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Effect of heat treatment on diffusion, internal friction, microstructure and mechanical properties of ultra-fine-grained nickel severely deformed by equal-channel angular pressing

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Abstract—Severe plastic deformation via equal-channel angular pressing was shown to induce characteristic ultra-fast diffusion paths in Ni (Divinski et al., 2011). The effect of heat treatment on these paths, which were found to be represented by deformation-modified general high-angle grain boundaries (GBs), is investigated by accurate radiotracer self-diffusion measurements applying the ^{63}Ni isotope. Redistribution of free volume and segregation of residual impurities caused by the heat treatment triggers relaxation of the diffusion paths. A correlation between the GB diffusion kinetics, internal friction, microstructure evolution and microhardness changes is established and analyzed in detail. A phenomenological model of diffusion enhancement in deformation-modified GBs is proposed.

Keywords: SPD; ECAP; Grain boundary diffusion; Microhardness; Internal friction

1. Introduction

Severe plastic deformation (SPD) is considered to provide a useful experimental tool for producing materials with a unique combination of enhanced mechanical and functional properties [1]. A large fraction of high-angle grain boundaries (GBs) and their specific deformation-induced state were suggested to be responsible for some of these properties [2,3].

In our previous study [4] we have demonstrated that short-circuit self-diffusion is greatly enhanced in ultra-fine-grained (UFG) Ni prepared by equal-channel angular pressing (ECAP) in comparison to that in the well-annealed coarse-grained material. Similar conclusions were previously drawn for ECAP Ni by monitoring the diffusion rate of Cu solute [5]. The diffusion enhancement was explained in terms of a specific deformation-induced state of general high-angle GBs. Moreover, the temperature dependence of the GB self-diffusion was found to exhibit non-linear Arrhenius behavior with a characteristic change (seen as a kink in an Arrhenius plot) around 400 K, which was related to the partial relaxation of the deformation-induced state of interfaces in UFG Ni [4].

The present paper aims to investigate the relaxation behavior of UFG Ni, highlighting a correlation of the changes in the kinetics of the short-circuit diffusion and the evolution of microstructure and mechanical properties including internal friction (IF) as a result of heat treatment.

2. Experimental details

2.1. Sample preparation

Pure Ni (99.6 wt.% purity) was severely deformed by ECAP via route B_C, i.e. by pressing the specimen through a 90° channel die four times and rotating it by 90° about the long axis in the same direction after each pass. Henceforth this processing route will be designated B_C4. The deformation was carried out at room temperature. The processing details were described in our previous paper [4].

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2.2. Microstructure analysis

The microstructure was investigated by transmission electron microscopy (TEM) using a Zeiss Libra 200FE instrument. A typical bright-field micrograph of the UFG microstructure induced by severe deformation is shown in Fig. 1a. The interiors of some grains display a strong contrast because of a large amount of strain therein. The density of dislocations is very high inside and near the GBs. Interference fringes are often observed across the GBs, indicating that they are heavily distorted. The grains are typically elongated, although equiaxed grains are also observed. A fraction of GBs (presumably high-angle GBs according to the variation of contrast on the corresponding bright- and dark-field images) are flat and almost straight. The observed UFG microstructure is similar to that found after SPD treatment of pure Ni (e.g. [6,7]). A lamellar microstructure with geometrically necessary GBs [8,9] can be recognized (Fig. 1a). The lamellar interfaces are represented by general high-angle GBs, whereas dislocation cells are typically separated by low-angle GBs. A detailed analysis of the microstructure was reported elsewhere [10].

Microstructure characterization was performed by scanning electron microscopy (SEM), using an FEI Nova NanoSEM 230 instrument. A typical large-scale microstructure of the as-prepared state is presented in Fig. 1b. Orientation imaging microscopy (OIM) employing electron backscatter analysis was used. The inverse pole figure map (the insert in Fig. 1b) was utilized for the color coding of grain orientations. The heterogeneity of the UFG microstructure of B_C4 Ni was more obvious in the SEM images, rather than in local TEM micrographs. The grains are mostly anisotropic and elongated along the shear direction of the final deformation pass. It is known that a larger number of ECAP passes (up to 12) results in a more homogeneous microstructure in Ni with almost equiaxed grains [11]. In the present study we limited the deformation to four passes in order to prevent the formation of so-called percolating porosity [12], which would complicate reliable measurements of the diffusion properties of the interfaces. Percolating porosity was absent in B_C4-processed Ni [4], but was found in ECAP-processed Ni after eight passes [13]. (For the effect of percolating porosity on diffusion see, for example, our publications on ECAP-processed Cu and Cu-based alloys [12,14–16].)

2.3. Microhardness

For microhardness measurements, a Buehler Indenta-Met 1105 Microindentation Hardness Tester and a Wolpert Wilson 402MVD device were used. The indentation force and the dwell time were set to 9.81 N and 10 s, respectively. Each microstructure state was characterized by performing at least 20 individual measurements distributed randomly over the whole polished surface of the sample (except near-edge areas), keeping the distance between individual indents as large as possible, and taking the average value of the microhardness.

2.4. Internal friction

A dynamic mechanical analyser DMA Q800 (TA Instruments) was used to study the damping characteristics of severely deformed Ni and its evolution with annealing treatment. The temperature-dependent internal friction (TDIF) was measured under strain control in the range of temperatures, T, from 298 K to 873 K. A periodic deformation was applied and an oscillating strain response, $\epsilon = \epsilon_0 \cos(\omega t + \phi)$, was measured, where $\omega = 2\pi f$, t is the time, and ϕ is the loss angle. An advantage of the DMA Q800 is that the same specimen can be tested over a range of frequencies, f, from 0.3 to 30 Hz by ramping the frequency. The IF, Q^{-1}, for the forced vibrations is given by $Q^{-1} = \tan\phi$. Specimens for damping tests were cut with long axis along the ECAP direction to a size of $1 \times 5 \times 25\ mm^3$.

2.5. Calorimetric measurements

Differential scanning calorimetry (DSC) was conducted using a Diamond DSC device (Perkin Elmer, USA), which was calibrated with indium, lead and tin standards. The samples were disc-shaped with a mass of about 160 mg. The DSC scans were recorded in the temperature interval from 323 to 723 K under Ar atmosphere using a heating rate of $5\ K\ min^{-1}$. Three identical DSC runs were performed for each sample. The second run was used as a baseline, which was then subtracted from the first run to obtain only the irreversible part of the heat flow signal. Within experimental uncertainties, the second and the third scans yielded nearly identical signals.

2.6. Diffusion experiments

The kinetic properties of the interfaces were determined via self-diffusion experiments which were carried out using the ^{63}Ni radioisotope (68 keV β-radiation; half-live 100 years). The samples were sealed in silica tubes in a purified Ar atmosphere following the application of a small amount, about 20 kBq, of water-diluted radioactive solution on the polished specimen's surface. After a given diffusion annealing treatment, the samples were reduced in diameter by at least 1 mm to eliminate the convoluted effects of surface and/or lateral diffusion. The penetration profiles were then determined using a precision parallel grinder. The section thicknesses were calculated by weighing the sample on a microbalance after each grinding step. The specific radioactivity of each section (which is proportional to the tracer concentration) was measured by a liquid scintillation counter (TRI CARB 2910TR).

Further details are similar to those described in Ref. [4]. The experimental uncertainties of the individual diffusion coefficients determined were estimated to be less than 20%.

3. Results and discussion

3.1. Microhardness and calorimetric measurements

The microhardness of as-cast and severely deformed UFG Ni is shown in Fig. 2a as a function of annealing temperature, T. The annealing time, t_a, was set to $t_a = 17$ h in order to introduce changes on a time scale comparable with the typical characteristic annealing time for the subsequent diffusion experiments.

ECAP deformation is seen to result in an almost doubling of the hardness with respect to its value in the initial (as-cast) state and it is three times larger than the hardness of the annealed coarse-grained material. ECAP-deformed