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Untangling dislocation and grain boundary mediated plasticity in nanocrystalline nickel

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Abstract

Nanocrystalline (nc) materials possess unique mechanical properties, such as very high strength. However, an understanding of the deformation mechanisms and the succession of related microscopic processes that occur during deformation is still incomplete. We used synchrotron-based in situ compression testing to investigate the sequence of deformation mechanisms emerging in bulk nc nickel with a grain size of 30 nm. The study was accompanied by high-resolution grain size analysis and crystal orientation mapping using transmission electron microscopy. Regardless of the initial microstructure, the deformation behavior of electrodeposited nc Ni is initiated by inhomogeneous elastic lattice straining and its accommodation within the grain boundary network, followed by the onset of dislocation plasticity, which was inferred from texture evolution, and stress-driven grain growth. This observation indicates that deformation in nc metals is governed by a succession of different, partly overlapping mechanisms. It is estimated that intragranular dislocation plasticity contributes only about 40% to the overall deformation.

Keywords: Nanocrystalline material; Mechanical properties; Texture; Synchrotron diffraction; Plastic deformation

1. Introduction

It is widely accepted that nanocrystalline (nc) metals (grain size $D \ll 100$ nm) show very high strength accompanied with a change in deformation behavior compared to their coarse-grained (cg) counterparts. However, despite considerable efforts, a detailed understanding of the relevant deformation mechanisms is still limited [1,2], but is required for the practical application of nc metals. The high strength of these metals is due to the large fraction of grain boundaries (GBs) that act as barriers to the motion of lattice dislocations, which in cg metals are carriers of plasticity. The interaction of dislocations leads to strain hardening and the typical ductility of metals. In contrast, the impeded intragranular dislocation plasticity in nc metals leads to reduced conventional strain hardening and ductility. However, the increased GB area per unit volume may lead to intergranular deformation mechanisms such as GB shear and slip, grain rotation or grain boundary migration; similarly, GBs may act as sources and sinks for lattice dislocations. In order to improve the mechanical properties

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of nc metals the relevant deformation mechanisms and their interplay need to be characterized in detail.

There has been a long-standing debate about the interplay of dislocation- and GB-mediated plasticity in the nc regime [3–10]. In fact, texture analysis can differentiate between both types of deformation mechanisms [10–12], since the combination of GB sliding and grain rotation maintains the random crystal orientation within a nc aggregate, while grain rotation based on dislocation plasticity promotes texture formation. More recently, the spectrum of possible deformation mechanism in nc metals was broadened by grain boundary migration [13,14].

Unraveling the deformation behavior of nc materials and accurately separating contributions from different deformation mechanisms calls for in situ synchrotron X-ray diffraction (XRD) [15]. We conducted in situ compression experiments on chemically and microstructurally well-characterized bulk electrodeposited Ni samples ($D = 30$ nm). We combine in situ XRD with high-resolution automated crystal orientation and phase mapping (ACOM) analysis using transmission electron microscopy (TEM) [16]. The combination of these methods enables us to correlate the in situ diffraction data with direct observation of the initial and deformed microstructures by high-resolution microscopy. Compression experiments enable the macroplastic regime ($\varepsilon > 10\%$) to be investigated in detail, while most mechanical testing experiments have been conducted in tensile mode [3,6,7,10], where fracture after a few per cent of plastic strain is commonly observed. In doing so, we have been able to differentiate between dislocation plasticity, interfacial deformation modes as well as stress-induced grain growth and could assign these modes to distinctive strain regimes. We observe coexistence of these three deformation modes even in the macroplastic regime ($\varepsilon > 10\%$). Moreover, we have been able to determine quantitatively the onset strain and relative share of the individual contributions to the overall deformation. As a result we found that interfacial plasticity plays a predominant role even at high plastic strains.

2. Experimental

2.1. Sample preparation

The bulk Ni samples (rectangular plates, 40 mm × 70 mm, thickness between 1.5 and 3 mm) from which the compression specimens ($1 \times 1 \times 0.6$ mm^3) were cut by spark erosion, were produced by pulsed electrodeposition (PED) from a Ni sulfamate electrolyte [17]. During this process, the microstructure of the deposit is controlled by the following deposition parameters: on-time, off-time, current density of the pulse function, and the concentration of organic grain refiners [18,19]. Severe texture formation is suppressed by adding butynediol to the electrolyte. In addition, grain size is also controlled by adjusting the current density. An increase in grain size promotes the formation of new nuclei rather than the growth of prevailing crystallites and thus also helps to suppress columnar growth [20,21].

Our nickel samples were prepared employing PED with a cathodic current density of 45 mA cm^{-2} and setting a pulse length of 5 ms followed by a 10 ms off-time. The electrolyte was based on nickel sulfamate (595 ml l^{-1}) with additives of nickel chloride hexahydrate (5 g l^{-1}) and sodium lauryl sulfate (0.2 g l^{-1}) [22]. Boric acid (35 g l^{-1}) was used to buffer the pH value. Furthermore butynediol (0.02 g l^{-1}) and saccharin (0.4 g l^{-1}) were added to refine the grain size.

Regarding sample purity, we detected using EDX an oxygen content of ~4.5 at.%, while heavy element impurities were beyond the detection limit of 0.5–1 at.%. In particular, we could not detect sulfur or chloride impurities from the electrolytic bath.

2.2. In situ synchrotron experiments

Synchrotron-based in situ compression testing is perhaps the most promising technique for investigations of deformation mechanisms in nc metals for the following reasons: (i) high plastic strains (up to 20%) can be induced in nc materials despite their inherent limited ductility; (ii) high penetration depth using high-energy synchrotron radiation permits experiments in transmission geometry and thus provides excellent statistics; (iii) fast (up to 2 patterns s^{-1}) and large area detectors allow collection of up to 1000 diffraction patterns during one mechanical test. Each pattern comprises several complete Debye–Scherrer rings and thus the evolution of peak shape and texture can be monitored with high accuracy and time resolution. We exploit this technique to penetrate 600 μm thick bulk nc electrodeposited Ni samples ($D = 30$ nm) during compression testing ($\dot{\varepsilon} = 7 \times 10^{-4}$ s^{-1}). The in situ XRD experiments were carried out at the High Energy Microdiffraction (HEMD) endstation of beamline ID15A of the European Synchrotron Radiation Facility (ESRF) (energy $E = 69.7$ keV, wavelength $\lambda = 0.178$ Å, beam size: 8 μm high × 20 μm wide). The setup (shown in Fig. 1) is based on a method for in situ lattice strain and stress measurements during tensile tests of thin metallic films on compliant substrates [23]. A mechanical testing device (Kammrath & Weiss, Germany) was mounted on the diffractometer, and the incident X-ray beam was oriented perpendicular to the sample surface. By scanning of the sample the X-ray beam was positioned at the center of the sample. Complete Debye rings were recorded on an area detector (Pixium 4700, Thales Electron Devices, Moiron, France) placed in transmission geometry (xy-plane) with fast data acquisition (2 patterns s^{-1}). The fast detector and the high X-ray flux allowed for continuous tests with a strain rate of 7×10^{-4} s^{-1}. The macroscopic strain of the sample is measured by a CMOS camera (Pixelink, Ottawa, Canada) using digital image correlation [24]. A homogeneous grid of markers was distributed over the entire sample. After processing unreliable markers were deleted.