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Magnetic characterization of $\text{Fe}_{32}\text{Co}_{40}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ alloy by the Transmission Mössbauer Spectroscopy technique

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This paper presents the results of magnetic characterization of the alloy of composition $\text{Fe}_{32}\text{Co}_{40}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ by the Transmission Mössbauer Spectroscopy technique. The amorphous alloy was obtained in the way of ribbon by the melt spinning technique, and then it was subjected to heat treatments at different temperatures in which crystallization process occurs. Mössbauer spectra showed the evolution of the crystallization process of the sample with the increase of the heat treatment temperature. From the fitting of spectra, the hyperfine field contributions and spectral area of amorphous and crystalline components were calculated. The nature of the crystallization products as well as the phase structure within the partial crystallization of the sample was determined using Mössbauer Spectrometry combined with XRD results.

Keywords: Soft magnetic properties; crystallization; Mössbauer spectroscopy.

Este artículo presenta los resultados de la caracterización magnética de la aleación de composición $\text{Fe}_{32}\text{Co}_{40}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ por la técnica de Espectroscopia Mössbauer de Transmisión. La aleación amorfa fue obtenida en forma de cinta por la técnica de la rueda fría y luego fue sometida a tratamientos térmicos a diferentes temperaturas en que transcurre el proceso de cristalización. Los espectros Mössbauer mostraron la evolución del proceso de cristalización de la muestra con el incremento de la temperatura de tratamiento térmico. Se calcularon los valores del campo hiperfino y área espectral de las componentes amorfa y cristalina a partir del ajuste de los espectros obtenidos. Tanto la naturaleza de los productos de cristalización como la estructura de las fases de la cristalización parcial de las muestras fueron determinadas utilizando Espectrometría Mössbauer en combinación con los resultados obtenidos por DRX.

Descriptores: Propiedades magnéticas blandas; cristalización; espectroscopia Mössbauer.

PACS: 81.40.Rs; 64.70.dg; 76.80.+y.

1. Introduction

In the last decades, Fe - based ferromagnetic amorphous alloys have been studied because they present good soft magnetic properties, which are required for a wide variety of technological applications. With the technology development it has been increased demanding of these materials for high temperatures and high frequencies applications [1].

It has been found that crystallization of Fe-based amorphous materials is the most useful method to improve the soft magnetic properties these alloys present. Crystallization process consists on heat treatments of the initial amorphous precursor at different temperatures. The new materials so obtained are called nanocrystalline soft magnetic materials.

FINEMET (FeSiBCuNb) and NANOPERM (FeMBCu, M = Zr, Hf, Nb) types alloys are among the softer magnetic materials known at room temperature. However, the soft magnetic properties of these alloys are lost for temperatures above the Curie temperature of the residual amorphous phase, and their applicability can not be extended over ~ 600 K [2]. Research on high temperatures applications of these systems yielded to the development of FeCo-based alloys (FeCoMBCu, M = Zr, Hf, Nb), called HITPERM. Partial substitution of Co by Fe extends the applicability of these alloys up to

higher temperatures than for FINEMET and NANOPERM alloys [3].

That is why, there have been several studies dedicated to the effect of Co substitution for Fe on the crystallization process, magnetic properties and hyperfine parameters [3]. For these studies, different structural and magnetic characterization techniques have been used.

One of the characterization techniques which give us magnetic and structural information about the material of study is the Mössbauer Spectroscopy technique (MS). This is a nondestructive and effective tool to probe environment in FeCo-based alloys [4].

In this work, the crystallization process and some hyperfine parameters of the alloy of composition $\text{Fe}_{32}\text{Co}_{40}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ ($x = 40$), is investigated using the Transmission Mössbauer Spectroscopy (TMS) and X- Rays Diffraction (XRD) as a complementary technique.

2. Experimental procedure

Amorphous ribbons, 1.77 mm wide and 46 μm thick, of nominal composition $\text{Fe}_{32}\text{Co}_{40}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$ were produced by the melt spinning technique. Crystallization treatments consisting on heating of amorphous ribbons, from room temper-

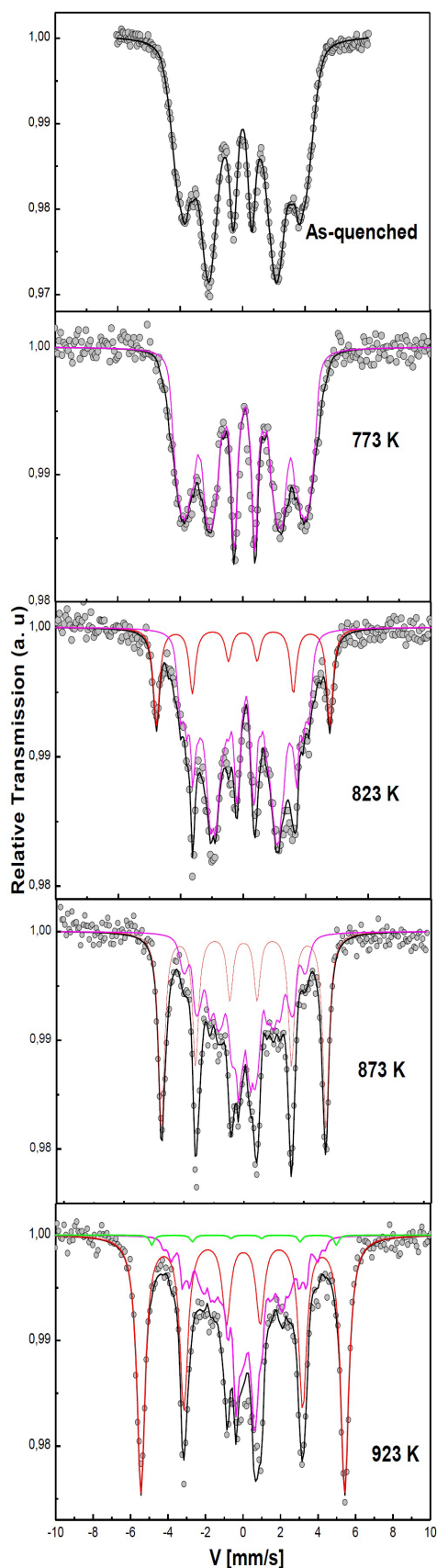


FIGURE 1. Mössbauer spectra for as-quenched and samples heated up to 773, 823, 873 and 923 K of the alloy $X = 40$.

ature up to temperatures 773, 823, 873 and 923 K. Heat treatments were performed at $5^\circ\text{C}/\text{min}$ under argon atmosphere in a vacuum tubular furnace OTX-1200X.

Mössbauer spectra were obtained at room temperature in a conventional multichannel spectrometer MS 1200, with a ^{57}Co source into a Rh matrix. Fittings were made with MOSFIT program and some hyperfine parameters were calculated.

XRD analysis were performed in a Diffractometer Bruker, using 2θ scan between 20° and 90° with 0.02 steps by second. Diffraction peaks were fitted by GSAS (General Structure Analysis System) program.

3. Results and discussion

Figure 1 shows the Mössbauer spectra for as-quenched and samples heated up to temperatures 773, 823, 873 y 923 K of alloy $\text{Fe}_{32}\text{Co}_{40}\text{Nb}_6\text{B}_{11}\text{Si}_{10}\text{Cu}_1$. From the figure, it is observed a broadened ferromagnetic spectrum attributed to the structural disorder of the amorphous state. For the sample heated up to 773 K, it also exhibits magnetic sextets with broadened peaks; which demonstrates that the sample is still found in amorphous state at this temperature. It is clearly observed a small crystalline contribution in the spectrum obtained at 823 K.

At 873 K, the spectrum shows a sextet of the crystalline phase with defined peaks, which are sharper at the temperature of 923 K; however, the sample is not fully crystallized at this temperature.

Figure 2 presents the hyperfine field distributions (HFD) for this alloy in its amorphous state and for samples heated up to temperatures of 773, 823, 873 and 923 K. In the figure, it is observed a maximum peak in the probability distribution for as-quenched and heat treated samples, corresponding to the most probable hyperfine field value of the total distribution. From the heat treatment temperature of 823 K, it can be notice the most intense peak is shifted at high hyperfine field contributions with the increase of the heat treatment temperature up to 923 K. The most probably hyperfine value of the intense peak at this temperature is nearly to the value of pure Fe (33 T).

It is also observed from the Fig. 2, the presence of a smaller peak at low hyperfine field values in the range from 7.5 to 15 T for the as-quenched and the sample heated up to 773 K. This low field peak can be ascribed to the presence of nonferromagnetic atoms of Nb in the neighborhood of Fe atoms, as it was reported in Refs. 2 and 5, as no evidence of such low values in $\text{FeCo}(\text{Si})\text{B}$ amorphous alloys.

The HFD shape corresponding to this alloy is very similar to that reported for the system $\text{Fe}_{78-x}\text{Co}_x\text{Nb}_6\text{B}_{15}\text{Cu}_1$ ($x = 18, 39$ and 60) HITPERM-type alloys [6] with 39 and 60 at. % Co at the heat treatment of 823 and 873 K.

The broadening of the maximum peak of the distribution for as-quenched and sample heated up to 773 K is ascribed to the amorphous state of the alloy, according to the obtained MS spectra for these samples. On the other hand, the crystalline contribution is clearly evidenced by the thinness of the

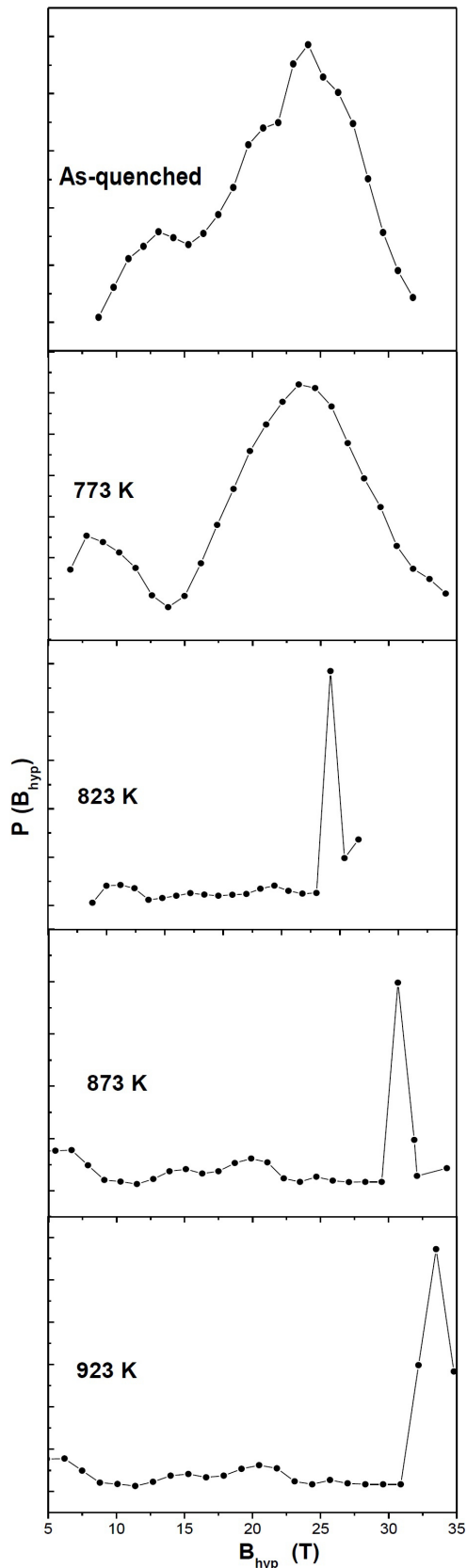


FIGURE 2. HFDs for as-quenched and samples heated up to 773, 823, 873 and 923 K of the alloy $x = 40$.

TABLE I. Spectral area (Λ) and hyperfine field (B_{hyp}) obtained from the fitting of the Mössbauer spectra of the sample $X = 40$.

| Heat treatment | $x = 40$ | | |
|----------------|-------------------|---------------|----------------------|
| | Component | $\Lambda(\%)$ | B_{hyp} (T) |
| As-quenched | Distribution | 100 | 20.9 |
| 773 K | Distribution | 98.9 | 22.2 |
| | α -FeCo | 1.07 | 31 |
| 823 K | Distribution | 79.2 | 28 |
| | α -FeCo | 20.81 | 32.2 |
| 873 K | Distribution | 53.68 | 30.4 |
| | α -FeCo | 46.32 | 35.5 |
| 923 K | Distribution | 42.21 | 29 |
| | α -FeCo | 56.63 | 33.5 |
| | Fe ₂ B | 1.16 | 23.3 |

maximum peak of the distribution, related to the main crystalline phase which is formed from the heat treatment temperature of 823 K.

It has been considered that the difference in the hyperfine field distribution of the alloy heated at the temperatures of 923 K, may be due to the formation of borides phases of the residual amorphous phase, as it will present later in the obtained XRD results for the sample heated up to 923 K.

Table I presents the hyperfine field and spectral area values obtained from the results of the Mössbauer spectra fitting for this alloy. The spectral areas represent the relative areas of the involved subspectra.

It is observed the hyperfine field values of the crystalline phase α -FeCo, increase at a higher heat treatment temperature, as a result of high interface atomic diffusion facilitated by the high temperature of heat treatment, as it was reported in Ref. 6 for FINEMET alloys annealed at different temperatures.

The Fe₂B crystalline phase was involved for the fitting in MS spectra according to the previous XRD results for the sample heated up to 923 K. The Fe₂B crystalline phase is one of the typical products of the secondary crystallization for FINEMET alloys [6].

With respect to the values obtained from the spectral area, it is observed that the values of the distribution (amorphous component) decrease from the sample heated up to 773 K, from 98.9 % to 42.21 % at the temperature of 923 K; whereas the spectral area values of the crystalline component, increase with the heat treatment temperature, from 1.07 % up to 57.79% for the sample heated up to 923 K.

The spectral area for the Fe₂B phase is lower than that obtained for FINEMET alloys (15 %), also reported in Ref. 6. The difference found in our results may be due to the presence of Co in the alloy of study.

It is evident that the values obtained from the spectral area and hyperfine field confirm the progress of the crystallization

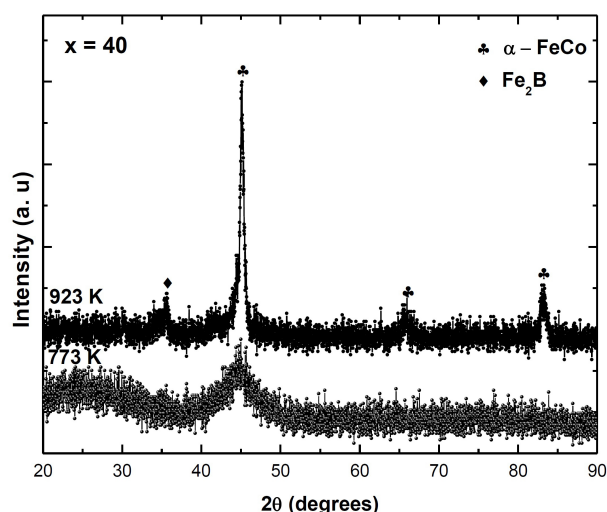


FIGURE 3. XRD pattern of the sample $x = 40$ heated up to 773 and 923 K.

process, with the increase of the heat treatment temperature at which is subjected the amorphous sample.

Figure 3 shows the XRD pattern of the sample under study that was submitted to heating treated at temperatures of 773 and 923 K.

In the XRD patterns is observed that the sample is still amorphous after heated up to 773 K, as it is shown by the broad peak, characteristic of the amorphous state. At the heat treatment temperature of 923 K, it appears the α -FeCo and Fe_2B crystalline phases. These phases were fitted to the experimental Mössbauer spectra according to the previous re-

sults of XRD. The presence of these crystalline phases also was reported by J. May [7] for HITPERM alloys.

XRD results are consistent with the MS results, because it is confirmed that the studied alloy is not fully crystallized at heat treatment of 923 K.

4. Conclusions

The progress of the crystallization process was evidenced by the increase of the hyperfine field of the FeCo crystalline phase and spectral area values of the crystalline component with the increase of heat treatment temperature.

It was verified by XRD that the sample is still in amorphous state at the temperature of heat treatment of 773 K and the presence of α -FeCo and Fe_2B crystalline phases at the temperature of heat treatment of 923 K.

Mössbauer Spectroscopy results as well as XRD results confirmed the evolution of the sample from an amorphous state to a crystalline state with the increase of the temperature of heat treatment.

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