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Original Article

Influence of Mo alloying on the thermal stability and hardness of ultrafine-grained Ni processed by high-pressure torsion☆

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ABSTRACT

The influence of Mo alloying on the thermal stability of grain size, dislocation density and hardness of ultrafine-grained (UFG) Ni alloys was studied. The UFG microstructure in alloys with low (~0.3 at.%) and high (~5 at.%) Mo contents was achieved by high-pressure torsion (HPT) performed for 20 turns at room temperature. The thermal stability of the two alloys was studied by calorimetry. A Curie-transition from ferromagnetic to paramagnetic state was not found for the Ni–5% Mo alloy due to the high Mo content. It was found that heating at a rate of 40 K/min up to ~850 K resulted in a complete recovery and recrystallization of the UFG microstructure in the alloy with 0.3% Mo. The same annealing for Ni–5% Mo led only to a moderate reduction of the dislocation density and the grain size remained in the UFG regime. Therefore, the higher Mo content yielded a much better thermal stability of the Ni alloy. The influence of the change of the microstructure during annealing on the hardness is discussed.

1. Introduction

Ni–Mo alloys possess high hardness and wear resistance which make them useful for various practical applications. For instance, these alloys can be used as hard coating materials [1]. In addition, they exhibit corrosion resistance to the reducing acids, such as hydrogen chloride. Thus, Ni–Mo alloys can be used as catalysts in the production of hydrogen as these acids induce reduction reactions and generally result in hydrogen evolution at cathodic sites [2–4]. These alloys also show high activity and long-term stability as hydrogen evolution reaction

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catalysts under alkaline conditions. They are also applicable as substrate material for superconducting coatings which can be produced by severe cold rolling and subsequent annealing [5]. The latter processing provides extremely sharp cube texture as required for epitaxial coatings.

Improvement in the mechanical properties of Ni–Mo alloys can be attained by the implementation of efficient and novel severe plastic deformation processes, such as equal-channel angular pressing (ECAP), cryorolling or high-pressure torsion (HPT). Over the years, there is an increasing interest in employing SPD techniques for production of ultrafine-grained (UFG) materials [6–11]. HPT is considered as the most effective SPD method in grain refinement and improvement of the strength of metallic materials [12–17]. The enhanced properties of UFG microstructures can be degraded due to grain growth at elevated temperatures [18–22]. The stability of UFG microstructures is an important aspect for their reliable performance in practical applications. Thus, a study of microstructure stability of UFG materials by annealing to high temperatures is crucial.

The addition of alloying elements further influences the development of the UFG microstructures during SPD as well as their thermal stability. Solute atoms have pinning effects on lattice defects, such as dislocations and grain boundaries, therefore they can stabilize the UFG microstructure [23–28]. The influence of concentrations of alloying elements on the thermal stability of face-centered cubic (fcc) metals (e.g., Ni and Cu) processed by SPD has been investigated in the literature [29,30]. However, despite the significant possible applications of Ni–Mo alloys, no investigations have been conducted to date to examine the influence of annealing on the microstructure and properties of HPT-deformed Ni–Mo alloys. Therefore, the present study was initiated to study the influence of heat-treatment on the microstructure and mechanical properties of UFG Ni alloys with two different Mo concentrations. The samples processed by a two-step combination of cryorolling and 20 turns of HPT were annealed to about 850 K and the microstructural parameters were compared with the HPT-processed state. In addition, the variation of the hardness during annealing was examined and correlated to the change of microstructure.

2. Experimental material and procedures

2.1. Processing of UFG Ni–Mo alloys

Two Ni alloys with low and high Mo contents were processed by induction melting and casting into a Cu-mold. The chemical compositions of the as-processed Ni alloys were determined by energy dispersive spectroscopy (EDS) in a scanning electron microscope (SEM). The alloys with ~0.28 and ~5.04 at.% of Mo were labeled as low-Mo and high-Mo, respectively. Although, in addition to Mo, other elements, such as Al (0.84–1.08 at.%), Fe (0.13–0.25 at.%) and Si (0.05–0.34 at.%) were also found in the samples, the major difference between the chemical compositions of the two samples was the much higher Mo content in the material labeled as high-Mo. Detailed chemical compositions for low-Mo and high-Mo alloys were given earlier [31]. The as-cast ingots with diameters of ~32 mm were hot-rolled at 1100 °C to a thickness of ~13 mm. The hot-rolled samples were then subjected to a combination of cryorolling and HPT. First, small strips cut from the hot-rolled materials were processed by cryorolling at liquid nitrogen temperature (LNT). This procedure resulted in a reduction of the thickness from ~13 mm to ~3 mm in multiple passes with a reduction of ~5% per pass. Then, disks with diameters of ~10 mm and thicknesses of ~1 mm were prepared from the cryorolled materials. In the second step of SPD, the samples were processed by the HPT technique under quasi-constrained conditions [32] with an applied pressure of 6.0 GPa and a rotating speed of 1 rpm at RT for 20 turns.

2.2. Differential scanning calorimetry

Our former study [31] revealed that for both alloys the microstructural parameters and the hardness were saturated between the half-radius and the periphery of the disks processed by 20 turns of HPT. Therefore, the thermal stability of the microstructures was investigated in the region between the half radius and periphery of the disks using differential scanning calorimetry (DSC). The DSC experiments were conducted on small samples cut from these regions of the HPT disks by a Perkin Elmer (DSC2) calorimeter at a heating rate of 40 K/min under an Ar atmosphere. Then, the HPT-processed samples were heated up to a characteristic temperature of the DSC thermograms. These specimens are regarded as annealed samples.

2.3. Microstructure from EBSD

The microstructures of the HPT-processed and the annealed Ni alloys were studied by electron backscatter diffraction (EBSD) using an FEI Quanta 3D SEM. Before EBSD, the specimens were mechanically polished first by SiC abrasive papers with 600, 1200, 2500 and 4000 grit and then by a colloidal silica suspension (OP-S) having a particle size of 1 micrometer. After mechanical polishing, the surfaces were treated by electro-polishing at 28 V and 1 A using an electrolyte with a composition of 70% ethanol, 20% glycerine and 10% perchloric acid (in vol.%). The step size in the EBSD study was ~30 nm and the grain sizes were evaluated using Orientation Imaging Microscopy (OIM) software. It is noted that only those regions in the EBSD images which were bounded by high-angle grain boundaries (HAGBs) with misorientations higher than 15° were considered as grains.

The distortions inside the grains were analyzed using Kernel Average Misorientation (KAM) maps prepared by the OIM software. In this evaluation process, a local misorientation angle value was assigned to each pixel, which was determined as the average misorientation between the studied central pixel and all pixels at the perimeter of the kernel around the investigated pixel. The radii of the kernels were ~50 nm for all images even if their pixel sizes were smaller in order to make their KAM maps comparable.