

## Fabrication of Nanostructured Fe-Co Alloy Powders by Hydrogen Reduction and its Magnetic Properties

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### Abstract

*Magnetic properties of nanostructured materials are affected by the microstructures such as grain size (or particle size), internal strain and crystal structure. Thus, it is necessary to study the synthesis of nanostructured materials to make significant improvements in their magnetic properties. In this study, nanostructured Fe-20at.%Co and Fe-50at.%Co alloy powders were prepared by hydrogen reduction from the two oxide powder mixtures, Fe<sub>2</sub>O<sub>3</sub> and Co<sub>3</sub>O<sub>4</sub>. Furthermore, the effect of microstructure on the magnetic properties of hydrogen reduced Fe-Co alloy powders was examined using XRD, SEM, TEM, and VSM.*

**Keywords :** Fe-Co alloy powder, hydrogen reduction, soft magnetic, coercivity

### 1. Introduction

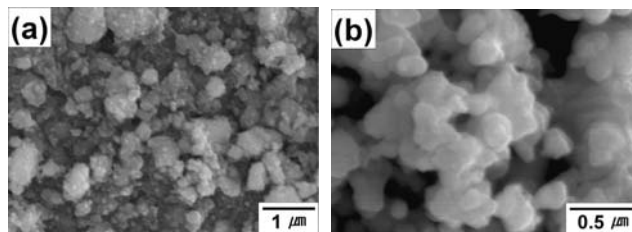
Nanostructured materials display different physical, chemical and magnetic properties such as higher permeability and lower coercivity. This is due to the single domain configuration [1-3]. Magnetic properties of nanostructured materials are also affected by other conditions, such as crystal structure and internal strain. There is a renewed interest in FeCo-based alloys because they exhibit high saturation magnetizations and high Curie temperatures. It is also well known that excellent softmagnetic properties are obtained when the alloy has the ordered B2 structure ( $\alpha'$  phase) [2]. It is necessary to have control of the microstructure, such as the internal strain, crystal structure and grain size (or particle size), for significant improvements of the magnetic properties.

In this study, nanostructured Fe-Co alloy powders with an ordered B2 structure or a disordered A2 structure ( $\alpha$  phase) were prepared by hydrogen reduction to study improvements in the softmagnetic properties.

### 2. Experimental and Results

Fe<sub>2</sub>O<sub>3</sub> and Co<sub>3</sub>O<sub>4</sub> powders were mixed with Fe-20at.%Co and Fe-50at.%Co using a Tubular (3-dimensional mixer). The materials were mixed at 62 rpm at Ar atmosphere for 1-10 h. Also, a SEM and TEM were used to characterize the microstructure.

To search for the reduction temperature of the milled oxides mixture, TG analysis was performed during the heating process up to 600°C at 10°C/min. Reduction of Co<sub>3</sub>O<sub>4</sub> primarily occurred near 300°C and reduction of Fe<sub>2</sub>O<sub>3</sub> occurred near 400°C. It was confirmed that the reduction of the oxides mixture was complete at above 500°C [4]. The microstructures of the milled oxide mixture powders and reduced Fe-Co alloy powders are shown in Fig. 1. When the ball milling was performed for 10 h, the particles of the oxide mixture powders conglomerated and the average particle size was about 300 nm. The oxides mixture was reduced at 450~600°C for 1 h in a H<sub>2</sub> atmosphere. X-ray diffraction (XRD) analysis in order to analyze the phase identification was performed and confirmed that the Fe- and Co- oxide was fully reduced.



**Fig. 1.** SEM micrographs of (a) milled oxide powders for 10 h and (b) hydrogen reduced Fe-50at.%Co alloy powders at 600°C for 1 h.

To further characterize the crystal structure, the selected

area diffraction (SAD) patterns of the hydrogen reduced Fe-20at.%Co and Fe-50at.%Co alloy powders are shown in Fig. 3. It was concluded that hydrogen-reduced Fe-20at.%Co alloy powders had disordered  $\alpha$  phases with lattice parameters of 2.867 Å. However, Fe-50at.%Co alloy powders had ordered  $\alpha'$  phases with lattice parameters of 2.858 Å.

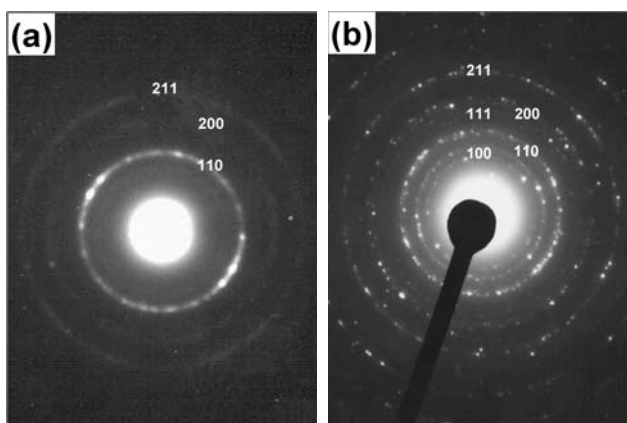


Fig. 3. SAD patterns of hydrogen reduced alloy powders ; (a) Fe-20at.%Co (b) Fe-50at.%.

The magnetic properties of the powders were estimated using a vibrating sample magnetometer (VSM) with an applied magnetic field up to  $\pm 15$  kOe. The saturation magnetization curves of Fe-20at.%Co and Fe-50at.%Co alloy powders prepared by hydrogen reduction are shown in Fig. 4. The saturation magnetization values are 214 and 216 emu/g which correspond to the data of the reference [5]. However, the magnetic susceptibility of Fe-50at.%Co alloy powders was higher than that of hydrogen reduced Fe-20at.%Co alloy powders. The coercivity of hydrogen reduced Fe-50at.%Co alloy powders was 40 Oe, but the coercivity of hydrogen reduced Fe-20at.%Co alloy powders was 80 Oe. As the hydrogen reduced Fe-50at.%Co alloy powders have the ordered BCC structure, they have a lower value of coercivity than the hydrogen reduced Fe-20at.%Co alloy powders. Generally, a Fe-Co alloy system has an ordered  $\alpha'$  phase with a B2-BCC structure in the composition range of 30~70 at.%Co and below 730°C. Moreover, it is well known that when a Fe-Co alloy undergoes an ordering transformation, excellent softmagnetic properties can be obtained compared to a disordered  $\alpha$  phase [5]. The Fe-Co alloy powders prepared by hydrogen reduction have an ordered  $\alpha'$  structure due to the relaxation of the internal strain during the hydrogen reduction process at 600°C for 1 h.

Therefore, it is reasonable to expect that hydrogen reduced Fe-50 at% Co alloy powders will have excellent softmagnetic properties compared to hydrogen reduced Fe-20at.%Co powders having a disordered  $\alpha$  structure. Therefore, the synthesis of Fe-Co alloy powders by hydrogen reduction with ball-milling and hydrogen

reduction using metal oxides is effective to obtain a suitable nano-sized powder with enhanced softmagnetic properties.

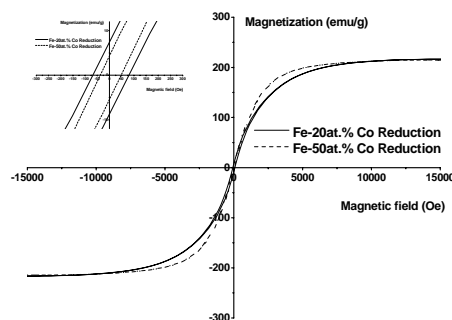


Fig. 4. Hysteresis loops of Fe-Co alloy powders measured at room temperature.

### 3. Summary

Nanostructured Fe-Co alloy powders were successfully fabricated by a ball milling and hydrogen reduction process. When the oxide mixture powders were milled for 10 h and reduced at 600°C for 1 h, the Fe-Co alloy powders were completely synthesized into the solid solution of Co into Fe. The SAD patterns showed that the synthesized Fe-50at.%Co alloy powders had the ordered  $\alpha'$  phase. With regard to magnetic properties, Fe-50at.%Co powders exhibited a coercivity value of 40 Oe, while the coercivity value of the Fe-20at.% Co powders was 80 Oe. This suggests that the enhanced magnetic properties of the hydrogen reduced Fe-50at.%Co powders were due to the formation of a more ordered structure than that found in the hydrogen reduced Fe-20at.%Co powders.

### 4. References

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