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MAGNETIC, MICROWAVE AND ABSORBING PROPERTIES Fe-Co OF ALLOY SYNTHESIZED BY MECHANICAL ALLOYING PROCESS

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ABSTRACT

Nanocrystalline $Fe_{(1-x)}$ Co_x mixtures have been prepared by mechanical alloying using a planetary ball mill under several milling conditions. Their structures and magnetic properties were investigated. Mechanical alloying is a non-equilibrium process for materials synthesis. The structural effects of mechanical alloying of powders were investigated by scanning electron microscopy, X - Ray diffraction analysis and bench of microwaves. Consequently, the alloy powder with average grain size 10-13 nm was obtained. Maximum saturation magnetization Ms was obtained at the composition of 65 % Co. Microwave measurements were performed on the mechanically milled $Fe_{(1-x)}$ Co_x powder. It has been shown that fine nanocrystalline Fe-Co alloy powders prepared by mechanical milling are promising for microwave applications.

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

1. Introduction

More than 20 years ago, mechanical alloying was developed as a way to circumvent the limitations of conventional alloying [1]. With this metallurgical process, powder particles are subjected to severe mechanical deformation due to collisions with balls and are repeatedly deformed, cold welded and fractured. Through recent years, mechanical alloying has emerged as a versatile technique for producing materials away from equilibrium, like for example, nanostructured alloys and amorphous alloys [2,4]. The mechanism of phase formation has been explained by an interdiffusion reaction of the components occurring during the milling process. Metastable phase formation has been observed as soon as a stable phase formation is suppressed under specific milling conditions. Intermetallic compounds could be energetically destabilized because of the chemical disorder, which is introduced by the deformation during the milling phase [5] particularly. Many nanostructured magnetic materials have exhibited excellent soft magnetic properties, which suit so many applications.

In the same way, it is well known that fine / ultrafine magnetic metal particles are promising candidates as microwave absorbers. Fe-Co alloys have been for some time the ideal materials for applications where a high magnetic saturation is a design parameter, particularly in the aerospace industries where volume and mass need to be minimized [6]. There has been a resurgence of interest in these alloys, particularly with the development of very strong variants [7,8]. The interest was aroused primarily by the soft-magnetic materials - needed for the construction of electric motors - which can be embedded in hot regions of aircraft engines [9-11].
Within this work, the structures and magnetic properties of mechanically alloyed $Fe_{(1-x)}Co_x$ powders have been investigated and the coefficient of reflection studied within a frequency range of 9, 9.5 and 10 GHz.

2. Experimental details

Highly pure initial Fe and Co powders have been used as starting materials and mixed at the desired compositions (15, 25, 35, 45, 65, 85 and 95% Co) in a planetary ball mill (Retsch PM 400). Initial powders, having an average particle size of 70 µms and a mean grain size of 30 nm, were introduced into a cylindrical tempered steel vial of capacity 25 ml. The materials were sealed under high purity argon atmosphere with ball to mass ratio 50:1. The milling was performed up to 36 hours with a planetary rotation speed at about 380 tr/mn. About 2 g of nanocrystalline powders of Fe-Co were compacted at ambient temperature at a 2 Gpa pressure during 2 hours in a cold uniaxial press (13 mm inner diameter, 25 mm outer diameter and 2.5 - 3 mm height).
The powder mixtures were characterized by a Siemens D500 diffractometer using Cu Ka radiation. Scanning electron microscopy was used for morphology and microstructure observations. Magnetic measurements were carried out with a Teslameter and an electronic oscilloscope. The coefficient of reflection at various frequencies was characterized by the use of a rectangular wave-guide.

3. Results and discussion

Fig.1 represents the evolution of saturation magnetization Ms and coercivity Hc according to the percentage of cobalt. Mechanical and magnetic properties might be affected by changes as well in the structure as in the percentage of cobalt, particularly coercivity and saturation magnetization Ms which remain important parameters in the industrial field [12]. One can notice that maximum saturation magnetization Ms and minimum coercivity Hc are obtained for the alloy to 65% Co. The coercivity Hc curve decreases down to the alloy 65% Co. Beyond that point, one can notice an increase of coercivity Hc, suggesting an important reduction of the crystallites size [13] and each grain behaves as a magnetic domain (monodomain grain) thus, eliminating the influence of Block's partitions. The decrease of saturation magnetization Ms is likely due to joints of grains whose fracture cannot be neglected.
Through recent years, with the development of radar technology, microwave communication and the need for anti-electromagnetic interference coatings, there has been considerable interest in the application of magnetic materials related to radar design absorber [14]. Magnetic powder materials with low values of the coefficient of reflection have been promising in the application of microwave absorption. The effective microwave of the composite depends on both intrinsic characteristics of the particles and their microstructural, electrical and magnetic parameters: such as particle size, saturation magnetization and magnetic anisotropy field. The coefficient of reflection has been studied within a frequency range of 9, 9.5 and 10 GHz.

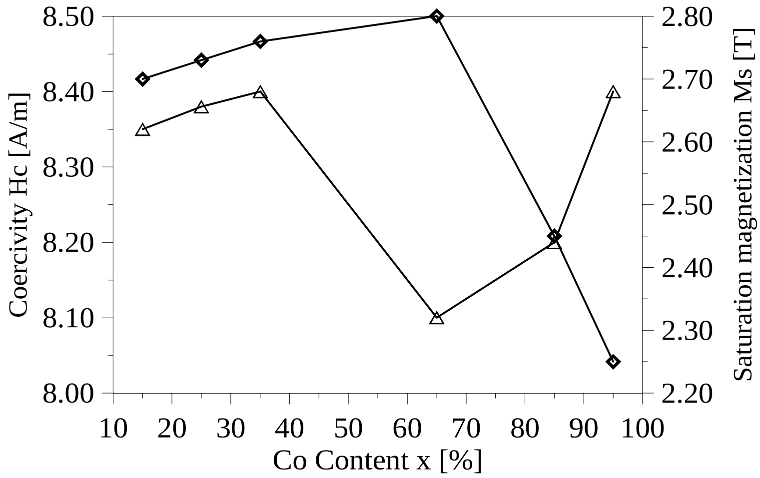

Fig. 1: Dependence of Co concentration on saturation magnetization Ms and coercivity Hc for MA Fe - Co powders after 36 hours milling time.

Fig. 2 represents the variation of the coefficient of reflection according to the concentration of the cobalt with various frequencies. One can notice that the coefficient of reflection depends highly on the frequency, thus decreasing when the frequency increases. Furthermore, it is noticeable that the smallest value of the coefficient of reflection is obtained with the alloy 65% cobalt. These results have, without any doubt, a link with the highest value of the saturation magnetization Ms. It is obvious that the weak value of coercivity might interfere in these results. At this stage, it is clear enough that the Fe_{35} Co_{65} alloy becomes absorbing.

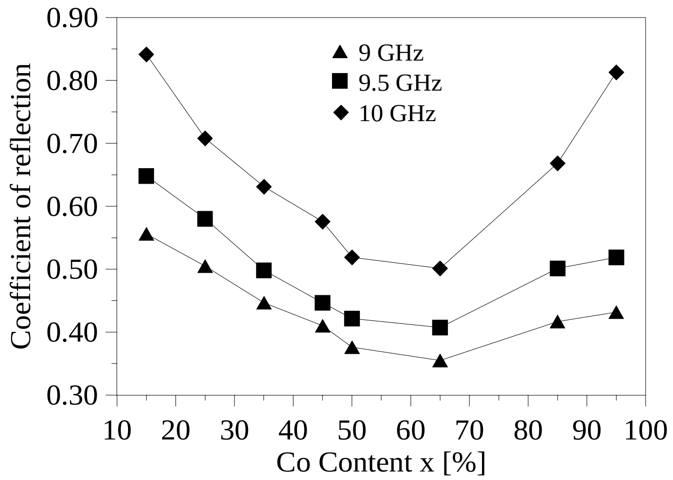

Fig. 2: Dependence of Co concentration upon the coefficient of reflection for MA Fe - Co powders after 36 hours milling time.

Some microstructural aspects of the powder according to the time of milling are shown. At the beginning, one can notice that the powders have a spherical shape (Fig. 3).

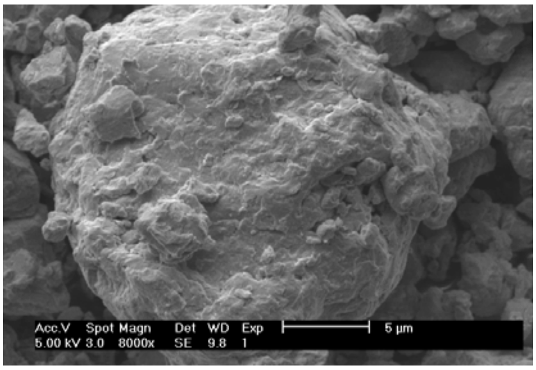

Fig. 3: Micrograph of unmixed Fe-Co powders.

After 2 hours milling, owing to the competition between fracture and welding phenomena, one can notice a small change in the morphology of the powders (Fig. 4) where compound particles are formed.

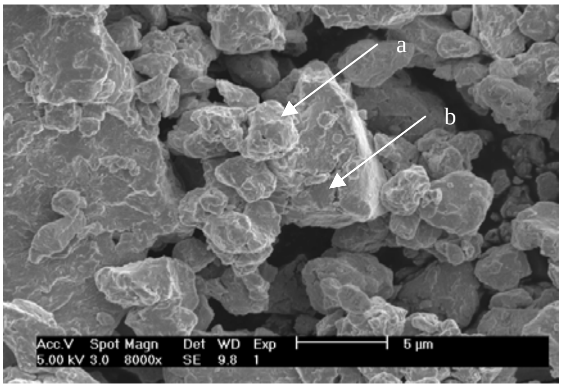

Fig. 4: Micrograph of Fe-Co powders shows welding (a) and fracture (b).

Beyond that stage, the process becomes more important and the plastic deformation leads to the formation of lamellar and multilayer structures (Fig. 5a). Milling, prolonged up to 36 hours, leads to a refinement of particles with agglomerates formation [14] (Fig. 5b) closely related to the existence of an equilibrium between fractures and welding phenomena.

a) b)

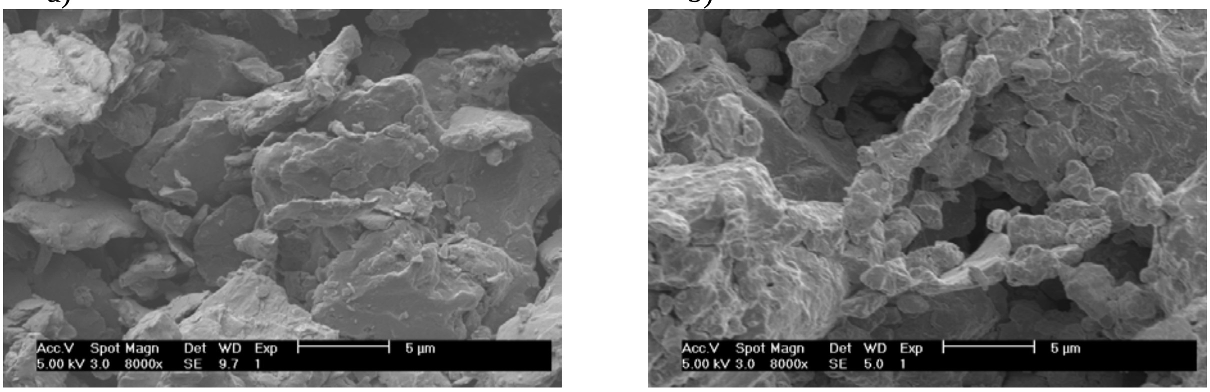

Fig. 5: SEM micrographs of Fe-Co powders prepared with mechanical alloying after milling time a) 12 hours: b) 36 hours.

4. Conclusions

The Fe-Co alloys powders have been prepared by a mechanical alloying process using high-energy ball milling. Intensive milling (36 hours) results in a non-equilibrium microstructure composed of refined grains ranging from 10 to 13 nm. Increased milling time leads to the refinement of the structures with agglomerates formation. Maximum saturation magnetization is observed in the composition of 65% Co while investigating the magnetic properties with varying Co concentration. Our microwave measurements have shown that small particle size tends to a low coefficient of reflection. The fine Fe_{35} Co_{65} powder having the smallest coefficient of reflection 0.35 at 10 GHz. These results are the indication that mechanically milled fine alloy powders are promising candidates to microwave applications.

5. References

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