

Effect of Crystallization Treatment on the Magnetic Properties of Amorphous Strips Based on Co-Fe-Ni-B-Si-Cr Containing Nitrogen

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Abstract Co-Fe-Ni-B-Si-Cr based amorphous strips containing nitrogen were manufactured via melt spinning, and then devitrified by crystallization treatment at the various annealing temperatures of 300°C~540°C for up to 30 minutes in an inert gas (N₂) atmosphere. The microstructures were examined by using XRD and TEM and the magnetic properties were measured by using VSM and B-H meter. Among the alloys, the amorphous ribbons of Co_{72.6}Fe_{9.8}Ni_{5.5}B_{2.4}Si_{7.1}Cr_{2.6} containing 121 ppm of nitrogen showed relatively high saturation magnetization. The alloy ribbons crystallized at 540°C showed dramatically reduced remanence and coercive force. A TEM micrograph showed that the grain size of Co_{72.6}Fe_{9.8}Ni_{5.5}B_{2.4}Si_{7.1}Cr_{2.6} alloy containing 121 ppm of nitrogen was about 5 nm, which exhibited paramagnetic behavior. The formation of nano-grain structure was attributed to the finely dispersed Fe₄N particles and the solid-solutionized nitrogen atoms in the matrix. Accordingly, it can be concluded that the nano-grain structure of 5 nm in size could reduce the core loss within the normally applied magnetic field of 300 A/m at 10 kHz.

Keywords : Co-based amorphous strip, Nitrogen and Fe additions, Core loss, Saturation magnetization

1. Introduction

In general, the soft magnetic properties and their frequency responses in amorphous and nanocrystalline alloys are superior to those of the conventional alloys, i.e. permalloy, sendust and ferrites, due to their low magnetic anisotropy and high resistivity¹⁾. Especially, Co-based amorphous alloys have ultra-high permeability and low coercive force, which are very useful for inductor, sensor and transformer core. However, Co-based amorphous materials still possess relatively low magnetic saturation and high core loss for high-frequency electronic applications. According to the reported study²⁾, the soft magnetic materials containing nitrogen has excellent high-frequency characteristics, which is due to the high electrical resistivity and magnetic anisotropy field. In this paper, the effect of the crystallization treatment

for a nano-grain structure on the magnetic properties of Co-Fe-Ni-B-Si-Cr based amorphous strips containing 103~121 ppm of nitrogen was studied in detail.

2. Experimental procedure

The Co_{72.6-53.2}Fe_{9.7-29}8N_{15.5-4.0}B_{2.4-1.8}Si_{7.1-6.5}Cr_{2.6-4.6} alloy ingots containing 116~121 ppm of nitrogen were prepared by arc melting, and then melt spun to make amorphous strips at a high cooling rate. Table 1 shows the chemical composition of the alloy strips.

The ribbons of ~20 μm in thickness and 6 mm in width were annealed at 300~540°C for 15 minutes and 30 minutes, respectively. The crystallization temperature was determined by differential scanning calorimetry (DSC). X-ray diffraction (XRD) with Cu Kα radiation was used to analyze the crystal structure.

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Table 1. The chemical composition of amorphous alloy strips.

Alloy Strips	Comp. (wt %)						
	Fe	Ni	B	Si	Cr	N	Co
Reference H	5.35	11.63	2.82	7.33	1.63	-	71.25
Alloy1	9.76	5.50	2.40	7.05	2.64	121ppm	72.64
Alloy2	13.2	4.82	2.26	7.06	3.15	103ppm	69.50
Alloy3	29.8	4.04	1.86	6.48	4.65	116ppm	53.16

Magnetic properties of the Co-based amorphous strips were measured with a VSM, and those of the crystallized strips were measured with a B-H meter within the maximum applied field of 300 A/m at 10 kHz. In order to examine the nano-grain structures, TEM micrography was performed on the specimens annealed at 540°C for 15 min .

3. Results and Discussion

With the increase of Fe content to 13.2%, the saturation magnetization increased up to 94.6 emu/g (Table 2). However, the residual magnetization and the intrinsic coercivity also increased considerably. Among the newly designed alloys, Alloy1 containing 121 ppm of nitrogen possessed not only low coercivity and retentivity but also relatively high saturation magnetization.

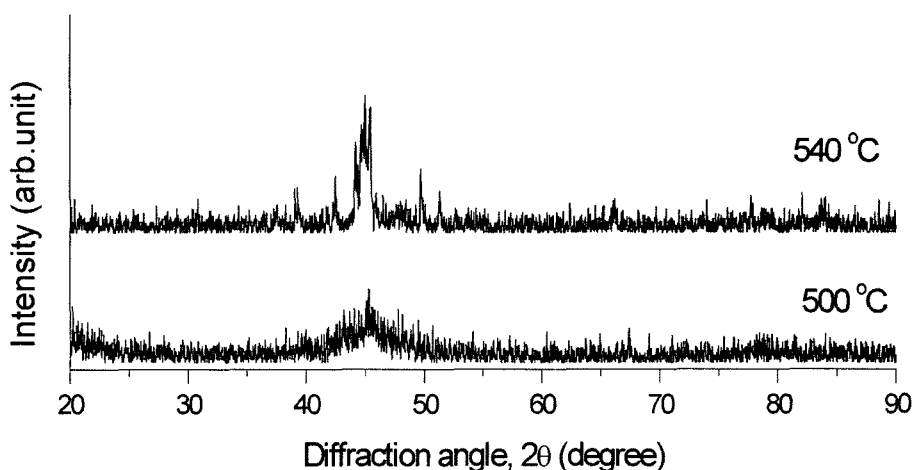
Fig. 1 presented the X-ray diffraction pattern of

Table 2. Saturation magnetization (Ms) and coercivity measured with a VSM.

	Ms (emu/g)	Hci (G)	Mr (emu/g)
Reference H	67.74	0.75	2.30
Alloy1	81.50	0.78	1.49
Alloy2	94.60	0.88	3.03
Alloy3	76.56	0.80	2.79

Alloy1 annealed at 500°C and 540°C, respectively. This showed that the amorphous Co-based alloy was crystallized after annealing at 540°C. The annealing up to 500°C did not change the remanence and coercive force considerably. However, the reduced remanence and the coercive force were obtained after annealing above crystallization temperature, as shown in Fig. 2 and Fig. 3. These figures showed that the crystallization temperature lies between 500°C and 540°C.

Above the crystallization temperature, the nitrogen

**Fig. 1. X-ray diffraction patterns of the annealed alloys.**

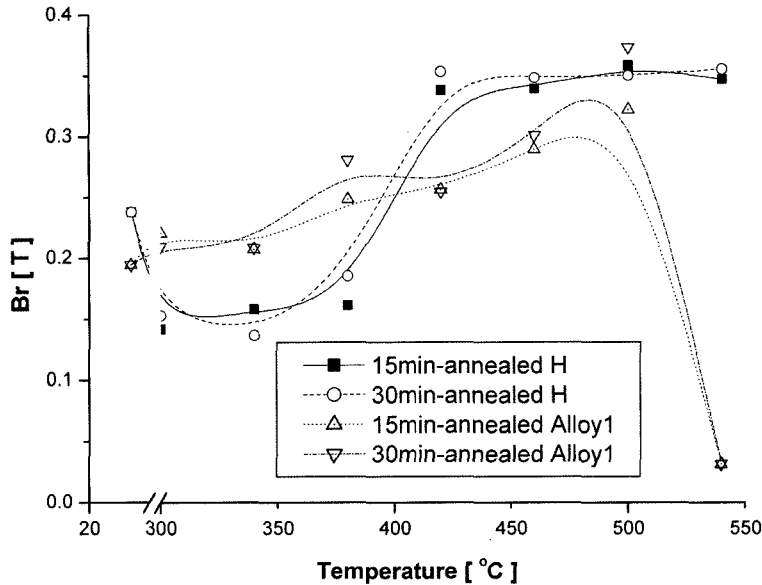


Fig. 2. The remanence changes of Reference alloy H and Alloy1 after annealing at 10 kHz.

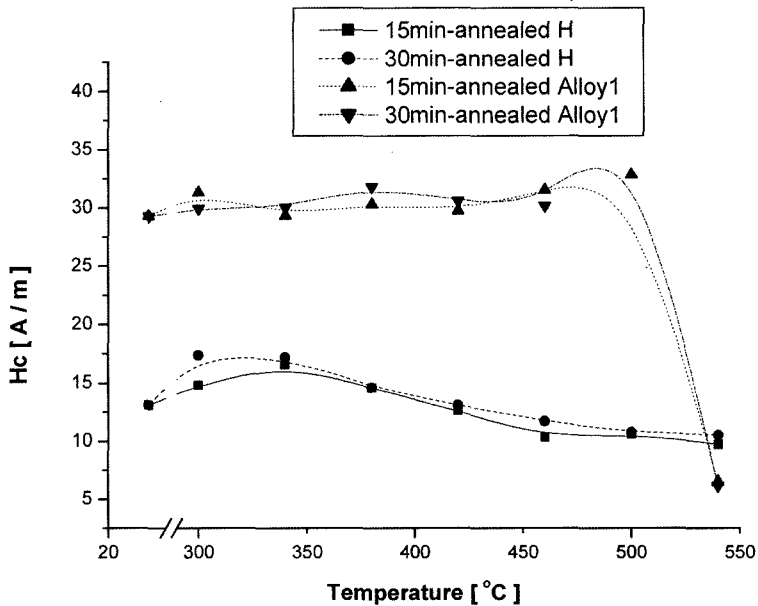


Fig. 3. Variation of the coercive force of Reference alloy H and Alloy1 with the annealing temperatures at 10 kHz.

exists as solid-solutionized atom in the matrix and as dispersed Fe_4N particles³⁻⁴⁾ along the grain boundary. The Fe_4N particles precipitated from the matrix with dilute nitrogen could suppress the grain growth of the crystallites. Therefore, the addition of nitrogen resulted in the decrease of the crystallized grain size

of the Co-based alloys⁵⁾. The size scale of the nanocrystallites was determined from TEM studies, which revealed that the grain size of Alloy1 was approximately 5 nm (Fig. 4). In the nanocrystalline Co-based alloy obtained by crystallization of the amorphous phase, the decrease of grain size generally

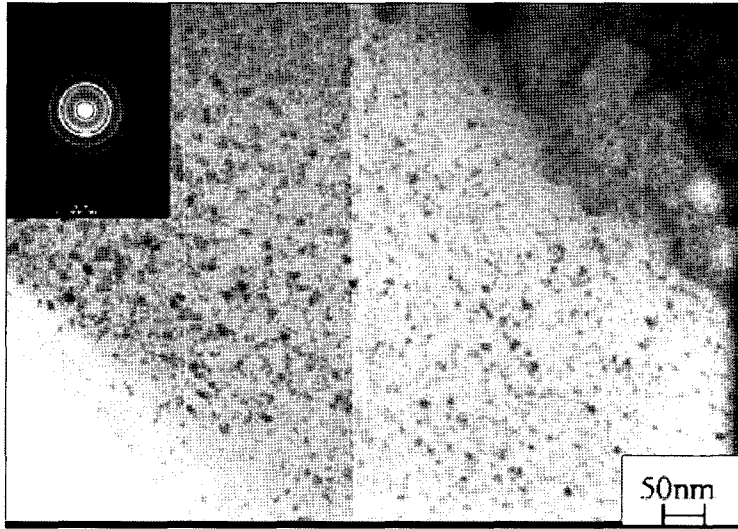


Fig. 4. TEM image of Alloy1 annealed at 540°C for 15 min.

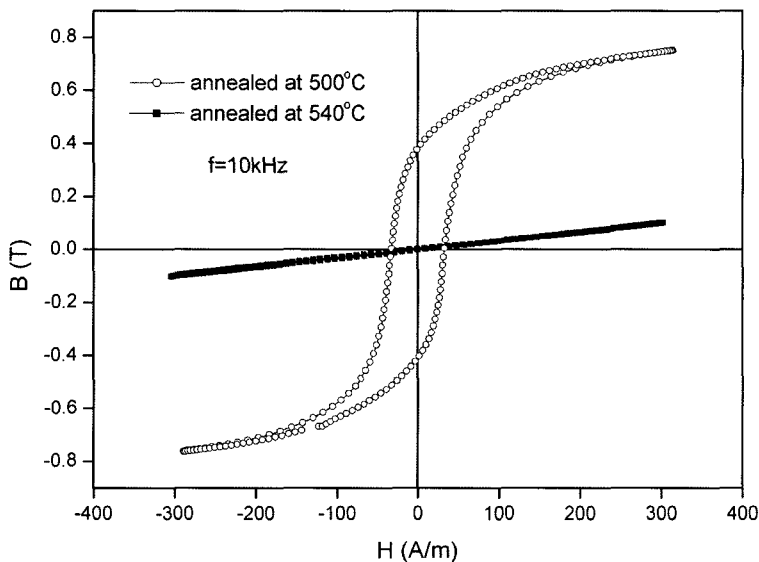


Fig. 5. Hysteresis loops of Alloy1 measured with the applied field of 300 A/m at 10 kHz.

implies a decrease in coercive force and an increase in magnetic permeability, and as a consequence the domain wall mobility is increased⁶⁾. However, the nanostructured Co-based alloy exhibited a paramagnetic behavior because the grain size was smaller than the single domain size of the Co-based alloy. The crystallites with a domain structure of ~ 5 nm were characterized as paramagnetism by their size

and by their magnetocrystalline anisotropy^{6,7)}. And, the residual intergranular amorphous phase was considered to be nonmagnetic.

Fig. 5 represented the linear-type hysteresis loop measured from Alloy1 annealed at 540°C at 10 kHz. This showed that the required magnetic field for saturation was noticeably increased, and the hysteresis loss would be reduced dramatically under

an application condition. The microstructure in Fig. 4 is closely related to this paramagnetic behavior. Hence, it can be concluded that the nano-structured Co-based alloy possessed the paramagnetism, which was attributed to the finely dispersed Fe_4N particles and the solid-solutionized nitrogen atoms inducing the nano-grain structures of ~ 5 nm in size.

4. Conclusions

The amorphous ribbons of $\text{Co}_{72.6}\text{Fe}_{9.8}\text{Ni}_{5.5}\text{B}_{2.4}\text{Si}_{7.1}\text{Cr}_{2.6}$ containing 121 ppm of nitrogen had higher saturation magnetization than the conventional Co-based amorphous ribbons. Especially, the alloy strips showed the improved magnetic properties with a reduced coercive force and remanence after a crystallization treatment at 540°C for 15 min. The grain size of the annealed alloy was approximately 5 nm,

which was probably attributed to the finely dispersed Fe_4N particles and the solid-solutionized nitrogen atoms in the matrix. As a result, the nano-structured Co-based alloy possessed the paramagnetism.

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