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Interrupted in situ EBSD study of texture evolution and mechanism of surface grains in electroformed Ni after annealing with an initially duplex ⟨100⟩ + ⟨111⟩ fiber texture during uniaxial tensile deformation

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ABSTRACT

The texture evolution process and mechanism of surface grains in electroformed Ni after annealing with an initially duplex ⟨100⟩ + ⟨111⟩ fiber texture during uniaxial tensile deformation were investigated by using interrupted in situ electron backscattered diffraction. The results showed that the initially duplex ⟨100⟩ + ⟨111⟩ fiber texture evolved into the metastable Cube orientation with a weak Cu orientation during the uniaxial tensile deformation. The volume fractions of ⟨100⟩ and ⟨111⟩ fiber texture decreased from 54.0% to 29.3% and 16.6% to 6.6%, respectively. The volume of the Cube orientation remained almost stable at 12%, whereas that of the Cu orientation increased from 1% to 2.9%. A simulation of the final preferred orientation based on the Taylor model was a cubic texture, which qualitatively agreed well with the experimental result. The simulated results indicated that the formed Cube and Cu textures evolved from the initial ⟨100⟩ and ⟨111⟩ fiber textures, respectively.

1. Introduction

Petroleum derivatives play a major role in the development of the modern society. In this regard, large amounts of reservoirs are required for the location and extraction of petroleum reservoirs [1]. Since Birkhoff [2] proposed a penetration theory about explosives with lined cavities in 1948, the pure metal shaped charge liner (SCL) has been successfully developed as a critical part of explosives with lined cavities and applied as an oil well penetrator in the oil industry [1]. As an important criterion to evaluate the damaging effect of SCL, penetration depth is affected by many factors, such as the density of SCL, standoff, and explosives [3]. Based on the assumption that the jet is continuous and jet properties remain constant throughout the penetration process, the penetration depth of SCL only depends on the jet elongation and density ratio between the jet and target, $P = l(\rho_j/\rho_t)^{1/2}$ [2], where P is the penetration depth, l is the jet original length, which is proportional to the plasticity of SCL, ρ_j is the jet density, which is determined by the density of SCL, and ρ_t is the density of the target. Pure Ni is an important material for penetrating high-density targets, owing to its high plasticity [4,5], high density [6,7], and good corrosion resistance [8]. Electroforming has attracted much attention as a unique method for preparing pure Ni liner, owing to its characteristics of high purity, controlled microstructure, and nearly uniform shape [6,9–12]. Yang et al. [6,12] prepared a Ni SCL using an electroforming technique and investigated the crystal defects and deformation behaviors of the electroformed Ni SCL.

In order to improve the penetration depth of the electroformed Ni SCL, the preparation of high-plasticity electroformed Ni SCL needs to be further investigated. Some researchers investigated the preparation and mechanical properties of the electroformed Ni working as a structural material [5,13–17]. Yang et al. [5] pointed out that an electroforming process without chloride ion activating the anode in the solution could be used to prepare high-plasticity Ni. Buchheit et al. [14] compared the microstructures and mechanical properties of Ni electroformed from a Watts bath and sulfamate bath. In addition, the effects of the interstitial elements [16,17], film thickness [13], and deformation temperature [4] on the fracture modes of the electroformed Ni were also carefully investigated. After the electroforming process, annealing is always used as an important heat treatment process to eliminate the inner stress in the electroformed part in order to improve the plasticity [10,14].

Annealing would cause an inevitable evolution of the texture. The initial texture has a significant effect on the microstructure evolution during uniaxial tensile deformation [18]. The ⟨100⟩ + ⟨111⟩ fiber texture of electroformed Ni after annealing [19,20] would have specific

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microstructural evolution behaviors. However, few investigations focused on the microstructure evolution of electroformed Ni after annealing with an initially duplex ⟨100⟩ + ⟨111⟩ fiber texture during uniaxial tensile deformation.

Owing to the free surface leading to stress relaxation [21], there are many fewer constraints for surface grains than for the interior grains of the same bulk material [22]. The surface grains have fewer independent slip systems and lower flow stresses [22]. Therefore, the deformation behaviors of the surface grains are affected by the free surface and are not entirely the same as the interior grains. In situ electron backscattered diffraction (EBSD) is usually used to investigate the deformation behaviors of surface grains [22,23], because it can only probe the near-surface volume [24]. To control the properties of thin materials such as microelectromechanical components [14] and aluminum beverage cans [22], it is essential to understand the deformation mechanisms of surface grains. For the electroformed shaped charge liner, it could also be regarded as a thin material when it consists of a few layers of large columnar grains.

In the present work, we investigated the texture evolution of surface grains in electroformed Ni after annealing during uniaxial tensile deformation by using an interrupted in situ EBSD. The results indicate that the initially duplex ⟨100⟩ + ⟨111⟩ fiber texture evolved into the metastable Cube orientation with a weaker Cu orientation during the uniaxial tensile deformation.

2. Experimental

2.1. Material Preparation

Pure Ni plates were electroformed from a nickel sulfamate bath to a thickness of approximately 1.3 mm. Table 1 shows the bath composition and operating conditions. The plating solution included sodium dodecyl sulfate (SDS) as the wetting agent. The pH was maintained between 3.3 and 4 in the sulfamate bath at 323 K. Pure electrolytic Ni coins (Falconbridge Limited) were used as the anode material for electroplating.

The electroformed Ni plate with columnar grains was peeled off from the Al substrate. Tensile test samples were cut into a shape with a gauge length of 6 mm and thickness of 0.6 mm by using electron discharging machining (EDM). Fig. 1a shows the definition of the specimen directions. As shown in Fig. 1a, RD, TD, and ND represent the tensile, transverse, and deposition directions, respectively. The tensile test samples were then annealed at 923 K for 1 h. The estimated sulfur and carbon contents of the electroformed Ni plate were 10 ppm and 30 ppm, respectively. Fig. 1b shows the dimensions of the tensile sample with a Vickers hardness indentation at the center point. The marked point acted as the reference point for further observations. Fig. 1c shows a photograph of the microtensile test machine used to perform the uniaxial tensile tests.

2.2. Mechanical Testing and Microstructure Analyses

The uniaxial tensile tests were performed by using a microtensile tester (TSL solutions), as shown in Fig. 1c. All the uniaxial tensile tests in this study were conducted at a rate of 0.003 mm·s^{-1}, corresponding to a strain rate of 5×10^{-4} s^{-1}.

Table 1
Composition of bath and electrolysis condition.

Bath composition	$Ni(SO_3NH_2)_2 4H_2O$	g·L^{-1}	450
	H_3BO_3		40
	$C_{12}H_{25}SO_4Na$		0.1
Operating conditions	Current density	A·dm^{-2}	4
	Temperature	K	323
	pH		3.3–4
	Cathode		Al (10 cm × 10 cm)
	Anode		Ni coins

The surface grains of the specimens were examined by electron backscattered diffraction (EBSD) using a Carl Zeiss Ultra 55 field emission scanning electron microscope (SEM) with the software package OIM 6. The EBSD observations were conducted with a working distance of 17 mm and an acceleration voltage of 15 kV. The scanning step size for the in situ experiments was 100 nm. Before and after the deformation of the samples, the same area including at least 300 grains was measured. In order to eliminate the effect of the indentation, the observation area was approximately 0.3 mm away from the marked center point. For regular (continuous) tensile experiments, the scanning step size is 400 nm, owing to the relatively larger scanning area. The orientation distribution functions (ODFs) are calculated from the EBSD data using the harmonic series expansion method for $l_{max} = 22$ and are presented in constant $\varphi_2 = 45^\circ$ sections in the Euler space defined by three Bunge Euler angles: φ_1, Φ, and φ_2 [25]. The Gaussian spread is set at 5°. Orthotropic sample symmetry has been used for the purpose of texture representation of the Ni samples. The specimens were first mechanically polished, followed by electropolishing in a solution of sulfuric acid (40%) and deionized water (60%) at room temperature. An ion milling machine (IM-3000, Hitachi) was also employed to polish the surface of the tensile test samples at an acceleration voltage of 2 kV and a tilt angle of 80° for 15 min.

3. Results and Discussion

3.1. Initial Microstructure before Deformation

Fig. 2 shows the EBSD orientation image maps of the texture of the electroformed Ni specimen after annealing from the plane and cross-sectional views. As presented in Fig. 2, the color represents the nearest ideal orientation at every location, with orientation spreading about ideal orientations of 15°. The Cube {100}⟨010⟩ orientation, ⟨100⟩ fiber orientation, and ⟨111⟩ fiber orientation are shown in red, cyan, and blue, respectively. The white region indicating random orientation components has orientations higher than 15° from the ideal orientations. The black area corresponds to the non-indexed zone. The black, grey, and green lines refer to misorientations of > 10°, 2°–10°, and Σ3 twin boundaries, respectively.

As shown in Fig. 2a, the shape of the grains from the plane view is approximately equiaxed after annealing at 923 K for 1 h. The recrystallized grains have an approximated average grain size of 3 μm. The texture mainly consists of the ⟨100⟩ fiber orientation, the Cube orientation, and the ⟨111⟩ fiber orientation.

From the cross-sectional view, columnar grains with the ⟨100⟩ fiber orientation could be found in Fig. 2b. A portion of the columnar grains evolved into equiaxed grains, which are distributed among the columnar grains as shown in Fig. 2b. The texture components of the cross-sectional area also contain the ⟨100⟩ fiber orientation, the Cube orientation, and the ⟨111⟩ fiber orientation, which is similar to that of the plane area, as shown in Fig. 2a.

Some annealing twins distributed in the recrystallized grains, as indicated by the green lines in Fig. 2b. For clarity, Fig. 2c only shows the boundary misorientations map of the same area as Fig. 2b. The length fraction of the Σ3 twin boundaries shown by the green lines in Fig. 2c is approximately 34.1%. The emerged weaker ⟨111⟩ fiber texture is probably caused by copious twinning occurring along the migration front of the abnormally growing grains [19].

3.2. Tensile Properties

Fig. 3 shows macroscopic stress–strain curves for the regular and interrupted tensile tests of the electroformed Ni specimens after annealing. As shown in Fig. 3, the sample of the interrupted tension experiment underwent the unload–reload processes in the strain control mode to perform the EBSD. Letters a to h represent the in situ