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EXAFS study for the effect of calcination temperature on the local structure of porous Ti-Nb Binary Oxide Capsule

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Introduction

A lot of investigations have been done to improve the efficiency of the metal oxide as a catalyst by increasing its surface area. We have prepared porous silica [1], alumina [2], titania [3], or niobium oxide [4] hollow capsules by use of oil gelling agent [5]. In this study, we investigated the local structure of Nb for Ti-Nb binary hollow particles calcinated at various temperatures.

Experimental

Ti-Nb binary hollow particles were prepared by the method in the previous paper [6]. Five-component solution of N-lauroyl-L-glutamic acid-α,γ-di-n-butylamido (LGBA; oil gelling agent), n-decane, ethanol, titanium tetraethoxide, and niobium pentaethoxide was used. Ti-Nb binary hollow particles were prepared when water mixed with drying control chemical additive was added to the five-component solution, followed by calcination at 853, 923 and 1273 K.

Ti-Nb binary metal oxide hollow particles were characterized by EXAFS, XRD and TG-DSC. EXAFS measurements at Nb K-edge were carried out using synchrotron radiation at BL-12C of KEK-PF.

Result and Discussion

The wall structure of Ti-Nb binary hollow particles was also investigated by XRD. Crystal structure was not observed in the case of Ti-Nb hollow particles before calcination and after calcination at 853 K. The crystal structure of TiO_2 (rutile) was observed after calcination at 923 K and the crystal structures of both TiO_2 (rutile) and $TiNb_2O_7$ were observed after calcination at 1273 K.

Figure 1 shows Fourier transforms (phase shift uncorrected) of Nb K-edge EXAFS spectra of Ti-Nb hollow particles before calcination and after calcination at 853, 923 and 1273 K. The FT peak at 0.35 nm in Fig 1 (c) showed that the long range order around Nb increased by calcination at 923 K though the crystal containing Nb was observed by XRD.

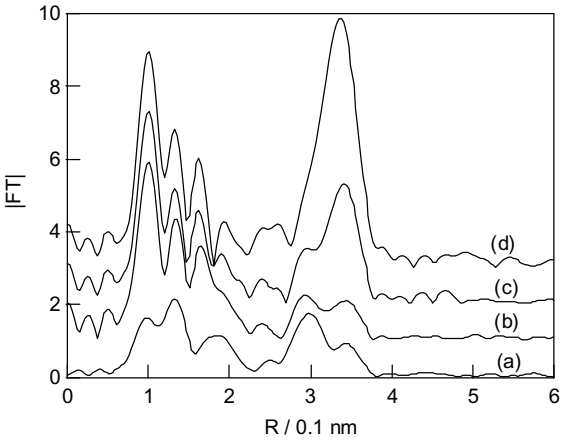

Figure 1. Fourier transforms (phase shift uncorrected) of Nb K-edge EXAFS spectra of Ti-Nb hollow particles (a) before calcination and after calcination at (b) 853, (c) 923 and (d) 1273 K.

References

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