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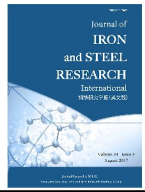

Evolution of microstructure, mechanical and magnetic properties of electrodeposited 50%Ni-Fe alloy foil after thermal treatment

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ABSTRACT

In order to expand the application of the electrodeposited Ni-Fe alloy foil, their mechanical and magnetic properties were studied after heat treatment. The development of grain growth during annealing was in-situ online investigated using a heating stage microscope, and the texture was analyzed via X-ray diffraction (XRD) and electron back-scattered diffraction (EBSD). The results indicated that abnormal grain growth usually occurred during annealing at 1000-1050 °C. The {111} oriented grains preferentially grew as the annealing temperature and holding time increased. The plasticities of the electrodeposited Ni-Fe alloy foils after heat treatment were better than those of the original samples. The excellent ductility was obtained without a loss in magnetic properties after annealing at 1100 °C for 6 h.

1. Introduction

With the rapid development of the maglev train, superconductive energy storage and precision electronics technologies, the demand for thin and light magnetic shielding materials is growing quickly. The 50% Ni-Fe alloy foil is an excellent soft magnetic material and has the superior magnetic shielding performance. Up to now, the methods of manufacturing Ni-Fe alloy foil mainly include casting and rolling method, quenching method and electrodeposition method. The casting and rolling is complex in procedures and high in energy consumption and production cost. The quenching can prepare amorphous foil with good performance, but the small width restricts its product specification, which limits its application. The electrodeposition is well known as a technological and economical method producing large scale and high purity nanocrystalline metals in one step, and the cost reduces as the thickness decreases. Therefore, the electrodeposition method has been attracting more and more attention. However, the achievement of these extraordinary properties is usually accompanied by a sacrifice in ductility[1-3] because the nature of nanoscale structures[4] restricts the generation and storage of dislocations inside grains, which limits the practical utility of these materials. Therefore, one challenge is to design new microstructures to achieve improved excellent soft magnetic properties without a loss in ductility.

Fortunately, it is possible to mitigate these problems by controlling the microstructure and texture of the material through heat treatment. Most of studies[5-11] solely focused on controlling the microstructure of nanocrystalline metals in nano-scale region. For example, Hou et al. [8] detailed the structure and mechanical properties of electrodeposited Ni-W alloy coatings after annealing at 500 °C. Specht et al. [11] investigated the stability of nanocrystalline Cr-Fe-Ni films after annealing at 200-800 °C. However, an improvement in magnetic and ductility properties can also be achieved by increasing average grain size in micro-scale region[12]. There have been few researches of thin electrodeposited magnetic films and foils on increasing grain size from nano-scale to micro-scale level through high temperature heat treatment.

Electrodeposited nanocrystalline material does not undergo rolling deformation and has no rolling strain, so the development of recrystallization of electrodeposited nanocrystalline metals differs from that of traditional cast-rolling metals. In this study, the primary goal was to improve the ductility with-

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out a great loss in magnetic properties. The evolution in microstructure and texture of the electrodeposited Ni-Fe alloy foil was also studied after high-temperature heat treatment. The results presented in this work will provide valuable insights for future researchers to expand the potential applications of Ni-Fe alloy foil.

2. Experimental Procedure

The experimental electrodeposited Ni-Fe alloy foil was supplied by an industrial enterprise in China. The foil dimensions were 50 μm in thickness, and 30 cm in width. Its chemical composition was Ni 50.03 wt.%, Mn 0.015 wt.%, Al$<$0.005 wt.%, Ti$<$0.005 wt.%, P$<$0.005 wt.%, S 0.042 wt.%, N 0.0042 wt.%, and Fe balance.

The first group of experiments was used to examine the effects of annealing temperature and holding time on the microstructures and properties of alloy foils. The annealing equipment is a tube furnace equipped with a vacuum system. Hydrogen gas was introduced at a flow rate of 200 mL · min^{-1} in order to purify the electrodeposited Ni-Fe alloy foil. The alloy foils were heat treated at the designed temperatures (900, 950, 1000, 1050 and 1100 °C) for 2, 4 and 6 h respectively at a heating rate of 5 °C · min^{-1}, and then the specimens were cooled to 500 °C at a rate of 5 °C · min^{-1}. Finally, the specimens were kept in the tube until reaching ambient temperature.

In order to further understand the evolution mechanism of microstructure, the second group of experiment was used to study the evolution of grain microstructure by in-situ online observation. The annealing equipment is a heating stage equipped with a high temperature thermo-stage microscope. The electrodeposited Ni-Fe alloy foil with a size of 5 mm×5 mm was heated to 1150 °C at a rate of 5 °C · min^{-1}.

The magnetic hysteresis loops of the alloy foils were measured with a soft magnetic direct current standard measuring device (NIM-2000S), and the mechanical properties of the alloy foils were tested with an electronic universal testing machine (WDW 3020). The micromorphology of heat-treated alloy foils was observed by optical microscopy and scanning electron microscopy (SEM, MLA-250). The crystal structure was analyzed via X-ray diffraction (XRD, MXP21-VAHF). Micro-orientation analysis was conducted using electron backscatter diffraction (EBSD, BRUCKER) with the associated equipment on the scanning electron microscope. The grain size of original electrodeposited Ni-Fe alloy foil was calculated by Scherrer formula:

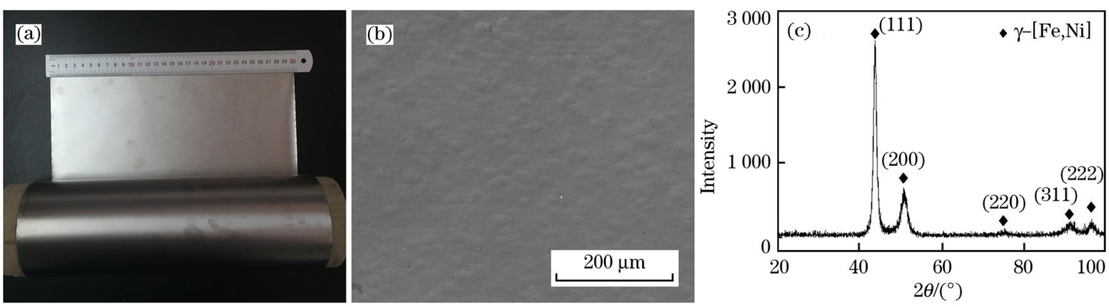
$$D = K\lambda / \beta\cos\theta \tag{1}$$

where, D is the grain size; λ is the X-ray wavelength, nm; β is the peak width of the diffraction peak profile at half maximum height resulting from small crystallite size in radians; θ is the diffraction angle; and K is a constant related to crystallite shape, normally taken as 0.9.

3. Results and Discussion

3.1. *Effect of heat treatment on microstructure*

Figs. 1(a) and 1(b) show the surface macroscopic and microscopic morphologies of the original electrodeposited Ni-Fe alloy foils, respectively. Fig. 1(c) shows the XRD patterns of the alloy foils. As compared with standard cards, the sample manifested γ-[Fe,Ni]. The diffraction peak was wide, which indicated that the grain size was very small. From the well-known Scherrer formula, the crystallite size was about 10 nm.



(a) Macro photo; (b) Surface microstructure; (c) XRD pattern.

Fig. 1. Original electrodeposited Ni-Fe alloy foil.

In order to examine the influence of heat treatment on the microstructure, the samples were heated at 900, 1000, and 1100 °C for 2−6 h. By comparison and analysis, the alloy foil had a fine-grain microstructure, mixed-grain microstructure, and coarse-grain microstructure as annealing temperature increased, as shown in Fig. 2. The grains were the finest and most uniform after annealing at 900 °C for 2 h, as shown in Fig. 2(a). When the holding time was prolonged to 6 h, abnormal grain growth was observed on the surface of the alloy foil and the maximum grain size (D_{max}) was 25 μm, as shown in Fig. 2(c). The number of abnormal grain was small, and the grain size was fine in the most of area after an-