Characterization by Non Destructive Testing Methods (NDT) of Nanomaterial Elaborated by Mechanical Alloying

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ABSTRACT
Mechanical alloying is one of powder metallurgical process. It consists to use the mechanical energy for reduce the grain size of material, realize a mixture to a scale grain and produce alloy. The procedure is based by successive welds and fractures caused by mechanical shock. So that, the end product being in the form of nanometers powder grain. The elaboration of nanomaterial by mechanical alloying improves the magnetic, physicochemical and mechanical properties of these materials. The present study treats experimental viewpoint the elaboration of material nanostructured form the basis of iron, cobalt by mechanical alloying (mechanical milling), the time of milling is the important parameters in this study, subsequently, and we have used the non destructive testing for the characterization of the nanomaterial.

Keywords: Mechanical Alloying, Mechanical milling, NDT, Fe-Co, Size of Crystallite, nanostructure, Magnetic control, hysteresis loops, eddy current, MEB, DRX

1. INTRODUCTION

Nanomaterials are a solid in which at least one parameter, such as the orientation of the crystal lattice or the atomic density varies on a scale of the order of several nanometers [1, 2]. These materials contain a significant proportion included in the atomic surfaces (nanoparticles), interfaces (multilayer's, nanocrystalline) and within the grain boundaries (nanostructured powders) [3], they have physical properties therefore very different from those materials microcrystalline or "massive". Their application is one of the technological challenges of the beginning of this millennium [4, 5]. Many methods of preparation of these materials have been developed in recent years. Mechanical alloying can be produced at industrial scale because of its low cost of implementation and the ease with which one can develop large amounts of materials [6, 7]. The work we will develop is the application of mechanical alloying in the development of the alloy $Fe_{80}Co_{20}$, In our study, we investigated their mechanism of formation, and this study treats in particular the evolution of physical and magnetic properties as a function of milling time.

2. ELABORATION BY MECHANICAL ALLOYING

Mechanical alloying is a powder metallurgy process. It consists to use the mechanical energy to reduce the grain size of the material and produce the alloy from this mixture. The procedure is based by successive welds and fractures caused by mechanical shock. In general, the final product comes is a nanometric powders. [8]

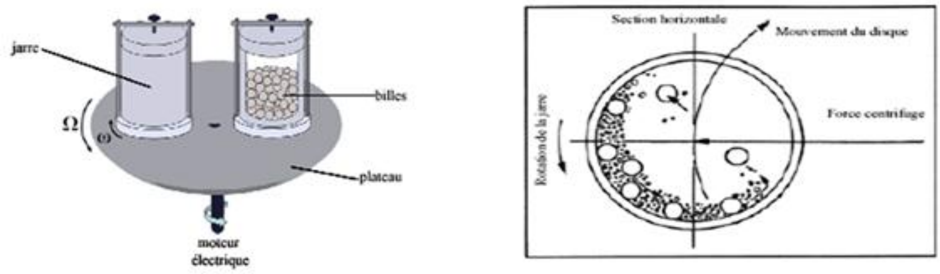

Fig.1: Mechanical milling

2.1. Synthesis and microstructural study

The observations by scanning electron microscopy were performed on samples of milled powder during the process. During the initial stage the powder particles are flattened by compressive force, the material is generally soft at this stage, Fig.2.

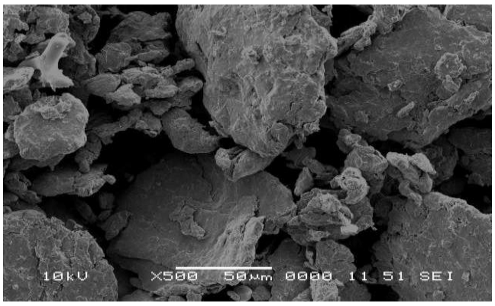

Fig.2: initial stage of mechanical alloying (Fe-Co after 5 h of milling).

During the intermediate stage, the welding process is important and plastic deformation leads to the formation a lamellar structure. The process of fracture and welding are dominant at this stage. The phenomenon of fracture causes the decrease of the thickness of the lamellae and dislocations can take place. At this point the chemical composition of the powder is always inhomogeneous see fig.3

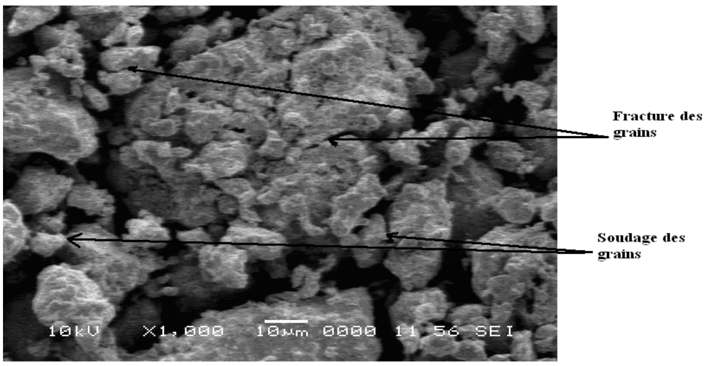

Fig.3: intermediate stage of mechanical alloying (Fe-Co after 10 h of milling).

During the final stage, a considerable refinement and a decrease in grain size are evident. The microstructure appears more homogeneous at the macroscopic scale as president of stages. No lamellar structure was observed and the alloy is already formed, Fig.4.

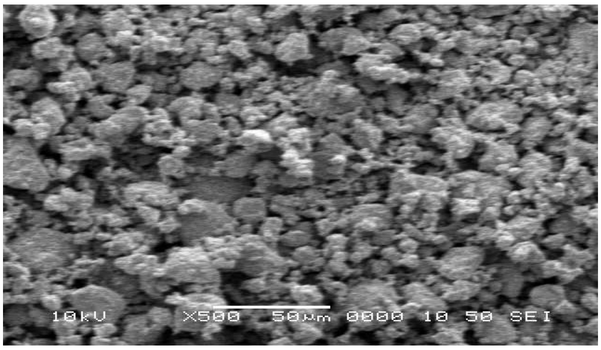

Fig.4: final stage de of mechanical alloying (Fe-Co after 15h of milling).

3. X-RAY DIFFRACTION

The measurements were performed using a Siemens goniometric assembly vertical axis with copper radiation KI, wavelength λ = 0.15406 nm. Fig.5 shows the kinetics of formation of the $Fe_{80}\,Co_{20}$ alloy considered during the milling of iron and cobalt. The peaks corresponding to cobalt decrease in intensity until they disappear completely, parallel iron peaks move to a new Bragg angles position. This displacement of the peaks of iron is due to the phenomenon of alloy. In parallel the cobalt atoms replace those of iron in the lattice of pure iron that deforms and gives rise to diffraction peaks of the new material. Once the solid solution is obtained, the peaks corresponding to pure cobalt disappeared

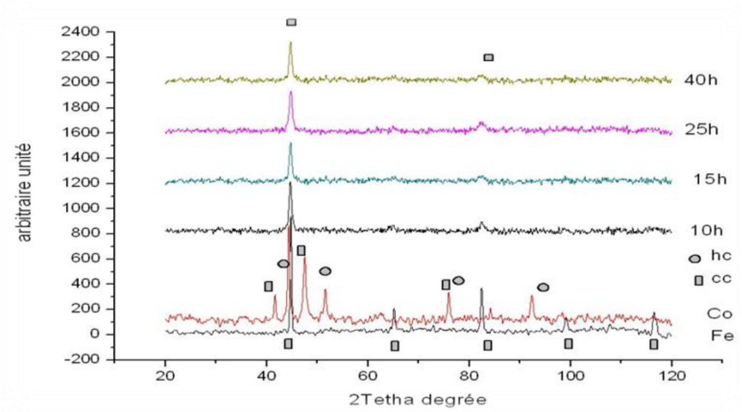

Fig.5: X-Ray Diffraction evolution of Fe-Co alloy during milling.

We used the method of Scherrer [9] to estimate the average crystallite size by measuring the width mid-height (FWHM) of diffraction peaks. This method is applicable only to crystallite size (d) between 10 and 150 nm and is done with great precision. More the larger the crystallite size is smaller; the width mid-height of the rays is large. We have:

$$d = K\lambda 360/2\pi.FWHM.\cos\theta$$

λ is the wavelength of X-rays and θ is the angle of diffraction. K is a factor (k = 0.89)

3.1. Lattice parameter

Fig.6 shows the evolution of the lattice parameter during milling. We note a rapid increase then stabilization from 15h. This behavior is explained in the first stage, by the diffusion of cobalt in the matrix of iron and / or deformations induced during milling. The decline in the second stage is explained by a dynamic balance between the effects of milling (defects, crystal lattice distortion, reduction of crystallite size, etc ...) and the restoration of the material (defect removal).

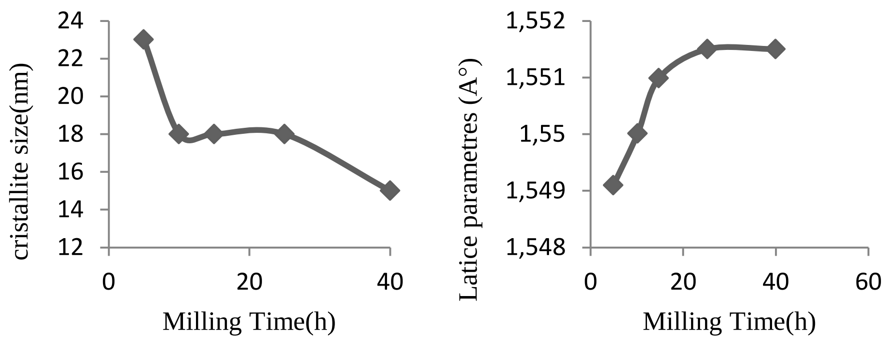

Fig.6: Evolution of crystallite size and lattice parameter during milling

4. Magnetic Measurement

The hysteresis loop is the response of a material to an applied field. We applied the magnetic measurements by the hysteresis loop to determine the magnetic properties of the alloy.

4.1. Influence of milling time on the Cœrcivity Hc

Cœrecitif field represents the characteristic of metal magnetic view point, because it also tells us the magnetic hardness of the material. We note from ig.7 that the milling time affects on the structure obtained.

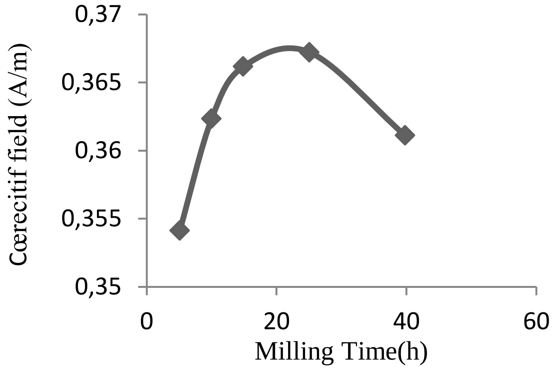

Fig.7: The magnetic saturation Bs Evolution during milling

4.2. Influence of milling time on the remanence field Br and the magnetic saturation Bs

The information returned from Br permits to determine the magnetic power of the material, this information is important because it also represents the quality of the magnetic power controlled. Fig.8 shows that the milling time increases, the material loses its ability and thus its magnetic polarization becomes difficult. The saturation field Bs, informs us that the ease of polarization of controlled materials.

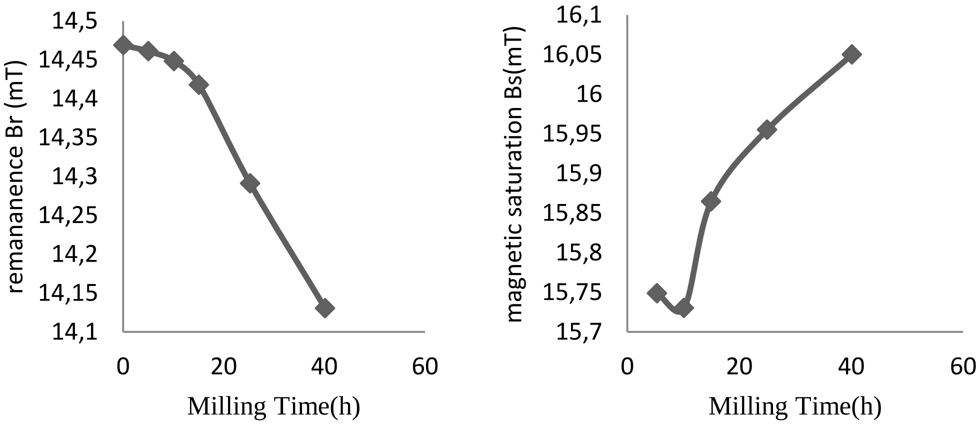

Fig.8: Br and Bs evolution during milling.

By comparing Fig.6 and Fig.8, we have shown that the lattice parameter "a" increases with the milling time because of the insertion of Co atoms in the lattice of Fe and this implies that the magnetization time becomes shorter and the Bs increases.

5. EDDY CURRENT MEASUREMENT

Recent analyzes of eddy current have shown that we can determine the microstructural state "nano structure" by impedance analyzing.

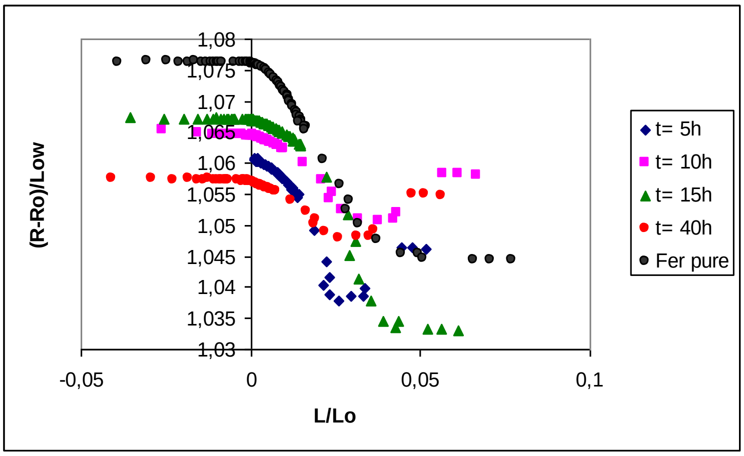

Figure (9): Eddy current measurement (Impedance diagram)

- Fig. 9 shows that the trajectories of Eddy Current obtained are different because of the variation of microstructural at different milling times:
  1 - Iron has a high impedance compared to Fe-Co
  2 - For the Fe-Co alloy, the milling time influences the impedance diagrams, we note that as the frequency increases, a bottleneck trajectory is obtained.

6.CONCLUSION

The confinement effect of the crystallite size can improve the magnetic and physical properties of materials. The formation of $Fe_{80}Co_{20}$ alloys is possible after 15 hours of milling. The hysteresis loops, from analysis of Hc, Br, and Bs, tells us about the appearance of magnetic structures obtained after milling, the extension of milling time affects the hysteresis loop. The phenomenon of saturation is obtained for any time longer than 40 hours; we get different trajectories in the impedance diagrams as a function of milling time. The low frequencies give us information on the formation of the binary alloy of the same way as the hysteresis loop; this is due to magnetic behavior of powders. The high frequencies used to analyze the forced onto the material. The decrease of the impedance corresponds to a drop in stress with a plateau around 15-25 hours explained by recrystallization of the material.

7. REFERENCE

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