Q-3

초음파를 이용한 철, 코발트-페라이트 나노입자의 합성
(Investigation of Iron and Cobalt Ferrite Nanoparticles by Sonochemical Method)

한국지질자원연구원 , 충남대학교[1]
김은희*, 이효숙, 소혜평[1]

1. INTRODUCTION

Nanostructured materials have been intensively studied in recent years particularly because the physical properties of these materials are quite different from those of the bulk [1-3]. A variety of chemical and physical preparative methods have been developed to produce materials with nanometer domain size, including metal evaporation, [4, 5] reduction of metal salts by borohydride derivatives [6, 7] laser pyrolysis, and thermal decomposition [9]. Recently, the sonochemical method has been applied to fabricate stable ferromagnetic colloids [10] and nano-sized ferrite powders [11, 12]. Sonochemistry arises from acoustic cavitation, the formation, growth, and implosive collapse of bubbles in a liquid. This method has good advantages to have a narrow size distribution [10] and control of particle size [11, 12]. We present here a sonochemical method for the synthesis of nanostructured iron (Fe_3O_4) and cobalt ferrite ($CoFe_2O_4$) particles with iron chloride and cobalt chloride.

2. EXPERIMENT

The mixed solution of 0.15M $FeCl_2$ (20ml, 0.003mol) and 0.30M $FeCl_3$ (20ml, 0.006mol) was prepared. The starting with irradiation of ultrasonic wave (ULSSO HITECH Co., LTD, model ULH700S, 10mm, Ti horn, 20kHz, 665W/cm^2) to the reaction mixture, NH_4OH (0.012mol) solution was rapidly added to the solution at room temperature. First of all, to consider the effect of ultrasonic waves, these nanoparticles were compared with other nanoparticles which were prepared by co-precipitation method without ultrasonic irradiation. And then, the dependence on ultrasonic power and irradiation time on the solution of the metal ferrite was investigated. All of these resulting black or dark brown particles were washed free of anions and were dried at 80°C in vacuum oven. The nanoparticles were characterized by various techniques, such as Differential Scanning Calorimetry (DSC), X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM) and Vibrating Sample Magnetometer (VSM).

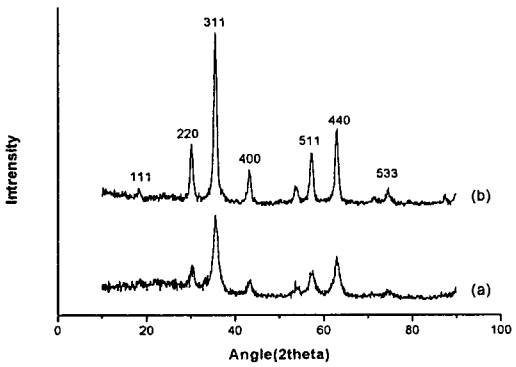


Figure1. XRD pattern of $FeOFe_2O_3$ nanoparticles (a)prepared by co-precipitation (b)exposed ultrasonic wave for 30min

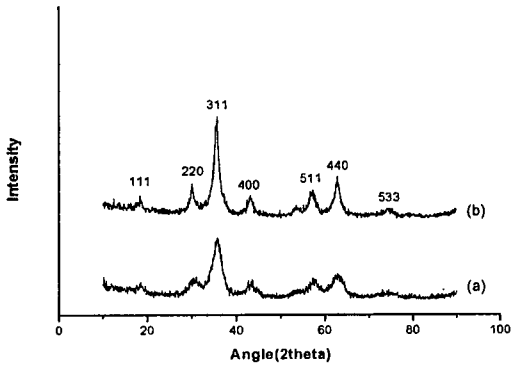
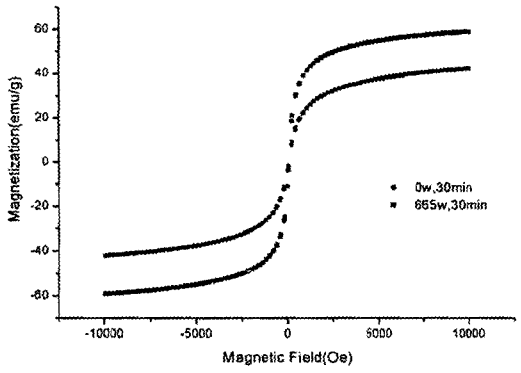


Figure2. XRD pattern of $CoOFe_2O_3$ nanoparticles (a)prepared by co-precipitation (b)exposed ultrasonic wave for 2h

3. RESULTS AND DISCUSSION

X-ray powder diffraction shows that the nanoparticles are $FeOFe_2O_3$ and $CoOFe_2O_3$ spinel ferrites. No impurity peak is observed in the XRD pattern (Figure 1-2). The diffraction peaks are clearly broadened, which can result of lattice strain and/or the reduced particle size. From the

result of XRD data, in both cases, the ferrite powder exposed to ultrasonic waves (Figure1.(b), Figure2.(b)) had higher crystallinity than that without ultrasonic irradiation (Figure1.(a), Figure2.(a)). As a result of TEM, particle sizes were increased when ultrasonic wave was irradiated. In case of iron ferrite which were prepared by sonochemical method, particle size was larger (10nm) than particles by co-precipitation method (3nm) for the same reaction time (30min). The magnetization values of iron ferrite were improved from 42emu/g to 59emu/g at a magnetic field of 10kG(Figure 3), as without and with ultrasonic irradiation. On the other hand, in case of cobalt ferrite, the particle size of was slightly larger than particles which are without ultrasonic irradiation. The magnetization (M) vs. field (H) curve does not reach saturation at a magnetic field of 10kG and the observed values of magnetization of two different experimental conditions, 33 and 17emu/g (without and with ultrasonic irradiation, respectively, Figure 4). Especially, after ultrasonic irradiation, magnetic coercivity (Hc) was disappeared.



Figur3.Room-temperature magnetization curves of $FeOFe_2O_3$

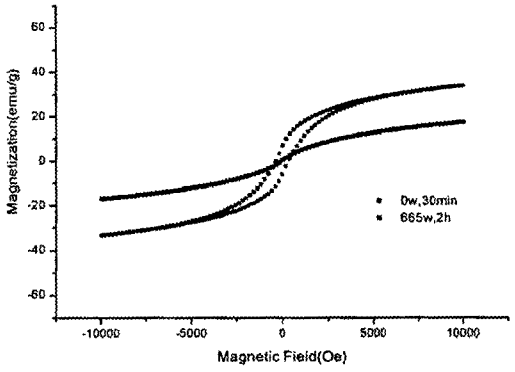


Figure4. Room-temperature magnetization curves of $CoOFe_2O_3$

4. CONCLUSION

A simple synthetic method has been discovered to produce nanosized metal ferrite using high-intensity ultrasound. And, in the sonochemical process, there might be some different mechanisms between iron and cobalt ferrites.

5. REFERENCES

[1] Schmid, G., Ed. *Clusters and Collids*, VH Press: New York, 1994.
[2] Weller, H. *Adv. Mater.* **1993**, *5*, 88.
[3] Gleiter, H. *Adv. Mater.* **1992**, *4*, 474.
[4] Hahn, H.; Averback, R.S. *J. Appl. Phys.* **1990**, *67*, 1113.
[5] Klabunde, K.J.; Zhang, D.; Glavv, G. N.; Sorenses, C.M. *Chem. Mater.* **1994**, *6*, 784.
[6] Bönnemann, H.; Brijoux, W.;Brinkmann, R.; Joussen, T. *Angew. Chem., Int. Ed. Engl.* **1990**, *29*, 273.
[7] Tsai, K.-L.; Dye, J. L. *J. Am. Chem. Soc.* **1991**, *13*, 1650
[8] Rice, G. W. *In Laser Chemistry of Organometallics*; Chaiken, J., Ed.; American Chemical Society: Washington, DC, 1993; p 273.
[9] Bradley, J.S.; Hill, E. W.; Klein, C.; Chaudret, B.; Duteil, *A. Chem. Mater.* **1993**, *5*, 254.
[10] Suslick, K.S.; Fang, M; Hyeon, T. *J. Am. Chem. Soc.* **1996**, *118*, 11960
[11] Shin, H. C.; Oh, J. H.; Lee, J. C.; Choi, S. C. *Phys. Stat. Sol. (a)* **2002**, *189*, No. 3, 735.
12] Shafi, K. V. P. M.; Gedanken, A. *Chem. Mater.* **1998**, *10*, 3445.