Formation and Structural Evolution of $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ Nanocrystalline Alloy

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Abstract

A 2.5-mm $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ glassy rod was successfully fabricated using copper mold casting. The introduction of Cu resulted in the formation of large quantities of α -Fe nanoparticles embedded in the glassy matrix after isothermal annealing. The $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ nanocrystalline alloy exhibited high saturation magnetization (~ 1.26 T) and a low coercive force (~ 0.8 A/m) after annealing at 833 K for 15 min due to the precipitation of ~ 15 -nm-sized α -Fe nanoparticles in the glassy matrix. The structural evolution of the FeBSiNbZrCu amorphous alloy during the annealing process was discussed using a dual-cluster model.

Keywords Nanocrystalline alloy · Magnetic property · Dual-cluster model · Structural evolution

1 Introduction

Fe-based nanocrystalline soft magnetic alloys, including Fe–Si–B–Nb–Cu (FINEMET), Fe–Zr–B (NANOPERM), (Fe,Co)–Zr–B–Cu (HITPERM), and Fe–Si–B–P–Cu (NANOMET), have been widely investigated due to their high saturation magnetization (B_s), high permeability (μ), low coercive force (H_c), and low core loss [1–5]. The FINEMET nanocrystalline alloy is commonly used as a magnetic component in high-frequency transformers, inductors, and sensors [6, 7]. The thickness of the nanocrystalline alloys is typically below 30 μm due to the low glass-forming ability (GFA) of their amorphous precursor, resulting in a low packing fraction of the magnetic core of nanocrystalline ribbons [5]. Therefore, the development of Fe-based nanocrystalline alloys with high GFA and good soft magnetic properties is required. Several attempts have been made

to synthesize Fe-based nanocrystalline alloys with a high GFA. Some researchers have reported on FeCoSiBNbCu, FeBPNbCu, FeCSiBPCu, FePCBCu, and FeSiBPNbCu alloys that exhibited high GFA and could be fabricated into nanocrystalline soft magnetic alloys under optimal annealing conditions [8–14]. There are few reports on bulk nanocrystalline alloys; therefore, the development of a new nanocrystalline alloy that combines a high GFA and good soft magnetic properties is required.

Cu atoms play an important role in the formation of nanocrystalline alloys. The addition of small quantities of Cu atoms (~ 1 at.%) to amorphous alloys may change the crystallization process of the amorphous precursors [15]. Hono et al. investigated the crystallization process of FINEMET alloys using a three-dimensional atom probe (3DAP) and a high-resolution electron microscope (HREM) [16]. The results indicated that the Cu cluster formation promoted the chemical segregation and the nucleation of the primary α -Fe(Si) phase. Nb and B have low solubility in Fe and are expelled from the body-centered cubic (BCC) Fe–Si crystal. The thermal growth of the nanocrystalline grains is suppressed since the Nb- and B-rich amorphous phases become thermally stable [17, 18]. The difficulty in controlling the size of the α -Fe nanocrystalline grains in the NANOMET alloys (requiring a high heating rate over 100 K/min) is primarily attributed to the lack of large-sized atoms, such as Nb, Ta, Zr, and Hf, which inhibit the growth of α -Fe nanoparticles [19, 20]. Zhang et al. found that stable precipitation

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of the α -Fe grains occurred in Fe–Si–B–Cu nanocrystalline alloys with high Cu concentration, even when large-sized atoms were not introduced [7]. These results suggest that the structural evolution of the nanocrystalline alloys is complex and requires further study.

In our previous research, Fe–B–Si–(Nb, Zr, Hf, or Ta) bulk glassy alloys with high GFA, good soft magnetic, and mechanical properties were developed using the cluster-plus-glue-atom model [21–24]. A dual-cluster model was proposed to understand the structure of the amorphous and nanocrystalline alloys [25–27]. In the current study, we developed a novel $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ nanocrystalline soft magnetic alloy with large GFA and good soft magnetic properties by adding Cu atoms to the FeBSiNbZr bulk amorphous alloy. The structural evolution of the nanocrystalline alloy was determined using the dual-cluster model.

2 Experimental

$\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ alloy ingot was prepared by arc melting a mixture of pure Fe and Si (99.999 wt%), B (99.5 wt%), Zr and Nb (99.95 wt%), and Cu (99.99 wt%) in a Ti-gettered argon atmosphere. Alloy rods with different diameters were prepared using copper mold suction casting. Amorphous ribbons (thickness = 20 μm , width = 1.5 mm) were fabricated using the single-roller melt-spinning method. The surface speed of the copper wheel was set to 40 m/s. The melt-spun ribbons were sealed in vacuum quartz tubes and isothermally annealed at various temperatures for 15 min. The structures of the as-prepared ribbon and the rod samples were examined using an X-ray diffractometer (XRD, Bruker D8 Focus, $\text{Cu-K}\alpha$ radiation, $\lambda = 0.15406$ nm) and a transmission electron microscope (TEM, JEM-2010F, 200 kV). Specimens for TEM observation were prepared via twin-jet electropolishing (20 vol% perchloric acid + 80 vol%

carbinol). The thermal dynamic characteristics of the glassy alloys were measured using differential thermal analysis (DTA) at a heating rate of 40 K/min. The B_s and H_c were measured with a vibrating sample magnetometer (VSM) under a magnetic field up to 1 T and a B – H loop tracer under a field up to 800 A/m at room temperature.

3 Results and Discussion

Figure 1a shows the XRD pattern and TEM image of the $\phi 2.5$ -mm $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ as-cast rod. The XRD results showed a typical amorphous structure. The high-resolution image showed a maze-like structure. The selected area electron diffraction (SAED) pattern consisted of two diffuse rings. The XRD and the TEM results confirmed that the $\phi 2.5$ -mm-diameter rod was fully amorphous [28]. The DTA curve for the 2.5-mm $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ glassy rod is presented in Fig. 1b. The glass transition occurred in the sample as the temperature increased, followed by an undercooled liquid temperature region ($\Delta T_x = T_x - T_g = 852$ K – 818 K = 34 K) and three exothermic peaks.

The XRD patterns of the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ melt-spun ribbon annealed at various temperatures are shown in Fig. 2a. The ribbon had an amorphous structure after isothermal annealing at 773 K for 15 min. Sharp peaks corresponding to the α -Fe phase emerged when the annealing temperature exceeded 833 K. When the annealing temperature was higher than 873 K, a mixture of α -Fe, Fe_2B , and Fe_3B -type phases was observed. Figure 2b shows the TEM image and the corresponding SAED pattern of the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ glassy alloy annealed at 833 K for 15 min. The nanocrystals with an average size of ~ 15 nm were embedded in the amorphous matrix in random dispersion. The SAED pattern (insert at the right top corner of Fig. 2b) clearly showed the diffraction rings of

Fig. 1 **a** XRD pattern, **b** DTA curve of the 2.5-mm $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ as-cast rods (inset: the high-resolution TEM image and the SAED pattern)

Fig. 2 **a** XRD patterns of the as-spun $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ glassy ribbon isothermally annealed at different temperatures for 15 min, **b** TEM image and corresponding SAED pattern of the glassy ribbon annealed at 833 K for 15 min

Fig. 3 Changes in the B_s and the H_c of the melt-spun $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ glassy ribbons as a function of the annealing temperatures (annealing for 15 min)

the (110), (200), (211), (220), (310), (222), and (321) α -Fe lattice planes.

The changes in the B_s and H_c of the melt-spun $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ glassy ribbons as a function of the annealing temperature (annealing for 15 min) are shown in Fig. 3. The B_s and the H_c of the melt-spun alloy after annealing at 773 K for 15 min were 1.17 T and 1.4 A/m, respectively. As the annealing temperature increased to 833 K, the B_s increased to 1.26 T and the H_c decreased to 0.8 A/m. The soft magnetic properties deteriorated when the annealing temperature further increased beyond 833 K, i.e., a sharp increase in the H_c and a decrease in the B_s occurred at 873–943 K. The B_s of the nanocrystalline alloy is defined as [29]:

$$B_s = B_{sc} V_c + B_{sa} (1 - V_c), \quad (1)$$

where B_{sc} and B_{sa} are the saturation magnetization of the α -Fe crystalline and the amorphous matrix, respectively. V_c is the volume fraction of the crystalline phases. The value of B_{sc} was higher than that of B_{sa} [30]. The B_s was dependent on the V_c . The increase in the B_s in the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ nanocrystalline alloy after isothermal annealing resulted from the high volume fraction of α -Fe. The decrease in H_c was ascribed to the refinement of α -Fe due to an increase in the number of heterogeneous nucleation sites and the suppression of grain growth. The deterioration of the magnetic properties in the nanocrystallized ribbons at annealing temperatures higher than 833 K was attributed to the α -Fe grain growth and the precipitation of the Fe_2B and Fe_3B phases [31].

Table 1 summarizes the GFA, the size of the α -Fe nanoparticles, the magnetic properties of the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ nanocrystalline alloy, and other typical soft magnetic nanocrystalline alloys reported in the literature. The $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ nanocrystalline alloy developed in this study exhibited a low H_c (~ 0.8 A/m, approaching that of the FINEMET nanocrystalline alloy) and a high GFA (critical diameter, $d_c = 2.5$ mm) [1–3, 11, 13–15, 18, 32].

A binary eutectic alloy liquid typically decomposes in a coupled growth mode into two eutectic phases with distinct compositional differences. The eutectic liquid contains two subunits that evolve toward their corresponding eutectic phases. The eutectic composition consists of a dual cluster, and glue atoms are described using the cluster-plus-glue-atom model, where the dual cluster belongs to the two liquid subunits and is inherited from the corresponding eutectic phases [33]. The glue atoms are located between the clusters. In the eutectic composition, the dual clusters have a ratio of

Table 1 GFA, size of α -Fe nanoparticles, and the magnetic properties of the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ nanocrystalline alloy developed in this study and Fe-based nanocrystalline alloys reported previously

Composition	d_c (nm)	Size of α -Fe nanoparticles (nm)	B_s (T)	H_c (A/m)
$\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$	2.5	~ 15	1.26	0.8
$\text{Fe}_{61.5}\text{Co}_{10}\text{B}_{13.5}\text{Si}_{10}\text{Nb}_4\text{Cu}_1$ [13]	1.5	~ 10	1.24	2.8
$\text{Fe}_{70}\text{B}_{22}\text{Y}_4\text{Nb}_2\text{Cu}_2$ [15]	2.0	~ 11	1.36	143
$\text{Fe}_{71}\text{B}_{22}\text{Y}_4\text{Nb}_2\text{Cu}_1$ [15]	2.0	~ 15	1.32	18
$\text{Fe}_{75.3}\text{C}_{7.0}\text{Si}_{3.3}\text{B}_{5.0}\text{P}_{8.7}\text{Cu}_{0.7}$ [14]	1.0	~ 5	1.79	11
$(\text{Fe}_{0.8}\text{P}_{0.09}\text{C}_{0.09}\text{B}_{0.02})_{99.3}\text{Cu}_{0.7}$ [11]	1.5	~ 19	1.60	–
$(\text{Fe}_{0.76}\text{Si}_{0.09}\text{B}_{0.1}\text{P}_{0.05})_{98.5}\text{Nb}_1\text{Cu}_{0.5}$ [18]	2.5	~ 21	1.46	2.8
$(\text{Fe}_{0.76}\text{Si}_{0.09}\text{B}_{0.1}\text{P}_{0.05})_{98.25}\text{Nb}_1\text{Cu}_{0.75}$ [18]	2.5	~ 22	1.51	2.2
$\text{Fe}_{73.5}\text{Si}_{13.5}\text{B}_9\text{Nb}_3\text{Cu}_1$ [1]	0.02	~ 20	1.21–1.24	0.5–1.4
$\text{Fe}_{88}\text{B}_4\text{Zr}_7\text{Cu}_1$ [2]	0.02	~ 13	1.52–1.64	2.4–4.5
$\text{Fe}_{44}\text{Co}_{44}\text{Zr}_7\text{Cu}_1$ [2]	0.02	~ 10	1.6–2.1	> 20
$\text{Fe}_{84.3}\text{Si}_4\text{B}_8\text{P}_3\text{Cu}_{0.7}$ [3]	0.02	~ 17	1.88–1.94	7–10
$\text{Fe}_{83}(\text{Co}_x\text{Ni}_y)(\text{B}_{11}\text{Si}_2\text{P}_3\text{C}_1)_{1-x,y/17}$ [32]	0.02	~ 5	1.65–1.72	5–13

1:1. When the composition deviates from the eutectic composition, a mixture of the two types of clusters exists with different ratios [25, 27]. This concept was used to describe the composition of the amorphous alloys based on inheriting the liquid–solid structure during rapid quenching [34].

The Fe–B-based amorphous alloys had a relevant eutectic point of $\text{Fe}_{83}\text{B}_{17}$, which was represented by the dual-cluster formula $[\text{Fe–Fe}_{14}]\text{B}_2\text{Fe} + [\text{B–B}_2\text{Fe}_8]\text{Fe} \approx \text{Fe}_{83.3}\text{B}_{16.7}$, where the $[\text{B–B}_2\text{Fe}_8]$ and the $[\text{Fe–Fe}_{14}]$ clusters were inherited

from the Fe_2B and the α -Fe eutectic phases, respectively [25]. The B and Fe glue atoms were located between the $[\text{B–B}_2\text{Fe}_8]$ and $[\text{Fe–Fe}_{14}]$ clusters. The structural stability of the clusters was considered when Si, Nb, and Zr were introduced into the $[\text{B–B}_2\text{Fe}_8]$ cluster to replace the center B and the shell Fe atoms [21, 22]; the Cu-free FeBSiNbZr amorphous alloys consisted of the dual-cluster $[\text{Fe–Fe}_{14}]$, the $[\text{Si–B}_2(\text{Fe,Nb,Zr})_8]$, and the glue atoms [26], as shown in Fig. 4b. The Cu exhibited positive enthalpies of mixing

Fig. 4 Structural evolution of **a** FeBSiNbZrCu , **b** Cu-free FeBSiNbZr glassy alloys during the annealing process

(ΔH) with Fe and Nb ($\Delta H_{\text{Cu-Fe}} = 13$ kJ/mol, $\Delta H_{\text{Cu-Nb}} = 3$ kJ/mol), zero ΔH with B, and weak negative ΔH with Si and Zr ($\Delta H_{\text{Cu-Si}} = -19$ kJ/mol, $\Delta H_{\text{Cu-Zr}} = -23$ kJ/mol) [33]. The Cu atoms were likely located in the glue atom position in the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ amorphous alloy (Fig. 4a).

In the FeBSiNbZr and $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ amorphous alloys, the $[\text{Si-B}_2(\text{Fe,Zr,Nb})_8]$ cluster exhibited high local structural stability because of the strong chemical bonding between the Si-Fe, Si-Zr, and Si-Nb pairs. The $[\text{Fe-Fe}_{14}]$ cluster did not have high local structural stability because of the weak correlation between the Fe-Fe pairs, which easily disintegrated. The released Fe atoms from the cluster diffused into the $[\text{Si-B}_2(\text{Fe,Zr,Nb})_8]$ cluster due to the strong attraction between the B-Fe, Zr-Fe, and Nb-Fe atom pairs; this was followed by the precipitation of the intermetallic phase during the annealing process of the Cu-free FeBSiNbZr amorphous alloy, as shown in Fig. 4b. The Fe atoms in the $[\text{Fe-Fe}_{14}]$ cluster did not approach the $[\text{Si-B}_2(\text{Fe,Zr,Nb})_8]$ cluster due to the repulsive effect between the Fe and Cu glue atoms in the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ amorphous alloy. The $[\text{Fe-Fe}_{14}]$ clusters favored the nucleation of the α -Fe phase, which easily grew from the amorphous matrix during the annealing process, as shown in Fig. 4a. The remaining clusters with high local structural stability and the Cu atoms (or Cu-rich clusters) around the α -Fe nanoparticles effectively slowed down the diffusion of Fe atom, thereby inhibiting the growth of the α -Fe grains. When the α -Fe grains reached an appropriate size (~ 15 nm), the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ nanocrystalline alloy exhibited the best soft magnetic properties with a high B_s of 1.26 T and low H_c of 0.8 A/m.

4 Conclusions

We successfully developed a $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ bulk glassy alloy, which exhibited good soft magnetic property after nanocrystallization. The structural evolution of the nanocrystalline alloy was determined using the dual-cluster model. The results are summarized as follows:

1. A $\phi 2.5$ -mm glassy rod was fabricated using copper mold casting. The glassy alloy exhibited highly stability and resistance to crystallization and a supercooled liquid region of 34 K.
2. The complex phase with a mass dispersion of α -Fe nanoparticles embedded in the amorphous matrix was obtained after isothermal annealing of the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ glassy ribbons at adequate temperatures. The obtained nanocrystalline alloy exhibited good soft magnetic properties with a high B_s (~ 1.26 T) and a low H_c (~ 0.8 A/m) at a α -Fe nanoparticle size of 15 nm.

3. The dual-cluster model was used to understand the underlying mechanism, and it was determined that the stable precipitation of the α -Fe nanocrystals in the $\text{Fe}_{72.5}\text{B}_{15.6}\text{Si}_{7.8}\text{Nb}_{1.7}\text{Zr}_{1.7}\text{Cu}_{0.7}$ alloy was primarily attributed to the existence of the weak stable $[\text{Fe-Fe}_{14}]$ clusters, the suppression of diffusion of the Fe atoms by the Cu glue atoms, and the strong stable clusters.

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