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Temperature dependence of the coercive field of gas atomized $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$

A. García-Escorial [a,*], M. Lieblich [a], A. Hernando [b], A. Aragón [b], P. Marín [b]

[a] CENIM-CSIC, Avda, Gregorio del Amo, 8, 28040 Madrid, Spain
[b] Instituto de Magnetismo Aplicado, IMA, P.O. Box 155, 28230 Madrid, Spain

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ABSTRACT

In this work, the dependence of the coercive field of $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ gas atomized powder with the temperature for different particle sizes has been studied, observing an anomalous behavior in the under 25 powder particle size fraction. This unusual behavior is related with the microstructure of the powder, and is attributed to the presence of a multiphase magnetic system, with non-magnetic regions decoupling the ferromagnetic domains.

1. Introduction

The alloy $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ has been the object of an enormous research interest due to its excellent soft magnetic behavior after partial crystallization [1,2]. This alloy has been produced and studied in many different forms, such as, melt spun ribbons, amorphous material, partially crystallized state, mechanically alloyed, ball milled and recently also gas atomization [3,4].

In a previous work, $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ was obtained as powder particles by gas atomization and ball milling of melt spun ribbons to show the analogies and differences between both powders [3]. Furthermore, gas atomized $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ and $Fe_{93}Si_7$ were investigated to study the influence of the alloying elements [4]. In these works an increase in the coercive field, Hc, as particle size decreased was obtained in both alloys [5]. Moreover, it was observed that for the same powder particle size and similar grain size the coercive fields for the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder were almost one order of magnitude higher than those of the $Fe_{93}Si_7$ alloy. This effect was attributed to Nb segregation, which could act as pinning center of domains walls.

This magnetic hardening is especially relevant nowadays due to the interest in obtaining hard magnetic materials without Rare Earths elements. Magnetic softening is also important since powders can be used as magnetic flux multiplier, forming nucleus of electromagnetic devices easy to form into different shapes.

In this work, with the aim of deepening our understanding of the high coercive fields presented by the $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ gas-atomized powder particles, the dependence of the coercive field with the temperature from 4 to 350 K has been investigated. The smallest and largest particles were specifically used to simplify the study. The big difference in size between those of less than 25 μm, the ones with higher cooling rate, and those in the range of 500–1000 μm, with slower cooling rate, implies very different solidification rates that lead to different phases, microstructure and grain and particle size, strongly correlated to the coercivity and its thermal dependence as outlined below.

2. Experimental techniques

$Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$, composition expressed in atomic per cent, was gas atomized at CENIM in a confined nozzle atomizer, already described in [3,4]. Gas atomization is a containerless process, where the liquid melt solidifies rapidly at high undercooling, with a cooling rate of the order of 10^3–10^5 K s^{-1}. The $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ at% alloy (83.4Fe–7.7 Si–1.2 B–5.66 Nb–1.3 Cu% in weight) was prepared from Fe–Si and Fe–Nb master alloys and Fe, B, and Cu elements. The resulting powder was allowed to cool down in the inert gas atmosphere of the atomizer. Afterwards, it was collected in air and sieved to achieve separation into different

* Corresponding author. Tel.: +34 91 5538900; fax: +34 91 5347425.
E-mail address: age@cenim.csic.es (A. García-Escorial).

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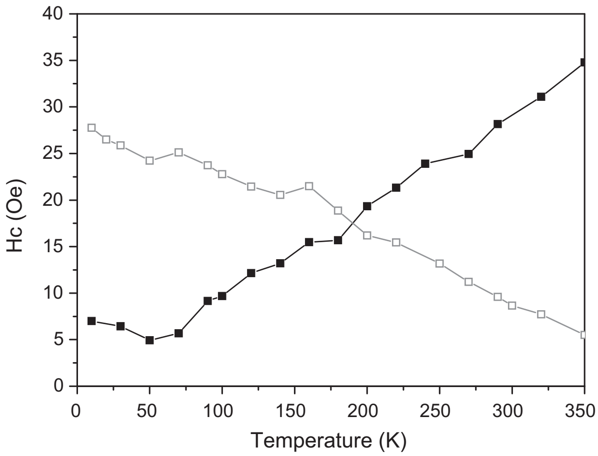

Fig. 1. Evolution of the coercive field with the temperature for under 25 μm powder, A (full symbols, black) and for a 500–1000 μm particle, B (open symbols, gray).

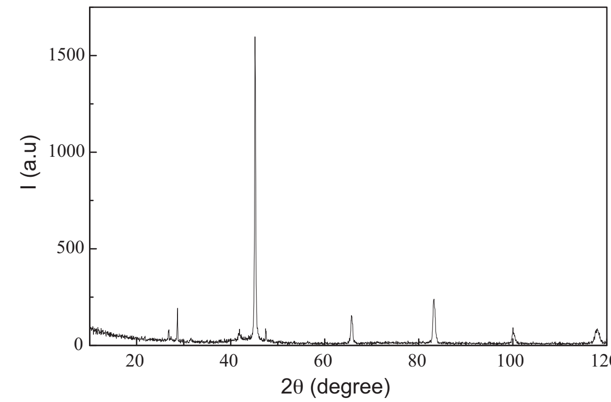

Fig. 2. XRD of as-atomized under 25 μm powder, A.

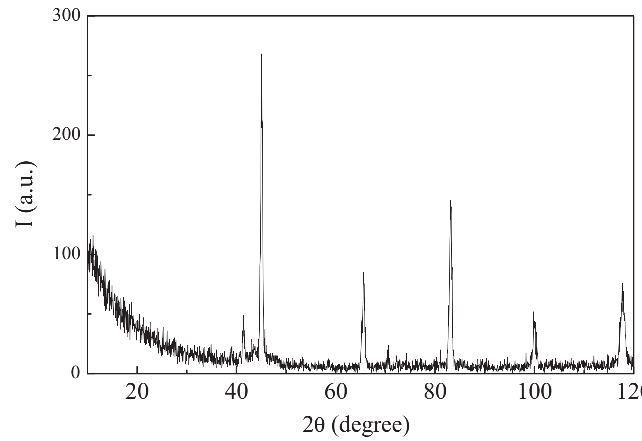

Fig. 3. XRD of as-atomized 500–1000 μm particles, B.

sizes ranges. This work has been focused on the small, less than 25 μm, and larger 500–1000 μm size ranges, labeled as A and B respectively.

Hysteresis loops at 300 K for the different particle size fractions were measured by using a vibrating sample magnetometer PPMS-VSM Quantum Design at 300 K and with a maximum field of 4 T, on encapsulated samples. The dependence of the coercive field of the gas atomized powder with the temperature from 4 to 350 K as a function of the powder particle size was measured.

The determination of the oxygen content was carried out in both size fractions by an infrared absorption method after fusion under inert gas of the powders. The smallest size fraction has an oxygen content of 0.2 wt% and the larger size 0.03 wt%, one order of magnitude less.

The $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ powder size fractions were studied by X-ray diffraction (XRD) using Cu-Ka radiation. The morphology and microstructure of particles as a function of size range were observed by scanning electron microscopy (SEM) equipped with an X-ray energy dispersive analysis unit (EDX). Electron channeling contrast images have been taken, that are produced from electrons which channel down the crystals planes, and thus, are sensitive to crystallographic orientation.

3. Results and discussion

Fig. 1 shows the influence of temperature on the coercive fields for both particle sizes, A and B, where an anomalous increase in the coercive field with temperature is observed in the smaller powder particles. In this graph, it can be seen that at room temperature the smaller powder is magnetically harder than the larger one, as has been reported in previous works [3,4]. However, this is not the case at temperatures below 180 K. For homogeneous ferromagnets the coercive field depends inversely on the exchange correlation length that generally increases with temperature [5]. In the present case, the observed thermal dependence indicates that an effect, different from the one associated with size, is affecting the overall behavior of the small $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ particles. A similar anomalous increase in coercivity with temperature, as shown in Fig. 1, was observed and reported for Fe–Cu alloys during its spinodal decomposition [6]. In heterogeneous microstructures some ferromagnetic phases could evolve during heating towards paramagnetic states so decreasing the exchange coupling between ferromagnetic regions which gives rise to a shortening of the exchange correlation length and consequently to an increase in the coercive field.

To correlate this magnetic behavior with the powder microstructure, particles were studied through X-ray

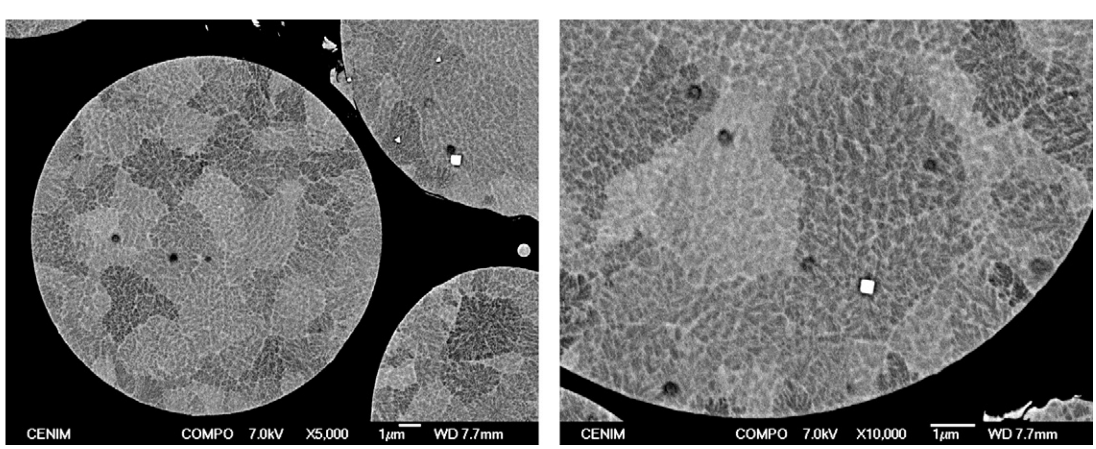

Fig. 4. SEM micrographs of as-atomized under 25 μm powder particles, A.