acta physica slovaca vol. 46 No. 2, 151 – 154 April 1996

CONNECTION BETWEEN AMORPHOUS AND NANOCRYSTALLINE FINEMET ALLOY PROPERTIES[1]

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Coercive force and hysteresis loss behave differently in amorphous and nanocrystalline finemet alloys depending on the thickness of the rapidly quenched ribbons. This can be interpreted by an atomic rearrangement which takes place before or during nanocrystallization in the thin ribbon and at higher temperatures in the thicker one.

1. Introduction

The finemet nanocrystalline alloy with the traditional composition $Fe_{73.5}Nb_3Cu_1Si_{13.5}B_9$ [1] is a very good soft magnetic material. This nanocrystalline alloy can be prepared from the amorphous one by suitable heat treatment. After such treatment the material contains Fe(Si) nanocrystals of ≈10 nm which are surrounded by an amorphous matrix. It was shown that the advantageous soft magnetic properties are due to the simultaneous disappearance of magnetic anisotropy and magnetostriction [2]. This balance between the magnetostriction of the amorphous matrix and the crystallites is very sensitive to the α-Fe(Si) phase composition and to the crystalline fraction both of which can be influenced by the thermo-mechanical history of the nanocrystalline material. If the Si content in α-Fe(Si) solid solution is increased both the lattice parameter and the (negative) magnetostriction decrease. Good balance can be achieved only at appropriate Si content (≈20 at %) and crystalline fraction (≈80 %) [3]. The thermo-mechanical history of the nanocrystalline finemet has two parts: the thermal history of the amorphous precursor and that of the crystallization process. In this paper we deal with both. The thermal history of the amorphous precursor was varied by changing the cooling rate during rapid quenching, i.e. the thickness of the ribbon. The crystallization process was varied using various T_{ann} annealing temperatures and t_{ann} annealing times.

[1] Presented at 9th Czech and Slovak conference on magnetism, Košice, Slovakia, August 28-30 1995
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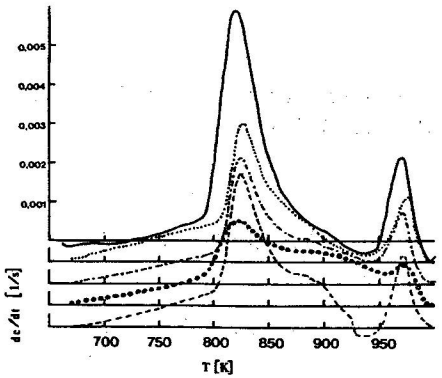

Fig. 1. DSC curves for as-quenched finemet ribbons with different ribbon thicknesses (different in-situ heat treatment): —— 16 μm; ..... 21 μm; -.-. 25 μm; o o o 30 μm; - - - 23 μm (from homogenized melt). Heating rate 20 K/min.

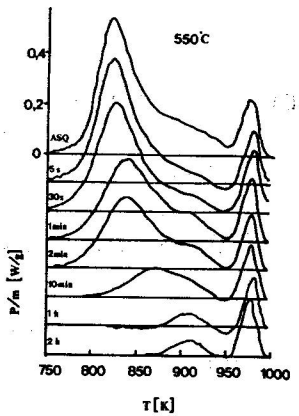

Fig. 2. DSC curves of as-quenched and at 823 K/ t[min] nanocrystallized, 30 μm thick samples; t[min] is the parameter. Heating rate during measurement 40 K/min.

2. Experimental methods

Amorphous ribbons 10 mm wide and of various thicknesses in the range 16 – 30 μm were rapidly quenched from the same alloy of $Fe_{73.5}Nb_3Cu_1Si_{13.5}B_9$ composition. The different ribbon thicknesses correspond to different in-situ heat treatments during quenching so these ribbons had different thermo-mechanical histories in the amorphous phase. In the as-quenched state all the ribbons were X-ray amorphous.

The thermally activated phase transformations of the amorphous ribbons were investigated by a Perkin-Elmer DSC-2 differential scanning calorimeter.

The short time heat treatments were performed in a tin bath for sheet samples of 6 cm length. Longer heat treatments were done on sheet samples and also on toroidal samples in protective Ar atmosphere. The overlap between the two types of heat treatment showed that the heat treatments were equivalent.

A Foner magnetometer was used for the magnetization measurements and the dynamical hysteresis loops were measured on toroids by a home-made, automated measuring system [4], at room temperature.

3. Results and discussion

Calorimetric study of the amorphous materials gives valuable information about the thermal properties of the material. In Fig.1 the DSC curves of as-quenched finemet alloy are shown for ribbons with different thicknesses. It can be seen that the curves differ from each other. The first peak corresponds to the nanocrystallization. In each curve this peak is asymmetric. In the thinner ribbons a shoulder can be seen at the higher temperature side from which a second peak was formed in the thickest sample.

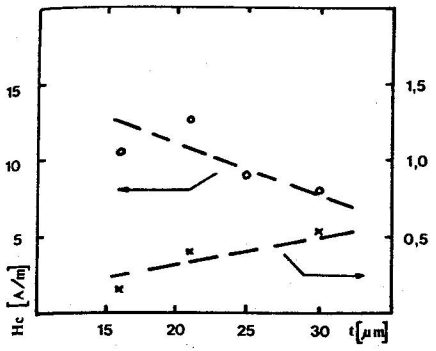

Fig. 3. Coercive force measured at room temperature in as-quenched amorphous ribbons, - o -, and in nanocrystallized ones (823 K/1h), - x -, versus of ribbon thickness.

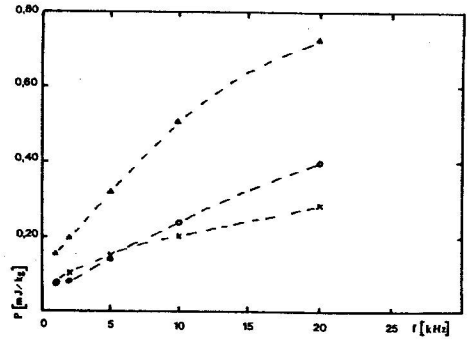

Fig. 4. Hysteresis loss, P [mJ/kg], versus measuring frequency, f [kHz] of 21 μm thick ribbon heat treated at 823 K for various t[h] annealing times, t: - o - 1h; - x - 5h; and of 30 μm thick ribbon heat treated at 823 K for 1h: - Δ -.

The same second peak can be seen in the curve of the "homogenized" ribbon (this ribbon was quenched from homogenized melt keeping the melt for some minutes in a high frequency field before quenching). The last peak corresponds to the crystallization of the amorphous matrix – which previously surrounded the nanocrystals – into Fe_2B. We postulate that the second peak in the thick ribbon corresponds to some atomic rearrangement in the amorphous or in the nanocrystalline phase.

To gain more information about this second peak another series of investigations were made on the 30 μm thick ribbon. The amorphous pieces of ribbon were heat treated at 550 °C for various annealing times from 5 sec to 2 hours and then the DSC curve was measured on each sample, see Fig. 2. After 1 min the first peak decreased noticeably – because of the onset of nanocrystallization – and the second peak became more evident. The second peak can be found even after 2 hours heat treatment at 550 °C. This strengthens our postulation that there is some atomic rearrangement in this temperature interval in the 30 μm thick ribbon. In the thinner ribbon this rearrangement may take place before or together with the nanocrystallization.

In order to see whether the differences in the thermal properties have any consequences on the magnetic behaviour in Fig.3. the coercive force, H_c, is shown as a function of the ribbon thickness in the as-quenched state and after a 550 °C/1h heat treatment. In the amorphous state H_c decreases with increasing ribbon thickness but in the nanocrystalline state it increases with increasing thickness. This shows that the thinner samples become softer than the thicker ones. Perhaps this was because the material did not go through the atomic rearrangement belonging to the second peak.

Optimum conditions are given in these 30 μm thick ribbons by the 550 °C/1h-2h heat treatments though better soft magnetic properties can be achieved in the thinner samples. This can be seen in Fig. 4 where the hysteresis loss is shown as a function of measuring frequency for 21 μm ribbon heat treated at 550 °C for 1 and 5 hours together with the hysteresis loss of a 550 °C/1h heat treated 30 μm ribbon. It is interesting that

hysteresis loss decreases with increasing annealing time in spite of the fact that with increasing annealing time the grain size increases from 8 nm to 12 nm [5]. It is explained by the fact that magnetostriction measured in this material continues to decrease even after 5 hours heat treatment [6].

4. Conclusions

The effect of thermo-mechanical history of the amorphous precursor and that of the crystallization process were investigated in the finemet nanocrystalline alloy. It was found that the coercive force and hysteresis loss are smaller in the thicker ribbons in the amorphous phase the situation is reversed but in the nanocrystalline phase. This is interpreted by an atomic rearrangement which takes place before or during nanocrystallization in the thin samples but only at higher temperatures in the thick, or homogenized sample. In the thinner ribbons which were quenched with higher cooling rate the amorphous structure is more inhomogeneous than in the thicker ribbons which have got an in-situ heat treatment during quenching resulting relaxation in their structure or in the homogenized ribbon in which the amorphous structure is quite homogeneous. The inhomogeneous structure promote any changes therefore in the thin ribbons the atomic rearrangement necessary for having more and more nanocrystalline nuclei can be carried out at lower temperature. These changes were demonstrated by TEM and the results will be published later. It is concluded that finemet ribbons of 16-20 μm thickness heat treated at 550 °C (823 K) for several hours have the best soft magnetic properties.

Acknowledgement The assistance of the Institute of Physics of the Slovak Academy of Sciences in preparing the ribbons is gratefully acknowledged. This work was supported in part by PECO Project Contract Nr. CIPA-CT93-0239 and by the OTKA Fund of Hungary through grant No. T-2954.

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