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Microwave-Assisted Synthesis of CoO and NiO Nanoparticles

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Introduction

Developing new methods for the preparation of metal oxide nanoparticles with various sizes and shapes and investigating their properties are of considerable interest. In particular, cobalt oxide (CoO) and nickel oxide (NiO) nanoparticles are significant owing to their potential applications based on magnetic, catalytic, and gas-sensing properties. In recent years, microwave-assisted synthesis [1, 2] is emerging as one of the efficient methods to produce nanomaterials with controlled size and shape, because of its characteristics of rapid volumetric heatings, short reaction time, high reaction rate, and energy savings. In this study we have demonstrated the development of microwave-assisted synthetic methodology and the structural analysis of CoO and NiO nanoparticles by the use of EXAFS measurements.

Experimental

In a typical experiment for the preparation of CoO nanoparticles, $Co(acac)_2$ (1 mmol) and oleylamine (10 mmol) were mixed and then heated in oil bath at 393 K for 30 min. After cooling to room temperature, 1-octanol (or 1-dodecanol) (50 ml) was added to the solution. This solution was exposed to high intense microwave irradiation (700W, 2.45 GHz) in a microwave apparatus (MICROSYNTH PLUS, Milestone General K.K.). The solution temperature was raised to 463 K (in the case of 1-octanol) or 533 K (in the case of 1-dodecanol) by heating the solution for 2 - 4 min, and it was maintained at this temperature for about 20 min. After naturally cooling down to room temperature, the colloidal solutions were collected for the EXAFS measurements. For the preparation of NiO nanoparticles, similar procedures are applied except the use of the starting material, $Ni(acac)_2$. The EXAFS measurements were carried out in a transmission mode at BL-9C. Data analysis was performed by REX2000 (Rigaku Co.).

Results and Discussion

Figure 1 shows the Fourier transforms (FTs) of the colloidal CoO and NiO nanoparticles and their reference compounds (CoO, $Co(acac)_2$, NiO, and $Ni(acac)_2$ powder). After the irradiation of microwave, FT spectra of the colloidal samples [(2) and (4)] in the Co K-edge or Ni K-edge are similar to those of the references (CoO or NiO). This obviously indicates the formation of CoO and NiO nanoparticles coordinated by oleylamine molecules. In particular, the formation of CoO nanoparticles more noticeably proceeded in 1-dodecanol than in 1-octanol,

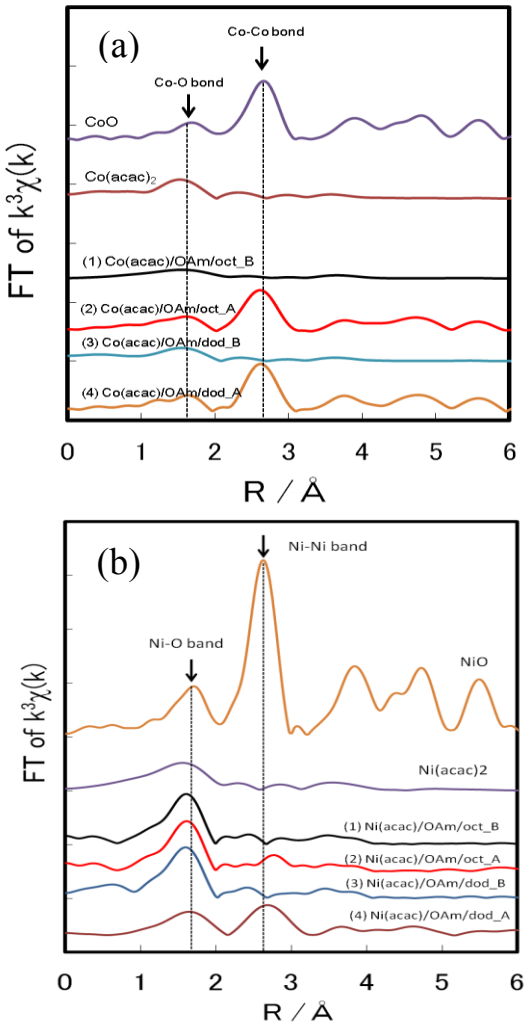

Fig. 1. Fourier transforms of (a) Co K-edge EXAFS spectra for the colloidal CoO nanoparticles and (b) Ni K-edge EXAFS spectra for the colloidal NiO nanoparticles. (1)&(3): before MW irradiation, (2)&(4): after MW irradiation. Fourier transforms of the reference compounds are also shown for comparison.

suggesting that higher reaction temperature accelerated the reaction efficiency. The dependence of reaction condition (irradiation time, starting compound, additives, solvent, etc.) on the structure of the finally-obtained oxide nanoparticles is also examined. The detailed analysis for the structural parameters is in progress.

References

[1] J.A. Gerbec et al., *J. Am. Chem. Soc.* **2005**, *127*, 15791. [2] T. Yamauchi et al., *Chem. Mater.* **2011**, *23*, 75.
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