Scripta Materialia 134 (2017) 95–99

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

In situ observation of stress induced grain boundary migration in nanocrystalline gold

Lihua Wang [a,d,*], Tianjiao Xin [a], Deli Kong [a], Xinyu Shu [a], Yanhui Chen [a], Hao Zhou [a], Jiao Teng [b], Ze Zhang [a,c], Jin Zou [d,e,*], Xiaodong Han [a,**]

[a] *Institute of Microstructure and Property of Advanced Materials, Beijing University of Technology, Beijing 100124, China*
[b] *Department of Material Physics and Chemistry, University of Science and Technology Beijing, Beijing 100083, China*
[c] *State Key Laboratory of Silicon Materials, Zhejiang University, Hangzhou 310008, China*
[d] *Materials Engineering, The University of Queensland, Brisbane, QLD 4072, Australia*
[e] *Centre for Microscopy and Microanalysis, The University of Queensland, Brisbane, QLD 4072, Australia*

ARTICLE INFO

Article history:
Received 12 January 2017
Received in revised form 1 March 2017
Accepted 3 March 2017
Available online xxxx

Keywords:
Plastic deformation
Grain boundary migration
Nanocrystalline metal
In situ
Transmission electron microscopy (TEM)

ABSTRACT

In this study, the plastic behaviors of nanocrystalline Au with an average grain size of 18 nm were investigated *in situ* using a home-made tensile device in a transmission electron microscope. We provide the direct experimental results revealed the process of grain boundary migration. The results show that dislocation behaviors are prevalent for large grains. However, for grain sizes below ~15 nm, grain boundary migration occurs frequently. The results of our statistical analyses show that grain boundary migration occurs more frequently than grain boundary sliding and rotation in nanocrystalline Au.

The deformation mechanisms of nanocrystalline (NC) materials have attracted intense interest because these materials exhibit greater strength than their coarse-grained counterparts [1,2]. However, the deformation mechanisms of NCs with grain sizes below ~15 nm are still under debate, though this topic has been extensively studied both theoretically [3–15] and experimentally [16–29]. Previous molecular dynamic (MD) simulations predict a transition in the deformation mode that occurs at a critical grain size of ~15 nm; at this point, the plastic deformation that is controlled by dislocation activities is transferred into grain boundary (GB)-mediated plasticity [3–6]. In other words, for grain sizes of less than ~15 nm, the plastic deformation in NC metals is controlled by GB-mediated plasticity mechanisms, such as GB sliding, grain rotation and GB migration [4–6]. Some MD simulation studies suggest that GB sliding and GB migration are the dominant deformation mechanisms [7–10] and that grain rotation is an accommodation mechanism during the deformation [15,28,29]. In addition, many theories and MD simulations have predicted that small grains are more likely to undergo GB migration than large grains because small grains exhibit relatively large GB curvatures [30–33]. However, very few direct experimental results confirm that this is the case.

Experimentally, many previous studies have observed obvious grain growth by postmortem examination. Based on these studies, grain growth is believed from GB migration [25,34,35]. Interestingly, Shan et al., discovered that GB sliding and grain rotation in ~10 nm NC Ni [18,19], while Kumar et al., and Hugo et al., reveal that dislocation-mediated plasticity plays a dominant role even in ~10 nm NC Ni [20,21]. Previous *in-situ* transmission electron microscopy (TEM) studies also observed the GB migration in Al [22–25] at grain sizes >50 nm. However, for NC Au, though grain rotations were observed both at low temperature [26] and during annealing [27], very few studies provide the quantity analyses on GB mediated plasticity; and at which grain size, there is a transition occurs from dislocation behavior to GB-mediated plasticity.

In this study, using a home-made device of our own design [36–38], the plastic behavior of NC Au with an average grain size of 18 nm was investigated *in situ* at room temperature using a TEM. We found an obvious transition in the deformation mode from dislocation behavior for

---

* Corresponding authors at: Materials Engineering, The University of Queensland, Brisbane, QLD 4072, Australia.
** Corresponding author.
E-mail addresses: wlh@bjut.edu.cn (L. Wang), j.zou@uq.edu.au (J. Zou), xdhan@bjut.edu.cn (X. Han).

http://dx.doi.org/10.1016/j.scriptamat.2017.03.003

large grains to GB migration-controlled plasticity for small grains. The results of our statistical analysis show that GB migration is more favored than other GB deformation mechanisms in NC Au.

NC Au thin films with a thickness of ~10 nm were deposited on single-crystal NaCl substrates by magnetron sputtering deposition. Our TEM examination shows that the grain size varies from 5 to 45 nm and that most grains have a diameter in the range between 10 and 20 nm, with an average diameter of 18 nm (see more details in Supplementary Fig. S1). The *in situ* tensile experiment was conducted using a specially designed double-tilt bi-metallic extensor [36–38], the real-time microstructural evolution of the TEM specimen was observed under a TEM operating at 300 kV and ~40 °C. The tensile strain rate was measured at ~10^{-3} s^{-1} by controlling the temperature increase rate (the method for measuring tensile strain is shown in Supplementary Fig. S2).

Fig. 1 shows a series of HRTEM images taken at different strain durations and shows GB migration between two neighboring grains with similar grain sizes of ~15 nm during tensile deformation. Fig. 1a is the HRTEM image captured before the GB migration process began. To show the GB motion, 4 different GBs are highlighted by white dashed lines and are marked as "G1", "G2", "G3" and "G4". As shown in Fig. 1a, the grain sizes of G1, G2 and G3 are ~15 nm, ~15 nm, and ~5 nm, respectively. For convenience, we define GB_{i-j} as the GB between grains G_i and G_j, the shape and position of GB_{i-j} change as the GB migration, and the misorientation angle change between two grains as grain rotation. With increasing strain, as shown in Fig. 1b, GB_{1-2} moved toward the interior of G2; meanwhile, GB_{1-4} moved toward the interior of G4. This process lead to an increase in the grain size of G1 and a decrease in the grain size of G2. As the deformation continues (Fig. 1c) no obvious motion of GB_{1-2} is observed; however, GB_{1-4} clearly moved toward the interior of G4, leading to an increased grain size of G1 from ~15 to ~18 nm. With extensive loading, both GB_{1-2} and GB_{1-4} underwent an obvious migration process, leading to changes in the shapes of G1 and G2; the grain size of G1 increases from ~15 to ~20 nm, while the grain size of G2 decreases from ~15 to ~10 nm (Fig. 1d). More interestingly, the small G3 grain underwent no obvious migration during the deformation, possibly because the local stress is not high enough to cause GB migration. This GB migration between two large grains is inconsistent with previous theoretical predictions and MD simulation results, which predicted that small grains are more likely to undergo GB migration [30–33]. This indicates that the driving force of GB migration is determined not only by the curvature of the GB but also by local stresses.

In addition to the GB migration between large grains, we also observed GB migration between large and small grains. Fig. 2 presents a series of HRTEM images of a region containing both large and small grains, showing that the small grains are absorbed by the surrounding large grains due to GB migration. Fig. 2a is a HRTEM image that was recorded before the GB migration process. Six grains are identified ("G1" to "G6"), and the corresponding GBs are highlighted using white dashed lines to show the GB migration process. As shown in Fig. 2a, the grain sizes of G2, G3, G4 and G5 are ~12 nm, ~6 nm, ~8 nm and 4 nm, respectively. As the strain increases (Fig. 2a–d) the GBs underwent obvious migration, which causes the grain sizes of G2, G3, G4 and G5 to decrease to ~8 nm, ~4 nm, ~2 nm and ~4 nm, respectively (Fig. 2d). As the deformation continued, the GB migration led to a reduction in the sizes of G4 and G5, while G2 and G3 decrease to 2 nm and 3 nm, respectively, as shown in Fig. 2e. With extensive loading (Fig. 2f), G2 and G3 diminished further due to the continuing GB migration. In this case, the small grains were absorbed by the surrounding large grains, consistent with previous theoretical predictions and MD simulations [30–33].

In addition to pure GB migration, GB migration accompanied by grain rotation was also observed. Fig. 3 presents a series of HRTEM images showing that the GBs not only undergo GB migration but also undergo grain rotation. Fig. 3a shows a TEM image that was captured before the deformation, and Fig. 3b shows an enlarged view of Fig. 3a at atomic resolution, in which five grains ("G1" to "G5") are highlighted. Fig. 3a shows that the grain size of G1 is ~14 nm and that the GB angle of GB_{1-3} is 18.52°. As the strain increases (Fig. 3b–d), GB_{1-3} and GB_{3-4} moved toward the interior of G3; at the same time, the angle of GB_{1-3} decreased from 18.52° to 10.77°. Under further deformation (Fig. 3d–f) GB_{1-2} and GB_{3-2} underwent migration, and the GB angle of GB_{1-3}

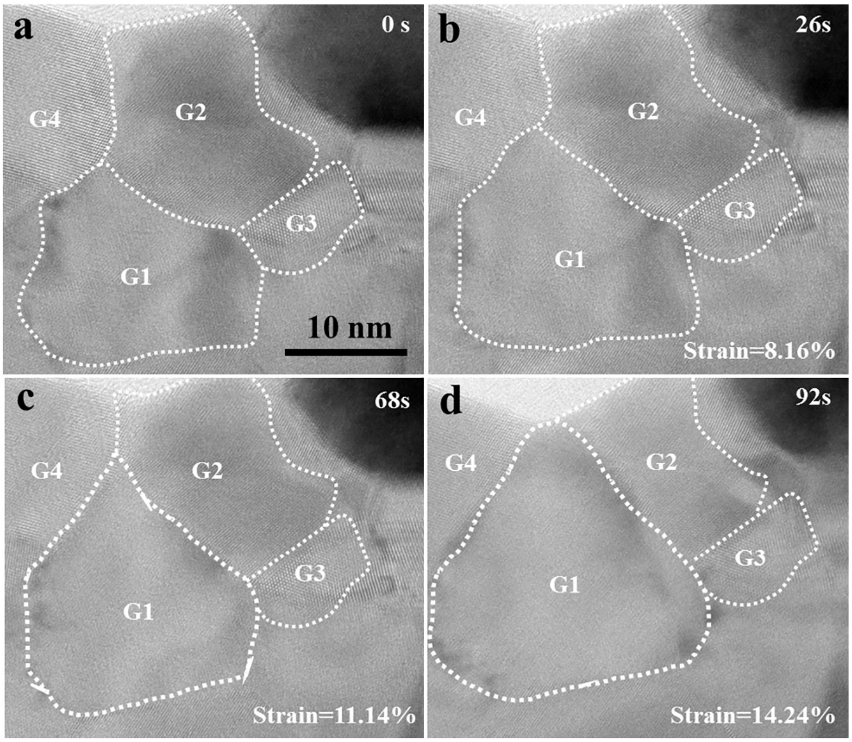

Fig. 1. (a–d) A series of HRTEM images taken at different times, showing that GB migration induces grain size reduction. The size of G1 increases as the size of G2 decreases (see also Supplementary Movie S1).