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Creep in nanocrystalline Ni during X-ray diffraction

S. Van Petegem, S. Brandstetter, B. Schmitt and H. Van Swygenhoven*

Paul Scherrer Institut, NUM/ASQ, CH-5232 Villigen PSI, Switzerland

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Stress-dip experiments followed by creep have been performed on nanocrystalline Ni during in situ X-ray diffraction. The signatures of the peak profile at different creep stress levels demonstrate the presence of a creep mechanism that broadens the diffraction peak width and of a creep mechanism that reduces the peak width. The balance between the two mechanisms is discussed in terms of the applied creep stress.

Keywords: Creep; Nanocrystalline materials; Nickel; X-ray diffraction (XRD)

Creep in nanocrystalline (nc) Ni has been studied during tension, compression or indentation [1–5]. The mechanism responsible for creep depends on the applied stress and temperature. For polycrystalline metals the stress dependence at steady-state creep at a particular temperature is described by a power-law where the creep rate $\dot{\varepsilon}$ is proportional to the stress σ^n. Here n is the stress exponent which is equal to $1/m$ where m is the strain rate sensitivity defined as $\partial \ln \sigma / \partial \ln \dot{\varepsilon}$. When n is close to 1, results are interpreted in terms of diffusional creep, e.g. Coble mechanism and grain boundary (GB) sliding [6]. When n is higher, creep is often attributed to dislocation climb [6] and cross-slip [7]. For nc metals, a range of results have been reported. Coble-type creep was reported in Ref. [5] whereas values of n between 2 and 10 were attributed to GB sliding and/or a dislocation-based creep mechanism [1–4]. On the other hand, determination of the strain rate sensitivity using strain rate jump tests resulted in values for m larger than 0.01 [8–10], values that are about one order of magnitude higher than those measured for coarse-grained materials. Recently Blum and Li [11] compared the literature data on creep with their own strain rate sensitivity measurements obtained via strain rate jump tests in compression, and demonstrated a remarkable stress dependency of n and m. This stress dependency clearly hints at the presence of a transition regime in terms of the creep behavior, where the stress exponent of the steady-state creep rate decreases rapidly with decreasing stress from values near 100 at $\sigma = 0.02$ G (where G is the shear modulus) to values near 8 at $\sigma = 0.012$ G, followed by a region that shows a very moderate stress dependence. A similar stress/strain dependence of m was observed by Vehoff et al. [4].

In this paper we report creep experiments performed in situ during X-ray diffraction (XRD) at the Materials Science beamline of the Swiss Light Source, and demonstrate the signatures of creep mechanisms at different stress levels on the peak profile. The experiments were carried out on electrodeposited nc-Ni purchased from Goodfellow. The material is characterized by a narrow grain size distribution with equiaxed grains having a mean grain size of ~30 nm. Ref. [12] provides more details on the microstructure determined by electron microscopy and conventional X-ray diffraction. Tensile samples were cut by electrodischarge machining in a conventional dog-bone shape as described in Ref. [13] followed by an electrolytic polishing procedure. Strain rate jump tests performed in tensile deformation resulted in strain rate sensitivities that varied between 0.03 at 1200 MPa and 0.014 at 1800 MPa.

During the creep experiments, the X-ray beam was focused on the center of the gauge section of the samples and had a spot size of less than $500 \times 500\ \mu m^2$. During straining of the specimens, high-quality diffraction spectra covering all diffraction peaks between {1 1 1} and {5 1 1} were continuously recorded within a period of 15 s. The mechanical data were recorded with a frequency of 10 Hz. More details on the technique and the fitting procedure of the XRD spectra are given in Ref. [13].

The in situ creep experiments are carried out on samples that have been deformed to 1200, 1600 and 1800 MPa followed by a drop in stress and a 20 min

* Corresponding author. E-mail: helena.vs@psi.ch

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creep period. Such high-tensile stresses have been chosen since previous experiments have demonstrated that nc-Ni exhibits an extended microplastic regime characterized by a recovery of RMS strain upon unloading, which has been ascribed to non-homogeneous onset of plastic deformation [14]. Therefore, deforming the sample first to a higher stress before the creep stress is applied is a way to guarantee the same initial microstructure for the samples. Stress reduction tests followed by a short 15 s creep period have demonstrated high values for the internal stress σ_i and the effective stress σ^* as well as the presence of negative creep at larger stress drops [15]. For instance, when the stress is dropped from 1800 MPa by more than 450 MPa, a negative strain is observed. The observation has been interpreted in terms of a dislocation mechanism where propagation is hindered by pinning at GB ledges [15]. In the present study, the stress drops which are carried out after the microplastic regime are at values for which a positive creep strain is observed.

Figure 1 displays a typical stress–strain curve involving two stress reduction tests at 1200 and 1600 MPa, where each was followed by a creep test period of 20 min. For the constant strain rate mode, a strain rate of $4 \times 10^{-5}\ s^{-1}$ was used. The stress reduction test were performed by imposition of a negative strain rate $\sim -10^{-2}\ s^{-1}$, resulting in a rapid drop in stress by an amount $\Delta\sigma$ followed by a constant stress creep test during which XRD patterns were continuously recorded. Note that the Young's modulus during unloading is about 160 GPa, which is somewhat lower than what is expected for Ni. This is due to the stiffness of the tensile machine. On a first series of five samples, stress reductions, $\Delta\sigma$, were performed at 1200 and 1600 MPa, resulting in creep at, respectively, 1200, 1190, 1050, 980 and 910 MPa after reductions at 1200 MPa, and at 1600, 1535, 1450, 1370 and 1310 MPa after reductions at 1600 MPa. Similar stress reduction tests were performed at 1800 MPa on another series of five samples, resulting in creep tests at stress levels of 1800, 1700, 1690, 1590 and 1350 MPa. The corresponding creep curves for the tests at 1600 and 1800 MPa are shown in Figures 2 and 3. Despite steady-state creep not being reached after 20 min [2], the experiment was stopped because of restrictions in beam time.

When the creep test is started without stress reduction the effective stress acting on dislocations is high enough

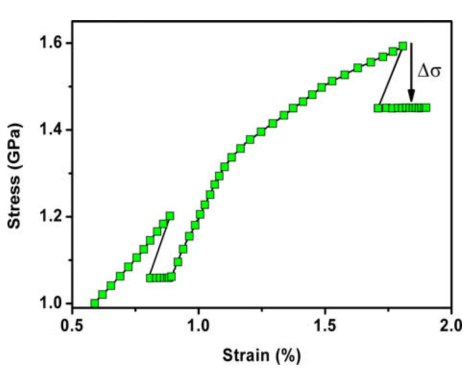

Figure 1. Stress–strain curve with two instantaneous stress reductions followed by creep test of 20 min.

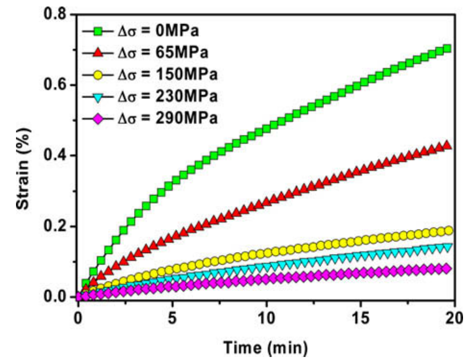

Figure 2. Creep curves after stress reduction at 1600 MPa. The legend indicates the values of the stress reductions.

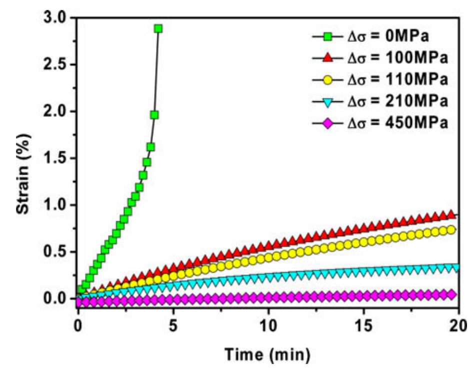

Figure 3. Creep curves after stress reduction at 1800 MPa.

to overcome local obstacles, resulting in a positive strain rate. The decrease of the strain rate with creep time is faster at 1200 MPa compared to 1600 MPa, in agreement with the larger strain-hardening rate. Note that the creep test at 1800 MPa (green squared curve in Figure 3) is performed very close to the maximum flow stress (~1850 MPa). This results in a creep rate that is close to the initially applied strain rate, which is in agreement with a work-hardening rate close to zero. Furthermore after a short period of creep enough plastic strain was accumulated such that the sample enters a regime of localized plastic deformation, resulting in a dramatic increase of strain rate and early failure. When the stress is reduced the creep rates reduce accordingly. For a stress reduction of 450 MPa almost no strain increase could be observed during the 20 min creep test, in agreement with earlier observations [15].

Figures 4 and 5 display the behavior of the full-width at half-maximum (FWHM) of the {311} diffraction peak as function of time for the stress reduction tests performed at, respectively, 1600 and 1800 MPa. The maximum broadening of the {311} peak that is reached at the maximum flow stress for this material during constant strain rate deformation amounts to 0.09° ($\Delta 2\theta$) (see also Ref. [15]). From 1600 MPa to the maximum flow stress the broadening increases by 60% and this for 2% additional strain. During the 0.7% strain obtained during 20 min creep at 1600 MPa, the broadening increases 30%, which is nearly the same amount that would be reached during a constant strain rate experiment for the same strain (Fig. 2). On the other hand,