

AC magnetic susceptibility and influence of heat treatment on obtaining the nanocrystalline structure for the amorphous alloy of $\text{Fe}_{37}\text{Co}_{35}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ composition

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Abstract. This work presents a study of AC magnetic susceptibility and the influence of heat treatment on obtaining the nanocrystalline structure for the amorphous alloy of $\text{Fe}_{37}\text{Co}_{35}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ composition. AC magnetic susceptibility at different frequencies and sample lengths, using a variable magnetic field was measured. The influence of heat treatment on obtaining the sample nanocrystalline structure was evidenced by means of XRD technique. The crystallite size, the crystalline volume fraction and the distance between grains for each heat treatment temperature were calculated using the XRD peaks refinement software GSAS. Samples were obtained in the amorphous state by melt spinning technique and all measurements by AC magnetic susceptibility and XRD were performed at room temperature.

1. Introduction

During the last 15 years, the importance of soft magnetic FeCo based alloys has increased due to their excellent soft magnetic properties, which allowed the development of improved technological applications such as magnets and sensors for high temperatures [1-3]. Nanocrystalline magnetic alloys show superior soft magnetic properties when their amorphous phase is submitted to a controlled heat treatment [1-4]. In Colombia, over the last decade, there has been a growing interest in research on Fe based soft magnetic materials, mainly because these alloys exhibit interesting magnetic phenomena [5-7]. However, AC magnetic susceptibility of FeCo-based soft magnetic alloys has not been studied yet. In this work, AC magnetic susceptibility, as a function of frequency and length of $\text{Fe}_{37}\text{Co}_{35}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ amorphous alloy, has been studied. The sample was prepared by the melt spinning technique and then was partially crystallized by heat treatments from room temperature up to different temperatures, in which crystallization process occur. The evolution of the crystallization process with the heat treatment temperatures increase was evidenced by XRD technique. From the XRD diffraction maxima refinement, the crystallite size, the crystalline volume fraction and the distance between grains [1] were obtained according to the heat treatment temperature. The evaluation of these parameters is crucial for the achievement of excellent soft magnetic properties.

2. Experimental procedure

Amorphous ribbons of $\text{Fe}_{37}\text{Co}_{35}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ composition were prepared by the melt spinning technique. AC magnetic susceptibility measurements was performed on an AC susceptometer at the Magnetism and Advanced Materials Laboratory from Universidad Nacional de Colombia, sede



Manizales, using a frequency range between 10Hz and 5kHz and sample lengths between 0.4 and 1cm, with a constant amplitude of about 1Vrms. The magnetic field was varied from -74 to 74Oe. To obtain the nanocrystalline structure, amorphous samples were heat treated from room temperatures up to 550, 600, 650, 700 and 750°C in a GSL-1500X (MTI) vacuum furnace. DRX analysis was performed on a PANalytical X'Pert diffractometer from the DRX Laboratory of Universidad del Valle. Measurements were made at room temperature using Cu radiation, over 30-90° 2 θ and 0.54°/2.22s step. The crystallite size and the crystalline volume fraction were estimated taking into account the XRD peak refinement software GSAS (General Structure Analysis System) and the distance between adjacent grains was calculated using the method of the literature [1].

3. Results and discussion

3.1. AC magnetic susceptibility measurements

Figure 1 shows real (a) and imaginary (b) components, χ'_{AC} and χ''_{AC} , of the AC magnetic susceptibility as a function of the applied magnetic field, for the $\text{Fe}_{37}\text{Co}_{35}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ alloy at a frequency of 40Hz at room temperature. A peak at zero magnetic field, corresponding to the maximum value of the AC magnetic susceptibility, is observed. This behaviour could be explained in terms of the domain walls movement, as it was indicated by O. Moscoso [7]. By the other side, the narrower peak observed in the χ''_{AC} imaginary part, is attributed to the minimum energy loss in magnetization processes of soft magnetic materials.

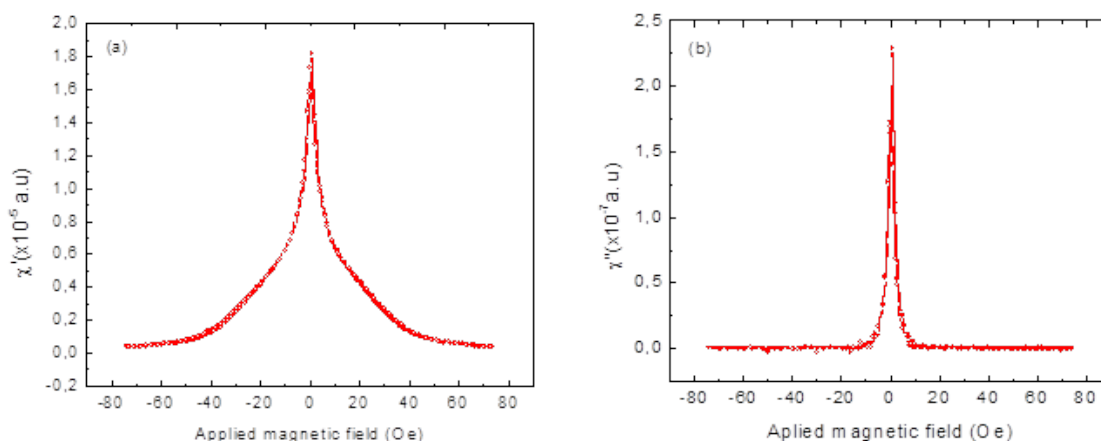


Figure 1. Real AC susceptibility (a) and imaginary AC susceptibility (b) Vs. Applied magnetic field at 40 Hz.

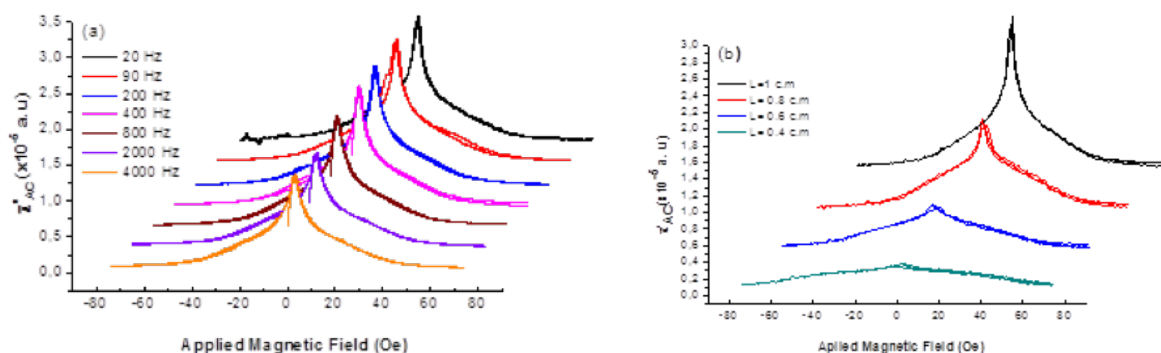


Figure 2. (a) Real part of AC magnetic susceptibility vs. external magnetic field at different frequencies and (b) at different lengths of the amorphous alloy of $\text{Fe}_{37}\text{Co}_{35}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ composition.

The AC magnetic susceptibility dependence on frequency and length of the sample is shown in Figure 2. It can be seen that the real AC magnetic susceptibility tends to widen as the frequency increases, while the AC magnetic susceptibility response enhances as the sample length increases; indicating the strong dependence of AC magnetic susceptibility on frequency and sample length. This dependence could be due to a possible magnetic hardening of the sample when the frequency is increased and the length is decreased, because the magnetic domain configuration is strongly influenced by the demagnetizing field, as it was explained by L. Goncalves et al. [8].

3.2. Structural characterization by XRD

In Figure 3, it is shown the XRD pattern obtained for the as-quenched sample and heat treated at different temperatures. It is observed that the sample is still amorphous at 550°C and from 600°C to 750°C, the sample is partially crystallized. This is evident by the appearance of the α -FeCo and $(\text{Fe,Co})_3\text{Si}$ primary crystalline phases and the $(\text{Fe,Co})_2\text{B}$ secondary phase. These crystalline phases were also reported for alloys of similar compositions in [1, 9].

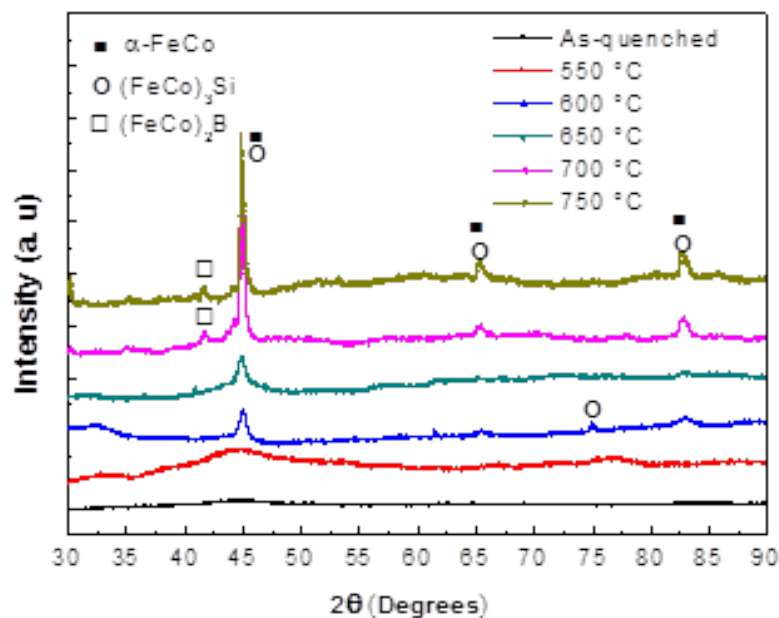


Figure 3. XRD pattern of the sample $\text{Fe}_{37}\text{Co}_{35}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ as-quenched and heat treated at different temperatures.

Table 1. Crystallite size (D), crystalline volume fraction of α -FeCo phase ($V_{\text{Cr-}\alpha\text{FeCo}}$), distance between grains (Λ) for the sample heat treated at different temperatures.

Heat treatment temperature (°C)	D (nm)	$V_{\text{Cr-}\alpha\text{FeCo}}$ (%)	Λ (nm)
550	-	-	-
600	9.9	3.5	20.7
650	27.4	4.1	56.7
700	25.6	11.4	27.5
750	21.1	24.9	13.0

From the principal diffraction maxima, the α -FeCo phase crystallite size (D) and the crystalline volume fraction ($V_{\text{Cr-}\alpha\text{FeCo}}$) as well as the distance between adjacent grains (Λ) were calculated, as shown in Table 1. It can be noticed that $V_{\text{Cr-}\alpha\text{FeCo}}$ increases with the heat treatment temperature increase,

indicating that the sample evolves from an amorphous state to a crystalline state. While, D increases from 9.9nm (600°C) up to 27.4nm (650°C), and tends to decrease slightly at higher heat treatment temperatures. Similar behaviour is observed for A , where at 650°C a maximum value is obtained. The variation in the obtained values D and A can be due to the formation of the α -FeCo nanocrystalline phase. Besides, the ordered (Fe, Co)₃Si phase and the (Fe, Co)₂B hard magnetic phase cause the magnetic decoupling between two adjacent grains at the heat treatment temperature of 650°C, as was reported in [10].

4. Conclusions

AC magnetic susceptibility depends strongly on the frequency and length of the sample Fe₃₇Co₃₅Nb₆B₁₁Si₁₀Cu₁ and its response is closely related to a minimum energy loss, which is characteristic for this type of alloys.

A nanocrystalline structure with the following heat treatment of the amorphous sample in the range 600–750°C was obtained. The α -FeCo and (Fe, Co)₃Si primary crystalline phases and the (Fe,Co)₂B secondary phase could be identified.

The partial crystallization of the sample was also confirmed by the increase of the crystalline volume fraction with the heat treatment temperature. Although, the crystallite size as well as the distance between two adjacent grains decreased, as a consequence of the existing crystalline phases.

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