Enhanced tensile ductility in an electrodeposited nanocrystalline Ni

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Abstract

A fully dense nanocrystalline Ni exhibited high strength of about 1200 MPa and enhanced ductility of 7.5–8.3% at strain rates of 1.04 × 10⁻⁴–1.04 s⁻¹ and room temperature. The dislocation activity in the strain hardening stage and collective grains rotation after instability are suggested as being responsible for the enhanced ductility.

Keywords: Nanocrystalline; Mechanical properties; Microstructure; Nickel

1. Introduction

Fully dense nanocrystalline (nc) materials are characterized by high yield stresses and limited tensile ductilities [1–10]. The decrease of ductility with the decrease of average grain size could be an inherent property of nc materials. It has been predicted that dislocation is difficult in such tiny grains where the distance between dislocation pinning points becomes very small, demanding very high stresses to activate dislocation sources [11]. In addition, the method of fabrication and the specimen size for tensile tests may also have effects on the ductility of nc materials [5,6]. So some efforts should be made to improve the tensile ductility of nc materials for structural applications, which often require both high strength and good ductility [2,12].

Wang et al. [13,14] proposed a thermomechanical treatment of Cu that could result in a bimodal grain size distribution, with micrometer-sized grains embedded inside a matrix of nc and ultra-fine crystalline (ufc) grains. This inhomogeneous microstructure can produce nanostructured Cu with a 30% uniform elongation and a 65% elongation to failure, which also retains its high strength. The reason for this behavior is that while the nc grains provide strength, the embedded large grains stabilize the tensile deformation of the material [13,15]. Other evidence for the importance of grain size distribution comes from the studies of the plastic deformations in ufc and nc Zn [16] and annealed nc Ni [17].

The electrodeposited nc Ni, usually with a grain size of below about 30 nm, has been investigated extensively and this material is usually low in ductility, with an elongation to failure of about 3–4% [1,5–10]. The current work shows a new electrodeposited nc Ni with special microstructures. Its tensile deformation behaviors are examined at strain rates of 1.04 × 10⁻⁴–1.04 s⁻¹ and room temperature (RT). The mechanisms underlying the mechanical response are probed using the results of analysis of the deformed specimens.

2. Experimental procedures

A sheet of fully dense (99.4 ± 0.5% of theory density) nc Ni with a thickness of about 350 μm was obtained by a direct current electrodeposition method from an electrolyte bath containing nickel sulfate, nickel chloride, boric acid and some surfactants which acted as stress relievers and grain refining agents. The crystallographic structure was analyzed using a X-ray diffractometer (XRD, D/max 2500PC). Microstructures of the nc Ni were observed with

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a transmission electron microscope (TEM, H-800). Chemical analysis by inductively coupled plasma atomic emission spectrometry (ICP-AES, Plasma/1000) and carbon/sulfur determinators showed that the nc Ni had a purity of about 99.8 wt.% with the main impurities of 120 ppm S, 660 ppm C, 511 ppm Pb, 230 ppm Co, and 133 ppm B.

The dog-bone shaped specimens (shown in Fig. 2(a)) with a gauge cross-section of 2.0 mm × 0.3 mm and a gauge length of 8.0 mm were cut from the as-deposited nc Ni sheet by using an electrodischarging machine and then polished to a mirror-like finish surface. The tensile tests were carried out on MTS-810 system at strain rates of 1.04 × 10^{-4}–1.04 s^{-1} at RT. Tensile ductility in this study was measured through the cross-head movement of the tensile machine. The morphologies of the fracture surface and deformed surface of specimens were observed using a scanning electron microscope (SEM, JSM-5600).

The surface texture of deformed specimen was studied by general area detector diffraction system (GADDS, Bruker D8 Discovery) with a Cu target at 40 kV and 30 mA. The scanning region 2θ and acquiring time were 32–66° and 60 s, respectively. The GADDS instrument could be used to identify and analyze small areas (usually determined by the collimator size) on the surface of the specimen which is mounted on an XYZ stage. In this study, the collimator size was 100 μm in diameter. The irradiation direction of the X-rays had angles of about 90° and 25° with respect to the tensile specimen axis and the plane surface of the specimen, respectively.

3. Results

3.1. Microstructures

The electrodeposited Ni is characterized by a mean grain size of about 40 nm, a strong preferential orientation along the {200} planes, and a microstrain of 0.39% revealed by XRD. Fig. 1(a) displays the TEM image of the nc Ni together with the corresponding selected area diffraction (SAD) patterns. No additional peaks could be measured in the inset SAD patterns, indicating that there is no second phase in the nc Ni. As shown in Fig. 1(a), there are many grain clusters with sizes of about 150–300 nm. The clusters are surrounded by nanograins with size of about 10–20 nm, or by fiber-like structures. The size of the grains in the clusters is about 30–80 nm. The grains in some clusters possess small misorientation angles, as shown in the higher magnification TEM image (Fig. 1(b)). Furthermore, a wide grain size distribution from 5 to 80 nm is shown in Fig. 1(c), which is another distinguishing characteristic of the present electrodeposited nc Ni. In Fig. 1(c), the distributions of the number fraction and the volume fraction vs. the grain sizes are illustrated. It can be seen that there are a large number of small grains with sizes typically less than 10 nm. The electrodeposited nc Ni also exhibits a low and uniform dislocation density.

![Fig. 1. TEM micrographs: (a) lower magnification, (b) higher magnification and (c) grain size distribution of the electrodeposited nc Ni.](image_url)
than 10 nm. The total volume fraction of the grains of sizes larger than 50 nm is estimated to be about 19%, which is far higher than their number fractions, at only about 4%. As to the volume fraction distribution, the predominant grain size of this Ni is about 40 nm. Typical cross-section TEM micrographs of the electrodeposited nc Ni [5,18] showed that the cross-sectional microstructure of the sample contains elongated grains. So this microstructure is also expected in the present nc Ni.

3.2. Mechanical behaviors

The specimen fracture direction has an orientation of about 60° with respect to the tensile axis, as shown in Fig. 2(a). Fig. 2(b) gives the nominal engineering stress–strain curves of the electrodeposited nc Ni performed at different strain rates and RT. Compared with previous studies in nc Ni [1,5–10], this nc Ni has a moderate yield strength \( \sigma_{0.2} \) and ultimate tensile strength \( \sigma_{UTS} \) of about 900 and 1200 MPa, respectively. However, this nc Ni exhibits prevalent high tensile ductility (7.5–8.3%). Its uniform strain \( \varepsilon_U \) and post-uniform strain \( \varepsilon_{PU} \) are 5.6–6.1% and 1.6–2.4%, respectively. The tensile properties of the nc Ni are also characterized by the significant strain hardening (\( \sigma_{UTS} - \sigma_{0.2} \approx 300 \) MPa), extended uniform plastic deformation and developed necking. In fact, the enhanced ductility in nc Ni was first reported by Li and Ebrahimi [19]. Our nc Ni and the reported nc Ni prepared by electrodeposition [19] are similar in average grain size (40 and 44 nm) and ductility values. However, the yield and ultimate tensile strength levels of the 44 nm Ni [19] are lower, about 446–544 MPa and 1076–1088 MPa, respectively. The strain rate sensitivity \( m \) of the flow stress (at 1% plastic strain) in this study is estimated to be about 0.012, as shown in Fig. 2(c), which is only slightly different from the reported values 0.01–0.03 [6] and 0.02 [10].

3.3. Morphologies of deformed and fracture surfaces

Fig. 3 presents the thickness surface morphologies of the broken specimen tested at a strain rate of \( 1.04 \times 10^{-3} \) s\(^{-1} \). The obvious necking extending about 400 \( \mu \)m in length is shown. The maximum thickness strain near the fracture region is about 50%, where the crossed micrometer shear bands (with widths in the range of 2–5 \( \mu \)m) exist on the thickness surface (shown in the up-right inset). However, there are no crossed shear bands but only strip deformation traces vertical to the tensile axis far from the large necking.
region, which is shown in the upper-left inset. In fact, shear bands are an indication of localized plastic deformation (instability), which is usually prevalent at large plastic strains (the necking region in our case or in compression tests [20–22]) of nc materials.

Some other interesting features are also observed on the sheet surface of the deformed specimen tested at a strain rate of $1.04 \times 10^{-3} \text{s}^{-1}$, which are shown in Fig. 4. The central picture of Fig. 4 is the half broken specimen. Fig. 4(a)–(d) shows SEM micrographs of the local regions marked by the white rectangles on the fractured specimen. Contrary to the polished smooth surface (Fig. 4(a)), an increasing roughness on sheet surface is observed from the uniform deformation to the necking region (Fig. 4). In particular, obvious surface relief (steps) is observed on the necking region (Fig. 4). This morphology looks quite similar to the surface relief that originate from grain rotation and grain boundary sliding, which usually occur during superplastic deformations [23]. In addition, significant slip traces have an orientation of about $60^\circ$ with respect to the tensile axis and are parallel to the fracture direction, as shown in Fig. 4(c) and (d). The similar deformation relief is also observed in the tensile tests of the SPD-processed 330 nm Ni [24].

Fig. 5 gives the typical tensile fracture surface of the nc Ni deformed at a strain rate of $1.04 \times 10^{-3} \text{s}^{-1}$. The

![Fig. 3. Thickness surface morphologies of the broken specimen tested at a strain rate of $1.04 \times 10^{-3} \text{s}^{-1}$ and RT.](image)

![Fig. 4. Sheet surface morphologies of the broken specimen tested at a strain rate of $1.04 \times 10^{-3} \text{s}^{-1}$ with different strains: (a) $\varepsilon = 0.0\%$; (b) $\varepsilon = \varepsilon_U = 5.5\%$; (c) $\varepsilon = 10–15\%$ and (d) $\varepsilon = 30–35\%$.](image)

![Fig. 5. Typical tensile fracture surface of the nc Ni deformed at strain rate of $1.04 \times 10^{-3} \text{s}^{-1}$. Some knobbly protuberances on the fracture surface are marked by an arrow.](image)
evidently dimpled morphology is observed on the fracture surfaces of all the tested specimens and these dimples have two main characteristics. One is that the dimple sizes have a wide distribution from 100 nm to about 2 µm. The other one is that the sizes of the observed knobby protuberances (about 150–300 nm) on the fracture surface (marked by an arrow in Fig. 5) are similar to those of the grain clusters (Fig. 1(a)), which implies that final fracture might initiate and evolve along the boundaries between the clusters of nanograins or between the small nanograins surrounding larger grain clusters. Also, it should be noted that the size of the dimples is similar to that of the surface relief (steps) observed on the surface of necking region (Fig. 3).

3.4. Results of the GADDS detection

The specimen for GADDS detections is chosen to be the one deformed at a strain rate of $1.04 \times 10^{-3} \text{s}^{-1}$. The dependence of the intensity ratios of \{111\}/\{200\} peaks on the thickness strains determined by SEM observation is shown in Fig. 6. The detections are undertaken on the thickness surface and the sheet surface of the fractured specimen, respectively. The typical regions for detections are marked with arrows plus numbers in the inset figure. The intensity ratio of \{111\}/\{200\} peaks in a standard powder diffraction pattern of Ni powder is about 2.38, which represents the value for the isotropic polycrystalline Ni. Interestingly, in this study, it is found that the intensity ratio of \{111\}/\{200\} peaks increases toward the isotropic direction with the increase in the thickness strain. In the necking region near the fracture front, the ratio reaches about 0.7, where a thickness strain of 48% is measured. This indicates that collective movement of grains or grain rolling would have occurred during the tensile deformation of the nc Ni. The rolling of grains should result in the variation from the \{200\} texture towards the random arrangement of grains.

4. Discussion and conclusion

For face-centered cubic metals with grain sizes of 30–50 nm, dislocation-mediated plasticity should be the dominant deformation mechanism [5,25]. A broad grain size distribution with some fraction of grains large enough to sustain dislocation activity has been shown to be effective in providing the needed strain hardening therefore increased ductility [4,13]. In our nc Ni, the grains in clusters should be deformed through dislocation activity and then provide the considerable strain hardening. The internal stress that develops owing to the strain incompatibility among grains with different grain sizes may also cause strain hardening [19]. Small sized grains also lead to reduced flaw sizes and increase difficulties for the imposed stress concentration at the flaw to exceed the critical toughness of the material, thus suppressing early crack nucleation and propagation [14].

In situ dynamic TEM observations on the nc Ni film with an average grain size of 10 nm showed that rotation of several grains as a group took place during tension [26]. Cooperative grain activity was also observed in the molecular dynamics simulation of 6 nm Ni [27]. The fully developed necking after the Considère instability in our nc Ni may be explained by the cooperative motions of grains, which leads to remarkable post-uniform deformation. Evident surface relief (steps) is observed on the sheet surface in necking region, which indicates that collective sliding or rolling of grains as a whole group take place during plastic deformation and is more evident at large deformation. The collective movement of grains results in the variation of the orientation of grains towards weakening the initial \{200\} texture revealed by the GADDS detections on the necking region.

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