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Crystallization and magnetic properties of $Fe_{40}Ni_{38}B_{18}Mo_4$ amorphous alloy

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Abstract

The crystallization behavior of heat treated amorphous $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy was studied by TEM in order to investigate the suitability of this material as a soft nanostructured magnetic alloy. Significantly, the phases observed during primary and secondary crystallization were nanosized Fe–Ni solid solution and $(FeNiMo)_{23}B_6$, respectively. The primary crystals had an unusual irregular morphology due to the presence of molybdenum. A twinned structure was observed by nanobeam diffraction within the Fe–Ni nanocrystals. The phase transformation mechanism was compared with that of $Fe_{40}Ni_{40}B_{20}$ and $Fe_{40}Ni_{40}P_{14}B_6$ alloys and the differences were analyzed from a thermodynamic point of view. The magnetic properties, M_s and H_c, were studied by VSM, it was found that improved soft magnetic properties were achieved by heat treatment. A mean crystal size of 10 nm and a volume fraction of 1% was found to be the microstructure which would yield the lowest coercivity.

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1. Introduction

Nanocrystalline materials are materials possessing grain sizes on the order of a billionth of a meter. They manifest extremely fascinating and useful properties, which can be exploited for a variety of structural and non-structural applications [1]. One family of nanocrystalline soft magnetic alloys were developed by Yoshizawa and coworkers and is now known as FINEMET ($Fe_{73.5}Si_{13.5}Nb_3B_9Cu_1$) [2] An explanation of the magnetic behavior of such alloys was provided by Herzer [3]. NANOPERM ($Fe_{88}Zr_7B_4Cu_1$) and HITPERM ($Fe_{44}Co_{44}Zr_7B_4Cu_1$) are other soft magnetic nanomaterials that are being developed for commercial applications [4,5]. The microstructure of nanocrystalline soft magnetic alloys consists of a nanocrystalline ferromagnetic phase surrounded by an amorphous ferromagnetic matrix, this microstructure may be produced by means of heat treatment of the amorphous precursor. A study of microstructural evolution of nanomagnetic materials can provide guidance to tailoring the material for an increased range of magnetic properties.

Crystallization can occur during heat treatment of amorphous alloys provided atomic diffusion is sufficient for nucleation and growth. The driving force for crystallization is the free energy difference between the glass and appropriate crystalline phase, the crystallization temperature is usually close to the glass transition temperature. A variety of equilibrium and metastable crystal structures and morphologies have lower energy than the amorphous structure, they compete as possible crystallization products, which cause different crystallization reactions, mechanisms and final microstructures in different alloy systems. There are typically three kinds of crystallization mechanisms: polymorphic, eutectic and primary crystallization [6].

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The iron-nickel based soft magnetic alloy, $Fe_{40}Ni_{38}B_{18}Mo_4$, was examined in this investigation. This alloy boasts excellent soft magnetic properties and the melt spun amorphous alloy is a commercial product, it is used commercially in the amorphous form. The microstructural evolution as a result of heat treatment and the magnetic properties were examined; the variation in magnetic properties with controlled partial nanocrystallization was also studied. Such crystallization can give rise to nanocrystals in an amorphous matrix, satisfying the requirement of the Herzer model [3].

The crystallization behavior of $Fe_{40}Ni_{40}B_{20}$ and $Fe_{40}Ni_{40}P_{14}B_6$ melt spun amorphous alloys has been extensively studied, but their crystallization behavior differs significantly from the $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy examined in the present investigation. Previous work on the $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy has been reported by Li et al. [7], Nunes et al. [8] and Biasi and D'almeida [9], but the structure of the crystals were studied mainly by the XRD technique, further, the conclusions drawn from their XRD results are not entirely consistent with each other. In the present study, DSC, XRD, TEM and nanobeam diffraction analysis was used, the magnetic properties were also studied by VSM.

2. Experimental procedure

Ribbons of the iron-based amorphous alloy $Fe_{40}Ni_{38}B_{18}Mo_4$ with a thickness of 30 μm were produced by melt spinning, the melt spun samples were kindly supplied by Honeywell Electronics, USA. Isothermal heat treatments were carried out in a vacuum furnace at temperatures of 350 °C, 380 °C, 400 °C, 420 °C and 450 °C for 10 min, 1 h, 4 h and 24 h. These temperatures were selected on the basis of the previously reported DSC results [10]. XRD was carried out on the heat treated samples to study the product of heat treatment. Crystals formed in the amorphous matrix of the heat treated samples, the structure of the crystals were carefully studied by bright field (BF), select area diffraction pattern (SADP) and nanobeam diffraction pattern (NBD) analysis using a transmission electron microscope. A JOEL 2010 Transmission Electron Microscope with an accelerating voltage of 200 kV was employed. The melt spun TEM sample was cut to a small disk by a punch, followed by ion milling. The magnetic properties were studied by vibrating sample magnetometer, a Lake Shore Vibrating Sample Magnetometer (VSM) Model 736 was employed to study magnetic properties.

3. Results

The DSC results under different heating rates are shown in Fig. 1 for the $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy. Two

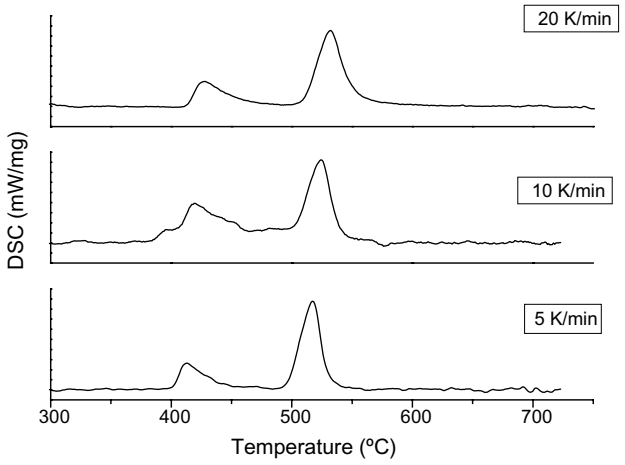

Fig. 1. DSC measurement curves of the $Fe_{40}Ni_{38}B_{18}Mo_4$ samples heated linearly at different heating rates: (a) 5 K/min, (b) 10 K/min and (c) 20 K/min.

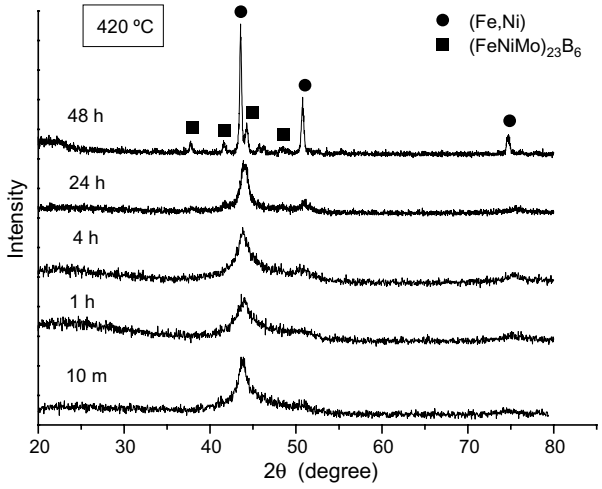

Fig. 2. XRD results for the $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy heat treated at 420 °C for 10 min, 1 h, 4 h, 24 h and 48 h.

main peaks at the position of 420 °C and 530 °C can be observed in the DSC results, which indicates that there are two phase transformations. Fig. 2 shows the XRD results of the heat treated alloy at 420 °C for different annealing time. The Fe–Ni phase is found to be the primary crystallization product, the $(FeNiMo)_{23}B_6$ phase formed after 48 h of annealing.

The heat treated samples were studied by TEM, Figs. 3–5 show the bright field and select area diffraction pattern of the melt spun alloy heat treated at 350 °C, 400 °C and 450 °C for 10 min, 1 h and 24 h respectively. Numerous fine irregular shape crystals within an amorphous matrix can be observed in the heat treated samples, the crystal size of the sample heat treated for 1 h is generally larger than that heat treated for 10 min but not much different from the sample heat treated for 24 h. The continuous lattice fringes observed from