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The mechanical behavior of a layered nanostructured Ni with an alternating growth of ultrafine grains and nano-sized grains fabricated by electrodeposition

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ABSTRACT

A layered nanostructured (NS) Ni with an alternating growth from the ultrafine-grains (UFG) layer with a thickness of 5 μm and the nano-sized grains (NG) layer with a thickness of 10 μm along the across-section is fabricated by periodically controlling the current density during electrodeposition. It is revealed by transmission electron microscope (TEM) and X-ray diffractometer (XRD) that the alternating growth of well-defined layers consists of the ultrafine-grains with the mean grain size of about 500 nm along a (200) preferential growth and the nano-grains with the mean size of 20 nm with a random growth. Tensile tests reveal that in comparison with the monolithic UFG and NG Ni, the layered NS Ni exhibits a higher tensile ductility of near 13% elongation to failure while maintaining a moderate ultimate tensile strength of near 1300 MPa. The improvement in the ductility for the layered NS Ni can be mainly attributed to two effects including the increased strain hardening ability sourced from the action of these specific microstructure involving the periodical distribution of ultrafine-grains, the existence of interface and the alternation of crystal orientation between the layers on the dislocation activities and alleviating the local stress concentration caused by the periodic coordination deformation of the UFG layer in the NG substrate. A larger homogeneous surface deformation trace with the uplift and collapse feature is observed for the layered NG Ni in comparison with the monolithic UFG and NG Ni, which further indicates a large uniform plastic deformation ability that the layered NG Ni holds. Additionally, the mutual restraint and coordination deformation between the UFG and NG layer also leads to the formation of a fracture feature with a smaller and uniform dimple structure.

1. Introduction

Nanostructured (NS) metals attracted considerable attentions over the past few decades due that their superior mechanical properties like the ultrahigh strength and hardness offer potential structural application [1–5]. However, the potential application of this new class of materials is limited due to their low ductility [5–10]. The low ductility of NS metals can be attributed to the intrinsic low strain hardening capacity sourced from the insufficient dislocation activities in the tiny grains [11–13]. Some strategies promoting the strain hardening ability mainly including tailoring an inhomogeneous microstructure with a bi/multimodal grain size distribution, introducing the twin structure or the second phase particles in a NS matrix, controlling the grain boundary character and changing the deformation conditions such as at low temperatures or high strain rates are attempted to enhance the ductility of NS metals in recent years [14–19]. Among these strategies, tailoring a bi/multimodal grain size distribution is the most potential method that could be applied to the practical engineering because its applicability is independent on the composition of the metals and the implementation condition is not harsh. An typical case of improving the ductility of NS metals by tailoring a bimodal or multimodal grain size distribution is the NS Cu earlier reported by Wang et al. [14], where an inhomogeneous microstructure of the micrometer-sized grains embedded inside a ultrafine-grained (UFG) matrix is created by the severe plastic deformed (SPD) followed by the thermo-mechanical treatment. It is believed that the small ultrafine grains retain the superior strength and the large micrometer-sized grains provide a necessary strain hardening capacity to stabilize the tensile deformation of material. Subsequently, similar technology is also applied to tackle the plastic instability of other metals [20–24]. Although these studies illuminated

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that the thermo-mechanical treatment can improve the ductility of NS metals, but this technology in some way only deal with soft and easily deformed metals. Furthermore, the typical mean grain size of the microstructure produced through this method is generally in a magnitude of near micrometers (i.e., 0.5–1 μm), which limits the application of materials in practical engineering.

Electrodeposition technique is not only suitable for manufacturing a wide variety of materials but also can adjust the grain size of material in a wide range by adjusting some simple electroplating parameters and has been a widely used process for preparing NS metals with a homogeneous structure [25–34]. Recently, several researchers began to try to tailor the inhomogeneous microstructure with a bimodal or multimodal grain size distribution in a small nanometer-sized range (< 0.5 μm) by using this technique to solve the low ductility of NS metals [35–38]. Gu et al. [35] fabricated an NS Ni with an inhomogeneous microstructure composed of 150–300 nm grains cluster surrounded by nano-grains of 10–30 nm, which shows the enhanced plastic strain of about 5–7%. Wang et al. [36] fabricated an NS Cu with a broad grain size distribution from 10 nm to 290 nm, which produced a nearly perfect plasticity with a large strain of close to 20%. Other studies on the microstructure with a bi/multi-modal grain size distribution constructed by the electrodeposition that contributes to a good ductility of NS metals also comes from the NS Nis with a broad grain size distribution reported by Shen et al. [37] and Xu et al. [38]. The above researchers all successfully construct an inhomogeneous microstructure with a bimodal or broad grain size distribution in the nanometer scale by the electrodeposition to improve the ductility of NS metals, but they don't provide how to construct such an inhomogeneous structure during the electrodeposition. Thus, these studies still do not provide a valuable guidance in optimizing the mechanical property of NS metals. Recently, Gu et al. [39] controllably fabricated a layered NS Ni with alternative UFG layer and NG layer by periodically adding the grain refinement agent according to the consumption cycle of additives during electrodeposition. By this method, the grain size of NS Ni is periodically tailored along the cross-section of the specimen, but the thickness of each layer reaches hundreds of micrometers due to the slow consumption rate of the grain refinement agent during the electrodeposition, which leads to the loss of the mechanism that the large-sized grains and the small-sized grains are coordinated to maintain the plasticity each other. Additionally, the application of additives that is used to tailor the microstructure also will produce extra byproduct, which is also not benefit to attain a high quality NS sample. As a result, the ductility of such a layered NS Ni fabricated by Gu et al. is not shown to be evidently increased. Thus, to truly optimize the mechanical property of NS metals, there is a need to find a flexible method that not only tailors the grain size but also controls the distribution and the proportion of the grains.

In our previous study, it has been shown that a high current density applied in the process of electrodeposition can produce a more small nanocrystal [34]. Thus, a method based on periodically changing the current density during the electrodeposition is designed to try to realize the flexible control of microstructure of NS metals in this paper. By such a method, a bulk layered NS Ni where not only the grain sizes for each layer but also the distribution and the proportion of grains are controllable by periodically adjusting deposition parameters is fabricated. The mechanical properties of this layered NS Ni are studied and compared with the counterpart with the homogeneous structure.

2. Experimental procedure

A bulk and compact layered NS Ni sheet with the microstructure composed of alternating UFG layer and NG layers was made by intermittently adjusting the current density at a direct current electrodeposition process from the sulfate bath containing nickel sulfate, nickel chloride, boric acid and a small amount of additives including saccharine and 1,4-butenediol at pH 5.0 and a temperature of 50 °C. Here two kinds of current densities, i.e., 1.5 A/dm^2 for 1 h and 20 A/dm^2 for 2 min, are intermittently used to modulate the microstructure, by which the thickness ratio of UFG layer and NG layer is controllably set to a fixed value. For comparison, two corresponding monolithic UFG and NG Ni with the homogeneous structure are also prepared under the above two current density. Chemical analysis by the inductively coupled plasma atomic emission spectrometry (ICP-AES, Plasma /1000) and Carbon/Sulfur determinators (CS-200) showed that three deposited NS Ni sheets all have a similar high purity of about 99.9 wt% with the main impurities of 130 ppm S, 410 ppm C, 330 ppm Co and 102 ppm B. These impurities are introduced from the used additives and chemicals. The cross-sectional morphology of layer NS Ni specimens was characterized by using the scanning electron microscope (SEM, JSM-5600). The cross-section of the layer NS Ni was eroded in a metallographic etchant which consists of 10 mL distilled water, 38 mL nitric acid, 100 mL glacial acetic acid for 30 s before SEM characterization. Microstructure and grain size and selected-area electron diffraction along the cross-section of layer NS Ni specimens were investigated by transmission electron microscope (TEM, a JEM-2100F). The thin specimen with the thickness of about 100 nm for TEM observation was cut by Tescan GAIA FIB-SEM with a Ga ion source at an accelerated voltage of 30 kV accompanied by eventually a polishing process at a voltage of 5 kV. The crystallographic structure of the layer NS Ni and that of the counterpart with the homogeneous structure also were performed by using the X-ray diffractometer (XRD, Bruker D8) with a Cu Kα radiation (0.154178 nm) operating at 40 kV and 40 mA over the 2θ range 5–90°. The hardness test was done along the eroded cross-section of the layer NS Ni sheet and on the surface of the monolithic UFG and NG Ni sheet by using an HXD-1000 micro-hardness machine with a Vickers indenter, at a load of 10 g and duration of 15 s. The tensile tests were conducted on a tensile testing machine (MTS Landmark 370.10) at a strain rate of $4.17 \times 10^{-2}\,s^{-1}$ at room temperature (RT). For tensile test, the dog-bone shaped tensile specimens with a gauge length of 8.0 mm and cross-section of $2.0 \times 0.3\,mm^2$ are machined from the as-deposited NS Ni plates by using the wire electrical discharge machining and then were polished to a mirror-like finish surface to avoid the influence of external defects (such as pits and cracks) to the intrinsic tensile properties. Additionally, the stress relaxation tests were also carried out to explore the asymmetric interior stress cumulated in three deformed NS Nis. The stress relaxation testing process was also described in our recent work [33]. Here it should be noticed that in order to avoid the difference of the experimental results caused by one single sample, three parallel specimens were adopted for the above each mechanical experiment. The morphology of the indentation of the layered NS Ni and the fracture surface morphology of the layered NS Ni and the counterpart with the homogeneous structure were also observed by SEM.

3. Result and discussion

Fig. 1 shows the cross-sectional SEM image of the layered NS Ni. It can be clearly seen that the cross-section of the layered NS Ni presents a surface with a well-defined layered structure composed of the smooth area with the thickness of about 5 μm and the rough one with the thickness of near 10 μm alternately. The appearance of such a cross-sectional morphology should be related to the alternation of corrosion resistance of the cross-section of layered NS Ni along the deposition direction. Recently, it has been illuminated that the corrosion resistance of NS Ni is obviously improved with the decrease of the grain size [40]. That is, such a cross-sectional feature reflects essentially the microscopic variation that the grain size of as-deposited sample alternately changes along the deposition direction. According to the above analysis, it can easily be concluded that the relative smooth area should belongs to the small grain sized layer and the rough one corresponds to the large grain sized layer. The refinement of grain size usually leads to the increase in the hardness of material. So, here the hardness test performed on the smooth area and the rough one along the cross-