JMEPEG (2006) 15:349-351
DOI: 10.1361/105994906X108675

1059-9495/$19.00

Formation and Magnetic Properties of Nanocrystalline $Fe_{60}Co_{40}$ Alloys Produced by Mechanical Alloying

S. Bergheul, H. Tafat, and M. Azzaz

(Submitted April 30, 2005; in revised form November 15, 2005)

The $Fe_{60}Co_{40}$ alloys were prepared by mechanical alloying of the Fe and Co powders using a high-energy ball mill. They were studied with respect to phase formation and magnetic properties using x-ray diffraction, scanning electron microscopy, and measurements of coercivity and remanence (B_r). The evaluation of Fe lattice parameters during milling showed that formation of a body-centered cubic solid solution occurred by mechanical alloying after 12 h of milling. Intensive milling of Fe-Co powders results in a nonequilibrium microstructure characterized by grain refinement to a minimum of 10-13 nm and the introduction of internal strain up to 0.5%.

Keywords iron-cobalt powder, magnetic measurements, mechanical alloying, nanoparticles

1. Introduction

Nanocrystalline materials, as a result of the considerable reduction of grain size and their significant volume fraction of grain boundaries and triple junctions, have exhibited many unusual mechanical, physical, chemical, and electrochemical properties compared with conventional polycrystalline or amorphous materials (Ref 1). Several studies demonstrated that the level of understanding of nanostructured materials has reached the point where a growing range of applications can be anticipated (Ref 2). Mechanical milling is one technique that is used to reduce grain size and to obtain nanocrystalline materials (Ref 3–5). Utilizing high-energy ball, or attritor, mills, nanoscale processing continues to grow in importance, at least as an experimental laboratory tool. Its attractiveness is its simplicity and low capital equipment cost.

Mechanical alloying of the starting $Fe_{100-x}Co_x$ powders leads to the formation of the body-centered cubic (bcc) solid solution for x up to about 70%, while for higher Co content, the face-centered cubic (fcc) structure is observed (Ref 6, 7). The Fe-Co system, with a −1 kJ/mol heat of mixing, is very interesting with respect to phase formation during mechanical alloying. In addition, its magnetic properties, such as coercivity and remanence (B_r), are very sensitive to alloy formation and microstructure, respectively (Ref 8).

In this work, the $Fe_{60}Co_{40}$ alloys were obtained by the mechanical alloying technique. The formation of the bcc solid solution is investigated as a function of milling time. The structural characteristics and magnetic behaviors of $Fe_{60}Co_{40}$ powders were also evaluated.

S. Bergheul, Department of Aeronautics, University of Blida, BP 270, Blida, Algeria; **H. Tafat** and **M. Azzaz**, Laboratory S.G.M., U.S.T.H.B., BP 32, Bab-Ezzouar, Algeria. Contact e-mail: sabergheul@yahoo.fr.

2. Experimental Procedures

Elemental Fe (particle size 50 μm) and Co (particle size 10 μm) powders were milled in a planetary ball mill (RETSCH PM 400) in Ar using 16 steel balls 20 mm in diameter. The ball-to-powder weight ratio was about 50:1. High-energy ball milling was performed for milling times up to 54 h (i.e., 2, 4, 8, 12, 36, and 54 h). The rotation speed (Ω) of the disc and vials was equal to 380 translations/min.

The microstructures of the resulting milled powder alloys were investigated using x-ray diffraction (XRD) (Siemens D 500 diffractometer) using $CuK\alpha_1$ radiation. The mean size of the crystallites was deduced from the line broadening. The morphology aspect of the samples was analyzed using a scanning electron microscope (SEM) with a field-emission gun as the electron source. About 2 g of nanocrystalline powder was compacted at 293 K and 2 GPa pressure for 90 min. The magnetic measurements were carried out using a Teslameter with attached electronic oscilloscope.

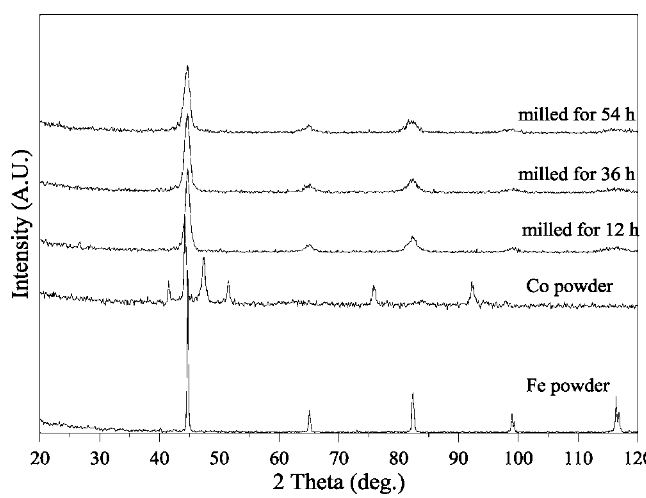

Fig. 1 XRD patterns of the powder mixtures at different stages of processing

3. Results and Discussion

The XRD patterns for the milled $Fe_{60}Co_{40}$ samples are shown in Fig. 1. After 2 h of milling, the peaks originating from both the elemental Fe (bcc) and Co [hexagonal close-packed (hcp)] powders are clearly seen in Fig. 1. The progressive mixing of the starting elements due to the high-energy ball milling process can be observed with the increase in milling time. The peaks related to pure Co vanish gradually and then disappear completely for milling times longer than 12 h. This shows that Co diffuses into the bcc structures and forms a disordered Fe-Co solid solution with a bcc structure. At the same time, the widths of the peaks related to the bcc phase increase gradually with the milling time due to the decrease in grain size and increase in internal strain.

The alloy formation in the solid state was also confirmed by the evaluation of the lattice parameter of Fe during milling. The Fe lattice parameter, as calculated from the XRD patterns using the Nelson-Killey method (Ref 9), increased with milling time (Fig. 2), and showed that Fe and Co form a solid solution through milling. Microstructural changes induced by extended

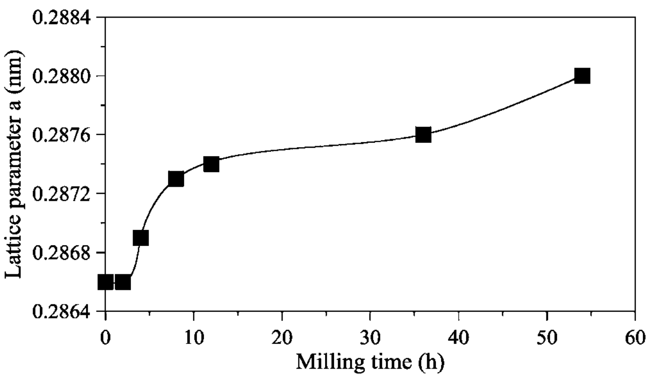

Fig. 2 Lattice parameter as a function of milling time for MA $Fe_{60}Co_{40}$ alloy

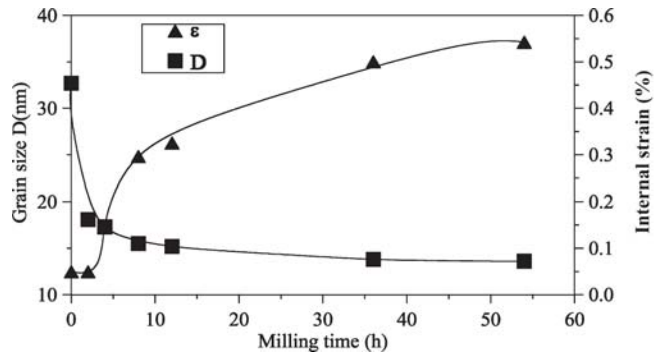

Fig. 3 Grain size and internal strain as a function of milling time for MA $Fe_{60}Co_{40}$ alloy

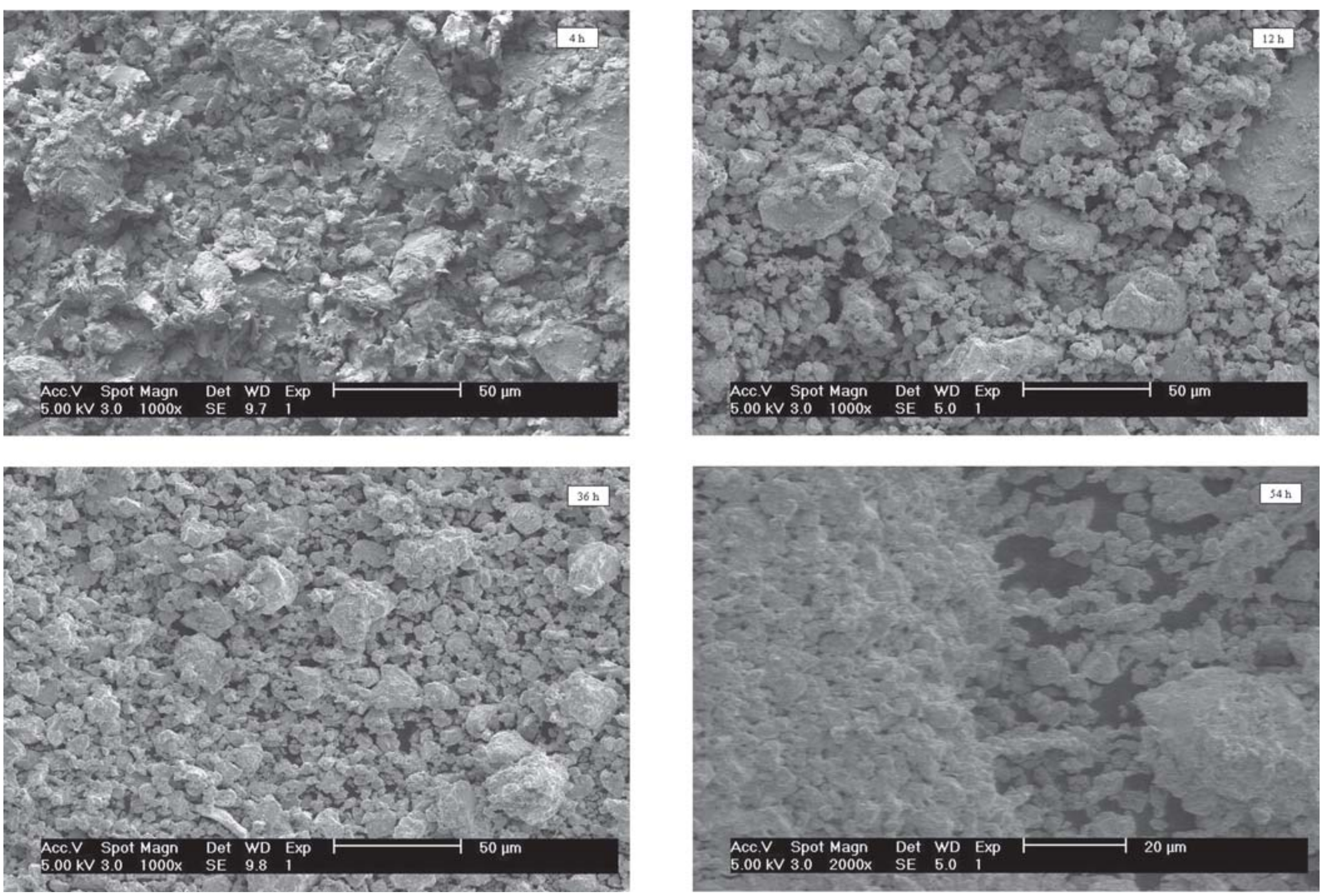

Fig. 4 Scanning electron micrographs of alloy structures in various milling times

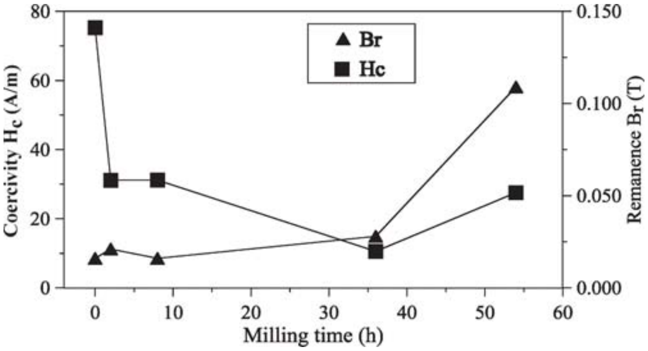

Fig. 5 Coercivity and remanence as a function of milling time for MA $Fe_{60}Co_{40}$ alloy

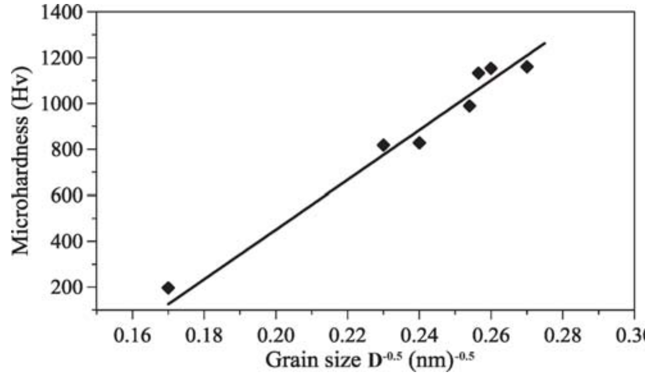

Fig. 6 Grain size dependence of the microhardness for MA $Fe_{60}Co_{40}$ alloy

milling, such as grain size reduction and increase of internal strain, are presented in Fig. 3. SEM micrographs (Fig. 4) show the alloy structures obtained after various milling times. Progressive refinement of the grains according to the time of milling can be noted. At the beginning of the mechanical alloying process, the particles are flattened by the plastic deformation caused by the compressive forces induced by the contacts between balls/powder/balls. The average grain size and the internal strain were calculated by the Hall-Williamson method. The grain size decreased sharply down to 13 nm, and the internal strain increased up to 0.5% due to the excessive deformation during milling. The grain size depends strongly on the chemical composition, the temperature of fusion, and the structure of the alloy (Ref 10, 11).

Figure 5 shows the milling time effect on the evolution of coercivity and remanence of $Fe_{60}Co_{40}$. The coercivity decreased sharply down to about 10 A/m while the remanence increased to 0.1 T due to the excessive deformation during milling. The increase in remanence (B_r) after 36 h of milling and the decrease in coercivity (H_c) at 36 h of milling are due to the small grain size developed during milling. The low and high values of H_c and B_r, respectively, indicate that the $Fe_{60}Co_{40}$ alloy can be considered as magnetic monodomains. However, the increase in H_c after 36 h of milling is caused by an increase of Fe content caused by the abrasion of Fe from the steel balls and container walls and its subsequent incorporation into the Fe-Co alloy.

The nonequilibrium microstructure of the $Fe_{60}Co_{40}$ alloys is achieved by grain size reduction and the introduction of internal strain after intensive milling. Finer grain microstructures are obtained at high milling intensities. The microhardness of the $Fe_{60}Co_{40}$ alloy as a function of grain size is shown in Fig. 6. The increase in microhardness at low grain size (13 nm), can be explained by means of the Hall-Petch relationship (Ref 12, 13). The yield stress required to deform a polycrystalline material by dislocation movement depends on the grain size *D*.

4. Conclusions

The effect of milling time on the microstructure and magnetic properties of the $Fe_{60}Co_{40}$ alloys prepared by mechanical alloying using high-energy ball milling has been studied. The main conclusions are as follows:

- The bcc Fe-Co solid solution was identified by XRD.
- Intensive milling of Fe-Co powders resulted in a small grain size (~13 nm on average) and the introduction of 0.5% internal strain.
- Increase in remanence (B_r) after 36 h of milling time and decrease in coercivity (H_c) at 36 h of milling are due to the small grain size developed during milling.
- Increase in microhardness of the $Fe_{60}Co_{40}$ alloy was explained by means of the Hall-Petch relationship.

Acknowledgment

The authors gratefully thank Professor M. Hadji from the Department of Mechanical Engineering of the University of Blida for his help.

References

1. C.B. Wang, D.L. Wang, W.X. Chen, and Y.Y. Wang, Tribological Properties of Nanostructured WC/CoNi and WC/CoNiP Coatings Produced by Electro-Deposition, *Wear,* 2002, **253,** p 563-571
2. B.H. Kear and G. Skandan, Report on the Third International Conference on Nanostructured Materials (Kona, Hawaii), July 7-12, 1996, *Nanostruct. Mater.,* 1996, **7,** p 913-917
3. S. Mi and T.H. Coutney, Processing Structure and Properties of Ni-W Alloys Fabricated by Mechanical Alloying and Hot-Isostatic Pressing, *Scripta Mater.,* 1998, **38,** p 637-677
4. R. Sundaresan and F.H. Froes, Mechanical Alloying, *J. Metals,* 1987, **8,** p 22-27
5. C.C. Koch and J.D. Whittenberger, Mechanical Milling/Alloying of Intermetallics, *Intermetallics,* 1996, **4,** p 339-355
6. M. Sorescu and A. Grabias, Structural and Magnetic Properties of $Fe_{50}Co_{50}$ System, *Intermetallics,* 2002, **10,** p 317-321
7. Ch. Kurt and L. Schultz, Formation and Magnetic Properties of Nanocrystalline Mechanically Alloyed Fe-Co, *J. Appl. Phys.,* 1992, **71,** p 1896-1900
8. T. Sourmail, Evolution of Strength and Coercivity During Annealing of FeCo Based Alloys, *Scripta Mater.,* 2004, **51,** p 589-591
9. B.D. Cullity, *Elements of X-ray Diffraction,* 2nd ed., Addison-Wesley, Reading, MA, 1978, p 356-359
10. N.E. Fenineche, R. Hamzaoui, and O. El Kedim, Structure and Magnetic Properties of Nanocrystalline Co-Ni and Co-Fe Mechanically Alloyed, *Mater. Lett.,* 2003, **57,** p 4165-4169
11. R. Elkalkouli, M. Grosbras, and J.F. Dinhut, Mechanical and Magnetic Properties of Nanocrystalline FeCo Alloys Produced by Mechanical Alloying, *Nanostruct. Mater.,* 1995, **6,** p 733-743
12. P.R. Soni, Mechanical Properties of Mechanically Alloyed Materials, *Mechanical Alloying,* Cambridge International Science Publishing, Cambridge, UK, 2000, p 65-67
13. G.E. Fougere, J.R. Weertman, and R.W. Siegel, On the Hardening and Softening of Nanocrystalline Materials, *Nanostruct. Mater.,* 1993, **3,** p 379-384