Materials Science

12C, NW10A/2007G095

XAFS study for Nb doped titania thin film

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Introduction

Nb-doped titania films are expected for the candidates of transparent conductive thin films for flat displays. However, the mechanism of conductivity is still unknown because it is difficult to study the structure for thin films. This study aimed to clarify the oxidation state and local structure of Nb and Ti for conductive thin films by XAFS technique.

Experimental

The Nb-Ti-O conductive films were obtained from Nb-doped titania nano particle by wet coating and calcination process on the quartz glass substrate. To make the carrier on Nb-Ti-O system, the obtained films were annealed in H_2/N_2 atmosphere at 750 degree C. The XAFS measurements were carried out at BL-12C of PF and NW10A of PF-AR. The X-ray was monochromatized with a Si(111) and Si(311) double crystal monochrometor for Ti K- and Nb K- edge respectively. The samples were set on the stage for total reflection measurements. The experiments were carried out by a total reflection fluorescent method.

Results and discussion

Figure 1 shows XANES spectra at (a) Ti K- and (b) Nb K- edge for conductive film that the ratio Nb / (Nb+Ti) = 0.10. Obvious difference was not observed in Ti K-XANES spectra between as-obtained and after anealing. The Ti K-XANES spectra seemed Ti on the film forms the anatase structure. Nb K-XANES spectra showed the shoulder structure at 18975 eV, that was characteristic structure showing the existence of Nb^{5+}. The Nb K-XANES of the sample after reduction process shows weaker structure at 18957 eV by comparison of the white line absorbance. The result means the reduction process leads Nb to low oxidation state as Nb^{4+} that shows shoulder structure at 18970-18980 eV. The content of reduction state of Nb seemed about 10% of total Nb.

Figure 2 shows Fourier transforms of Ti K- and Nb K-EXAFS oscillation for the as obtained sample on figure 1. The local structures around Ti and Nb were significantly different. It means that the doped Nb did not substitute the Ti of Ti-O skeleton with anatase structure. The doped Nb was seemed to make Nb_2O_5 structure, and form nano-size particle because no niobium oxide phase was detected by X-ray diffraction technique.

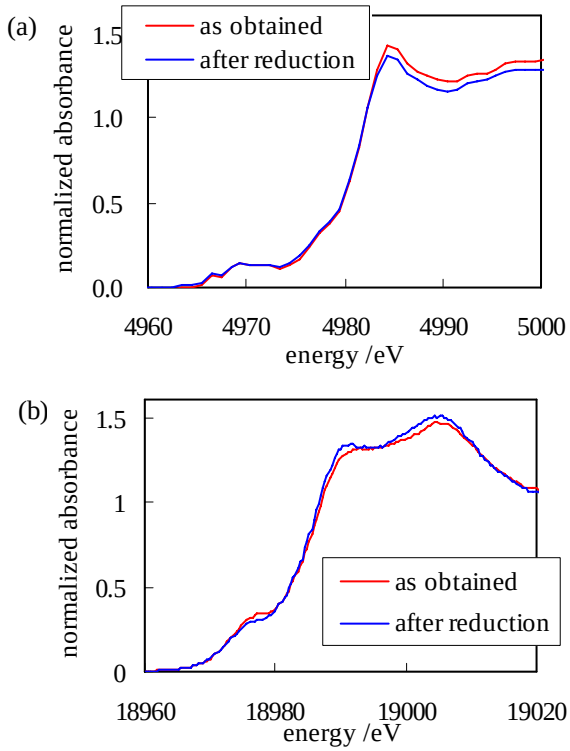
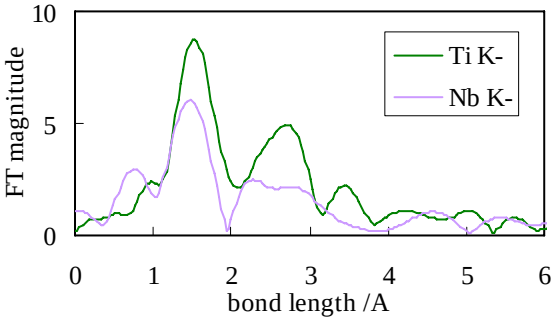


Figure 1. XANES spectra for Nb-doped titanate films. (a) Ti K-, and (b) Nb K- edge. The absorbance of shoulder structure at 18975 eV decreased after reduction.



Firure 2. Fourier transforms of Ti K- and Nb K-EXAFS for as obtained sample of figure 1. The single peak at about 2.6A is characteristic for anatase structure.

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