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# Studies of crystallization and soft magnetic properties of FeNiMoB(Si) alloys

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**Abstract**

Crystallization behaviors of $Fe_{40}Ni_{38}Mo_4B_{18}$, $Fe_{38}Ni_{35}Mo_4Si_5B_{18}$, and $Fe_{39}Ni_{36}Mo_2Si_5B_{18}$ (at%) amorphous alloys as well as magnetic properties of the $Fe_{40}Ni_{38}Mo_4B_{18}$ alloy, annealed in the temperature range from 400 to 520 °C for 1 h, have been investigated. The results show that for the samples of $Fe_{40}Ni_{38}Mo_4B_{18}$, annealed in the temperature range of 430–450 °C, a single-crystalline FCC phase with composition near $Ni_3Fe$ solid solution and grain size of about 10 nm precipitates in the amorphous matrix, and the samples exhibit good soft magnetic properties. As the samples are annealed at $T_a \geqslant 470$ °C, the magnetic properties drop rapidly, this is mainly due to the formation of Fe–B and Ni–B compounds. For the samples of $Fe_{38}Ni_{35}Mo_4Si_5B_{18}$ and $Fe_{39}Ni_{36}Mo_2Si_5B_{18}$, a single FCC crystalline phase structure cannot be formed in the amorphous matrix, where more than single-crystalline phase such as Fe–B compounds as well as γ-(Fe, Ni) phase are precipitated almost simultaneously by the annealing treatment.
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## 1. Introduction

Nanocrystalline soft magnetic materials produced by a partial devitrification of Fe-based amorphous metallic alloys have been widely investigated because of their fundamental scientific interest as well as excellent soft magnetic properties since they were observed by Yoshizawa et al. [1] in the alloy with composition $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$. Later on Suzuki et al. [2] developed a new kind of alloy Fe–Zr–B nanocrystalline material with higher magnetic flux density in 1990. After that, hundreds of papers have been published, concerning with the structure and magnetic properties of these nanostructured soft magnetic materials [3–7]. However, almost all of these studies concentrated on the Fe-based Fe–Cu–M–Si–B (M = Nb, Mo, V, W, Ta) and Fe–M–B (M = Zr, Nb, Hf) alloy systems, because it is difficult to find out a proper composition in Ni- or Co-based amorphous alloys in which a single-crystalline phase with nanometer grain size and large volume fraction can be formed by partial devitrification. It is hopeful to develop a new kind of Ni-based or (Ni, Fe)-based nanocrytalline soft magnetic alloy with good quality, since the traditional excellent soft magnetic crystalline material is the Supermalloy with composition 79Ni–5Mo–Fe. Hasegawa et al. [8] have studied the crystallization and magnetic properties of $Fe_{82-x-y}Ni_xMo_yB_{18-z}Si_z$ $(0<x<12,\ 0<y<6,\ 0<z<6)$ amorphous alloy system. They found that the star-shaped dendritic BCC Fe crystals with sizes of 100–300 nm started to precipitate around 380–400 °C and the volume fraction of the Fe crystal was less than 1%. It means that any

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nanocrystalline materials with grain size of about 10 nm cannot be found in this composition range. The formation of nanocrystalline structure in amorphous alloys $Ni_{58.5}Mo_{31.5}B_{10}$ and $Ni_{70}Mo_{10}B_{20}$ was studied by Abrosimova et al. [9]. They found that the crystallization of the first alloy follows the primary cystallization mechanism and the primary phase is FCC-Ni crystals with the grain sizes about 10–30 nm. The second alloy crystallized according to the eutectic crystallization mechanism, and nanocrystalline structure was not observed in this alloy. However, the amorphous matrix of these two alloys is paramagnetic, any soft magnetic material could not be developed on it.

The object of this paper is to develop a new kind of soft nanocrystalline magnetic materials in the (Ni, Fe)-based alloy by experiments. In the design of the research experiments, we keep in mind the two principles as follows: (1) since the Curie points of the Ni–Fe amorphous alloys with very high Ni-content ($>60$ at%) are lower than room temperature [10], the new alloy must be a (Ni, Fe)-based alloy without high Ni content. (2) In the light of developing a soft magnetic material, the new (Ni, Fe)-based alloy should have a low magnetic anisotropy; therefore, the structure of the crystalline phase produced by crystallization in the amorphous matrix should be cubic symmetric, BCC or FCC structure. It is well known that one of the important condition for the Fe-based nanocrystalline alloy (as mentioned above) to obtain excellent soft magnetic properties is the formation of a single-crystalline phase with BCC structure. If the annealing temperature is higher than 600 °C, the formation of tetragonal structure compound $Fe_2B$ with big magnetocrystalline anisotropy detracts the soft magnetic properties [11,12].

Under the guidance of the two principles mentioned above, the crystallization behavior of some (Fe, Ni)-based amorphous alloys was studied in the preceding stage of our work. The composition of these alloys was $Fe_{75-x}Ni_xNb_3Si_9B_{13}$($x = 35$, 45, 55, 58). The results showed that we could not obtain a single-crystalline phase in the Fe–Ni–Nb–Si–B amorphous matrix at any annealing temperature, whereas many phases such as γ-(Fe, Ni) solid solution, $(Fe, Ni)_{23}$ $B_6$ and $Ni_{31}Si_{12}$ crystals precipitated almost simultaneously [13] and the permeability decreased substantially after crystallization.

In the present work, the crystallization behavior and magnetic properties of new designed amorphous alloys have been investigated. The compositions of these alloys are $Fe_{40}Ni_{38}Mo_4B_{18}$ (No. 1), $Fe_{38}Ni_{35}Mo_4Si_5B_{18}$ (No. 2), and $Fe_{39}Ni_{36}Mo_2Si_5B_{18}$ (No. 3).

## 2. Experimental

Amorphous ribbons with 5 mm width and 25 μm thickness were prepared by single roller rapid quenching method. The crystallization temperature $T_x$ was measured by DSC under heating rate of 10 °C/min. Amorphous ribbons and toroid samples were annealed at 400–520 °C for 1 h in the argon atmosphere. The structure of the samples was measured in X-ray diffratometer (D/max-γ A) with copper radiation. The magnetic properties of the toroidal samples were measured with CD-4 type magnetic parameter measuring equipment.

## 3. Experimental results and analyses

Fig. 1 shows the DSC curves of $Fe_{40}Ni_{38}Mo_4B_{18}$ (No. 1), $Fe_{38}Ni_{35}Mo_4Si_5B_{18}$ (No. 2), and $Fe_{39}Ni_{36}Mo_2Si_5B_{18}$ (No. 3) amorphous alloys. It can be seen that there are two separated exothermic peaks in the DSC curve of sample No. 1, $T_{x1} = 435\,°C$, $T_{x2} = 505\,°C$. This curve is similar to that of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy [14], where $T_{x1} = 505\,°C$, $T_{x2} = 630\,°C$. In the DSC curve of sample No. 2, only one sharp exothermic peak appears, $T_x = 522\,°C$. For the sample No. 3, there are two overlapped peaks, the left one is very high and sharp and the right one is low and blunt, $T_{x1} = 505\,°C$, $T_{x2} = 520\,°C$ (at top position).

Fig. 2 is the XRD spectra of amorphous $Fe_{40}Ni_{38}Mo_4B_{18}$ alloy annealed at different temperatures. It shows that, at the $2\theta = 40$–$50°$, there is a diffused diffraction peak, and shows that the alloy annealed at 400 °C is still in the amorphous state. In the spectrum for $T_a = 430\,°C$, an FCC phase of the diffraction peaks appears. The Miller indexes of these peaks, from left to right, are (1 1 0), (2 0 0), (2 2 0), and (3 1 1). As the increase of the annealing temperature $T_a$, the intensity of the diffraction peaks enhances gradually, this indicates that the volume fraction of the crystalline phase increases with the increasing of $T_a$.

The grain size $D$ of the crystalline phase in the samples annealed at different temperatures was estimated by using the Scherrer formula according to the width of (1 1 0) peak. The values of $D$ in the samples annealed at 430, 450, 470, and 520 °C are 8.6, 9.6, 10.1, and 13.7 nm, respectively. It is illustrated in the XRD spectra that a single-nanocrystalline phase with an FCC structure is formed in the amorphous matrix after the samples are annealed for an hour at 430

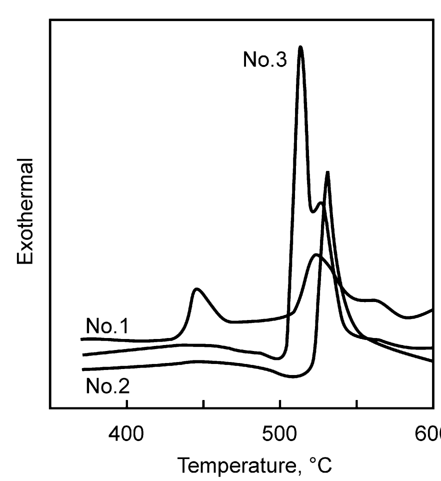


Fig. 1. DSC curves of the $Fe_{40}Ni_{38}Mo_4B_{18}$ (No. 1), $Fe_{38}Ni_{35}Mo_4Si_5B_{18}$ (No. 2), and $Fe_{39}Ni_{36}Mo_2Si_5B_{18}$ (No. 3) alloys with a heating rate as 10 K/min.

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