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ScienceDirect
Acta Materialia 91 (2015) 91–100
Acta MATERIALIA
www.elsevier.com/locate/actamat

Dynamic recovery in nanocrystalline Ni

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Received 2 February 2015; revised 27 February 2015; accepted 2 March 2015
Available online 30 March 2015

Abstract—The constant flow stress reached during uniaxial deformation of electrodeposited nanocrystalline Ni reflects a quasi-stationary balance between dislocation slip and grain boundary (GB) accommodation mechanisms. Stress reduction tests allow to suppress dislocation slip and bring recovery mechanisms into the foreground. When combined with in situ X-ray diffraction it can be shown that grain boundary recovery mechanisms play an important role in producing plastic strain while hardening the microstructure. This result has a significant consequence for the parameters of thermally activated glide of dislocations, such as athermal stress and activation volume, which are traditionally derived from stress/strain rate change tests.

Keywords: Stress reduction; X-ray diffraction; Recovery by GB accommodation; Quasi-stationary state; Constant flow stress

1. Introduction

Nanocrystalline (nc) materials, i.e. polycrystalline structures with grain sizes below 100 nm have been extensively studied during the last three decades because of their interesting properties such as high strength [1,2]. A typical stress–strain curve of a nc metal is characterized by a high yield stress, a relatively short work hardening period followed by a regime with a constant flow stress. The deformation mechanisms at play can be dislocation based i.e. dislocation emission and absorption at grain boundaries, or purely grain boundary based where mechanisms such as diffusion, climb of grain boundary dislocations, and stress driven grain boundary migration are at the focus of attention. Details on these deformation mechanisms have been gathered by extensive research combining deformation experiments, electron microscopy analysis and computational modeling (such as atomistic simulations and dislocation dynamics) [1,2]. Due to the extremely small mean free dislocation paths limited by the boundaries of nano-sized grains, the rate of defect generation inside grains and at boundaries must be very high [3]. The high resulting defect density (point defects, dislocations, grain boundaries and their reaction products) provides a high driving force for dynamic recovery. Therefore, it can be expected that a quasi-stationary state where some (but not necessary all) structure parameters have saturated, will be attained in a relatively small strain interval. Such a quasi-stationary state is reflected in a constant flow stress. In order to model the quasi-stationary state of a material, the kinetics of dislocation generation and recovery must be known. There is little information available in this regard. The aim of the present work is to gain more information on recovery processes and their relationship to the strength of electrodeposited nc Ni, a nc material known to have predominantly high-angle grain boundaries with enough impurities to stabilize the grains against fast coarsening during uniaxial deformation [4–6].

To study recovery processes, stress reduction tests have been combined with in situ X-ray diffraction. In short-term reduction tests the immediate creep rate after a sudden reduction in stress is studied as a function of the magnitude of the stress drop. This allows to study the magnitude of the internal stress [7–9]. In a long-term stress reduction test the full transient response of the strain rate after a stress reduction is recorded [10–16]. The basic principle behind this test is to suppress regular dislocation glide by fast reduction of the stress, while dynamic recovery of all kinds of defects goes on. This technique has been applied to study recovery mechanisms in single crystals [13,16] and coarse-grained

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http://dx.doi.org/10.1016/j.actamat.2015.03.033
1359-6462/

polycrystals [10–12,14] that after deformation contained subgrains with (mainly) low-angle boundaries.

In literature, stress reduction tests have been carried out on electrodeposited nc Ni in situ while performing X-ray diffraction. The samples were initially deformed to the plastic regime, followed by a relatively mild stress drop and a creep period of 20 min [17]. The signatures of the peak profile at different creep stress levels suggest the presence of two competing deformation mechanisms with opposite effects on the diffraction peak width.

In the present work stress drops of various amounts are carried out on electrodeposited nc Ni pre-deformed to the plastic regime. The full transient response of the strain rate from the initial microstructural state to a new microstructural state corresponding to the changed conditions of deformation is recorded. We demonstrate that recovery of defects plays an important role in the generation of plastic strain. To identify the deformation mechanisms that may be essential to the development of a constant deformation resistance, several tests are carried out in situ during X-ray diffraction, allowing to distinguish between mechanisms that increase and decrease the width of a diffraction peak.

2. Experimental

2.1. Sample

Bulk nc Ni was synthesized by pulsed electrodeposition (PED) at the University of Erlangen-Nürnberg. PED was done at a pulse current density of 40 mA cm^{-2}, a pulse repetition rate of 20 Hz, and a duty cycle of 50% at a bath temperature of 50 °C. The electrolyte was based on nickel sulfate (280 g l^{-1}) with additives of nickel chloride (60 g l^{-1}) and boric acid (30 g l^{-1}) as a buffer. Furthermore, sodium dodecyl sulfate (SDS) (0.2 g l^{-1}) was used as a surfactant, and saccharin (0.5 g l^{-1}) was added for grain refinement. It is well known that under these conditions S, C and O impurities are present in both grain interiors and at grain boundaries. This technique has the advantage of producing a pore-free nanostructure. However, it generally leads to columnar grains along the growth direction [5,18], while in the deposition plane of grain growth the grains are equi-axed [18]. For the mechanical tests 3 mm mini dogbone-shaped specimens with a cross-section of 200 × 200 μm^2 [19] were cut by wire electrical-discharge machining, and the resulting recast-layer was later on removed by electro-chemical polishing. Table 1 displays structural parameters obtained by X-ray powder diffraction at the Swiss Light Source (SLS). The ratio of the integral intensities I(111)/I(200) is clearly lower than expected for a randomly textured fcc metal (~2), which indicates a (100) texture. This is in agreement with findings from literatures for these kind of samples [5,20–22]. A measure of the average grain size and the root-mean-square (rms) strain is obtained by a Williamson–Hall analysis of the {111}-grain family. We find a grain size of 35 nm (average column length along the grain growth direction) and a rms-strain of 0.32%, both in agreement with earlier work on electrodeposited nc Ni [20,22]. Fig. 1 is a bright-field TEM micrograph of as-prepared nc Ni; a TEM lamella prepared by Focused Ion Beam (FIB) gives the in-plane view (deposition plane).

Table 1. Structural parameters of as-prepared nc Ni determined by X-ray powder diffraction.

Material	Ratio of diffraction intensities I(111)/I(200)	Average grain size/nm	RMS microstrain/%
PED nc Ni	0.97	35	0.32

2.2. Mechanical testing

All mechanical tests were performed with a miniaturized tensile machine [19]. For the in situ tests the machine was mounted at the powder diffraction station of the Materials Science Beam line at the SLS. Diffraction patterns are acquired every 10 s with the Mythen detector [19]. The diffraction peaks are fitted by a split Pearson VII function, yielding information on peak broadening (in terms of the full-width at half-maximum – FWHM), peak position, peak intensity, and peak asymmetry. In this work we concentrate on the behavior of the FWHM during the creep period.

Table 2 provides an overview of the stress reduction experiments that have been done in the present work. A series of standard stress reduction tests in tension has been performed during in situ X-ray diffraction. Fig. 2a provides the schematic representation of the basic principle. 12 specimens were first strained to a fixed stress level σ_0. For all tests a constant total strain rate $\dot{\varepsilon}_{tot}$ of 10^{-3}/s was used except mentioned otherwise. Upon reaching σ_0 the stress is suddenly reduced to a new level σ followed by a 30 min period of creep. Such an operation on the machine is realized in the following way: at a pre-defined stress σ_0, the crosshead is moved backward (opposite to the loading direction) by a certain distance and stops; then the average of the stress value σ is determined immediately and kept constant via a proportional-integral-derivative (PID) controller in a LabView program. The ratio between both stress levels is denoted by the relative stress reduction $R = \sigma/\sigma_0$. Although a constant flow stress can be attained

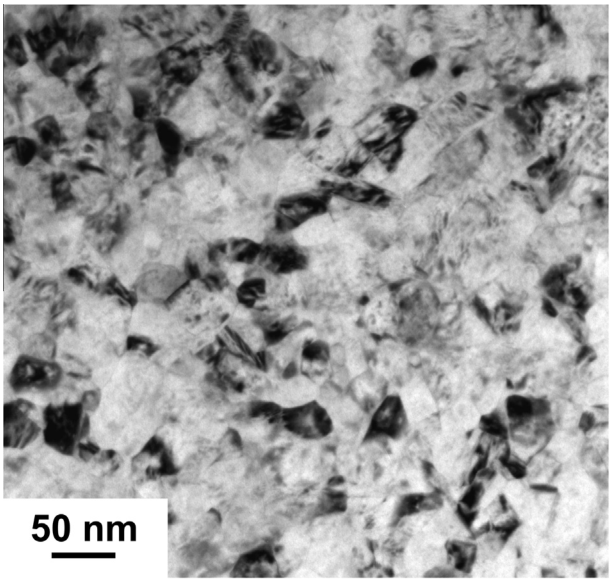

Fig. 1. Bright-field transmission electron micrograph of as-prepared nc Ni in the deposition plane.