

Microwave-Assisted Synthesis of Monodispersed Cobalt Ferrite Nanoparticles

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1 Introduction

Spinel ferrite nanoparticles, MFe_2O_4 ($M = Mn, Co, Ni, Fe, \text{etc.}$), are a class of inorganic oxide nanoparticles with remarkable magnetic, catalytic, optical, and electrical properties. In particular, cobalt ferrite nanoparticles ($CoFe_2O_4$) is one of the most widely studied ferrites, due to its room temperature coercivity and high magneto-crystalline anisotropy [1, 2]. In recent years, microwave (MW)-assisted rapid synthesis has been an efficient methodology for the preparation of oxide nanoparticles. In this study we have examined the MW-assisted rapid synthesis of magnetite (Fe_3O_4) and cobalt ferrite nanoparticles in the presence of oleylamine, oleic acid, and trioctylphosphine oxide as protective reagents, and characterized the structures using EXAFS measurements.

2 Experiment

In a typical preparation procedure of $CoFe_2O_4$ nanoparticles, $Co(acac)_2$ (0.333 mmol), $Fe(acac)_3$ (0.667 mmol) and oleylamine (10 mmol) were mixed and heated in oil bath at 393 K for 30 min. After cooling to room temperature, 1-dodecanol (50 ml) dissolving oleic acid (3 mmol) and trioctylphosphine oxide (TOPO, 1 mmol) was added to the solution. The solution was heated to 523 K, and kept at this temperature for 20 min (or 60 min) under MW irradiation. The MW reactor was a focused single-mode MW synthesis system (CEM Discover SP). The temperature and pressure was controlled by regulation of MW power output. After naturally cooling down to room temperature, the colloidal solutions were collected for the EXAFS measurements. For the preparation of Fe_3O_4 and CoO nanoparticles, similar procedures were applied. The EXAFS measurements were carried out in a transmission mode at BL-9C. Data analysis was performed by REX2000 (Rigaku Co.).

3 Results and Discussion

Figure 1 shows the Fourier transforms (FTs) of Fe K-edge EXAFS for the colloidal Fe_3O_4 and $CoFe_2O_4$ nanoparticles in addition to the Fe_3O_4 reference powder. Even after the shorter irradiation time of 20 min, FT spectra of the colloidal Fe_3O_4 samples show the first peak assigned to Fe-O bond (around 1.5 Å) and the second peak assigned to Fe-Fe bond (around 2.6 Å and 3.1 Å), which are similar to those of Fe_3O_4 reference compound. However, in the case of $CoFe_2O_4$ nanoparticles, the second peak becomes broad without splitting into two. This obviously indicates the formation of ferrite nanoparticles possessing Fe-Co as well as Fe-Fe bond. Furthermore, as shown in Fig. 2, the FTs of Co K-edge EXAFS reveals the formation of $CoFe_2O_4$ nanoparticles,

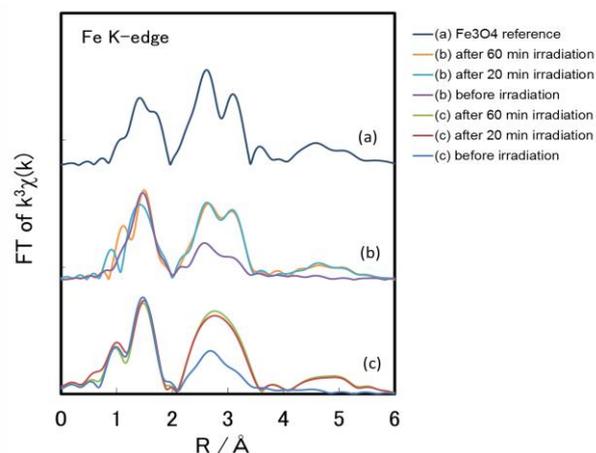


Fig. 1. Fourier transforms of Fe K-edge EXAFS spectra for (a) Fe_3O_4 powder (reference), those for (b) the colloidal Fe_3O_4 nanoparticles prepared by MW irradiation of 0 min, 20 min, and 60 min, and those for (c) the colloidal $CoFe_2O_4$ nanoparticles prepared by MW irradiation of 0 min, 20 min, and 60 min.

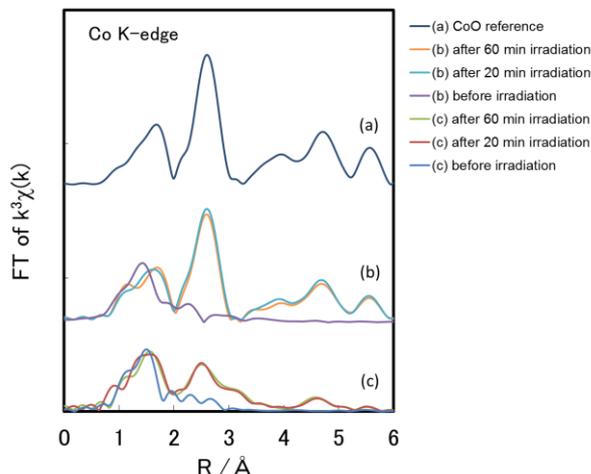


Fig. 2. Fourier transforms of Co K-edge EXAFS spectra for (a) CoO powder (reference), those for (b) the colloidal CoO nanoparticles prepared by MW irradiation of 0 min, 20 min, and 60 min, and those for (c) the colloidal $CoFe_2O_4$ nanoparticles prepared by MW irradiation of 0 min, 20 min, and 60 min.

apparently different from the FT of CoO nanoparticles. The dependence of reaction conditions (kinds of precursors, metallic compositions, additives and solvents, and so on) on the structure of the ferrite nanoparticles is also examined. The detailed analysis is in progress.

References

- [1] D. Carta et al., *J. Phys. Chem. C* **2009**, *113*, 8606.
- [2] E. Solano et al., *J. Appl. Cryst.* **2014**, *47*, 414-420.

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