DÉTERMINATION DES NANO STRUCTURE PAR LES MÉTHODES MAGNÉTIQUES

NANOSTRUCTURE DETERMINATION BY MICROMAGNETIC METHODS

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Résumé

La mécano synthèse est une des méthodes pour diminuer la taille des poudres. Généralement la taille des particules diminue avec le temps de broyage. Les méthodes de contrôle non destructif utilisant les techniques avancées de magnétisme permettent d'avoir un ensemble d'information concernant ces nanomatériaux. Dans ce papier nous avons étudié l'apport des méthodes magnétiques dans la caractérisation nano structurale. Des mélanges Nanocristallines $Fe_{(1-x)}$ Co_x , Fe ,Fe-Co-Cu ont été préparées par broyage suivant plusieurs conditions expérimentales. Les méthodes de contrôle non destructif ont fourni des informations concernant ces poudres et leurs comportements.

Abstract

Milling time is one of the most important factors for achieving the desired particle size of milled powders. Generally, the particle size of milled powders decreases with increasing milling time. However, long milling time results in agglomeration of small particles for some powders [9]. The using of the NDT method gives lot information about these microstructures in particular the magnetic NDT techniques [1-2]. In this paper, we examine the contribution of micromagnetic techniques in-the characterisation of nanostructure materials. Nanocrystalline $Fe_{(1-x)}$ Co_x , Fe ,Fe-Co-Cu mixtures have been prepared by mechanical alloying using a planetary ball mill under several milling conditions. The residual, the coerecitivity, the saturation moment and the eddy current methods can give the most result about the nanostructure determination Eddy currents has proved their efficiently for characterization a microstructure maters. In this study we try to make a new approach for application eddy current testing. The morphology of the nanostructure powders can be described by an assembly of nanocrystalline grains directed at random, knitted to each other by joints of grains. The exploitation of the experimental results was leaded jointly and has permitted a microstructural average description of these powders. In other respect, the use of these techniques permitted a comparison of experimental results and knowledge of structural and magnetic properties of nanostructure powders. This permitted to arrive to a conclusion of an average microstructure and an average magnetically behaviour of powders.

Keyword: NDT, nanostructure, powders, BN, remanence, corecitivity

INTRODUCTION

During the last 20 years, the mechanical alloying / ball milling process has been successfully used to prepare a variety of alloy powders including powders exhibiting supersaturated solid solutions, quasicrsytals, amorphous phases and nano-intermetallic compounds. During the mechanical alloying process, the powder particles are periodically trapped between colliding balls and are plastically deformed. Such a feature occurs by the generation of a wide number of dislocations as well as other lattice defects. Furthermore, the ball collisions cause fracturing and cold welding of the elementary particles, forming clean interfaces at the atomic scale. Further milling lead to an increase of the interface number and the sizes of the elementary component area decrease from millimeter to submicrometer lengths. As is true for most metallic materials, a good understanding of magnetic or mechanical properties often requires knowledge of the microstructure. This review therefore covers the microstructural aspects of FeCo alloys, attempting to sum up the knowledge available to date and identifying areas where further work is required. The contribution of NDT in the nanostructure characterisation will be important. The aim of the present study was to make a new approach for application eddy current testing for testing nanostructure mater [6-7.]

MAGNETIC THEORY

Mechanical alloying (MA) is a process that has proven to be a versatile tool to produce nanostructured composites and alloys from normally immiscible materials. The process parameters can be varied to control the degree of alloying and the microstructure characteristics, so it is possible to produce magnetic materials with novel properties.

As will be discussed later, many of the mechanical and magnetic properties of FeCo based alloys are conditioned by the grain size. It appeared therefore appropriate to discuss the problem of re crystallisation and grain-growth.Because of the ordering reaction, re crystallisation and grain-growth do not follow behaviour. Using X-ray, Borodkina *et al.]* studied the recovery and re crystallisation of FeCo alloys of different stoichiometries.

The magnetic properties of a material are generally very sensitive to the local atomic environment the hysteresis properties are governed by a combination of the intrinsic properties of the material, such as saturation polarisation, magnetic exchange and magneto crystalline anisotropy and the influence of the microstructure on the magnetization reversal process. The role of intergranular structure between the grains plays a significant role determining the magnetic properties, especially if the grain diameter is in the nanometer scale [6].Our interest in this system comes from the fact that granular binary alloys of FeNiCo and alloy exhibit giant magneto resistance (GMR), so the electric resistance of the alloy can change highly by applying of magnetic field. This kind of materials has potential application in magnetic sensors. Furthermore, we want to know if the addition of each elements (Co, Cu.)to the system Fe can be a way to control its microstructure in such a way that it improves magnetoresistance properties because of the magnetic properties of the FeNi system.

NDT APPROACH

According the state of art the eddy current testing (ET) has a strong application in the microstructure evaluation... This work discussed here is a study to materials structure characteristics and especially the nanostructure analysis using eddy current techniques. The design of eddy current probes was optimized in order to increase their sensitivity and their resolution. Studies of the magnetic fields in the vicinity of a probe are suitable to characterize the field activity and to optimize the controlled measuring process and the probe size [3].

The magnetic Barkhausen noise (MBN) technique has attracted considerable attention in recent years as a possible non-destructive technique for evaluation of microstructural parameters such as grain size, carbide precipitates, inclusions, dislocation and

characterizing the magnetic properties of steel. Barkhausen noise shows magnetizing discontinuities of ferromagnetic materials, such as alloys containing iron, Co, Ni [4].
In the application of variable magnetic field on the ferromagnetic material, many processes such as the creation and the annihilation of domain and the domain wall motion's are observed. Each domain is strained along its direction of magnetisation , the magnetostriction phenomena occurs, if the domain wall motion's are sufficiently rapid, the abrupt changes in local strain give rise to the generation of elastic waves, This phenomenon is observed for certain crystals when, one records the hysteresis loop B(H) which appears made up of a succession of marches.

EXPERIMENTAL PROCESS

SAMPLE REALISATION

The technique called mechanical alloys consists generally of grinding micrometric powders of several elements or alloys to incorporate them. The essential characteristic of this technique is to permit the obtaining of (nano-precipites or nano-objets) scattered homogeneously or heterogeneously within the matrix. The Fe, Ni, Co and Cu powders are mixed in jars filled with 16 steel tempered balls, within a controlled atmosphere (argon); the jars are fixed on the plate of the planetary grinding of the type Retsch PM400. The jars and the plate are mechanically coupled or attached. With 200 tr/min speed and varying the grinding time of each sample, the structure of the composites are determined by x-rays diffraction. The nanocristallites' sizes after mechanical treatment and reheating or tempering treatment are calculated. Powder particles are subjected to severe mechanical deformation due to collisions with balls and are repeatedly deformed, cold welded and fractured...
The powders densification made visible many stages. During the first to the much weak stress or strain, we take part in a putting into position of the grains; it is principally checked by the morphology of particles. The plastic deformation is produced during the second stage the roughness presence could play the part of an amplifier of strains or stress located in contact joints between the particles. On this other hand, the high proportion of grains joints let to think the mechanism of joints or similar to those intervening in the amorphous matters could also take part in the deformation.

MEASUREMENT AND ANALYSIS

The grinding with great energy is a complex process and implies an optimisation of a number of variables to obtain the final product. Certain of these parameters have an impact over the final product's nature. The production of nanostructure powders requires more precautionary measures.
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.X-ray diffraction measurements were performed for all mixed powders at the different stages of milling. The grains size result obtained by X-ray for different sample are represented in next table

Times		0	2	4	8	12	24	36	54	100
Fe	Size (nm)	32.7	-	27.57	18.55	15.79	14.35	13.19	12.69	-
FeNi		27	22.40	12.30	12.20	11.60	11.2	10.71	-	9.82
FeCo		32.7	18.1	17.3	15.5	15.2	-	13.8	13.6	-
FeCuCo		33.2	-	16.05	13.26	15.02	15.59	16.40	14.04	-

The result obtained by the NDT techniques, in particular a magnetic process can give an idea on crystallographic aspect of simple.

The originality of this work is giving by the eddy current analysis. For the nanostructure we have shown the relation between impedance and micro hardness measures. The evolution of the trajectory is the same according to the milling time and then it is possible to have some mechanical information about this nanostructure by NDT technique as eddy current, figure 5.d, [14].The Eddy Current method allows linking the impedance variation to the crystallographic aspect of sample. The figures 1-4, show the variation impedance according to the milling time and we observe a maximum for each mixture. These maximum is in relation with crystallographic analysis in the material. This characterization is verified for $Fe_{(1-x)}$ Co_x, Fe, Fe-Co-Cu mixtures, also we notice that Z get an extreme after a certain hour of milling. The XRD patterns for the Fe_{60} Co_{40} powders, as received and for different milling times, are shown in Fig. 6. After milling for 12 h, Co peaks disappear completely and Fe peaks shift to the low-angle side. Based on the examination of the lattice parameter of Fe, calculated from the XRD patterns using the Nelson-Riley method [10], it is indicated that the alloying formation occurs in a solid state during milling. These extreme is also verified when we proceed at the add the Co rate (figure), this maximum is situated around 36 hours, this result is confirmed by the X diffraction. Figure5 ,as the same of eddy current analysis, show that a micro magnetic measurements as remanence Br (t) , field corecitive Hc (t) , give a similar variation according to the milling time .The figure give the X-ray diffraction patterns of the powder mixtures at different stages of processing.

The different contributions of magneto crystalline anisotropy strongly differ of those of microcrystalline system. Besides, the wall concept of domain disappears because the grains become monodomain. However, the magnetocrystalline anisotropy or of the shape of an assembly of crystalline nanograins distributed at random has an influence on the magnetic answer of manometers as indicated by the variation of (crystallography parameters) founded after 100 hours of grinding, the $Fe_{80}Ni_{20}$ (crystallography parameters) grows from 2,8664 A° to 2,87111A° in the plane (110), figure 8.This result is important .The NDT micro magnetic measure allow the possibility to have information on the crystallographic aspect of material, this results is obtained for $Fe_{(1-x)}$ Co_x , Fe, Fe-Co-Cu mixtures, figure 8.

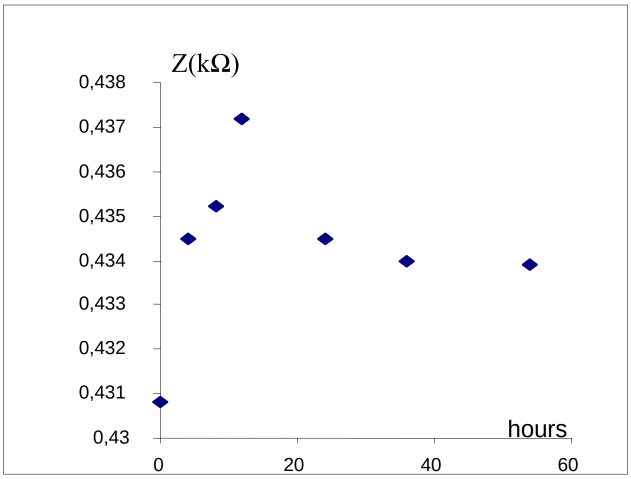


Figure1: comportment of impedance CuFeCo according milling times

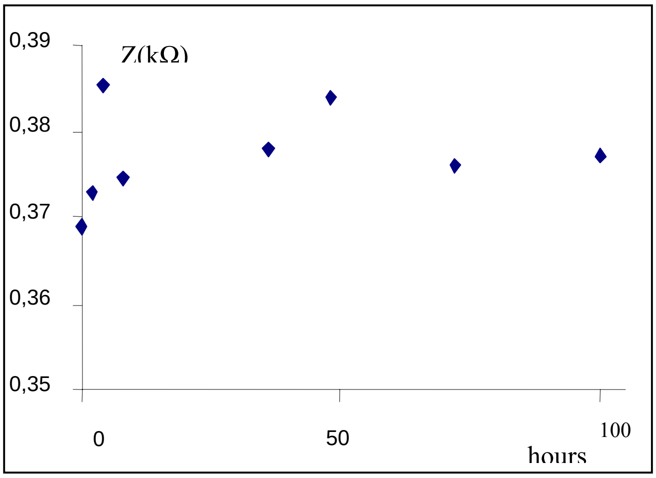


Figure2: comportment of impedance Fe80Ni20 according milling times

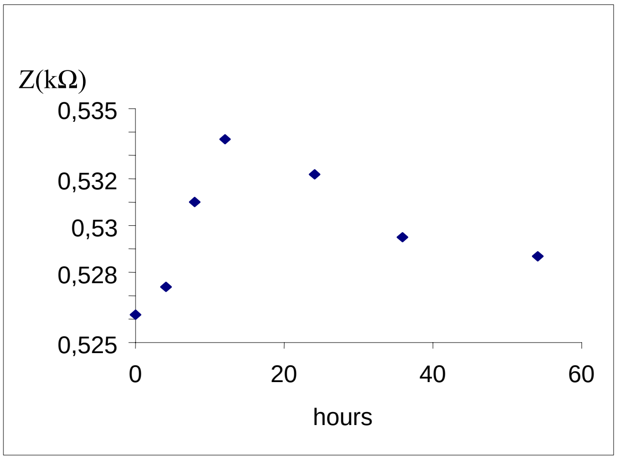


Figure 3: impedance Variation according to the milling times

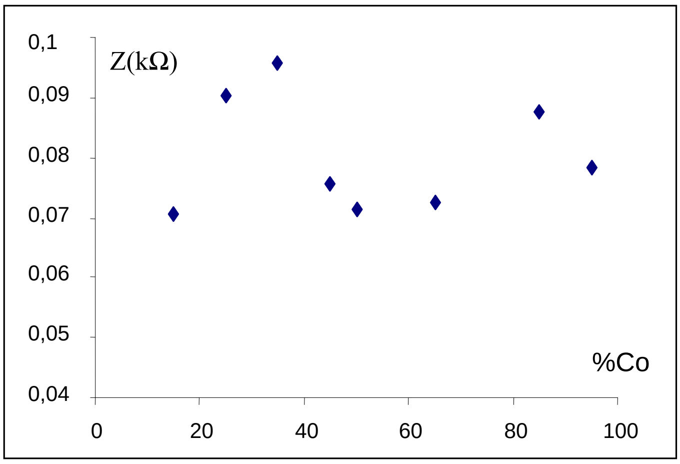
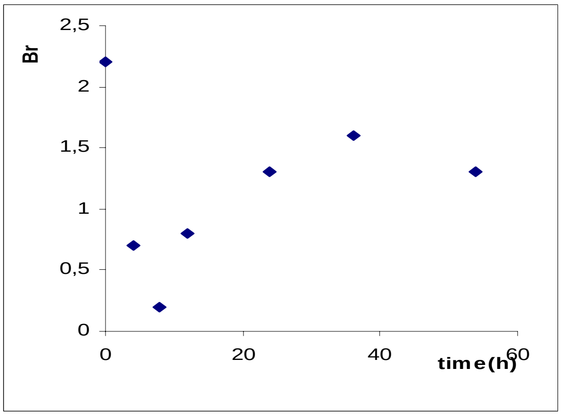
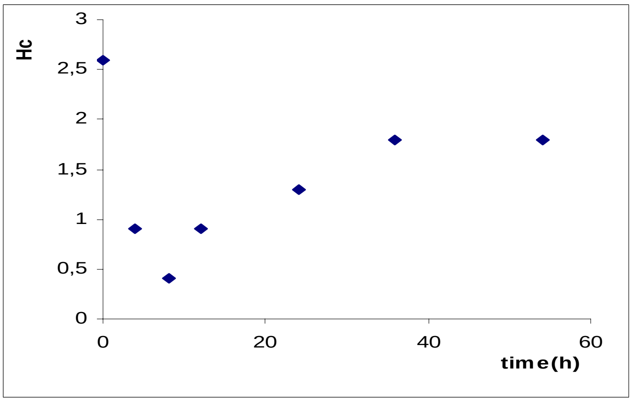


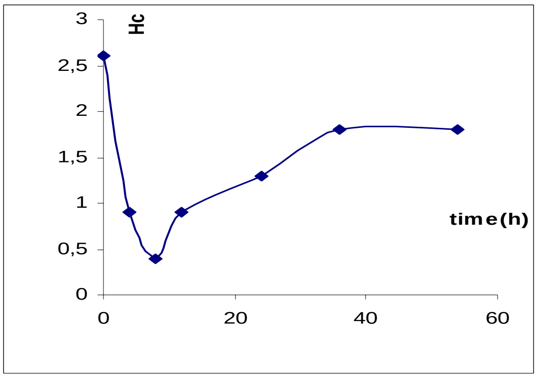
Figure 4: impedance Variation according to the %CO



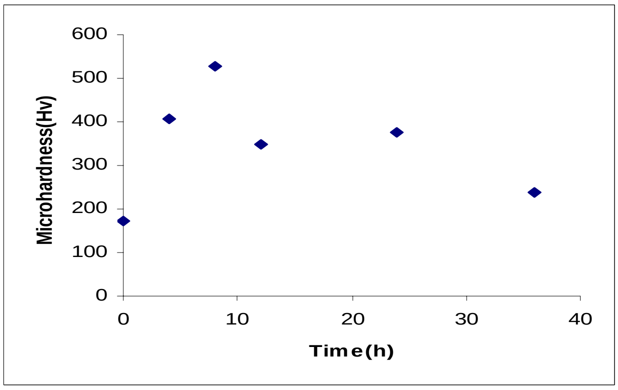
a: Remanence according to the milling time



b: Coercivity according to the milling time for



c: Impedance according to the milling time



d: micro hardness according to the milling time

Figure 5: NDT Measurements for the CuFeCo $_{\text{alloy}}$

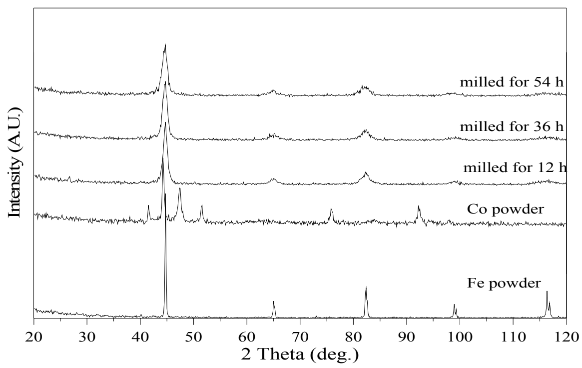


Figure 6: X-ray diffraction patterns of the powder mixtures at different stages of processing

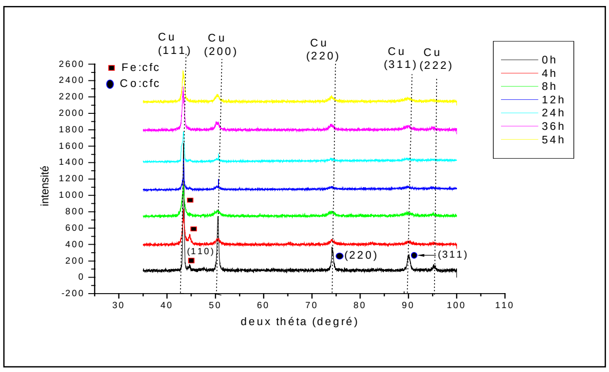
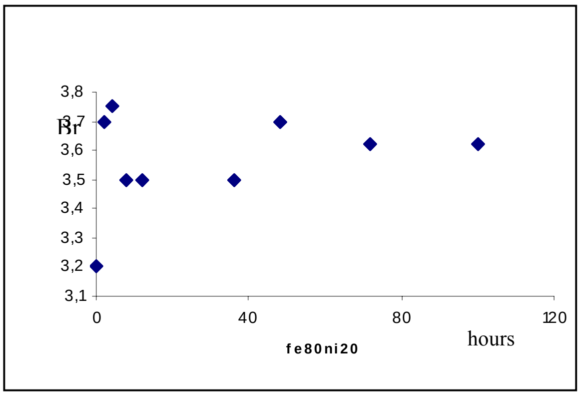
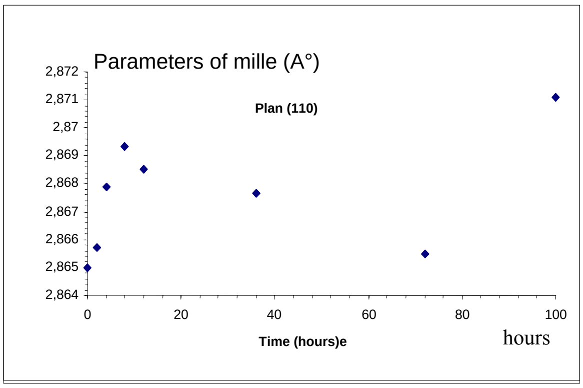


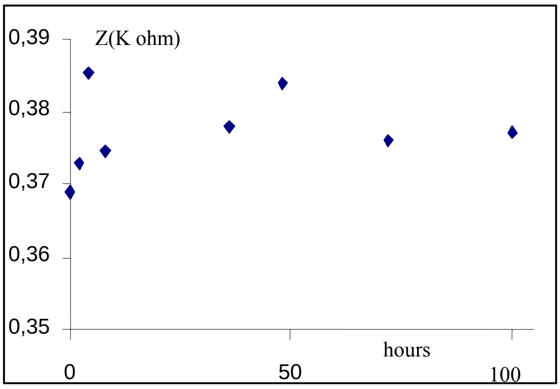
Figure 7: X-ray diffraction patterns of the powder mixtures at different stages of



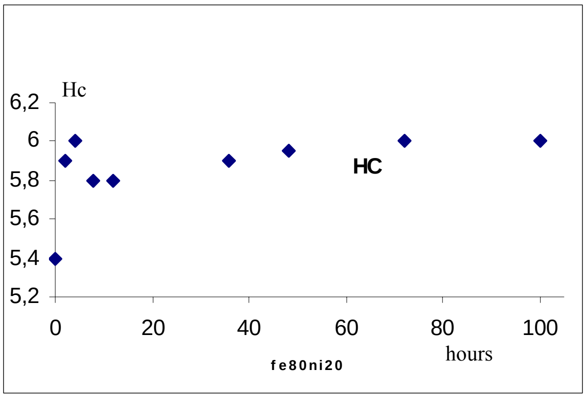
a:Remanence according to the time milling



c: crystallography parameters in the plane (110) in different grinding times.



f: Impedance according to the time milling



d:Coercitivity according to the time milling

Figure 8: Micro magnetic methods and crystallography as a function of milling time for Fe_{80} NI_{20} alloy.

CONCLUSION

This work is about the correlations between the grinding conditions (Temperature and intensity), the structural propriety and electromagnetic study of the compounds $Fe_{(1-x)}$ Co_x , Fe, Fe-Co-Cu mixtures, elaborated by mechanical alloying process with high energy.
The use of the characterization methods magnetic measure (ET……) gave a fine description of nanostructure proprieties.The X-ray has confirmed all result obtained by NDT methods.
The micro magnetic NDT in metal powders is influenced by variation both of grains size and the new mixture (new crystallography parameters). The morphology of the nanostructure powders can be described by an assembly of nanocrystalline grains directed at random, knitted to each other by joints of grains. The coercive field and the residual magnetism were deduced from hysteresis curves, they are represented according to grinding duration.
We have obtained some information about crystallography specimen. The use of these techniques permitted a comparison of experimental results and knowledge of structural and magnetic properties of nanostructure powders.

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