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Effect of Annealing Temperature on Magnetic After-Effect in FeCuNbSiB Alloys

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Abstract. The microstructure and magnetic properties i.e. the initial permeability, magnetic after-effect and coercivity for the as-quenched and annealed at different temperatures $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ samples are studied. After annealing the samples at 573 K for 1 h the decrease of the initial permeability and increase of the disaccommodation amplitude are observed. These are connected with a local anisotropy induced during this treatment. The increase of the annealing temperature causes the improvement of soft magnetic properties. However, after annealing the toroidal samples at 723 K for 1 h a magnetic hardening is observed. It seems to be connected with the appearance of $\alpha\text{Fe-Si}$ nuclei and clusters enriched in Cu. Moreover, it was stated that the cooling conditions of the samples influence also their magnetic properties. Therefore, the values of coercivity obtained for annealed strips in comparison with corresponding results of μ' obtained for toroids are shifted towards higher temperature by 50 K.

1. INTRODUCTION

Nanocrystalline $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ alloys are prepared by controlled crystallization of initially amorphous ribbons. The best annealing conditions (typically 823 K for 1 h) allow to obtain nanocrystalline alloys consisted of the $\alpha\text{Fe-Si}$ fine grains of about 10 - 20 nm in size randomly distributed in an amorphous matrix. This ultra-fine grain structure is achieved by combined addition of Cu and Nb [1]. This material exhibits excellent soft magnetic properties due to averaging the magnetocrystalline anisotropy and very low saturation magnetostriction [2].

The aim of this paper is to study the magnetic properties, i.e. initial relative permeability, its disaccommodation, coercivity and microstructure of the $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ alloy in the as-quenched state and after annealing in the temperature range from 573 to 873 K for 1 h.

2. EXPERIMENTAL PROCEDURE

The ribbons of the $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ alloy were about 20 μm thick and 2 mm wide. The initial permeability was measured for toroidal samples of 2 cm diameter using a completely automated set-up at room and nitrogen temperature. From these results the disaccommodation was calculated according to:

$$\Delta(1/\chi) = 1/\chi_2 - 1/\chi_1 = f(T) \quad (1)$$

where $1/\chi_1$ and $1/\chi_2$ are reciprocal magnetic susceptibilities at times $t_1=2$ s and $t_2 = 120$ s after demagnetization, respectively. The amplitude and frequency of the magnetizing field were equal to 0.24 A/m and 2 kHz, respectively.

The coercivity was measured for the strips 10 cm long by an automated induction method. The microstructure of the samples was investigated by X-ray diffractometry and differential scanning calorimetry.

The investigations were carried out for the samples in the as-quenched state and after annealing the toroidal samples and strips for 1 h at the temperatures: 573, 623, 673, 723, 748, 773, 798, 823 and 848 K.

3. RESULTS

The evolution of X-ray diffraction patterns with annealing temperature (T) is plotted in Fig.1. The samples in the as-cast state and after annealings below 750 K are characterized by the typical amorphous halo. Annealing temperature higher than 773 K gives rise to the precipitation of a randomly textured α Fe-Si phase with mean grain diameter, obtained through Scherrer formula, around 12 nm.

Figure 1: X-ray diffraction patterns obtained for the as-cast (curve 1) and annealed for 1 h at the temperatures: 573, 623, 673, 723, 748, 773, 798, 823 and 848 K samples (curves 2-10, respectively)

The results of the magnetic properties measurements are shown in Figs 2-4. Experimental points in these figures are linked to show the tendency of the magnetic properties changes.

Fig.2 shows the relative permeability $\mu' = \mu/\mu_0$ (where μ is the permeability measured at $H_m = 0.24$ A/m and $t_1 = 2$ sec after demagnetization and μ_0 is the vacuum permeability) and the amplitude of disaccommodation $\Delta(1/\chi) = f(T)$ for as-quenched and annealed samples obtained during measurements at room and nitrogen temperatures. A slight decrease of the permeability after annealing the sample at 573 K for 1 h can be detected; then it increases with annealing temperature (T) up to 848 K except the samples annealed at 723 K where μ' drops to about 400. The dependence of the relative permeability on annealing temperature obtained at nitrogen temperature is similar to the previous one (Fig.2a). The disaccommodation reaches the maximum after annealing the sample at 573 K; the next smaller maximum is observed after annealing at 723 K for 1 h. After annealing the sample above 773 K the disaccommodation drops almost to zero. Similar behaviour exhibits the disaccommodation during measurements at nitrogen temperature (Fig.2b).

In Fig.3 the coercive field (H_c) versus annealing temperature is shown. The coercivity slightly increases after annealing the samples at 623 K and subsequently shows the distinct maximum after

annealing the samples at $T=773$ K. The further increase of annealing temperature leads to the drop of H_c .

4. DISCUSSION

The magnetic properties observed for the $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ alloy are highly influenced by their microstructure which depends on annealing conditions. During annealing the amorphous samples of these

Figure 2: The dependence of $\mu'=\mu/\mu_0$ (a) and $\Delta(1/\chi)$ (b) on annealing temperature (T) for as-cast and annealed samples obtained at room (curve a) and nitrogen temperatures (curve b), respectively.

Figure 3: The coercive field (H_c) versus annealing temperature (T) obtained for the $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ alloy.

alloys below crystallization temperature, mainly two effects occur: inducing of the local magnetic anisotropy and annealing out of free volumes which caused the stress-relief. When the sample is annealed at temperature $T < T_c$ (T_c - Curie temperature) the domain structure exists and the internal field within each domain can induce the local anisotropy. The second effect is dominant during annealing the samples at temperatures $T \geq T_c$.

The samples in the as-quenched state exhibit rather poor soft magnetic properties, i.e. low relative permeability (about 400) and high coercivity (about 4 A/m) due to internal stresses frozen-in during the ribbon's preparation. After annealing the toroidal samples at 573 K and strips at 623 K for 1 h the decrease of permeability and increase of coercivity, respectively, is observed (Figs 2 and 3). This behaviour may be explained by local induced magnetic anisotropy which leads to domain wall stabilization. According to results obtained by P.Allia and F.Vinai [4], the after-effect of magnetic induction at $B=0$ may be described by

$$\left(\frac{\Delta B}{B}\right)_{B=0} = \frac{5 \pi^2 K_u \mu_0 L}{8 M_s A} \quad (2)$$

where L, A, M_s are the distance between walls, mean wall thickness and saturation magnetization, respectively. Thus the increase in induced anisotropy (K_u) should increase the disaccommodation amplitude after annealing the samples at 573 K, which is experimentally observed (Fig.2b). Annealing at higher temperatures leads to the increase of the permeability and decrease of the disaccommodation amplitude and coercivity. This is caused by annealing out of free volumes and stress relief of the samples. However, a magnetic hardening is observed after annealing the toroidal samples at 723 K and strips at 773 K for 1 h (Figs 2 and 3).. It seems to be connected with the appearance of $\alpha\text{Fe-Si}$ nuclei and clusters enriched in Cu as X-ray diffraction and differential scanning calorimetry show [4-6]. The further enhancement of annealing temperature up to 848 K leads to the increase of μ and decrease of H_c due to stress-relief of samples and more effective averaging of magnetocrystalline anisotropy caused by occurrence of $\alpha\text{Fe-Si}$ fine grains. As for disaccommodation, its amplitude decreases despite the volume fraction of the crystalline phase increases and is connected with annealing out more and more free volumes in the amorphous matrix [7]. This result indicate that the processes occurring in the amorphous matrix are the main source of the magnetic after-effect in nanocrystalline alloys [8, 9].

It is worth noticing that during measurements at nitrogen temperature (77 K) a decrease of μ and disaccommodation amplitude is observed, which is connected with freezing spins in some regions of the samples [10].

5. CONCLUSIONS

1. After annealing the toroidal samples of the $\text{Fe}_{73.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ alloy at 573 K and strips at 623 K for 1 h the deterioration of the soft magnetic properties i.e. the decrease of the initial permeability and increase of coercivity and disaccommodation amplitude are observed.
2. The magnetic hardening occurs after heat treatment the toroidal samples and strips, respectively, at 723 and 773 K for 1 h.
3. The appearance of the $\alpha\text{Fe-Si}$ grains leads to the increase of the magnetic permeability and decrease of the coercivity and disaccommodation amplitude.

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