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Effect of Ni on the microstructure and precipitate phases of $Fe_{78}Si_9B_{13}$ glassy alloy

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ABSTRACT

The $(Fe_{0.78}Si_{0.09}B_{0.13})_{100-x}Ni_x$ glassy alloys (x = 0, 2, 3 and 5) have been investigated by X-ray diffraction (XRD), differential scanning calorimetry (DSC), Vickers microhardness test, transmission electron microscopy (TEM) and vibrating sample magnetometer (VSM). The experimental results show that the addition of Ni has not only promoted the growth of α-Fe phase and the precipitate of Fe–B compounds but also facilitated the dissolution of silicon in the α-Fe phase. For the as-spun ribbon with x = 5, the microhardness (H_v) of the ribbon with super heating (SH) treatment is lower than that of the ribbon without SH; and the H_v of the free side is lower than that of the wheel side of the same ribbon. The saturation magnetization (M_s) of the as-spun $(Fe_{0.78}Si_{0.09}B_{0.13})_{95}Ni_5$ ribbon with SH is higher than that of the ribbon without SH. The variation of the H_v and M_s is explained by the free volume model which is related to the two structure model in Fe-based glassy alloys, as well as the change of the B content in the amorphous matrix.

1. Introduction

As the first and major amorphous alloys widely utilized in industry, $Fe_{78}Si_9B_{13}$ glassy alloy attracts great interest because of its low cost, good mechanical and soft-magnetic properties [1]. In the last three decades, people have tried adding transition metal and metalloid element into Fe–Si–B system in order to improve the properties of the Fe–Si–B glassy alloys. The most important discovery is the FINEMET ($Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$), which was synthesized by Yoshizawa et al. [2], and it was found that the bcc Fe solid solution phase with ultrafine grain size around 10 nm after annealing led to the excellent soft magnetic properties [3]. However, the structure and precipitate mechanism of the precipitate phase of this series nanocrystalline alloys are still not clear. Hono found that the nanocrystalline particles in the FINEMET were DO_3 prototype Fe_3Si phase containing 20–25 at.% Si with little Nb and Cu [4]. Zhang and Ramanujan reported that one of the primary precipitates is Fe_3Si phase in Fe–Si–B–Nb alloys [5,6].

Recently, a lot of Fe–Si–B based glassy alloys were synthesized such as $(Fe_{0.75}B_{0.15}Si_{0.1})Nb_4$ [7], $Fe_{79-x}Mo_xP_{10}C_4B_4Si_3$ (x = 0–6) [8] and $\{[(Fe_{0.5}Co_{0.5})_{0.75}B_{0.2}Si_{0.05}]_{0.96}Nb_{0.04}\}_{100-x}Cu_x$ (x = 0 and 0.25) [9]. Those newly developed Fe-based glassy alloys not only possess good soft magnetic properties, but also exhibit excellent mechanical properties. Consequently, it shows an attractive prospect of the utilization of these alloys in functional and structural material domains. However, to authors' knowledge, the mechanism of microstructure evolution with the addition of transition metal in the Fe–Si–B based glassy alloys is still not clear.

Recent reports show that the addition of small amount of Ni could improve the soft magnetic properties and plasticity of Fe-based bulk metal glasses [10,11]. In present work, we chose the $Fe_{78}Si_9B_{13}$ alloy as the starting alloy and added small amount of Ni to it. The alloys with nominal composition of $(Fe_{0.78}Si_{0.09}B_{0.13})_{100-x}Ni_x$ (x = 0, 2, 3 and 5) were fabricated by rapid solidification at two melt-spinning conditions; the effect of Ni on the microstructure evolution, precipitate phases, mechanical and magnetic properties of Fe–Si–B based glassy alloys was investigated by different experimental techniques.

2. Experimental procedures

The master alloy ingot with a nominal composition of $Fe_{78}Si_9B_{13}$ was supplied by the National Amorphous Nanocrystalline Alloy Engineering Research Center of China. The ingot and nickel (99.9 mass%) were put into a quartz tube and were spun using a single copper roller. Four ribbons were fabricated by spinning the melt at about 1400 °C directly; other four ribbons were fabricated by spinning the melt at 1400 °C after a super heating (SH) treatment at about 1500 °C. The diameter of the copper roller is 35 cm, and the circumferential speed is 36.6 m/s. The obtained ribbons with nominal composition of $(Fe_{0.78}Si_{0.09}B_{0.13})_{100-x}Ni_x$ (x = 0, 2, 3 and 5) were 20–50 μm in thickness and 3–5 mm in width. The samples were investigated

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by X-ray diffraction (XRD, Cu Kα, λ = 0.15405 nm). The thermal behavior of the amorphous ribbons was measured by means of a Netsch 404 differential scanning calorimetry (DSC) with a heating rate of 20 K/min. The microstructure observations were obtained by transmission electron microscopy (TEM). The magnetic properties were measured by vibrating sample magnetometery (VSM) under a field of 3000 Oe. We tested the Vickers microhardness (H_v) at room temperature with a microhardness tester at a load of 1.96 N and a holding time of 15 s, and the H_v was averaged from 100 tests.

3. Results

Fig. 1 shows the X-ray diffraction spectra of the free side of the as-spun $(Fe_{0.78}Si_{0.09}B_{0.13})_{100-x}Ni_x$ (x = 0, 2, 3 and 5) ribbons. For x = 0, 2, 3 and 5, the ribbons melt-spun with SH, the XRD patterns show solely a typical diffusive maximum, indicating the almost amorphous structure of the ribbons (Fig. 1a), which may be related to the homogeneous distribution of the addition element after SH. Meanwhile, for the ribbons melt-spun without SH, the XRD patterns (Fig. 1b) exhibit the sharp peaks corresponding to the crystalline phases, which are identified as α-Fe for x = 0, 2, 3 and 5; $Fe_{23}B_6$ for x = 5; Fe_3B for x = 3 and 5; Fe_2B for x = 2, 3 and 5, respectively. Here, the (2 0 0) plane of α-Fe cannot be found in the ribbon with x = 0, owing to the significantly low volume fraction of the α-Fe phase. Compared Fig. 1b with a, it is shown that the SH results in a larger amorphous volume fraction in the samples and the minor Ni additions promotes the formation of Fe–B compounds especially at x = 3.

Moreover, for the ribbons without SH, the intensities of the α-Fe peak vary with the Ni content (c_{Ni}) (Fig. 1b). Fig. 2 presents the variation of the intensities and positions of (1 1 0) and (2 0 0) planes

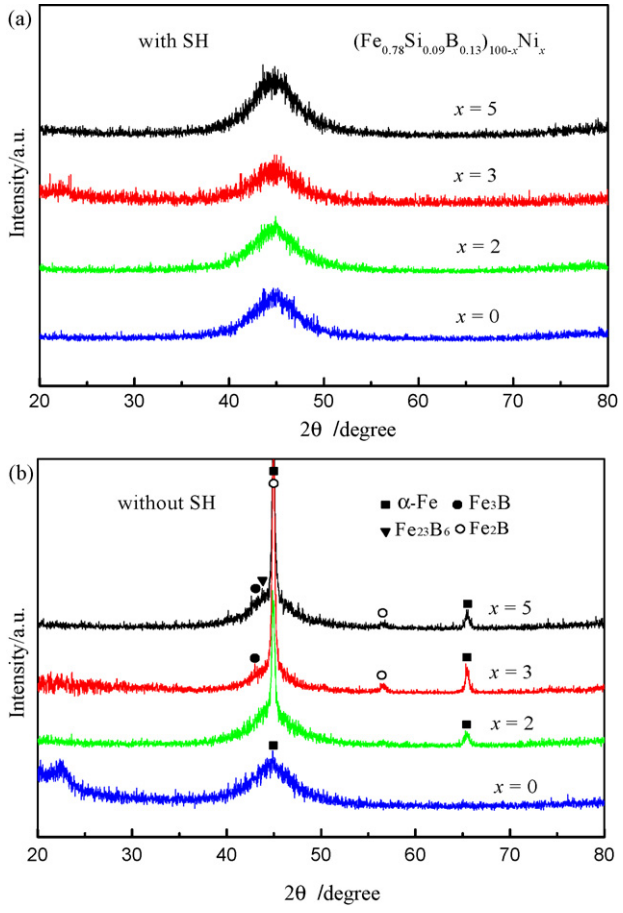

Fig. 1. XRD spectra of the free side of as-spun $(Fe_{0.78}Si_{0.09}B_{0.13})_{100-x}Ni_x$ (x = 0, 2, 3, 5) ribbons with SH (a) and without SH (b).

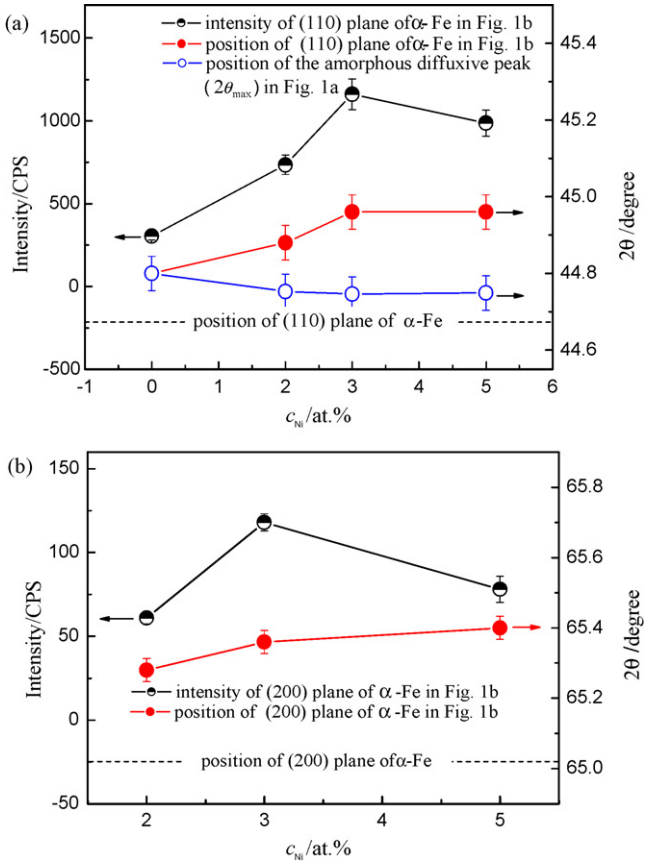

Fig. 2. Correlation of Ni content with intensity and position of diffraction α-Fe peak in the as-spun $(Fe_{0.78}Si_{0.09}B_{0.13})_{100-x}Ni_x$ (x = 0, 2, 3, 5) ribbons: (a) intensity and position (2θ) of (1 1 0) plane, position of amorphous diffusive peak ($2\theta_{max}$); (b) intensity and position (2θ) of (2 0 0) plane.

of α-Fe phase in the ribbons without SH, as well as the positions of the amorphous diffusive peak of the ribbons with SH. Here, the intensity of (1 1 0) plane in the ribbons without SH has an increasing tendency with increasing c_{Ni}, and the position of (1 1 0) plane shifts towards the higher angle. However, the position of the amorphous diffusive peak $2\theta_{max}$ of the ribbons with SH shifts towards the lower angle with increasing c_{Ni}, and the varying degree is smaller than that of (1 1 0) plane of α-Fe in the ribbon without SH (Fig. 2a).

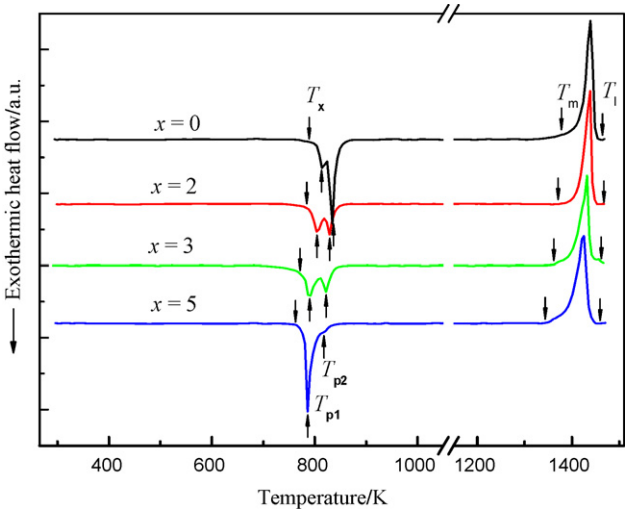

Fig. 3. DSC scans of the $(Fe_{0.78}Si_{0.09}B_{0.13})_{100-x}Ni_x$ (x = 0, 2, 3, 5) ribbons with SH.