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EFFECT OF FLASH ANNEALING ON MAGNETIC PROPERTIES OF Fe-BASED NANOCRYSTALLINE ALLOYS

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Abstract- A heat-treatment method of pre-annealing and then flash annealing(FA) has been used to improve the soft magnetic properties of nanocrystalline $Fe_{76}CuSi_{13}B_{10}$ and $Fe_{74}CuNb_3Si_{12}B_{10}$ alloys. Outstanding magnetic properties of nanocrystalline $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy were attained by flash-annealing in air after annealed at 500°C for 0.5hr below the crystallization temperature. The same results were obtained for $Fe_{74}CuSi_{13}B_{10}$ alloy. The measurment of relief of stress and X-ray diffraction were used to analyze the effect of flash-annealing.

I. INTRODUCTION

A recent example of nanocrystalline amorphous materials is the devitrified Fe-Cu-Nb-Si-B. The alloy is initially casted as an amorphous ribbon. A subsequent heat-treatment for longer time above the crystallization temperature produces an ultrafine structure of Fe-Si embeded in a disordered matrix. In the nanocrystalline stata, the material exhibits outstanding soft magntic properties, even better than the results for the corresponding amorphous materials.

For nanocrystalline amorphous materials Fe-Cu-Nb-Si-B, heat-treatment for a long time causes possibly disparity of structrue, especially grain size, and makes soft magnetic properties to be worse. There were some reports(1)(2)(3) which found that nanocrystalline can be created in Fe-based amorphous alloy by flash annealing. But in our previous experiments this phenomenon did not happen for $Fe_{75}Mn_6Si_8B_{11}$ alloy(4), and only stress of amorphous alloy cxan be relieved completely. In order to make crystalline nucleus form easily and subsequently to crystallize, the method of pre-annealing for a loge time at the lower temperature than the crystallization one, and flash annealing was developed(5). Outstanding magnetic properties were obtained by this method for $Fe_{75}Mn_6Si_8B_{11}$ alloy. In this paper, the same method was applied to investigate how effect there were on soft magnetic properties of $Fe_{76}CuSi_{13}B_{10}$ and $Fe_{74}CuNb_3Si_{12}B_{10}$ nanocrystalline amorphous alloys.

II. EXPERIMENT

Amorphous $Fe_{76}CuSi_{13}B_{10}$ and $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy ribbons were prepared by the single roll melt spinning metyhod. The samples were 7mm wide and 20 m thick. They were wound into toroidal cores and annealed at below their crystallization temperature in N2 gas atmosphere, and flash annealed by causing pulse current of 10A for varied time in air. The FA temperature was roughly eastimated according to the caculated Joule's heat. The structure of the samples was examined by X-ray diffractometer using Cu Kα radiation. The M-T curve were obtained by means of vibrating samples magnetometer(VSM). Their static and high frequency magnetic properties were measured by using B-H loop tracer.

III. RESULTS AND DISCUSSION

The crystallization process may consist of the formation of crystalline nucleus and the subsequent crystalline growth. The curie and crystallization temperature is 415°C and 445°C for $Fe_{76}CuSi_{13}B_{10}$ alloy, and 335°C and

520°C for $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy according to M-T curves. The temperature of preannealing for formation of crystallin nucleus was fixed at 420°C For $Fe_{76}CuSi_{13}B_{10}$ alloy, 400°C and 500°C for $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy respectively.

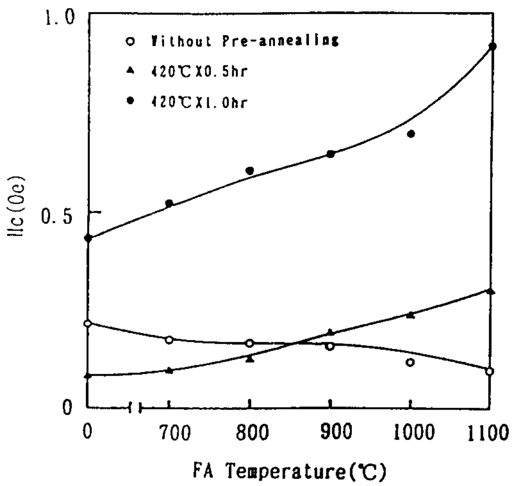

Fig.1 Relationship of flash-annealing temperature and coercive force Hc for variously pre-annealed $Fe_{76}CuSi_{13}B_{10}$ (Hm=80A/m)

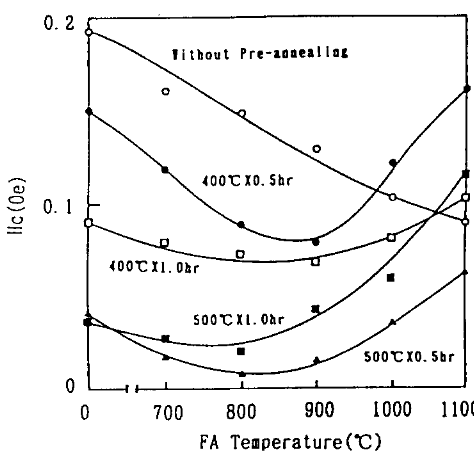

Fig.2 Relationship of flash-annealing temperature and coercive force Hc for variously pre-annealed $Fe_{74}CuNb_3Si_{12}B_{10}$ (Hm=80A/m)

The FA temperature dependence of coercive force Hc under maximum field of 80A/m for variously pre-annealed $Fe_{76}CuSi_{13}B_{10}$ and $Fe_{74}CuNb_3Si_{12}B_{10}$ amorphous alloys are shown in Fig.1 and Fig.2 respectively. From Fig.1, coercive force Hc of $Fe_{76}CuSi_{13}B_{10}$ alloy without pre-annealing decrease slowly with increase of FA temperature. But for alloy with pre-annealing, Hc increases with raising FA temperature. It is concluded that magnetic properties of this alloy can not be improved by means of this kind of way.

From Fig.2 Hc of $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy without pre-annealing decrease until at 1100°C of temperature. Differently from $Fe_{76}CuSi_{13}B_{10}$ alloy, Hc of this alloy with pre-annealing reaches to the minimum value at optium FA temperature and increases with raising FA temperature. The lowest Hc value as low as 0.8A/m was obtained for the sample which had been annealed at 500°C for 0.5hr, and then was subsequently flash annealed at 800°C.

The variation of effective permeability at 0.8A/m and 1kHz andcore losses Pc at 0.2T and 100kHz with raising FA temperature for sample pre-annealing at 500°C for 0.5hr are shown in Fig.3. It can be seen that μ and Pc of $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy reaches to maximum and minimum

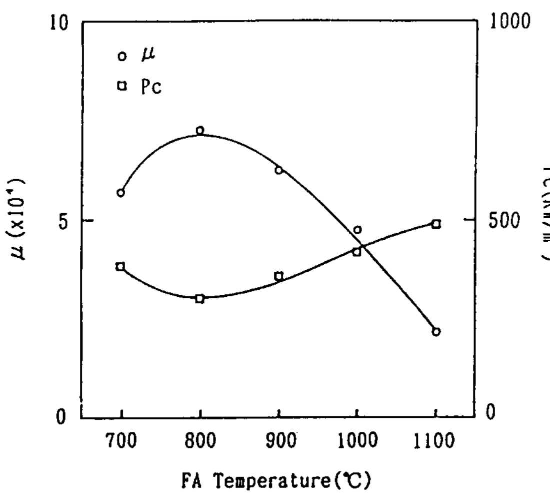

Fig.3 Effect of flash-annealing temperature on effective permeability μ (Hm=0.8A/m, f=1kHz) and core losses Pc (Bm=0.2T, f=100kHz) of $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy pre-annealed at 500°C for 0.5hr

respectively when FA temperature is 800°C at which Hc is also the lowest shown in Fig.2.

The ratio of circle radius(r_o) of wounnd ribbon to that(r) of loosed ribbon after flash-annealing $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy at 500°C for 0.5hr or not and $Fe_{76}CuSi_{13}B_{10}$ alloy at 420°C for 0.5hr with relation to FA temperature are shown in Fig.4. For $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy, the stress relife process for pre-annealed sample is nearly accomplished after flash-annealing at 800°C.

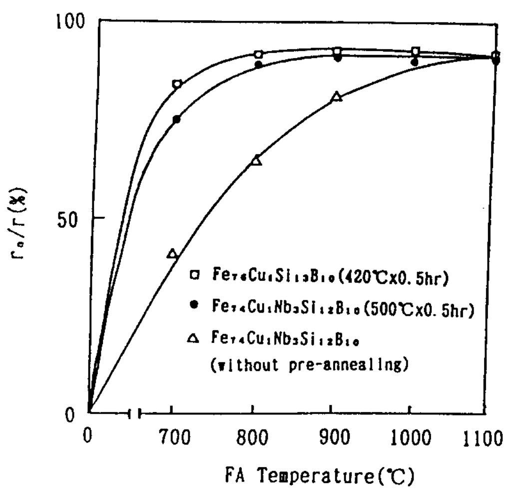

Fig.4 Flash-annealing temperature dependence of the ratio of circle radius r_0 of wound ribbon to that r of loosed ribbons with and without pre-annealing

However, FA temperature higher than 1000°C is needed for the sample without pre-annealing to relieve the stress. Compared with $Fe_{76}CuSi_{13}B_{10}$ and $Fe_{74}CuNb_3Si_{12}B_{10}$ alloys, velocity of stress relief for the former is quicker than that for latter.

The results of X-ray patterns of $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy with pre-annealing and then flash-annealing are shown in Fig.5. The reduction of Hc with FA temperature up to 800°C for the sample pre-annealed at 500°C for 0.5hr may be attributed to the stress relief with flash-annealing. The increase of Hc withraising FA temperature higher than 900°C may be based on the

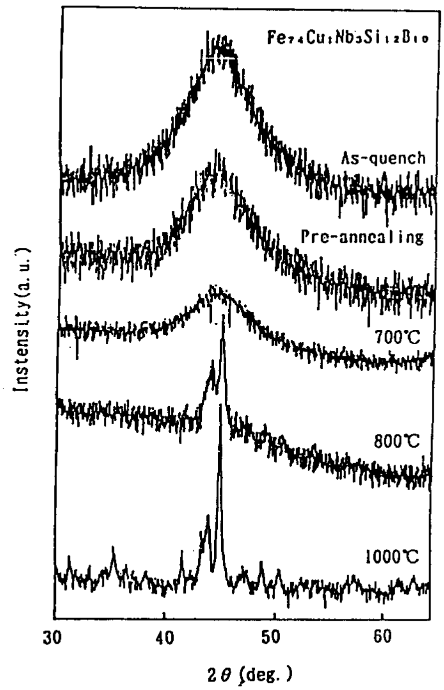

Fig.5 X-ray patterns of $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy with various states

crystallization and growth. The lowest Hc at FA temperature 800°C is consided to be based both the complete stress relief and the formation of nanocrystalline.

Ⅳ.CONCLUSION

(1) The magnetic properties of $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy were improved by means of pre-annealing and then flash-annealing. But this method was not applicable fór $Fe_{76}CuSi_{13}B_{10}$ alloy.
(2) Best D.C. and A.C. magnetic properties for $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy were obtained after pre-annealing at 500°C for 0.5hr and flash-annealing at 800°C.
(3) The reason of chang of magnetic properties in $Fe_{74}CuNb_3Si_{12}B_{10}$ alloy may be consideed to be attributed by complete stress relief and formation of nanocrystalline.

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REFERENCE

[1] A.R.Yavari et al, J.M.M.M., 69(1987)43.
[2] J.Gonzalez et al, J.Phys., D.21(1988)162.
[3] M.A.Escobar et al. IEEE Trans.Mag., MAG -28(1992)1991.
[4] B.Quan & K.Narita, Materials Science and Engineering, A181(1994)969.
[5] X.Yu & K.Narita, J.M.M.M., 133 (1994)344.