Enhanced tensile ductility and strength of electrodeposited ultrafine-grained nickel with a desired bimodal microstructure

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ABSTRACT

This work aims to use surfactant-assisted direct current electrodeposition technique to prepare four types of bimodal nickel, under different current densities. Bimodal Ni is obtained with different grain size and spatial distribution of CG and UFG areas showing a big disparity in mechanical properties. As a result of small population of coarse-grained surrounded by quite a lot of ultrafine-grained forming a unique shell-and-core bimodal structure, bimodal one present the best comprehensive mechanical properties with an ultrahigh tensile strength (~847 MPa) and a considerable plastic strain (~16.7%). Deformation initial, bimodal structures display more positive strain hardening to meaningful strains than unimodal structure of UFG and CG. Particularly bimodal one work-hardening rate is the highest thanks to its structure (UFG occupy 76.7% in total number fraction) and the distribution of growth twins. Growth twins in this article are referred to $\Sigma3(111)$ coherent twins playing an important role in improving high strength, enhancing uniform plastic deformation ability.

1. Introduction

Scientists and engineers have, for centuries, focused on obtaining materials with both high strength and ductility. This can be attributed to the problem that strength and ductility are mutually exclusive [1–16]. High-strength materials are needed, particularly in addressing current challenges, such as environmental pollution, global warming, and energy crisis. Strong materials reduce the weight of vehicles, thereby improving their energy efficiency. In industrial processes, good ductility is needed to prevent catastrophic failures. Nanocrystalline (NC, $d_{grain} < 100$ nm) and ultrafine-grained (UFG, 100 nm $< d_{grain} < 1$ μm) metal materials exhibit high strength. However, their ductility, particularly the uniform elongation in tension, is quite low and, in most cases, nowhere closes to that of normal metals [17]. This drawback has been an insurmountable hurdle in bringing NC/UFG materials into industrial application.

Previous studies reported that the poor ductility of NC and UFG metal materials primarily result from the restrain of dislocation-mediated mechanism in small grain size, leading to the low strain-hardening capability. As a result, the appearance of early necking can induce the materials' fracture, which can be attributed to the lack of limitation to plastic instability. A variety of strategies, such as the multi- and bimodal microstructures, nanolaminated structure, and gradient nanostructure, aimed at improving the poor ductility of NC and UFG materials have been reported [8,18,19]. Among them, the bimodal grain size distribution microstructure, in which volume fraction of micron-sized grains are introduced into the NC and UFG matrix, is considered as an efficient strategy.

Strength-to-ductility ratio strongly depends on grain size distribution [20–22]. For instance, experimental evidence presented by Wu et al. demonstrated that bimodal grain size distribution could balance increased strength and high ductility in metal materials [18]. One strategy suggested to enhance the ductility of NC/UFG materials is to process a bimodal grain structure, in which the fine grains provide strength and the coarse grains improve ductility. Zhao et al. used cryomilling and, subsequently, quasi-isostatic forging processes (before the famous Ceracon forging was developed) to prepare the bimodal UFG/coarse-grained (CG) Ni. Average grain size of the bimodal Ni was 2.1 μm, yielding at 312 MPa with 49% ductility [7]. Tao Qian et al. prepared the bimodal UFG/CG Ni through severe plastic deformation and annealing technologies. With the increase of the volume fraction of CG, the ultimate tensile strength (UTS) of the specimen decreased from
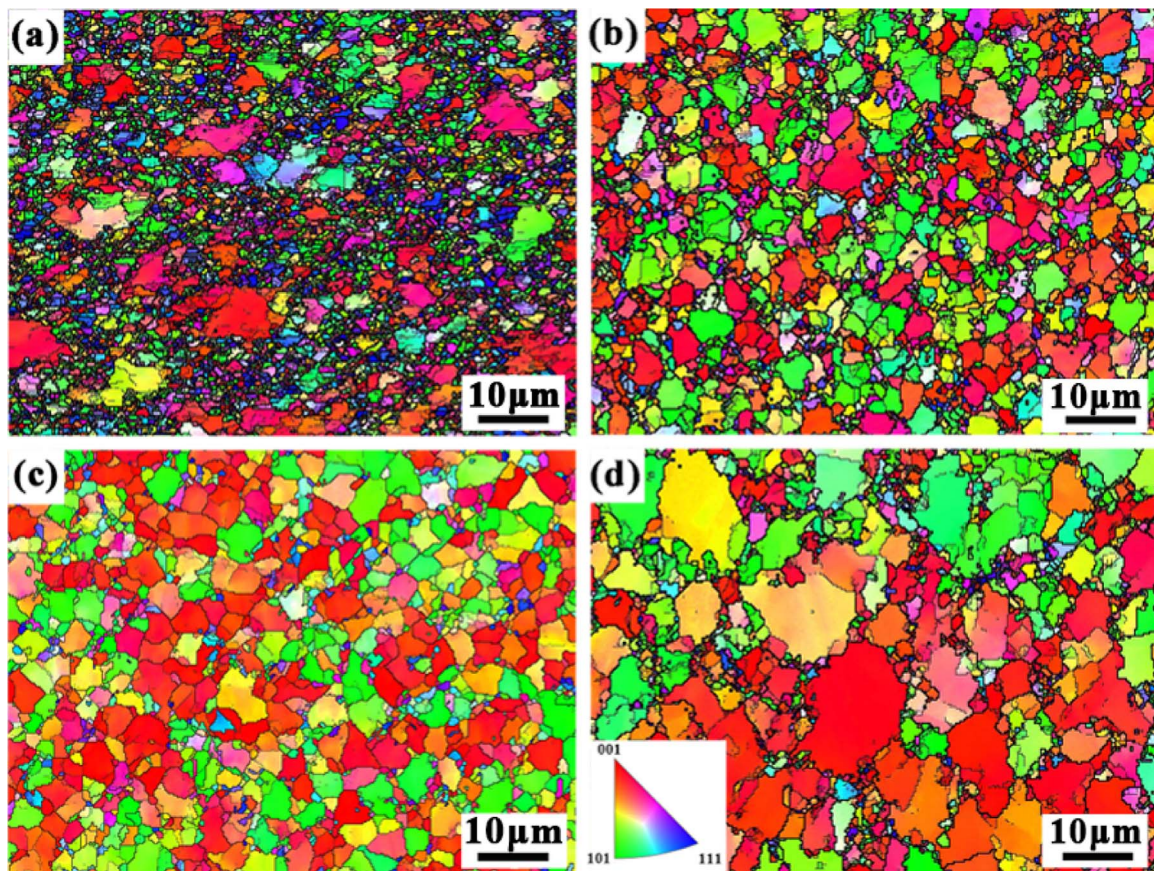
Fig. 1. EBSD images of different samples: (a), (b), (c), (d) show the microstructure of bimodal one, two, three and four.

470 MPa to 380 MPa, and uniform elongation increased from 1.5% to 20% [23]. These results reveal that the plastic instabilities can be overcome by the additional strain-hardening capability of micron-sized grains, increasing the tensile ductility while minimizing loss of strength. However, some problems persist. First, the bimodal grain size distribution microstructure is obtained from two- or multistep processes accompanied by secondary recrystallization, and the annealing process is usually difficult to control because of different influencing parameters. Second, only a narrow area fraction of micron-size grained aggregate was investigated during the heat treatment. Despite enormous efforts in the past decade, quantitative understanding of the grain size, spatial distribution, and the mechanical behaviors of bimodal Ni remains poor. The present investigation is undertaken with the specific objective of preparing high-performance Ni with desired bimodal grain size distribution through a simple method and evaluating microstructural changes occurring during the tensile process. Herein, bimodal Ni with a different grain size distribution has been obtained through direct current electrodeposition in an improved traditional electroplating bath. The microstructure, mechanical properties, and deformation mechanism of the as-synthesized samples are investigated and discussed.

2. Experimental

2.1. Preparation of the bimodal Ni

The dense and bulk bimodal Ni was synthesized through direct current electrodeposition, with a focus on adjusting the current density and the type of additives. All reagents used were analytical grade, and deionized water was utilized to prepare the plating bath. The electrolyte consisted of $NiSO_4.6H_2O$ (208 g/L), $NiCl_2 \cdot 6H_2O$ (30 g/L), H_3BO_3 (30 g/L), and a small amount of sodium dodecyl benzene sulfonate. The

pH of the solution was adjusted to 4.0–4.2 through diluted hydrochloric acid, and the temperature was controlled at 55 ℃. To eliminate the effect of impurities further, the electrolyte was first purified at a low current density of 0.1 A/dm^2 for two days. The bulk Ni with a thickness of approximately 0.28 mm was synthesized on the austenitic stainless steel sheets (40 mm × 50 mm × 1 mm) under the direct current density ranging from 0.35 A/dm^2 to 1.12 A/dm^2. Then, all samples were peeled off from the stainless steel substrate.

2.2. Microstructure characterizations

The crystallographic structure of the as-deposited bulk Ni samples was investigated through X-ray diffraction (XRD, D/max 2500PC, Co-Kα radiation, λ = 0.179 nm) with a step of 0.05˚. Microstructure and grain size distribution of the specimens were characterized under an electron back scatter diffraction (EBSD) using a TSL OIM system on a Philips XL30 FEG SEM with step sizes of 0.25 μm. The EBSD samples were vibratory polished using diamond suspension, and a final particle size of 0.2 μm was achieved, then electropolished through an etchant of 30 vol% nitric acid and 70 vol% ethanol for several seconds at 20 V and room temperature.

2.3. Mechanical property tests

Mechanical property tests were composed of tensile tests and Vickers hardness. The dog-bone-shaped specimens with a gauge cross-section of 2.5 mm × 0.28 mm and a gauge length of 14.0 mm were cut from the as-deposited bimodal bulk Ni sheet using a wire-cut electrical discharge machine, and then the surface was polished to a mirror-like finish. The tensile tests were carried out on an Instron 5582 testing machine at a strain rate of $1.2 \times 10^{-4} s^{-1}$. Moreover, three identical specimens were measured to ensure sufficient experimental data are