11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25
ICAM2009

Production of nanocrystalline $Fe_{74,3}Si_{14,2}Cu_1Nb_3B_{7,5}$ alloy for magnetic sensors

G. Perez[(1)]*, G. Solorzano[(1)], L. C. C. Benyosef[(2)]

(1) DCMM, Pontifícia Universidade Católica do Rio de Janeiro, Rua Marques de São Vicente 225, Gavea, Rio de Janeiro - RJ 22453-900, Brazil.
(2)Observatório Nacional, Rua General José Cristino, 77 , São Cristovão, Rio de Janeiro - R.J. 20.921-400 Brazil.

Abstract - Ribbons of amorphous alloys of $Fe_{74,3}Si_{14,2}Cu_1Nb_3B_{7,5}$ were prepared by vacuum melt-spinning. These amorphous ribbons were annealed at 554 °C for 1 h, this annealing produce the formation of the DO_3 FeSi nanostructured phase embedded within an intergranular amorphous phase. X ray diffraction (XRD) was used to verify the structure, before (as spun) and after of the annealing. It was observed by transmission electron microscopy (TEM) that the size of the nanocrystals is around 10 nm. The magnetic characterization of the ribbons, before and after annealing, was made by vibrating sample magnetometer (VSM).

The family of FeSiBCuNb alloys, also known as FINEMET, developed in 1988 by Yoshizawa et al, has been showing excellent soft magnetic properties. These alloys combine both high permeability and high saturation. This material have a nanocrystalline structure composed of bcc Fe-Si supersaturated solution, the α-Fe(Si) phase, embedded in the remaining amorphous matrix. The nanostructure is produced by annealing at temperatures around 550 °C of amorphous ribbons obtained by melt-spinning [1]. The local content of Si into bcc nanograins is bigger than the mean content of alloy. The composition of the nanograins is around Fe_3Si, that result on DO_3 order [2]. The size of the DO_3 order region and the degree of order of the α-Fe(Si) phase increase with increasing annealing temperature [3].

The material was produced as amorphous ribbons using the melt-spinner. The melt spinner was set with the follow parameters: The quartz crucible used had a hole of 1,7 mm of diameter, the distance between wheel and crucible was 0,3 mm. The ejecting temperature of alloy was 1350 °C, near 200 °C above of the melting point of alloy. The speed of the wheel was 48 m/s, the pressure of the crucible was 100 mbar and the camera pressure was 20 mbar. The ribbon obtained had a thickness of 40 μm and the width is 1,8 mm. Later the amorphous ribbons were annealed in an programmable electric furnace with a heating rate of 10 °C/min and kept at 554 °C for 1 h to obtain de nanocrystalline structure.

The material was examined by transmission electron microscopy (TEM) to determine grain size. Figure 1 shows nanograins in the order of 10 nm. Most of nanocrystals have a spheroidal shape. Faceted nanocrystals were not observed. This spherical equilibrium shape is due of the presence of intergranular amorphous phase between the grains. Figure 2 shows two diffractograms, one before (a) and the other after (b) annealing. The difractogram of sample as spun (a) is typical of amorphous structures, while the difractogram of annealed sample (b) shows typical peaks of the Fe_3Si crystalline structure.

The soft magnetic properties were enhanced due to formation of nanocrystalline structures produced by annealing. The annealed sample show high saturation and low coercivity.

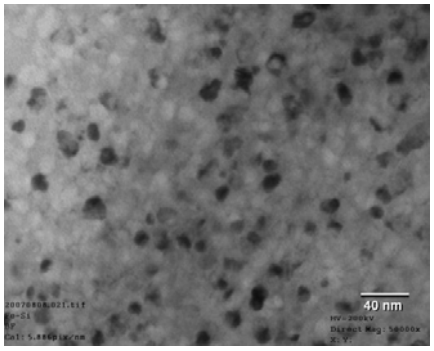

Figure 1: TEM image of nanocrystals of diameter around 10 nm (50.000 x).

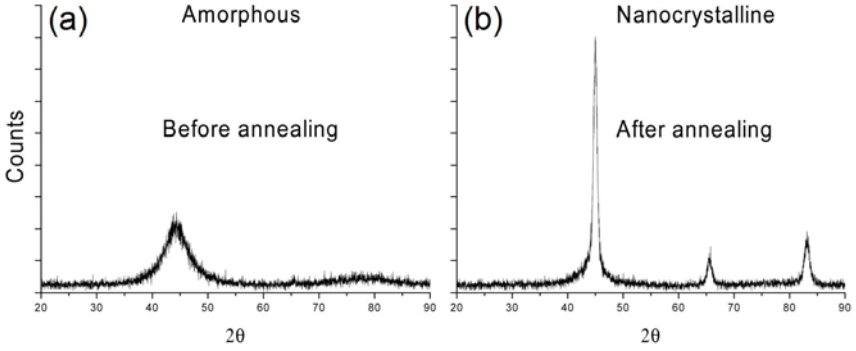

Figure 2: XRD graphic of material before and after annealing. The graphic of not annealed sample (a) don't show peaks, typical for amorphous structure. The annealed sample (b) show peaks of α-Fe(Si) phase.

References

[1] Y. Yoshizawa, S. Oguma, and K. Yamauchi, New Fe-based soft magnetic alloys composed of ultrafine grain structure, J.Appl. Phys. 64 (10), 15 November 1988.
[2] G. Herzer, Nanocrysalline soft magnetic alloys, Handbook of Magnetic Materials, Vol. 10, Ed. K.H.J. Buschow, 1997.
[3] X. Y. Zhang, J. W. Zhang, F. R. Xiao, J. H. Liu, R. P. Liu, J. H. Zhao, Y. Z. Zheng, Ordering of the crystalline phase á-Fe(Si) in annealed $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy, Materials Letters 34 85-89, 1998.