

## Synthesis of Nanostructured Fe-Co Alloy Powders from Metal Salts

Young Jung Lee, Jea Sung Lee, Young Ik Seo and Young Do Kim\*

*Division of Materials Science and Engineering, Hanyang University, Seoul 133-791, Korea*

(Received September 1, 2006; Accepted October 2, 2006)

**Abstract** Magnetic properties of nanostructured materials are affected in complicated manner by their microstructure such as grain size (or particle size), internal strain and crystal structure. Thus, studies on the synthesis of nanostructured materials with controlled microstructure are necessary for a significant improvement in magnetic properties. In the present work, nanostructured Fe-Co alloy powders with a grain size of 50 nm were successfully fabricated from the powder mixtures of (99.9% purity)  $\text{FeCl}_2$  and  $\text{CoCl}_2$  by chemical solution mixing and hydrogen reduction.

**Keywords :** Fe, Co salt, Fe-Co alloy, slurry mixing and hydrogen reduction, nanocrystalline, magnetic.

### 1. Introduction

Fe-Co alloys are important soft magnetic materials that have been widely and commercially used in industry. Compared to its polycrystalline counterpart, these nanostructured materials have showed superior magnetic properties, such as higher permeability and lower coercivity due to the single domain configuration<sup>1-3</sup>). It is also well known that excellent soft magnetic properties are obtained when the Fe-Co alloy has the ordered B2 structure ( $\alpha'$  phase)<sup>4</sup>). In addition, its magnetic properties such as the saturation magnetization and the coercivity are very sensitive to the alloy formation and the microstructure. For the improvement of the magnetic properties during the fabrication of nanostructured materials, it is needed to control the microstructure like the internal strain, crystal structure and grain size.

In the present work, chemical solution mixing followed by hydrogen reduction were used to fabricate the nanostructured Fe-Co alloy powders. The manufacturing process developed in the present study have several advantages over the conventional methods for the fabrication of nanostructured powders

because these methods do not cause internal strain or introduce impurities during processing<sup>5</sup>). In this study, nanostructured Fe-xCo ( $x=10, 50, 90$ ) alloy powders were prepared from the powder mixtures of  $\text{FeCl}_2$  and  $\text{CoCl}_2$  by chemical solution mixing and hydrogen reduction. The microstructure and the magnetic properties were observed for the fabricated Fe-Co alloy powder.

### 2. Experimental Procedure

$\text{FeCl}_2$  and  $\text{CoCl}_2$  powders with 99.9% purity (Kojundo Chem. Lab. Co., Japan.) were used as the starting materials to fabricate the nanostructured Fe-xCo ( $x=10, 50, 90$ ) alloy powders.  $\text{FeCl}_2$  and  $\text{CoCl}_2$  powders were dissolved in ethyl alcohol for homogeneous mixing and dried at 70°C for 24 h. The thermogravimetry analysis was performed to evaluate the hydrogen reduction process of the salts mixture. And then, the salts mixture was reduced at 450~600°C for 1 h in  $\text{H}_2$  atmosphere. Fig. 1 shows the schematic diagram of chemical solution mixing and hydrogen reduction process. Phase identification of prepared powders was determined by X-ray diffrac-

\*Corresponding Author : [Tel : +82-2-2220-0408; E-mail : ydkim1@hanyang.ac.kr]

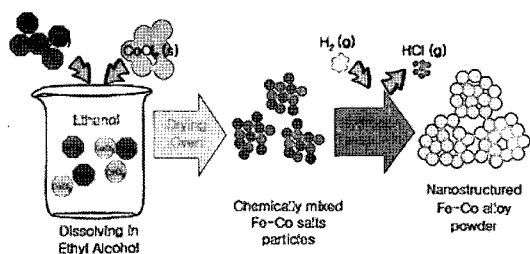


Fig. 1. The schematic diagram of chemical solution mixing and hydrogen reduction process.

tion analysis and TEM. Microstructural characteristics were analyzed by SEM and TEM. The magnetic properties of the powders were measured by using a VSM with an applied magnetic field up to  $\pm 15$  kOe at room temperature.

### 3. Results and Discussion

Fig. 2 shows the thermogravimetry curves during heating up to  $1000^{\circ}\text{C}$  with a heating rate of  $10^{\circ}\text{C}/\text{min}$  in  $\text{H}_2$  atmosphere. As shown in Fig. 2, the reduction reaction of the salts mixture was started at approximately  $150^{\circ}\text{C}$  and was completed at  $530^{\circ}\text{C}$ . In Fig. 2, peak A and B are the peaks occurred by evaporation of the remaining ethyl alcohol and moisture in salt mixture powders. And then, weight loss of peak C was due to the oxidation of the salt mix-

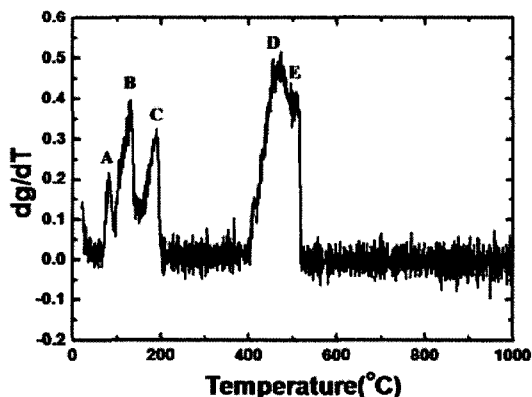


Fig. 2. Thermogravimetry (TG) curves for the hydrogen reduction process of Fe and Co salts powders during heat-up from room temperature to  $550^{\circ}\text{C}$  with a heating rate of  $10^{\circ}\text{C}/\text{min}$ .

ture powders by the evaporated moisture. Oxides produced at the peak C were reduced at the range of  $400\text{--}450^{\circ}\text{C}$ . The peak D and E were the reduction peaks of  $\text{FeCl}_2$  and  $\text{CoCl}_2$ , respectively. Reduction reaction was assumed to proceed simultaneously with the following process.

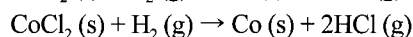
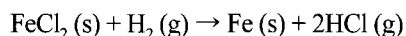


Fig. 3 shows the XRD patterns of Fe- $x$ Co ( $x=10, 50, 90$ ) alloy powders reduced at  $550^{\circ}\text{C}$  for 1 h in  $\text{H}_2$  atmosphere. It was shown that  $\text{FeCl}_2$  and  $\text{CoCl}_2$  salt mixture powder was completely reduced after the reduction at  $550^{\circ}\text{C}$ . In both case of the Fe-10at.% Co and Fe-50at.% Co alloy powders, the Co peaks were not shown after the hydrogen reduction over  $550^{\circ}\text{C}$ . However, Fe peak was invisible in XRD patterns of the Fe-90at.% Co alloy powder after the hydrogen reduction at  $550^{\circ}\text{C}$  and Co peaks with HCP and FCC were observed. This difference of the XRD peaks with the Co composition was regarded as the difference of matrix. In the case of Fe-10at.% Co and Fe-50at.% Co alloy powder, the matrix of alloy powders was Fe based matrix. It would be considered that Co atoms formed a solid solution into the Fe based matrix.

In the case of Fe-90at.% Co alloy powder, the based matrix is Co. Small amount of the Fe atoms

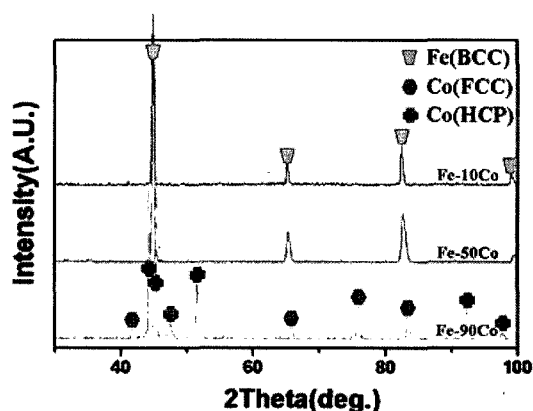


Fig. 3. XRD patterns of reduced Fe- $x$ Co ( $x=10, 50, 90$ ) alloy powders at  $550^{\circ}\text{C}$  for 1 h.

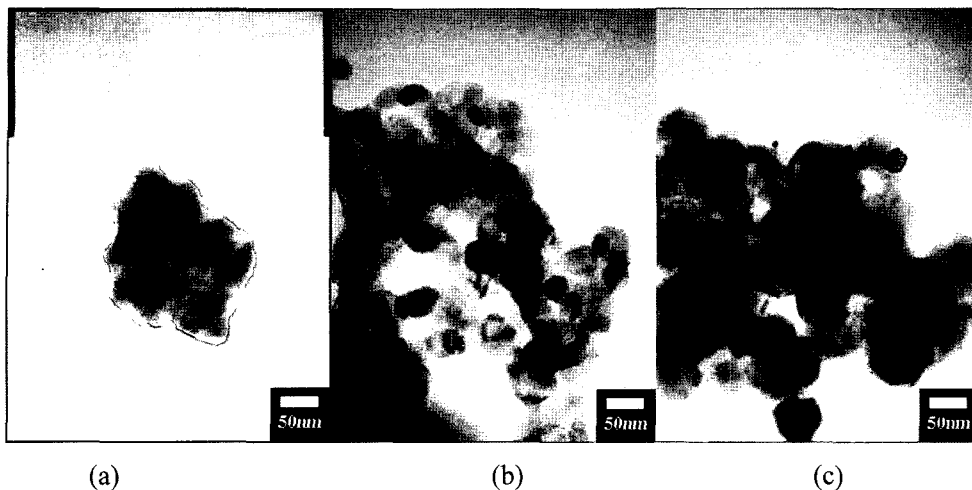


Fig. 4. TEM micrographs of reduced Fe-xCo ( $x=10, 50, 90$ ) alloy powders with Co contents: (a)  $x=90$ , (b)  $x=50$  and (c)  $x=10$ .

formed a complete solid solution in the Co based matrix. Although the Fe atoms formed a Fe-Co alloy phase in the Co matrix, that the Co peak was only observed at Fe-90at.% Co alloy powders was due to the overlapping the Co peak and Fe-Co alloy peaks. Also, it was well known that Co had the  $\alpha$ -Co structure with HCP at room temperature and transformed to the FCC structure above the Neel temperature ( $477^{\circ}\text{C}$ )<sup>7</sup>. These peaks with FCC crystalline structure were considered that  $\beta$ -Co with FCC could not fully transform to  $\alpha$ -Co of HCP during cooling.

In order to further characterize the microstructure, microstructures of the hydrogen reduced Fe-Co alloy powders were observed by TEM analysis. Fig. 4 shows the bright field images for Fe-Co alloy powders with Fe-xCo ( $x=10, 50, 90$ ) prepared after 1 h of hydrogen reduction at  $550^{\circ}\text{C}$ . Fe-Co alloy powders were aggregated each other and the average grain size of powders synthesized by chemical solution mixing and hydrogen reduction process was determined less than 50 nm from the bright field image.

The hysteresis loop of the Fe-50at.% Co alloy powders prepared by chemical solution mixing and hydrogen reduction process was shown in Fig. 5. The saturation magnetization value of Fe-50at.% Co alloy powders is about 214 emu/g. Also, the Fe-50at.% Co alloy powder had a low coercivity value

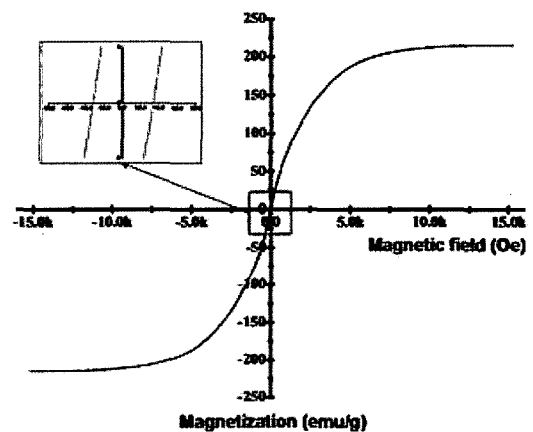


Fig. 5. Hysteresis loop of reduced Fe-50at.% Co alloy powders measured at room temperature.

of 32.5 Oe and it can be expected that Fe-50at.% Co alloy powder produced by chemical solution mixing and hydrogen reduction process has better soft magnetic properties compared with mechanical alloying powders<sup>3,8-10</sup>.

Enhanced magnetic properties of powder synthesized by chemical solution mixing and hydrogen reduction are due to the lack of impurities and internal strain compared to mechanically alloyed powders. In addition, the low coercivity and high saturation magnetization of nanostructured Fe-Co alloy powder could be predicted over their mechanically alloyed powders.

#### 4. Conclusion

The synthesis of nanostructured Fe-xCo (x=10, 50, 90) alloy powders was successfully performed by chemical solution mixing with hydrogen reduction at 550°C for 1 h. In case of Fe-10at.% Co and Fe-50at.% Co alloy powders, the Co peaks were not shown after the hydrogen reduction over 550°C. However, Fe peak was invisible in XRD patterns of the Fe-90at.% Co alloy powder after the hydrogen reduction at 550°C. This was regarded as the difference of the matrix. It was confirmed that the average grain size of the Fe-xCo (x=10, 50, 90) alloy powders were less than 50 nm by TEM analysis. The better soft magnetic properties at the powder synthesized by chemical solution mixing and hydrogen reduction process were due to the lack of impurities and internal strain. Synthesis of nanocrystalline Fe-Co alloy powders from chemical solution mixing of the metal salts and hydrogen reduction seem to be suitable for the low cost production of an alloy with

enhanced soft magnetic properties.

#### References

1. D.L. Lesile-Pelecky, R.D. Rieke, *Chem. Mater.*, **8**(8) (1996) 1770.
2. G. Herzer, *J. Magn. Magn. Mater.*, **157/158** (1996) 133.
3. B.-H. Lee, B. S. Ahn, D.-G. Kim, S.-T. Oh, H. Jeon, J. Ahn and Y. D. Kim, *Mater. Let.*, **57** (2003), 1103.
4. Z. Turgut, J.H. Scott, M.Q. Huang, S.A. Majetich, M.E. Mchenry, *J. Appl. Phys.*, **83** (1998) 6468.
5. J. Park, K. An, Y. Hwang, J.-G. Park, H.-J. Noh, J.-Y. Kim, J.-H. Park, N.-M. Hwang and T. Hyeon, *nature mater.*, **3** (2004) 891.
6. B.S. Ahn, B.-H. Lee, K.H. Lee, Y.D. Kim, *J. Korean Powder Metall. Inst.*, **12** (2002) 490 (*Korean*).
7. H. Schumann, *Metallographie, trans.*, Korean, S. R. Kim, (1993), 193.
8. B.-H. Lee, S. S. Hong, K. H. Lee, Y. D. Kim, *J. All. Comp.*, **385** (2004) 264.
9. Y.D. Kim, J.Y. Chung, J. Kim, H. Jeon, *Mater. Sci. Eng. A*, **29** (2000) 17.
10. H. Moumeni, S. Alleg, J.M. Greneche, *J. All. Comp.*, **386** (2005) 12.