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X-RAY STUDY OF NANOCRYSTALLINE RIBBONS FeCuNbSiB SUBJECTED TO THERMOMECHANICAL AND THERMOMAGNETIC TREATMENTS

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An attempt to discover the structure peculiarities giving rise to induced magnetic anisotropy in a finemet subjected to thermomechanical or thermomagnetic treatment has been undertaken. Grain size, internal stresses and texture in FeCuNbSiB ribbons were investigated. It was concluded that the induced magnetic anisotropy must have another reason *e.g.*, directional ordering of Si atoms.

Keywords: Finemet; Thermomechanical treatment; Induced magnetic anisotropy; Structure peculiarities

INTRODUCTION

The nanocrystalline alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ (at.%) is produced by annealing a quenched amorphous ribbon at a temperature of 530°C during 1 hour (Finemet). This alloy shows high soft magnetic properties. The constant of magnetic anisotropy is close to zero.

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In the nanocrystalline alloy subjected to annealing under load at a temperature equal to or above the crystallization temperature, magnetic anisotropy is induced. The magnetic anisotropy constant is maximum when the thermomechanical treatment proceeds simultaneously with the crystallizing annealing (Glazer *et al.*, 1991). A weak magnetic anisotropy is induced after the crystallizing annealing in a constant magnetic field applied in the direction perpendicular to the long axis of a ribbon (Tab. I).

The reasons for the induced magnetic anisotropy in a nanocrystalline alloy haven't been clarified, yet. One can suggest different structure changes upon the treatments mentioned above. Firstly, annealing under load or magnetic field might favor aligning the crystallite directions (texture). Secondly, external actions could cause internal lattice microdistortions which promote magneto–elastic interactions inside the FeSi grains and could thus be responsible for the induced magnetic anisotropy. Finally, such kinds of treatments can lead to lining up of pairs of Si atoms along a definite direction (Neél, 1954).

Contradictory data on texture formation are presented in the literature. Thus, Hofmann and Kronmüller (1996) consider texture in the nanocrystalline alloy FeCuNbSiB to be absent after thermomechanical treatment. However, their conclusion is based only on the fact that the grains in the structure are equiaxial. On the contrary, Lu and Szpunar (1994) discovered a texture in the nanocrystalline alloy FeCuNbSiB after a conventional annealing without a load. They even discuss the variations of texture between different sides of the ribbon. Such discrepancy is possibly related to the peculiarities of texture analysis in case of nanocrystalline alloy, which requires application of special methods. The purpose of this work was to study different structure changes that accompanied the origination of induced magnetic anisotropy.

TABLE I Magnetic anisotropy constant for nanocrystalline ribbons after different variants of treatment

Treatment	K_u, J/m^3
1 (CRYS)	0
2 (TMT1)	−4000
3 (TMT2)	−2000
4 (TMagT)	−100

EXPERIMENTAL

Amorphous FeCuNbSiB ribbons 2 mm wide and 20 μm thick were produced by quenching on a wheel and then were treated by the following regimes:

(1) Crystallization at 530°C during 1 hour (CRYS);
(2) Crystallization at 530°C during 1 hour under a load of 200 MPa (Thermomechanical treatment 1 – TMT1);
(3) Thermomechanical treatment at 530°C during 1 hour of the preliminary crystallized (by regime 1) sample (Thermomechanical treatment 2 – TMT2);
(4) Crystallization of 530°C during 1 hour under a constant magnetic field, H, of 150 A/m directed perpendicular to the long axis of the ribbon.

During the treatments by the last three modes, an induced magnetic anisotropy arose (see Tab. I). After the regime 2 (TMT1) the sample was plastically elongated by 20%, after the regime 3 (TMT2) no elongation was revealed.

Texture investigation was carried out by X-ray diffraction with Co-radiation using a position sensitive detector (Wcislak and Bunge, 1996). Three incomplete (up to $\alpha = 70°$) pole figures of the integral intensity {110}, {200} and {112} were measured. A pole figure of the integral intensity is obtained by using the intensity data from the whole diffraction profile colleted with a position sensitive detector and multichannel analyzer. This technique was chosen because the diffraction peaks from the nanocrystalline substance are rather broad and the texture variations expected could be small.

The determination of grain size and internal elastic microdistortions was carried out by the Warren – Averbach method (linear version).

TEXTURE AND STRUCTURE INVESTIGATIONS

The texture analysis with a position sensitive detector of the samples after treatments by all four methods did not reveal any texture. Figure 1 shows some measured diffraction peaks for the samples with maximum and minimum magnetic anisotropy. They look very similar.

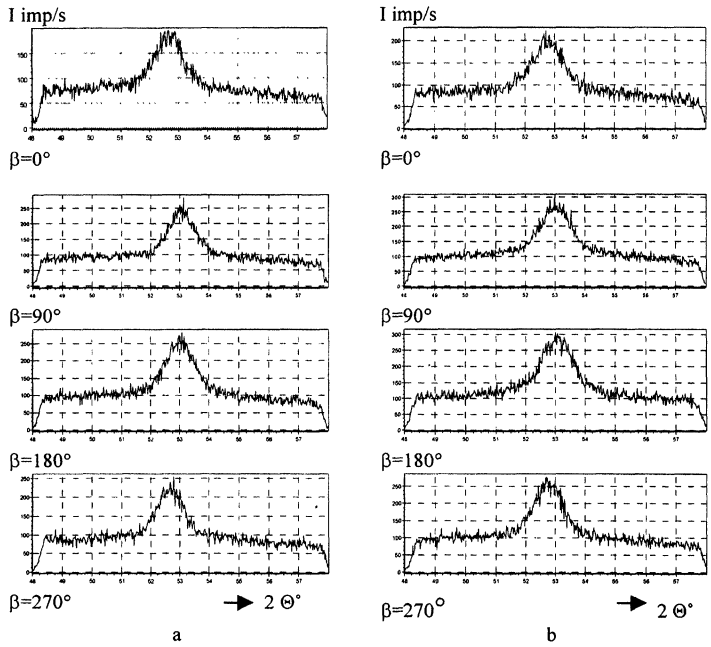

FIGURE 1 Diffraction peaks {110} measured at the tilt angle $\alpha = 45°$ at different rotation angles β as indicated in the figure. (a) the crystallized sample CRYS, (b) the sample after TMT1.

X-ray diffraction patterns were also measured with Mo-radiation for a standard sample (Fe-powder), for the crystallized ribbon (CRYS), and for the ribbons after TMT1 and TMT2. From these, grain size (domains of coherent scattering) and internal elastic microdistortions were calculated using the Warren–Averbach method. The results are represented in Table II.

One can see that the grain size after the treatments 1–3 is almost the same (5–6 nm). It is worth noting that the value of grain size (domains of coherent scattering) measured by X-ray diffraction should be less than that obtained in the electron microscope because of broad distorted boundary areas. So, these data are in good agreement with the data of TEM investigations of the same alloy, where the grain size was found to be about 10–12 nm (Hofmann and Kronmüller, 1996; Yoshizawa, Oguma and Yamauchi, 1988). The value of the internal elastic microdistortions increases in the case of the combined

TABLE II Grain size (d) and internal elastic microdistortions (e) for the samples after different regimes of treatment

Treatment	*d, nm*	*e, %*
1 (CRYS)	5	2.7
2 (TMT1)	6	3.1
3 (TMT2)	6	1.2

treatment TMT1, but in the case of sequential treatment TMT2, this value is even less than in the conventionally crystallized sample (CRYS). This seems strange at the first sight, but it can be explained with a glance to the conditions of the treatment of this sample. The sample subjected to TMT2 was held at the temperature of crystallizing annealing during two hours while the conventionally crystallized one (CRYS) was exposed to 1 hour annealing. During this additional annealing, the residual amorphous phase continued crystallizing and the elastic microdistortions in the conventionally crystallized sample with the zero magnetic anisotropy exceeds that in the sample after TMT2 which shows a rather high value of the induced magnetic anisotropy. This means that the internal stresses are not the main cause for the induced magnetic anisotropy.

CONCLUSIONS

Using a highly sensitive method of texture analysis, texture could be excluded as a possible reason for the magnetic anisotropy induced in FeCuNbSiB alloys after thermomechanical or thermomagnetic treatment. Also, grain size and internal elastic microdistortions did not correspond to the values of induced anisotropy and may thus be also excluded as possible reasons. Hence, it is concluded that directional ordering of Si atoms may be the most probable explanation for this effect, but this requires further investigations.

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