

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