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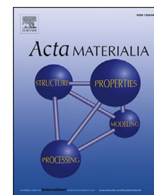

Full length article

Time, stress, and temperature-dependent deformation in nanostructured copper: Stress relaxation tests and simulations

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In the present work, stress relaxation tests, high-resolution transmission electron microscopy (HRTEM), and molecular dynamics (MD) simulations were conducted on coarse-grained (cg), nanograined (ng), and nanotwinned (nt) copper at temperatures of 22 °C (RT), 30 °C, 40 °C, 50 °C, and 75 °C. The comprehensive investigations provide sufficient information for the building-up of a formula to describe the time, stress, and temperature-dependent deformation and clarify the relationship among the strain rate sensitivity parameter, stress exponent, and activation volume. The typically experimental curves of logarithmic plastic strain rate versus stress exhibited a three staged relaxation process from a linear high stress relaxation region to a subsequent nonlinear stress relaxation region and finally to a linear low stress relaxation region, which only showed-up at the test temperatures higher than 22 °C, 22 °C, and 30 °C, respectively, in the tested cg-, ng-, and nt-Cu specimens. The values of stress exponent, stress-independent activation energy, and activation volume were determined from the experimental data in the two linear regions. The determined activation parameters, HRTEM images, and MD simulations consistently suggest that dislocation-mediated plastic deformation is predominant in all tested cg-, ng-, and nt-Cu specimens in the initial linear high stress relaxation region at the five relaxation temperatures, whereas in the linear low stress relaxation region, the grain boundary (GB) diffusion-associated deformation is dominant in the ng- and cg-Cu specimens, while twin boundary (TB) migration, i.e., twinning and detwinning with parallel partial dislocations, governs the time, stress, and temperature-dependent deformation in the nt-Cu specimens.

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1. Introduction

Creep and stress relaxation behaviors of metallic materials are extremely important to their applications in industries. The behaviors depend highly on time, stress, and temperature and are the responses of microstructures of metallic materials to external loading conditions of time, stress, and temperature. Plastic deformation in polycrystalline metals can be mediated by dislocation activities, grain boundary (GB) and twin boundary (TB) movement, and GB diffusion. GBs are strong obstacles against dislocation movement, and therefore refining grain size is an important approach to strengthen metallic materials as indicated by the famous Hall–Petch relation [1,2]. Too many strong dislocation-motion obstacles of GBs might lead to the brittleness in nanograined (ng) metals. The TB resistance against dislocation motion is much more moderated, especially in face-centered cubic (fcc) metals, thereby simultaneously strengthening and toughening nanotwinned (nt) metals [3–8]. When the grain size is small, however, GB diffusion or Coble creep [9–11], GB sliding [12–14], grain rotation [15], etc., might become the predominant modes of the time-dependent deformation [16–18]. In general, the GB-diffusional deformation and the dislocation-mediated deformation are respectively dominant at low and high strain rates/stresses

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[14]. Since coherent TBs are not fast diffusion channels [19–21], the nominal dislocation-mediated plasticity might switch to the twining and detwining mechanism in nt-Cu, leading to the change from normal Hall–Petch relation to revise Hall–Petch relation [22].

Deformation activation energy and activation volume might reflect the mechanism of thermally activated plastic deformation [23–26]. The activation volume represents the generalized change in volume caused by flow stress and the generalized area swept over by mobile dislocations in dislocation mediated plasticity. Deformation activation energy and activation volume can be determined by stress relaxation tests at various temperatures. For example, the data of stress relaxation tests on Al–Zn–Mg–Cu alloy at temperatures of 120 °C, 160 °C, and 200 °C [27] were illustrated in curves of logarithmic strain rate versus logarithmic stress, showing (i) initial high stress stage, (ii) middle stress transition stage, and (iii) last low stress stage. The obtained activation energy and the stress exponent suggested that dislocation creep was the dominant deformation mechanism in the initial high-stress and subsequent medium-stress stages; whereas diffusional creep was the mechanism in the last low-stress stage. Although repeated stress relaxation tests and strain rate jump tests were conducted over temperatures (77–373 K) on the electrodeposited (ED) ng-Ni with an average grain size of about 30 nm [28], the apparent activation volume and the physical activation volume were determined only at a given temperature [29], thereby giving temperature-dependent values. The difference in the time, stress, and temperature-dependent deformation behavior between the ng-Ni and the coarse-grained (cg) Ni counterpart might indicate that the thermally activated process in ng-Ni was controlled by the GB-mediated dislocation process. The seven-hours-long stress relaxation tests on ng-Ni with an average grain size of 27 nm at room temperature (RT) [16,17] determined the activation volume, which values were over 23 b^3, about 15 b^3, and around 1.7 b^3 in the near linear rapidly stress delayed, lumber nonlinear stress delayed, and near linear slowly stress delayed regions, respectively, where b denotes the magnitude of Burgers vector. The change in the activation volume implied the deformation transition from the initial dislocation-dominated plasticity to the mixture of dislocation motion and diffusion-based GB activity and finally to the entire diffusion-based GB activity. The stress relaxation tests [30] on ED ng-Ni with grain size of 21 nm and high pressure torsion (HPT) produced ng-Ni with grain size of 105 nm at RT showed a decreased activation volume with increasing stress in the ED ng-Ni and an opposite trend in the HPT ng-Ni. The indentation stress relaxation tests [31] on the rolled and annealed ng-Ni with an average grain size of 20 nm at RT showed that the activation volume in the rolled ng-Ni was decreased first and then increased as the rolling strain increasing, and the activation volume in the annealed Ni was continuously increased with the increase of annealing temperature. Stress relaxation tests on nt-Cu with twin lamellae thicknesses of 35 and 15 nm at RT [32] indicted that the apparent activation volume and the physical activation volume were about 20 b^3 and 15.5 b^3, 36 b^3 and 30 b^3, and 350 b^3 and 309 b^3 for the nt-15, nt-35, and cg specimens, respectively, which were correspondingly attributed to the TB-mediated plasticity and the intra-twin dislocation activity.

The above description indicates that the apparent activation energy depends on the stress level. To experimentally determine the apparent activation energy, one must have the experiment data of the plastic deformation rate under a certain stress level at various temperatures. Then, the activation volume can be extracted from the stress-dependent apparent activation energy. Clearly, to acquire more information about the time, stress, and temperature-dependent deformation, stress relaxation tests must be conducted at various temperatures. In the present work, stress relaxation tests were conducted on cg, ng, and nt copper at temperatures of RT (~22 °C), 30 °C, 40 °C, 50 °C, and 75 °C to investigate the time, stress, and temperature-dependent deformation. We clarify the relationship between the strain rate sensitivity parameter, stress exponent, and activation volume. Together with the evidence from the high-resolution transmission electron microscopy (HRTEM) and molecular dynamics (MD) simulations, we suggest the predominant deformation mechanisms under different stress levels in the cg, ng, and nt-Cu specimens.

This paper is organized as follows. Section 2 gives the experimental methods that include the synthesis methods of the used copper specimens with different microstructures, the procedure of stress relaxation tests, and the simulation methodology. Section 3 summarizes experimental and simulation results of stress relaxation, from which the activation parameters in the different stress levels are determined. The results suggest a theoretical formula to describe the time, stress, and temperature-dependent deformation. The relationship between the strain rate sensitivity parameter, stress exponent, and activation volume is also discussed in Section 3. Based on the determined activation parameters, and the evidences from the HRTEM and MD simulation observations, Section 4 mainly discusses the transition in the deformation mechanism from the high stress level to low stress level in the three types of specimens. Finally, concluding remarks are given in Section 5.

2. Methods

Commercial pure Cu sheet of ~3 mm thickness was annealed at 400 °C for 4 h to get a cg microstructure. High-pure ng and nt-Cu sheets with dimensions of about 12 cm × 6 cm × 500 μm were synthesized with $CuSO_4$ electrolyte [33,34]. The density of the synthesized sheets was determined by the Archimedes principle to be 8.92 ± 0.02 g/cm^3. Dog-bone shaped tensile specimens of with 33 mm overall length, 8 mm gage length and 2.5 mm gage width were cut by an electro-discharge machine from the synthesized sheets. Double surfaces of specimens were polished with SiC papers of 400–1200 grits and alumina suspensions of 0.1 μm, and the final thickness of specimens is ~300 μm. The micro/nanostructure of the cg, ng and nt-Cu before and after tests was characterized by using an optical microscope (OM, Olympus BH2-MJLT) and a transmission electron microscope (TEM, JEOL 2010F) operated at 200 kV.

Stress-relaxation tests were carried out on a Universal Testing Machine (UTM) with Furnace (MTS SINTECH 10/D) at RT, 30 °C, 40 °C, 50 °C, and 75 °C with accuracy ± 1 °C in air. After the specimens were stretched at the strain loading rate of 10^{-2} s^{-1} to a designed stress level, which was 115 MPa for the cg-Cu, 350 MPa for the ng-Cu, and 550 MPa for the nt-Cu, the cross-head was stopped there to allow stress relaxation under sustained strain for 300 s and the stress decay was recorded as a function of time. Then, the relaxed specimen was reloaded to the same stress level with the strain rate 10^{-1} s^{-1} to conduct the second cycle of relaxation test. In total, four cycles of repeated stress relaxation tests were carried out on each specimen with the same procedure.

Atomistic simulations were performed with the Large-scale Atomic/Molecular Massively Parallel Simulator (LAMMPS) code [35] and an embedded-atom method (EAM) potential for Cu atoms [36]. The twin-free ng-Cu sample contained 27 randomly orientated Voronoi grains with averaged grain size about 10 nm. The nt-Cu sample was constructed by inserting nts into the ngs and the nt thickness was 1.25 nm on average. The twin plane orientations in the nt sample were also randomly arranged on {111} planes. Each of the ng and nt-Cu, as shown in Fig. 7 (a, e), contained approximately 2,200,000 atoms, with dimensions of about 30 × 30 × 30 nm^3. Following the setting-up of the simulated sample, energy minimization was performed at a designed temperature by

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