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Effect of the Crystallization on the Electromagnetic Wave Absorption Behavior of a Fe-based Nanocrystalline Alloy

S K Koo[1], C D Yim[2], B G Moon[2], Y S Song[3], W W Park[1] and K Y Sohn[1]
[1] School of Nano Engineering, Inje University, 607, Obang-dong, 621-749, Gimhae, Korea
[2] Advanced Materials Research Division, Korea Institute of Materials Science, 531, Changwondaero, 641-831, Changwon, Korea
[3] Research Institute, Amosense Co., 185-1, Sucham-ri, Tongjineup, 415-860, Kimpo, Korea

E-mail: ksohn@inje.ac.kr

Abstract. The crystallization effect of Fe-based nanocrystalline on the electromagnetic (EM) wave absorption behavior has been investigated. Amorphous ribbons were annealed at temperatures ranging from 500°C to 650°C for 1 hour to investigate the crystallization behavior. The grain size was in the range from 10 to 20nm as the specimen was annealed under 600°C, while it significantly increased when annealed above the temperature. The initial permeability increased with volume fraction of bcc-Fe(Si) phase and showed a maximum at f_c~0.77. The optimized magnetic properties of nanocrystalline ribbons can be acquired when they were crystallized at 550-560°C for 1 hour. However, the EM wave absorption properties in a sheet made of the nanocrystalline powder was greatest in the specimen crystallized at 510°C for 1hour annealing, indicating that the pulverization and milling process lowered the optimum crystallization condition.

1. **Introduction**

The recent spread of high-frequency electronic and communication devices has led to a rise in the amount of electromagnetic (EM) waves, causing harmful effects on human body and other nearby devices to malfunction. As concern about the effect of EM wave grows, the devices are required to have electromagnetic compatibility (EMC) [1-2]. Fe-based nanocrystalline magnetic materials such as Finemet alloys have excellent soft magnetic properties including large saturation magnetization and high relative permeability in the high frequency range. One application of the Finemet type alloy is an EM wave absorber, which absorbs the generated EM waves to transform into heats. FeSiBNbCu alloys exhibit excellent soft magnetic properties when nanocrystalline bcc-Fe(Si) phases that was formed by the crystallization annealing were embedded uniformly in the amorphous matrix [3-5]. Numerous studies have been made on the effect of grain size of crystalline bcc-Fe(Si) phase on the magnetic properties of FeSiBNbCu alloy, in which the optimum magnetic properties can be acquired when the grain size is controlled to the range 10~15nm [6-7]. However, the effect of volume fraction of crystallized bcc-Fe(Si) phase on the magnetic properties of FeSiBNbCu alloy has not been studied in detail. The objective of this study is to investigate the effect of the crystallization annealing conditions on the EM wave absorption behavior of a FeSiBNbCu alloy. The relative volume fractions

[1] Corresponding Author : [Tel : +82-55-320-3714; E-mail : ksohn@inje.ac.kr]

of nanocrystals produced at various crystallization conditions were quantified using a differential scanning calorimeter (DSC) method.

2. Experimental procedure

Amorphous ribbons with a nominal composition of $Fe_{73}Si_{16}B_7Nb_3Cu_1$ (at%) alloy were annealed at temperatures ranging from 500°C to 650°C for 1hour under nitrogen atmosphere to investigate the crystallization behavior. The DSC analysis was carried out using as-fabricated ribbons of 21mg at temperatures ranging from 300°C to 720°C and heating rates ranging from 5 to 2°C /min.

After annealing, the ribbons were pulverized and sieved to several classes of particle size. The powder with sizes of <45, 45~53, and 53~75 μm were mixed uniformly with the volume ratio of 1:2:7, respectively. Subsequently, the mixture was formed to 2.79mm thick inductor cores of 6.35mm outer diameter and 2.79mm inner diameter under a pressure of 18 ton/cm^2 without binder. The permeability was measured under the frequency range of 10~1000 kHz.

In order to identify the interrelations between the crystallization behavior and electromagnetic wave absorption, the ribbons were pulverized prior to crystallization annealing. Crystallization annealing was carried out at 500~650°C for 1hour, followed by a tape-casting to produce a thin sheet of 0.5mm thick after mixing with a binder. The EM wave absorption properties were measured by a two –port coaxial method using a network analyzer (Agilent, N5260A).

3. Results and discussion

3.1. Crystallization behavior

Figure 1 shows the variation of initial permeability of annealed FeSiBNbCu alloy on annealing temperature. The initial permeability increased to 540°C and decreased thereafter. It has been reported that the magnetic properties of FeSiBNbCu alloy are significantly dependent on the grain size of bcc-Fe(Si) phase. The optimized magnetic properties can be acquired when the grain size is controlled to the range of 10~15nm. Figure 2 shows the variations in average grain size measured from XRD results on annealing temperature. In the temperature range of 500~600°C, the average grain size was about 13nm and did not significantly change on the annealing temperature, while it dramatically increased to 38nm in the ribbon annealed at 650°C. This result indicates that the change in permeability with annealing temperature is not attributed only to the change in the grain size under given annealing conditions.

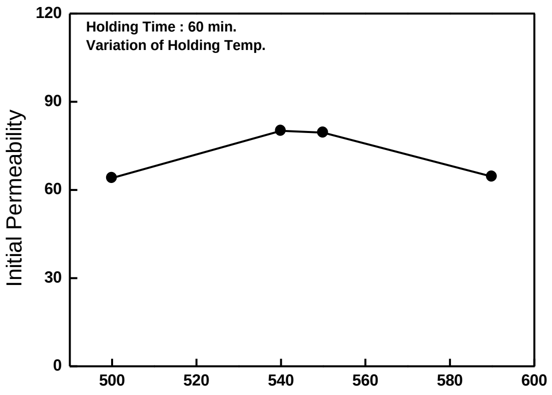

Figure 1. The variation of initial permeability with annealing temperature.

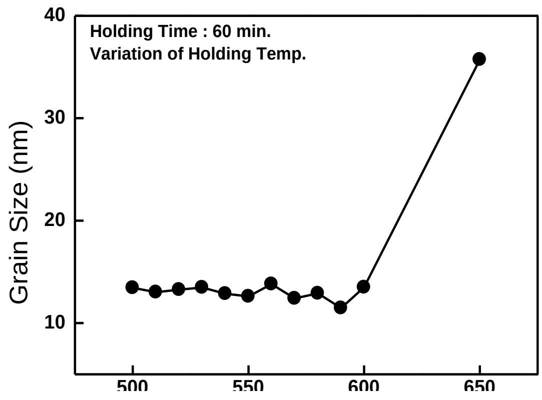

Figure 2. The variation in average grain size with annealing temperature.

One of the factors to affect the magnetic properties of FeSiBNbCu alloy is the volume fraction of bcc-Fe(Si) nanocrystalline phase, which is embedded in the amorphous matrix after annealing. Figure 3 shows DSC curves of the amorphous FeSiBCuNb alloy observed at various heating rates. The crystallized fraction of the annealed specimens can be estimated using the equations (1) and (2) [8-9],

$$d\left[\ln\left(\frac{dx}{dt}\right)_p\right] / d\left(\frac{1}{T_p}\right) = -\frac{E}{R} \quad (1)$$

$$\left(\frac{dx}{dt}\right)_p = 0.37nK_p \quad (2)$$

where R is the gas constant, T_p is the peak temperature in absolute unit, K_p is the rate constant that equals to E/RT_p, $(dx/dt)_p$ is the maximum crystallization rate and $x(t)$ is the volume fraction of crystallized portion at time, t. Table 1 summarizes the kinetic parameters estimated from the DSC curves shown in Fig. 3.

The crystallized fraction was identified to increase with increasing annealing temperature. Figure 4 shows the variation of initial permeability of FeSiBNbCu alloy on volume fraction of bcc-Fe(Si) phase (f_c). The initial permeability increased with increasing volume fraction of bcc-Fe(Si) phase to f_c = 0.77 where it showed a maximum and maintained its value without significant decrease until f_c = 0.96. This result indicates that the partially crystallized FeSiBNbCu alloy has better magnetic properties than that of fully amorphous or fully crystallized alloys. The optimum volume fraction of nanocrystals in the amorphous matrix showing the best initial permeability appears to be at range of f_c = 0.8~0.9.

Table 1. DSC results and kinetic parameters.

Margin	1^{st} **Peak**	2^{nd} **Peak**
T^a_{onset} (^oC)	509	690
T^a_{peak} (^oC)	528	701
ΔH (kJ/kg)	79	15
E (eV)	3.3	4.3
n	1.42	2.4

[a] 10 ^oC /min

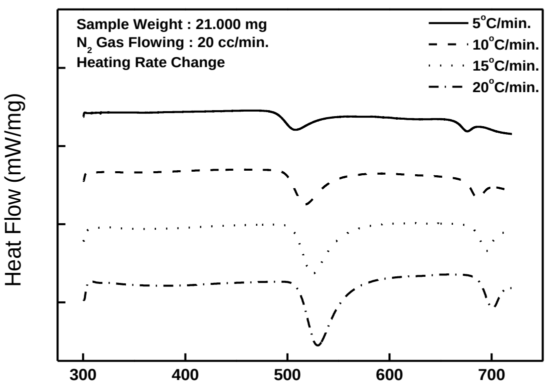

Figure 3. The Results of DSC analysis of amorphous FeSiBNbCu alloy at different heating rates.

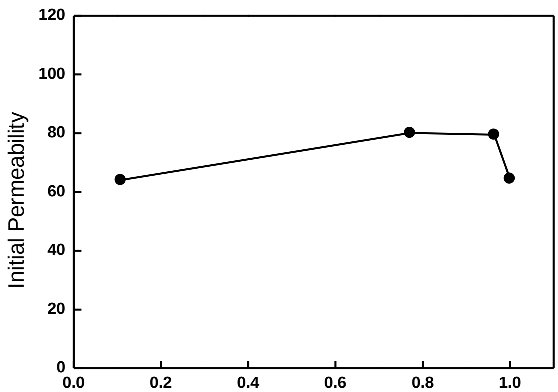

Figure 4. The variation of initial permeability of FeSiBNbCu alloy on volume fraction of bcc-Fe(Si) phase(f_c).

3.2. EM wave absorption behavior

Figure 5 and 6 show the variation of the complex permeability and the EM wave absorption behavior on the frequency for the sheet crystallized at different conditions. All of the specimens were oxidized at 400°C for 7 hours after crystallization annealing to insulate the surface of particles. The results on the real part and imaginary part of the relative permeability over the frequency ranges from 10MHz to 5GHz for the specimens crystallized at different temperature represented that the alloy crystallized at 510°C for 1hour showed the highest relative permeability in both parts, especially at the high frequency range. Finemet type of nanocrystalline alloys has been reported to exhibit best soft magnetic properties when annealed for crystallization at 550~560°C for 1hour. However, the EM wave absorption sheet made of FeSiBNbCu powder clearly showed that the EM absorption efficiency was greater for the specimen crystallized at 510°C for 1hour as shown in Fig. 6. This result indicates that the significant level of deformation or impact such as pulverizing and subsequent milling during the processing somewhat shifted the optimum annealing temperature to lower side because of the accumulated internal energy during fragmentation.

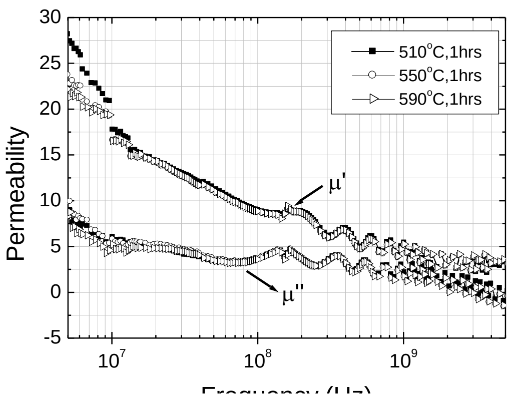

Figure 5. The complex relative permeability curve at various annealing conditions of FeSiBNbCu alloy.

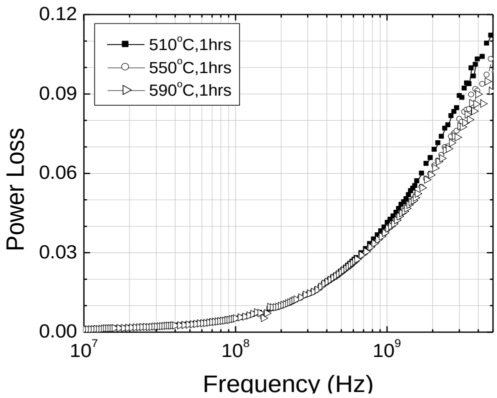

Figure 6. The EM wave absorption curve at various annealing conditions of FeSiBNbCu alloy.

4. Conclusions

1. In the temperature range of 500~600°C, the average grain size was about 13nm and did not significantly change with annealing temperature. However, it dramatically increased in the ribbon annealed above 650°C.
2. The initial permeability increased with increasing the volume fraction of nanocrystals (f_c) in amorphous matrix to 0.77. The partially crystallized FeSiBNbCu alloy has better magnetic properties than that of fully amorphous or fully crystallized alloys. The optimum volume fraction of nanocrystals in the amorphous matrix showing the best initial permeability appears to be at range of f_c = 0.8~0.9.
3. The EM wave absorption efficiency of the sheet made of FeSiBNbCu alloy powder was greatest for the specimen crystallized at 510°C especially at high frequency ranges.

5. References

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