

# Synthesis of Nano Metal Powder by Electrochemical Reduction of Iron Oxides

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## Abstract

*Synthesis of iron nanopowder by room-temperature electrochemical reduction process of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanopowder was investigated in terms of phase evolution and microstructure. As process variables, reduction time and applied voltage were changed in the range of 1~20 h and 30~40 V, respectively. From XRD analyses, it was found that volume of Fe phase increased with increasing reduction time and applied voltage, respectively. The crystallite size of Fe phase in all powder samples was less than 30 nm, implying that particle growth was inhibited by the reaction at room temperature. Based on the distinct equilibrium shape of crystalline particle, phase composition of nanoparticles was identified by TEM observation.*

**Keywords :** Electrochemical reduction, Nano Metal Powder, Iron Nanopowder

## 1. Introduction

Over the years, nano metal powders have been focused on their chemical, magnetic, electronic and optical applications from their specific characteristics, such as high surface area and quantum-size effect [1]. Hence, many researches have been carried out on the development of synthesis methods for nano metal powders, such as sol-gel, co-precipitation, and mechanical alloying (MA) [2,3]. Recently, Lee and his co-workers reported on the successful fabrications of nano metal alloy and composite powders by hydrogen reduction of ball-milled metal oxide nanopowders [4,5]. In spite of the processing advantage, however, thermal process of hydrogen reduction inevitably accompanies grain growth during the process at elevated temperatures.

In order to solve this problem, the authors suggested a modified method to inhibit grain growth by applying an electrochemical reduction process at room temperature. This process can be the modification of conventional electrochemical reduction process which gives rise to an abrupt particle growth by thermal energy. As a target material, we chose iron nanopowder system which has a great potential for electronic and magnetic applications.

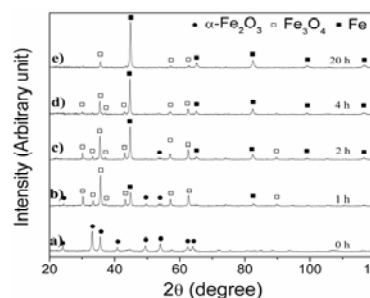
## 2. Experimental and Results

Electrochemical reduction was conducted in the reduction system where reduction stage consisted of Pt electrodes in the NaCl reducing electrolyte. In order to enhance reduction reaction by enlarging contact surface area between electrode and powder, the cathode was prepared in a ribbon-typed stainless steel plate. From the result of XRD analysis, the mean crystallite size of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanopowder was calculated by 24 nm.

First, a sample holder including  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanopowder was immersed in 0.5 mol NaCl solution at 298 K in air atmosphere. The power source for electrochemical reduction was direct current (D.C.) power of 27 V. Under this power condition, the reduction time was changed from 1 to 20 h.

Fig. 1 shows XRD patterns of nanopowders electrochemically reduced at 27 V for various reduction times. It is seen that  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> phase is reduced firstly to Fe<sub>3</sub>O<sub>4</sub> and then finally to Fe phase with increasing time. After 20 h, the reduction process is almost completed with small amount of residual Fe<sub>3</sub>O<sub>4</sub>. Fig. 1 also reveals that the crystallite size of Fe phase is less than 30 nm for all powder samples regardless of reduction time. These results indicate that electrochemical reduction at room temperature effectively suppresses grain growth of nanopowder. Simultaneously, the microstructure reveals that the prolonged reduction process resulted in agglomeration of nanoparticles due to sintering effect. It is thus concluded that the low temperature reduction by electrochemical reaction is very efficient in making nano metal powder.

In other to check the effect of electrical power on reduction behavior, the applied voltage was changed with 30, 35 and 40 V.



**Fig. 1.** XRD patterns of nanopowders reduced at 27 V for a) 0 h, b) 1 h, c) 2 h, d) 4 h, and e) 20 h.

Fig. 2 shows XRD patterns of the nanopowder samples reduced at each applied voltage for 1 h. As seen, the oxide powder was almost completely reduced under all voltage conditions. The crystallite size of Fe phase was 23.9 nm, 24.5 nm, and 25.3 nm at 30, 35 and 40 V, respectively. Also no apparent evidence for growth of the nanoparticles was observed under these electrical power conditions. Fig. 3 shows a typical example of TEM micrograph of the nanopowder reduced at 35 V for 1 h in which the mean particle size is 25 nm. This result is consistent with the crystallite size calculated from XRD result of Fig. 2(b). Interestingly, the various types of particle shape are observed in Fig. 3 showing that nanoparticles with spherical or faceted shape coexist. Based on the distinct equilibrium shape of crystalline particle we can identify the phase of nanoparticles approximately. It was reported that the equilibrium shape of  $\text{Fe}_3\text{O}_4$  and Fe phases are a prism and a sphere, respectively in two-dimension. In this respect, it is obvious that nanoparticles in Fig. 3 consist of  $\text{Fe}_3\text{O}_4$  and Fe phases.

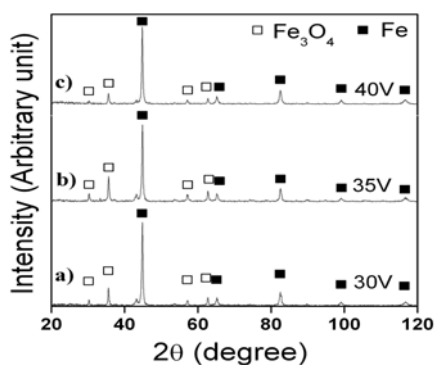


Fig. 2. XRD patterns of electrochemically reduced powders at a) 30 V, b) 35 V, and c) 40 V for 1 h.

In addition, from the area ratio of  $\text{Fe}_3\text{O}_4$  (square) and Fe (near sphere) the phase composition of the powder was calculated by 25%  $\text{Fe}_3\text{O}_4$  and 75% Fe. This result seems to correspond to the phase ratio calculated from the XRD pattern in Fig. 2(b).

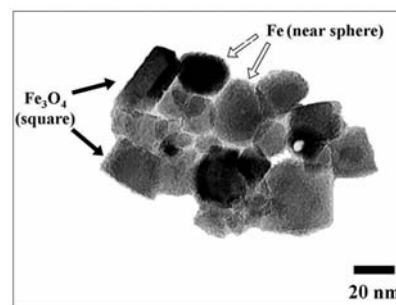


Fig. 3. TEM morphology of nanopowder electrochemically reduced at 35 V for 1 h.

### 3. Summary

Iron nanopowder was successfully synthesized by room temperature electrochemical reduction of  $\text{Fe}_2\text{O}_3$  nano-powder. XRD analyses revealed that ball-milled nano-powder of  $\alpha\text{-Fe}_2\text{O}_3$  phase was reduced firstly to  $\text{Fe}_3\text{O}_4$  and then finally to Fe phase. It was found that the volume of Fe phase in nanopowder increased with increasing time and applied voltage, respectively. Also, the crystallite size of Fe phase in all powder samples was less than 30 nm, regardless of time and voltage. TEM observation revealed that nanoparticles with spherical- or faceted shape coexisted. Based on the distinct equilibrium shape of each crystalline particle ( $\text{Fe}_3\text{O}_4$  (square) and Fe (near sphere)), the phase composition of nanoparticles was identified with 25%  $\text{Fe}_3\text{O}_4$  and 75% Fe.

### 4. References

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