



# On the influence of Joule heating induced nanocrystallization on structural and magnetic properties of $\text{Co}_{64}\text{Fe}_{21}\text{B}_{15}$ alloy

S.N. Kane<sup>a,\*</sup>, M. Coisson<sup>b</sup>, P. Tiberto<sup>b</sup>, F. Vinai<sup>b</sup>, F. Mazaleyrat<sup>c</sup>

<sup>a</sup>School of Physics, D. A. University, Khandwa Road Campus, Indore 452001, India

<sup>b</sup>INRIM, Electromagnetism Division, Strada delle Cacce 91, 10135 Torino, Italy

<sup>c</sup>SATIE UMR 8029 CNRS, ENS de Cachan, 61, Av. du Pdt. Wilson, 94235 Cachan cedex, France

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## ABSTRACT

Structural and magnetic properties of Joule heated  $\text{Co}_{64}\text{Fe}_{21}\text{B}_{15}$  alloy have been studied. Crystallization of the specimens starts after Joule heating with 1.6 A current, leading to the formation of nanocrystalline  $\text{Co}_{80}\text{Fe}_{20}$  phase with grain diameter ranging between 19 and 25 nm. Crystallization of the specimens is found to deteriorate the soft magnetic properties. Observed decrease of the hyperfine field of the amorphous component upon Joule heating suggests the increase of the boron near-neighbours to iron and the changes in the hyperfine field of the crystalline component are attributed to the changes in the environment around Fe atom in the specimen. The coercivity is found to obey a domain wall pinning process instead of random anisotropy, as expected in soft magnetic nanocrystalline magnetic materials.

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## 1. Introduction

The soft magnetic properties of amorphous ferromagnets are generally improved by low-temperature anneals, which produces relaxation of the atomic structure and a reduction of the quenched-in internal stresses [1]. Conversely, the development of a dominant crystalline fraction by performing high-temperature thermal treatments is generally seen to destroy the soft magnetic properties of amorphous alloys [2]. However, the soft magnetic properties of certain Fe-based alloys have been improved by early crystallization stages [3]. In the eighties, in addition to conventional furnace treatment having heating rate of the order of 0.1–1 K/s, a new method exploiting the heat released to the sample by an electrical current flow has been employed [4,5]. The current annealing techniques are characterised by rather high heating rates ( $10^4$ – $10^6$  K/s) allowing the sample to crystallize at higher temperatures with respect to furnace annealing. As a consequence, fast heating techniques [6] are currently employed in order to obtain metallic materials with attractive physical properties starting from amorphous ribbons. Among these, dc Joule heating is a convenient technique to induce structural changes [7] in a shorter time with respect to conventional furnace annealing treatments [8]. Typically, electrical current density values ranging in the interval  $1 \times 10^7$  to  $1 \times 10^8$  A/m<sup>2</sup> for time varying between 1 and 1000 s are exploited.

This technique allows to perform annealing under a self-generated magnetic field and, consequently, to induce a magnetic anisotropy simply by using low-current densities in order to reach temperatures below the Curie temperature of the studied alloy [9]. In this technique, heating rate reaches  $\sim 100$  K/s, which is lower than the maximum values provided by pulse heating technique; however it remains two orders of magnitude higher with respect to conventional annealing treatment. The amorphous-to-crystalline transformation occurs several seconds after reaching a steady-state temperature, and allowing to carry out proper measurements of selected physical properties (e. g. variation of the resistance of the specimen by passing dc current through it, providing information on crystallization of the specimen) during the structural transformation [10]. The time scale of the process is made suitable for on-line physical measurements by using an electrical current of the order of few amperes. In this case, sample heating has been monitored by measuring a structure sensitive property such as the electrical resistivity.

Both X-ray diffraction (XRD) and Mössbauer spectroscopy provide structural information on the specimen under investigation. It is of value to note that, X-rays can not differentiate between Fe and Co atoms, as their scattering amplitudes are very similar. Thus, XRD essentially provides information about the average TM–TM pair correlation function (TM – Transition Metal). On the other hand, transmission Mössbauer spectroscopy measurements are sensitive to the Fe atoms only and hence they provide information about the local environment around Fe atoms. Furthermore, the Fe hyperfine field is more sensitive to the boron near neighbour

\* Corresponding author.

E-mail addresses: [kane\\_sn@yahoo.com](mailto:kane_sn@yahoo.com) (S.N. Kane), [m.coisson@inrim.it](mailto:m.coisson@inrim.it) (M. Coisson).

(both number and the distances) as compared to the Co near-neighbours and thus hyperfine field would provide information about the variations in the metalloid near-neighbour environment.

In the present work, we investigate the influence of dc Joule-heating induced nanocrystallization on structural and magnetic properties of  $\text{Co}_{64}\text{Fe}_{21}\text{B}_{15}$  amorphous ribbons, using X-ray diffraction (XRD), Mössbauer spectroscopy and hysteresis measurements.

## 2. Experimental details

Amorphous  $\text{Co}_{64}\text{Fe}_{21}\text{B}_{15}$  ribbons (width  $\approx 6$  mm and thickness  $\approx 19$   $\mu\text{m}$ ) were obtained by rapid solidification technique on a rotating drum. In order to carefully evaluate the electrical current value, which induces the amorphous-to-crystalline transformation, one sample has been submitted to a current ramp and the electrical resistance was continuously monitored. The electrical resistance evolution as a function of electrical current  $I$  shows an increase of resistance up to  $I \approx 1.9$  A and decreases for higher currents, indicating the onset of microstructural changes within the amorphous matrix (i. e. sample crystallization). Selected strips with a length of 10 cm have been submitted to dc Joule-heating in vacuum [7] with  $I$  ranging from 0 to 2 A for 30 min in order to induce structural changes in the specimens. Hysteresis loops were measured using a conventional induction technique at 10 Hz, which were used to obtain hysteresis parameters and the relative permeability. X-ray diffraction (XRD) measurements were carried out at room temperature using Cu- $K\alpha$  radiation. XRD patterns were analyzed by fitting a crystalline and one amorphous component using pseudo-Voigt profiles. The average grain size  $D$  is obtained by Scherrer's formula using integral width of the (110) line and the lattice parameter  $a$  is calculated using Nelson-Taylor-Sinclair correction in order to take into account the peak shift due to sample offset. For the amorphous phase, the first nearest neighbour distance  $X_m$  is calculated using the expression:  $X_m = 1.227 \lambda/2 \sin\theta$  [11]. Apparent area is obtained using expression

$$A^* = \frac{A_c}{A_c + A_a} \quad (1)$$

where  $A_c$  – area of the crystalline phase (110) peak,  $A_a$  – area of amorphous phase main halo.

Volume of the crystallites is given by

$$V_x = \frac{1}{\frac{1 - A^*}{A^*} \frac{f_c}{f_a} + 1} \quad (2)$$

where  $f_c$  – scattering factor of the crystalline component and  $f_a$  – the scattering factor of the amorphous component. These factors having dependence on the concentration of electrons, we can consider in first approximation  $f_c/f_a \approx 1/(1-C_B)$ , where  $C_B$  is the concentration of Boron in residual amorphous matrix. Because  $C_B$  and  $V_x$  are linked, it is initially considered that  $V_x = A^*$ . The composition of the amorphous phase is then calculated and a new value of  $V_x$  is decreased. After 3 to 4 iterations, it converges to better than 1%.

Volume fraction of the crystalline component is thus calculated by

$$V_{XRD} = \frac{A^*(1 - C_B)}{1 - A^*C_B} \quad (3)$$

where  $C_B$  is the concentration of Boron in residual amorphous matrix.

Transmission Mössbauer spectra were recorded at room temperature in constant acceleration mode using  $^{57}\text{Co}:\text{Rh}$  source

and were fitted assuming a distribution of hyperfine fields using NORMOS program [12]. For Mössbauer measurements,  $X$  – the Co % in Fe-Co phase ( $\text{Co}_x\text{Fe}_{1-x}$ ) is deduced as follows :

$$\frac{1}{X} = \frac{A^* C_{\text{Fe}}}{V_x} + 1 \quad (4)$$

where  $V_x$  is the volume fraction of crystallites obtained from XRD measurements,  $A^*$  is the area of the crystalline phase obtained from Mössbauer measurements,  $C_{\text{Fe}}$  is the concentration of Fe atoms in the parent alloy.

The apparent area is obtained using expression

$$A^* = \frac{C_{\text{Fe}}^{\text{Cr}}}{C_{\text{Fe}}^{\text{aq}}} \quad (5)$$

where  $C_{\text{Fe}}^{\text{Cr}}$  is the concentration of Fe in crystalline phase and  $C_{\text{Fe}}^{\text{aq}}$  is the concentration of Fe in as-cast specimen. Volume fraction of the crystalline component from Mössbauer measurements  $V_x^{\text{Möss}}$  is thus obtained by

$$V_x^{\text{Möss}} = \frac{A^*}{1 - x} \quad (6)$$

where  $x$  – Co % deduced from average hyperfine field.

## 3. Results and discussions

Fig. 1 depicts the XRD patterns of the selected Joule heated specimens. Perusal of Fig. 1 shows that after annealing with a Joule heating current of 1.6 A for 30 min, the crystallization starts and for higher Joule heating currents, crystalline fraction increases as evidenced by the presence of sharp crystalline peaks at  $45.2^\circ$  and  $65.8^\circ$  (on two theta scale). Parameters obtained by analysing XRD data are depicted in Table 1. For the studied specimens, within experimental errors the  $X_m$  ( $0.246 \pm 0.001$  nm) remains unchanged, so no definite conclusion can be drawn about the change in the mass density of the specimen from the XRD data. Table 1 also shows that the grain size of the nanocrystalline phase ranges between 19 and 25 nm and the corresponding volume fraction varies between 3.4 and 31.4%. Volume fraction shows linear dependence with increase in the Joule heating current. Grain size of the studied samples is of the order of nanometer, although the studied samples do not contain Cu, which is a well-known nucleating element [13]. It is worth noting that in a recent paper Ping et al. [14] have shown that in Co-rich alloy systems, Fe-Co nanocrystalline grains do not need



Fig. 1. X-ray diffractograms of selected Joule heated specimens.

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