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Cyclic deformation of nanocrystalline and ultrafine-grained nickel

S. Cheng [a,b,*], J. Xie [c], A.D. Stoica [b], X.-L. Wang [b], J.A. Horton [d], D.W. Brown [e], H. Choo [a,d], P.K. Liaw [a]

[a] Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN 37996, USA
[b] Neutron Scattering Science Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA
[c] State Key Laboratory of Nonlinear Mechanics, Institute of Mechanics, Chinese Academy of Sciences, Beijing 100190, China
[d] Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA
[e] Los Alamos Neutron Science Center, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

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Abstract

The cyclic deformation behavior of ultrafine-grained (UFG) Ni samples synthesized by the electrodeposition method was studied. Different from those made by severely plastic deformation, the UFG samples used in this study are characterized by large-angle grain boundaries. Behaviors from nanocrystalline Ni and coarse-grained Ni samples were compared with that of ultrafine-grained Ni. With in situ neutron diffraction, unusual evolutions of residual lattice strains as well as cyclic hardening and softening behavior were demonstrated during the cyclic deformation. The microstructural changes investigated by TEM are discussed with respect to the unusual lattice strain and cyclic hardening/softening.

Keywords: Ultrafine-grained; Cyclic deformation; Neutron diffraction; Cyclic hardening/softening; Lattice strain

1. Introduction

With in-depth investigations of the plastic deformation of nanocrystalline (NC) metals in recent years, many distinctive deformation behaviors were reported for these materials, and accordingly new deformation mechanisms were proposed [1–7]. For example, under certain deformation conditions, NC Ni could deform by partial dislocations and twinning [8,9]. Moreover, in situ X-ray diffraction studies demonstrated fully reversible peak broadening in NC Ni during tensile loading/unloading, suggesting that dislocation debris might not be left over upon unloading [10]. Considerable efforts were also devoted to the deformation mechanism investigations of ultrafine-grained (UFG) metals. In situ X-ray diffraction showed irreversible peak broadening after unloading in UFG Ni, indicating that the deformation resulted in a build-up of dislocation networks, as in conventional coarse-grained (CG) metals [11]. However, our recent in situ synchrotron X-ray diffraction studies suggest that, in addition to dislocation activities, deformation twinning could also play a significant role in the tensile deformation of UFG Ni [12]. Thus, as in NC Ni, the deformation mechanisms in UFG are also complex and not well understood. Furthermore, cyclic deformation of NC and UFG metals has been shown to be very different from their CG counterparts [13–20]. However, so far, only limited investigations were carried out on cyclic deformation of UFG metals, and most of them were focused on the fatigue response and damage characterization. The underlying deformation mechanism was rarely addressed.

One the other hand, to date, the UFG metals studied were mostly produced by severely plastic deformation (SPD)

* Corresponding author. Address: Department of Materials Science and Engineering, The University of Tennessee, Knoxville, TN 37996, USA. Tel.: +1 865 974 2683; fax: +1 865 974 4115.
E-mail address: scheng1@utk.edu (S. Cheng).

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method, e.g., equal-channel angular pressing (ECAP) or rolling, etc. The microstructures of those UFG metals are usually very different from those of well annealed samples. Firstly, the grain boundaries of the UFG metals by SPD are often of small-angle type and non-equilibrium, far from the stable large-angle grain boundaries usually known in the polycrystalline materials [21,22]. Secondly, the grains are usually filled with pre-existing defects (e.g., high density of dislocations) [23–25]. Thus, the deformation behaviors reported in the literature are quite different from the samples with large-angle grain boundaries. In recent years, electrodeposition has found its way of making metals and alloys with very fine grain size [26]. To compare the deformation of UFG samples with that of conventional alloys, it is a prerequisite to have large-angle grain boundaries in UFG metals. In this study, we study the cyclic deformation behavior of electrodeposited UFG Ni with large-angle grain boundaries. With in situ neutron diffraction, the lattice strain and the macroscopic strain evolutions are investigated during cyclic deformation. We also compare the samples with different grain sizes. To understand the deformation mechanism, we characterize the microstructure changes from the as-received samples to the deformed samples by detailed transmission electron microscopy (TEM) observations. Combining the TEM observations with the evolutions of both the residual lattice strains and the macroscopic strain, the deformation pathways of UFG Ni were identified, and their role will be discussed with regard to the strain evolutions. With these model materials mimicking the structure of typical polycrystalline metals, we aim to clarify the intrinsic mechanism of cyclic deformation for this emerging group of materials.

2. Experimental

Nickel samples with four different grades of grain sizes, which cover a full range from nanocrystalline to the conventional coarse-grain regime, were used for comparison. The NC and UFG Ni samples were synthesized by a pulsed electrodeposition technique at Integran Technologies Inc. (Canada). The dimensions of the as-received NC and UFG Ni sheets were approximately 75 mm × 75 mm in planar directions, and ~1 mm in thickness. Flat dog-bone shaped pin-load samples were fabricated using an electrical discharge machine. The overall length of a fatigue sample was 75 mm, with the gage length of 28 mm, gage length of 4 mm, and 0.6 mm in thickness. CG Ni foils with commercial purity were purchased from Alfa Aesar. Since the as-received CG Ni was cold rolled, recrystallization annealing was preformed at 450 °C for 3 h [27]. In situ neutron diffraction with a time-of-flight method was conducted using SMARTS hydraulic load-frame at Los Alamos Neutron Science Center. The loading axis on the samples was at 45° angle to the incident neutron beam, and the two detector banks were at ±90° to the incident beam. With this setting, the lattice strains in the loading direction and the transverse direction were determined using the ±90° detector banks, respectively. The lattice strains, defined as the relative lattice spacing change, i.e., $d^{hkl} - d_0^{hkl}/d_0^{hkl}$, are calculated from the peak position shift, where d^{hkl} and d_0^{hkl} are the d-spacings at the applied load and the reference load, respectively. Single-peak profile fitting was carried out using the general structure analysis systems (GSAS) software.

The cyclic deformation was conducted with a load-controlled mode, with the maximum stress, σ_{max} and the minimum stress, σ_{min} both maintained at constant levels. The cyclic loading was conducted at a frequency of 1 Hz. To ensure a plastic deformation, all the samples were loaded with the σ_{max} greater than the yield stress ($\sigma_{0.2}$), see Table 1 for details. The macroscopic strain evolution during cyclic loading was recorded using a strain extensometer clipped on the sample gage section. The grain misorientations and texture of the as-received materials were measured by electron backscattering diffraction (EBSD). The microstructures of the as-received samples and deformed samples were examined by TEM. The TEM specimens were prepared by twin-jet electropolisher using an etching solution of 25% nitric acid plus 75% methanol at −45 °C. The TEM observation was performed using a Hitachi-800 microscope operated at 200 kV.

3. Results and discussions

The microstructures as well as the grain size histograms of the as-received electrodeposited Ni are shown in Fig. 1a–c. The average grain size of the NC Ni is around 20 nm. For the Ni sample with a nominal grain size of 100 nm (Ni100), the grains have a rather broad grain size distribution, ranging from tens of nm to 1200 nm. For the UFG Ni sample with a nominal grain size of 1000 nm (Ni1000), the grains range from 300 nm to 2 μm, but most of them are around 700–900 nm. The grain boundaries of the UFG Ni are mostly clear and sharp, indicative of large-angle type.

Fig. 2 shows the grain-misorientation distributions of the as-received UFG Ni samples (both Ni100 and Ni1000) measured by EBSD. One can see that the grains are mostly orientated with large-angle boundaries in both Ni samples, as it is normally considered as a large-angle boundary when the misorientation is greater than 15° [28]. Particularly, a predominant majority of misorientations appears at the angle of ~60°. Note that the misorientations exhibited in our UFG samples are significantly superior to those made by SPD methods, in which the grain orientations were mostly below 15° despite the grain size

Table 1
Parameters used in the in situ cyclic loading.

Samples	σ_{min} (MPa)	σ_{max} (MPa)	$\sigma_{0.2}$ (MPa)
$d = 20$ nm	50	850	~820
$d = 100$ nm	50	620	~510
$d = 1000$ nm	50	460/560	~450
CG Ni	50	575	~550