Physical property evaluation of a niobium oxide thin film

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We have studied and reported the preparati--on and catalysis of various mesoporous transition metal oxides, represented by tantalum and niobium oxides [1]. In spite of some advantages such as high specific surface area as compared with ordinary transition metal oxides, the poor thermal stability was a serious problem. On the other hand, SBA-15 mesoporous silica is widely used as a catalyst support because of its high specific surface area and thermal stability. In this study, we attempted to synthesize a porous transition metal oxide with high thermal resistance by preparing thin films of transition metal oxide film on SBA-15.

SBA-15 was prepared referring to the report [2]. The transition metal oxide film was obtained by the reaction of the support (0.5 g)to niobium (V) pentaethoxide in toluene (50 ml) by refluxing for 3 hours. After filtration drying, the obtained powder and was hydrolyzed with water (30 ml) by stirring for 30 minutes. Finally, it was calcined at several temperatures for 5 hours to prepare a niobium oxide film. Further, by repeating a series of the above preparation, multilayered niobium oxide thin films were prepared.

The small-angle XRD patterns of SBA-15 with niobium oxide films were coincident with that of the original SBA-15. Thus, the ordered mesoporous structure was preserved. N₂ adsorption-desorption isotherms of all samples exhibited type IV patterns, with adsorption uptake at around 0.6 to 0.7, confirming the preservation of homogeneous mesopores. The pore size distribution curve, pore diameter and pore volume were decreased by repeating the treatment with niobium species.

Figure 1 compares UV-vis. absorption spectra of niobium oxide films with 1-5 layers prepared by calcination at 500 °C together

with that of bulk-type niobium oxide. The energy gaps estimated from the absorption edge wavelength of each spectra were 3.1 eV in the bulk, whereas the niobium oxide film continuously decreased from 4.0 to 3.7 eV. It was suggested that the niobium oxide film gradually became multilayered by repeating the preparation, approaching the bulk state.

Figure 2 shows wide-angle XRD patterns of the niobium oxide film calcined at 500 °C, as well as that of the bulk-type niobium oxide. Interestingly, the niobium oxide films were not crystallized even at the temperature of crystallization in the bulk state. More detailed physical and chemical properties as well as their catalytic activity are reported.

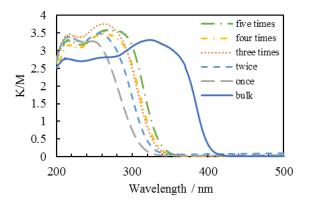


Fig.1 UV-vis. spectra of niobium oxide samples.

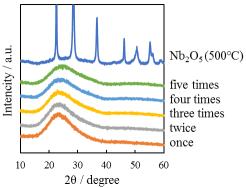


Fig.2 Wide-angle XRD patterns of niobium oxide films.

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