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Magnetic, Microstructure Characterization of $Fe_{65}Co_{35}$ Nano Crystalline Alloy Synthesized by Mechanical Alloying Process

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Abstract

An investigation was conducted to explore the applicability of magnetic and microwave techniques to characterize grains size variation during mechanical alloying. A series of Nano crystalline Fe65Co35samples have been prepared, these structures are prepared using mechanical alloying based on planetary ball mill under several milling conditions. Mechanical alloying is a non-equilibrium process for materials synthesis. The structural effects of mechanical alloying of powders were investigated by X-Ray diffraction analysis, microwaves and VSM magnetic technique. Consequently, alloy powder with an average grain size about of 8 nm was obtained. Experimental results show that fine Nano crystalline alloy powders prepared by mechanical milling are very promising for microwave applications.

Keywords: Fe-Co powder; Mechanical alloying; Magnetic properties; Microwave

Introduction

Materials and Nanomaterial's are experiencing a growing interest in many areas; in particular that of nanotechnology because of their physical properties; often better than those of conventional materials and have been developed to overcome the limitations of conventional materials [1]. In recent years, they have emerged as a versatile solution for producing new materials such as nanostructured and amorphous alloys [2,3]. They may be in the form of nanoparticles and multilayered thin films of nanostructured powders, Nano crystalline alloys etc.

Nanomaterial's have a high concentration of material at their surfaces (nanoparticles); in interfaces (multilayer: Nano crystalline) and grain boundaries (nanostructures alloys of powders) [4]. This characteristic gives a specific physicochemical property very different from those of ordinary materials. Their preparation is based on a variety of methods such as soft chemistry method, consolidation of atom clusters, partial devitrification of the amorphous phase and the high energy milling.

In the last method, high energy milling, the powder particles are subjected to severe mechanical deformation due to collisions with balls and deformed repeatedly, and then they are fractured and welded cold. The phase-forming mechanism is an interdiffusion reaction of the components, it occurs during the milling process. The formation of the metastable phase is observed as a stable phase is suppressed in specific milling conditions [5].

The Fe-Co alloys are presented as an ideal material for applications where high magnetic saturation is a design parameter, particularly in aerospace where the volume and mass must be minimized [6]. There has been a renewed interest in these alloys, particularly with the development of strong variations [7,8]. The interest was sparked primarily by the construction of electric motors, which can be incorporated in warm regions of aircraft engines [9,10], their new application in high frequency magnetic.

Experimental Details

The particles have an average size of 70 um and a particle means crystallite size of 35 nm, they are introduced in a cylindrical container (walls) of hardened steel with a capacity of 25 ml. The materials were sealed under high purity argon atmosphere with mass ratio of balls 32:1.

Milling was performed up to 54 hours with a rotation speed of 380 r/min. About 2 g of Nano crystalline powders of $Fe_{65}Co_{35}$ were compacted at room temperature under a pressure of 2 GPa for 2 hours in a cold uniaxial press (13 mm inner diameter, outer diameter 25 mm and 2.5 to 3 mm in height).

The powder mixtures were characterized by a Siemens D500 diffract meter using Cu Ka radiation in the 2θ range of 30° to 120°. The magnetic measurements were performed with a VSM equipment "micro-sense EV9".

Results and Discussion

An electron microscopic observation is carried out on powder samples milled at different times. Figure 1 shows an overview of the powders used before grinding. One can still note a spherical dominant form. Some facets of the particles are still observable.

Figure 2 shows the powder after 12 hours of milling with an enlargement of 8000, there is the presence of a large number of particles of platforms and angular. Indeed, the flat particles are produced by the collisions between the balls and the walls of the vase. The ground powder is then projected and sometimes it sticks to the walls and undergo successive collisions.

When the grinding is continued after 36 hours, larger particles disappear and the particle size in dispersion is considerably reduced. At this stage, we notice that the particles are arranged in a string form of aggregates. We can say that the small particles have a single domain and are aligned because of their spontaneous magnetization (Figure 3).

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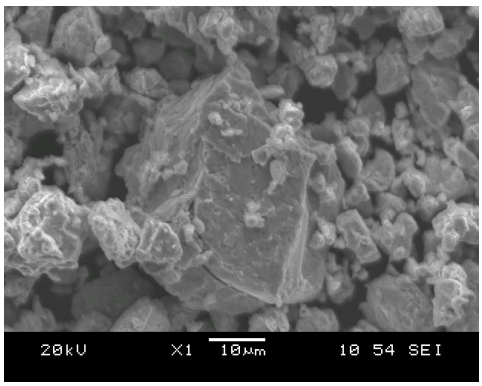

Figure 1: Presentation of non-milled powders.

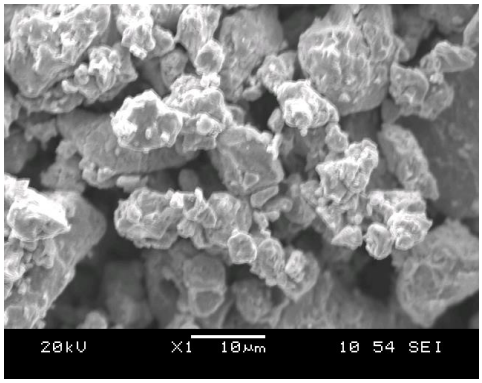

Figure 2: Morphology of powders after 12 hours of milling.

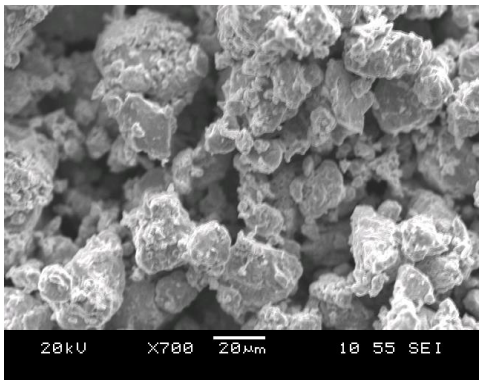

Figure 3: Morphology of powders after 36 hours of milling.

The results of the X-ray diffraction for the powder $Fe_{65}Co_{35}$ are shown in Figure 4. The peaks associated with pure Co disappear completely for the time of upper grinding to 4 hours. This demonstrates the substitution of Co in Fe structures and the formation of disordered solid solution of Fe-Co with a face-centered cubic structure (CFC). At the same time, the width of the peak associated with the CFC phase is gradually reduced with the time of crushing due to the decrease of the grain size.

The estimate of crystallite size was obtained from the refinement of X-ray patterns with the Williamson-Hall method, shown in Figure 5, during the first phase of milling, the crystallite size decreases quickly and then slows and gradually becomes smaller about 8 nm.

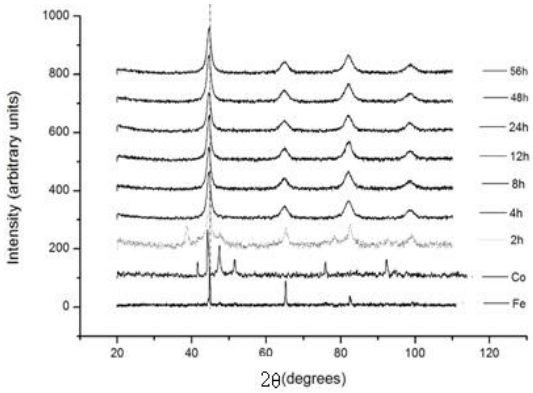

Figure 4: The XRD results (Cu-Kα) for powder $Fe_{65}Co_{35}$ milled at room temperature.

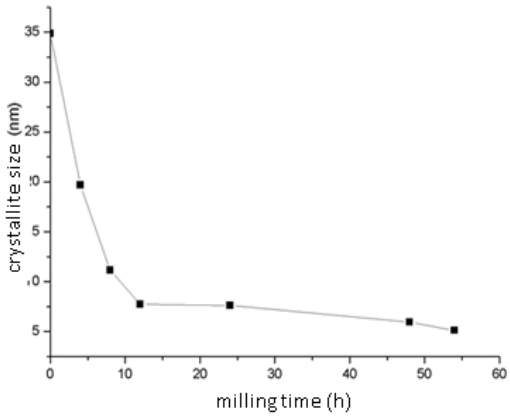

Figure 5: Evolution of crystallite size of ($Fe_{65}Co_{35}$) on several milling times.

Magnetic Study

The change in the coercive force Hc and residual Br according to the milling time was shown in Figure 6, the curves show a similar steady decrease of the two parameters when the milling time is increased. After 8 h, Hc and Br have remained almost unchanged. However, a slight increase of Br and Hc after 24 h suggests that the cobalt alloy formation proceeds to a simultaneous transformation Co (PHC) in Cooperation [11].

The variation of the reflection coefficient "Agilent N5222A" and "Agilent E8257D" based grinding times the frequency of 9 GHz illustrated in Figure 7. We can see that the coefficient of reflection decreases as milling time increases resulting in a decrease in grain size. In addition, it is noted that the smallest value of the reflection coefficient is obtained with 54 h of milling. These results are unrelated to the induction field remanence Br or coercive field Hc. At this stage, it is quite clear that the alloy $Fe_{65}Co_{35}$ becomes less absorbent.

The grinding process always involves a contamination by the grinding elements. More grinding time is more important contamination increases. In our case, this contamination was not detected by X-ray against it could be demonstrated by an EDX analysis. This analysis demonstrates the presence of Chrome (Figure 8).

Only three elements have been identified with varying concentrations: iron with 58.06%, cobalt with 30.02% and oxygen with 2.12%. This highlights the relative good experimental conditions

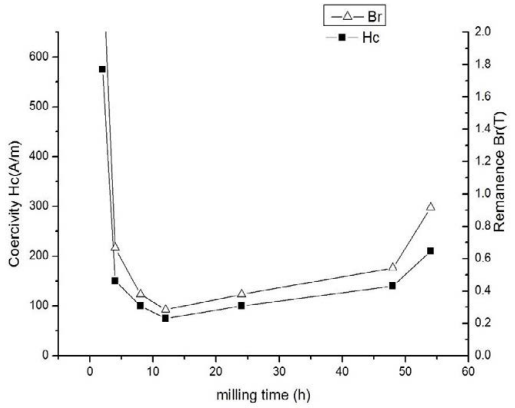

Figure 6: Evolution of remanence Induction Br and Coercivity Hc for MA $Fe_{65}Co_{35}$ powders for several milling times.

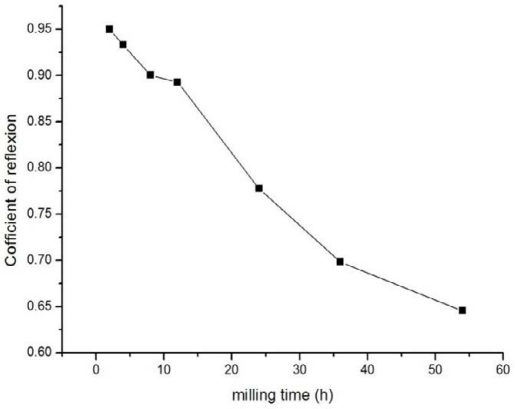

Figure 7: The coefficient of reflection for MA $Fe_{65}Co_{35}$ powders for several milling times.

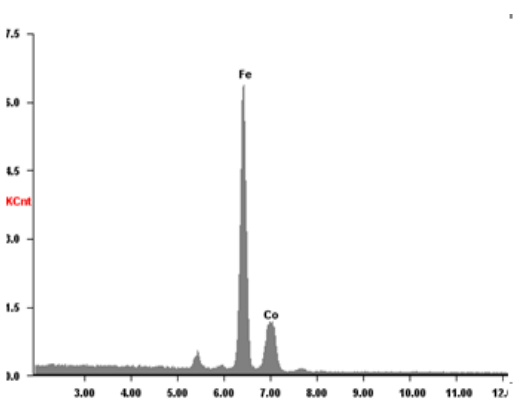

Figure 8: Average compositions atomic samples $Fe_{65}Co_{35}$ analyzed by electron microprobe.

in which the synthesis operation was made, because no contaminant element was detected, except for the inevitable incorporation of atmospheric oxygen, despite of manipulations in argon atmosphere. The contamination by the balls and the walls of jars is 2.03%. Finally, we can say that the contamination of the resulting alloy appears after 36 hours of grinding.

Conclusion

The first objective of this study is the development of iron-cobalt Nano composites different cobalt percentage mechanosynthesized. The mass ratio balls/powder mass is equal to 50:1, regardless of the alloy concerned. The grinding time is between 2 and 54 hours.

In the first part, we characterized the samples obtained by X-ray diffraction, scanning electron microscopy, laser particle size. The second part concerns the study of properties of magnetism and microwave.

The formation of binary alloys $Fe_{100-X}Co_X$ is possible after 12 hours of milling pure iron and cobalt elements. Their lattice parameter reaches 0.2866 nm after 54 hours of milling.

The size analysis shows a logarithmic distribution of the particle clusters. Their sizes are between 4 microns and 46 microns.

The study of the microstructure of powders helped highlight the different stages of the formation mechanism of Fe-Co alloys.

First, cobalt agglomerates in grains of iron. Secondly, the structure is refined by a phenomenon of fracture and of weld.

The distribution of cobalt inclusions is homogeneous

We showed the need not to extend the grinding time to prevent contamination.

Nanostructured materials Fe-Co alloys have higher values to those conventional. This fact is attributed to:

- Reduction of particle sizes.
- A super paramagnetic behavior.
- Increasing the interfacial region (grain boundary).
- Different kinds of anisotropy.

The reflection coefficient of the Fe-Co alloys nanostructured decreases and as the milling time increases. This fact can be attributed to the refinement of particle size. The refinement of the latter is accompanied by surface effects that cannot be neglected (the surface atoms can represent up to 70% of the total number of atoms).

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