

Preparation and characterization of Fe₅₀Co₅₀ nanostructured alloy

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Abstract. Nanostructured Fe₅₀Co₅₀ alloy was prepared by mechanical alloying of Fe and Co powders in a planetary high energy ball milling. The microstructure and structural evolution of the alloy have been investigated as a function of milling time (0 h, 8 h, 20 h and 35 h) by scanning electron microscopy (SEM) and X-Ray diffraction (XRD) characterization techniques. SEM micrographs showed different powder particles morphologies during the mechanical alloying stages. By XRD analysis it could be identified the structural phases of the alloy and the crystallite size was calculated as a function of the milling time.

1. Introduction

FeCo-based soft magnetic materials have the largest saturation magnetization of all known magnetic alloys. These alloys exhibit high saturation magnetization and high Curie temperatures [1], that make them potential candidates for the high temperatures applications such as new generation turbine engines as well as in recording media [2]. In particular the Fe₅₀Co₅₀ alloy has a considerably large permeability, low magnetocrystalline anisotropy and low electrical resistivity [3]. All these properties are shown to be superior at the nanoscale. That's why there has been a great interest in researching on processing and characterization of nanostructured magnetic alloys.

Mechanical Alloying (MA) is one of the most important methods for preparation of nanostructured magnetic materials, such as equiatomic iron-cobalt alloy [4]. This method is an intensive energy process of mechanical grinding for the preparation of alloyed powders. It involves a repeated fracturing and welding of particles, leading to size reduction of nanometer scale [3].

Nanostructured Fe₅₀Co₅₀ alloys prepared by high energy mechanical alloying method at different milling times and characterized by SEM and XRD have been reported in the literature [1-6].

In this work, a study of morphological and structural characterization of nanostructured Fe₅₀Co₅₀ alloy prepared by high energy mechanical alloying (MA) is presented.

2. Experimental procedure

Fe (99.5 %, 6-9 μm) and Co (99.8 %, 2 μm) powders supplied by Sigma Aldrich, were separately weighted and mixed to get the desired composition. The mechanical alloying process was performed



in Fritsch Pulverizette 5 (P5) planetary high energy ball mill, belonging to Physical Metallurgy and Phase Transitions Theory Group of the Universidad Del Valle (Cali, Colombia). The mixed powders were introduced in stainless steels containers of 250 ml with balls of the same material, making previously vacuum. The ball to powder mass ratio was about 20:1 and the milling intensity was 305 rpm. Un-milled (0 hours) and milled samples during 8, 20 and 35 hours were prepared. To avoid excessive heating during milling each milling time was followed by a pause of one hour.

For morfological characterization of samples by SEM a Jeol JSM - 5600 microscope from the Universidad Del Norte (Barranquilla, Colombia) was used. SEM micrographs for un-milled powders (0 hours) and milled during 8, 20 and 35 hours were taken.

Structural characterization was performed on a PANalytical X'Pert diffractometer, from the Universidad Del Valle (Cali, Colombia) XRD laboratory. Measurements were carried out at room temperature; using Co radiation over $40\text{--}130^\circ 2\theta$ and $0.54^\circ/2.22\text{s}$ step. The mean crystallite size was estimated by the Scherrer formula [7], taking into account the diffraction peak integral width calculated from the diffraction peaks refinement software GSAS.

3. Results and Discussion

3.1. Morphological characterization by SEM.

Figure 1 shows the SEM micrographs of the $\text{Fe}_{50}\text{Co}_{50}$ alloy at different milling times. Figure 1 (a) displays the un-milled powder, the different shapes of the alloying elements particles can be seen. The Co particles have spherical shape while the Fe particles have cylindrical shape. Similar results were found by Akkouche et al. [3] for nanocrystalline powders of the same composition prepared by mechanical alloying.

Figure 1 (b) shows the micrograph obtained from the $\text{Fe}_{50}\text{Co}_{50}$ milled during 8 h. It is observed the agglomeration of powder particles, as consequence of powders that are repeatedly flattened, cold weld, fractured and rewelded, what is typical of mechanical alloying process for ductile elements as Fe and Co, as it is reported in the literature [1-6]. The powder size distribution and the particles shape are not uniform, as indicated by N. Yepes [8].

SEM micrograph for the powders milled during 20 h is shown in Figure 1 (c). It can be seen the flattening and welding of powders, resulting in a narrower distribution of particle size, also reported by Chermahini et al. [5] for powders of similar compositions.

The cold welding and fracture events continue to take place for milled powders during 35 h, leading to microstructural refinement, as it is shown in Figure 1 (d), which is a characteristic of ductile-ductile systems.

3.2. Structural characterization by XRD.

3.2.1. Phase identification.

Figure 2 shows the evolution of the XRD patterns for $\text{Fe}_{50}\text{Co}_{50}$ alloy as a function of the milling time. For the initial powders mixture the XRD pattern (0 h) confirms the presence of body-centred cubic Fe (bcc), hexagonal closed-packed (hcp) Co and face-centred cubic (fcc) Co phases. Also, it is observed a slight broadening of the bcc Fe-Co diffraction peaks between 8 and 35 hours of milling, due to the deformation processes and internal stresses occurring at macroscopic level during the milling process [3, 5]. After 8 hours of milling, the hcp and fcc Co diffraction peaks disappear, the Co atoms diffuse into the Fe bcc lattice leading to the onset of alloy formation with iron. Analogous results were found by Chermahini et al. [5] for powders of similar compositions milled during 45 h. These results differ from those found by Akkouche et al. [3] for alloys of the same composition, where a bcc Fe (Co) solid solution is formed after 100 h of milling. The formation of a crystalline solid solution is typical for all FeCo alloys prepared by MA [1-5]; unlike FeNi alloys in which an amorphous structure is formed by MA [1-2].

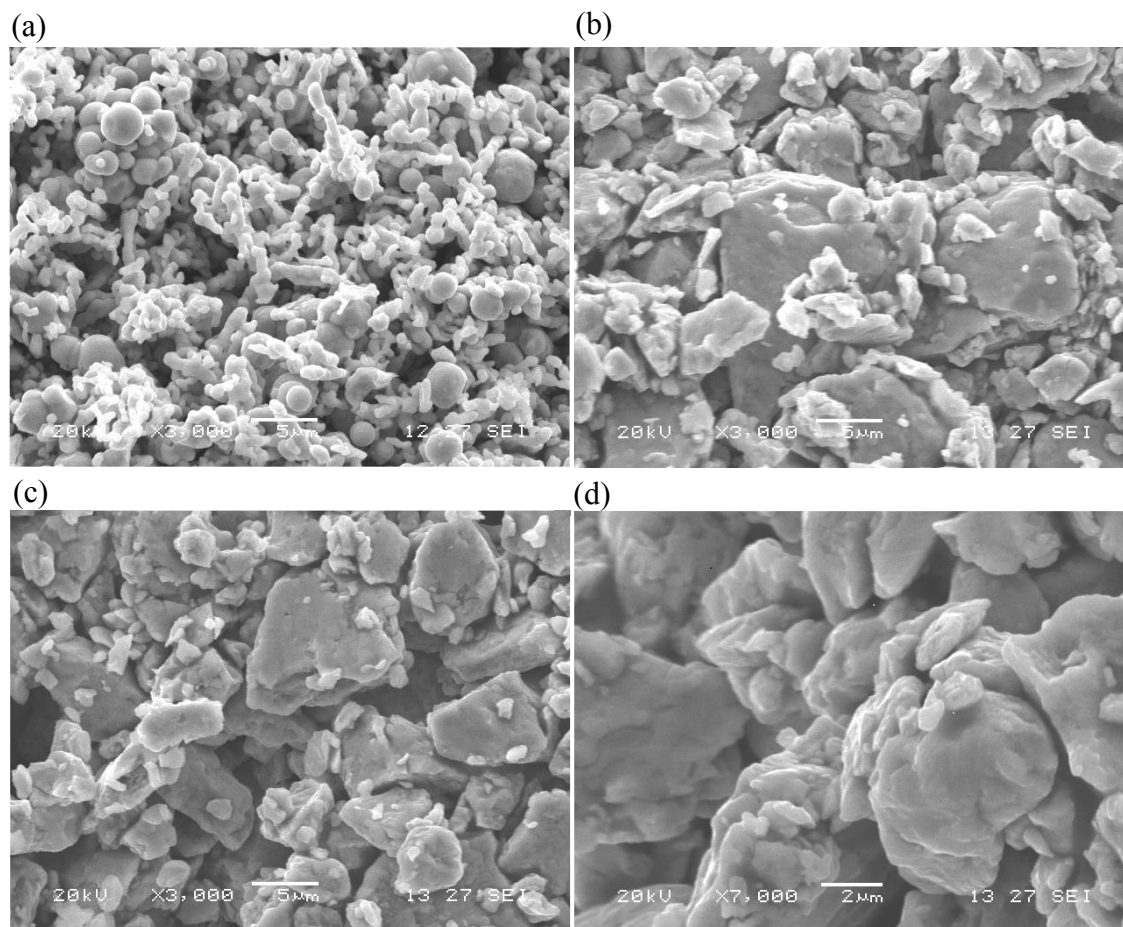


Figure 1. SEM micrographs of $\text{Fe}_{50}\text{Co}_{50}$ powders for various milling times: a) 0 h (un-milled), b) 8 h, c) 20 h and d) 35 h.

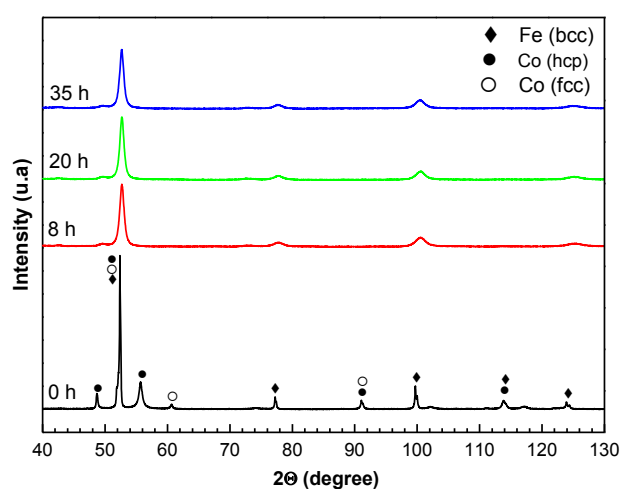


Figure 2. XRD of mechanically alloyed $\text{Fe}_{50}\text{Co}_{50}$ for various milling times.

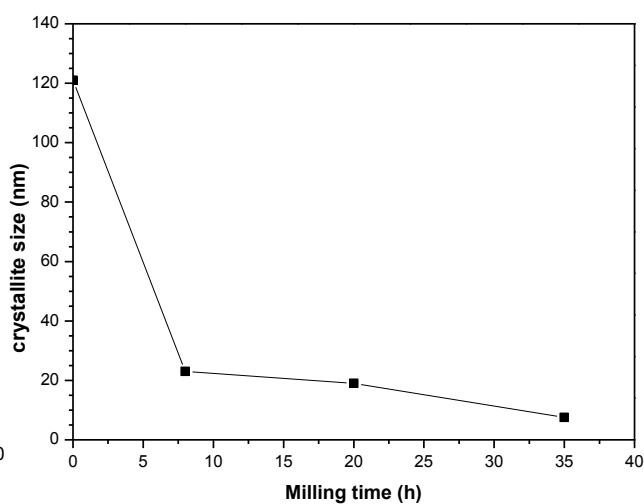


Figure 3. Evolution of crystallite size as a function of milling time.

3.2.2. Crystallite size.

Figure 3 shows the evolution of the crystallite size as a function of the milling time. It is observed that the crystallite size decreases with increasing milling time, from a value of 121 nm for un-milled powders (0 h, corresponding to Fe powders) to a value of 7, 5 nm for milled powders during 35 h. It is also noticed that the crystallite size decreases rapidly from 121 nm (0 h) to 23 nm (8 h) followed by a relatively slower one at 20 h of milling. These results are comparable with those obtained by Chermahini et al. [5] for nanostructured $\text{Fe}_{1-x}\text{Co}_x$ (x between 0, 2 and 0,7 at. % of Co) alloys prepared by MA using very similar milling times. Nevertheless, the crystallite size between 20 and 35 h changed slower, reaching a value of 12 nm. Different results were obtained by Akkouche et al. [3], for alloys of the same composition prepared by high energy MA, whose average crystallite size values were 150 ± 2 nm for un-milled powders (0 h) and 14 ± 2 nm for milled samples during 200 h. These differences are due to the used methods and the conditions for preparing the alloy.

The decreasing crystallite size with milling time behavior is attributed to dislocation generation caused by severe plastic deformation [4, 5].

4. Conclusions

Nanostructured alloys of $\text{Fe}_{50}\text{Co}_{50}$ in powder form were prepared by mechanical alloying method by means of a high energy planetary ball milling P5, in dried milling, making previously vacuum and with a milling intensity of 305 rpm. The morphology powders evolution with milling time was revealed by SEM. The formation of nanostructured alloy $\text{Fe}_{50}\text{Co}_{50}$ was confirmed by XRD. The crystallite size decreased with the increasing milling time; the lowest value in the crystallite size (7, 5 nm) was obtained at 35 hours of milling. The evolution of powders morphology as well as the crystallite size is attributed to deformation processes that powders undergo when they are mechanically alloyed within a high energy ball milling.

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