Preparation and Characterization of Al_2O_3 Thin Films from Liquid Phase.

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Introduction

Al_2O_3 thin films are known as a material having excellent properties such as heat resistance, corrosion resistance, and friction resistance, etc. Various methods for preparing thin film, e.g. ion plating, sputtering, sol-gel method, have been carried out. Preparation of thin film by sol-gel method has attractive features, such as its simplicity, and the fact the process doesn't need expensive equipment.

In this experiment, we prepared the Al_2O_3 precursor solution using the advanced sol-gel process and made a Al_2O_3 thin film. To make hard-coating films, trials of doping Co or Fe to Al_2O_3 were performed.

Experiment

The Al_2O_3 precursor solution was prepared as below: First, aluminum butoxide was prepared by reacting $AlCl_3$ with butanol in toluene solvent, then reacting it with water dissolved in butanol so that hydrolysis and polymerization reaction occurred. The Al_2O_3 precursor solution was condensed and diluted to 1mol/l. To make a harder film, Co or Fe, which was supplied by $CoCl_2$ or $FeCl_3$ dissolved in butanol, was added to Al_2O_3 precursor solution. This was dried under an IR lamp, and then the powdered product was heat-treated at several temperatures. The structures of powders were measured by XRD.

The thin film was coated by dipping on glass or quartz substrate. After drying the film under IR lamp and then heat-treatment, the transmittance of the thin films was measured by UV-vis. Surface roughness and morphology of films were measured by AFM and FE-SEM.

Results and Discussion

XRD patterns of Al_2O_3 after heat-treatment are shown in Fig.1. At about 700 to 800°C, γ- Al_2O_3 structures appeared. At about 1000℃, a mixture of g α- Al_2O_3 and γ- Al_2O_3 structurntified. XRD result of 3-atom % Co doped to Al_2O_3 thin filmes can be ide is shown Fig.2. The XRD peak of α- Al_2O_3 crystallized at 1000°C shifted to a lower angle. This peak shift is thought to be the formation of solid solution. Al_2O_3 and Co or Fe doped films obtained from precursor solution are transparent. The presence of dissolved Co or Fe ion in Al_2O_3 is detected by UV-vis spectra. From surface morphology in Fig.3, the grains grow to about 30nm in diameter at 1000°C. X-ray intensity becomes greater at higher temperature. AFM image also shows the same trend.

Preparation of Al_2O_3 thin film is very easy by this method. Moreover, the doping process is a very simple step, that of mixing the material to Al_2O_3 precursor solution.

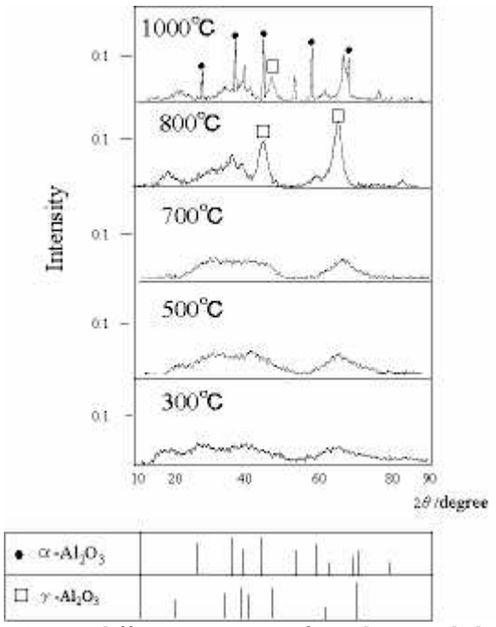

Fig1X-ray diffraction patterns of powder annealed at various temperatures

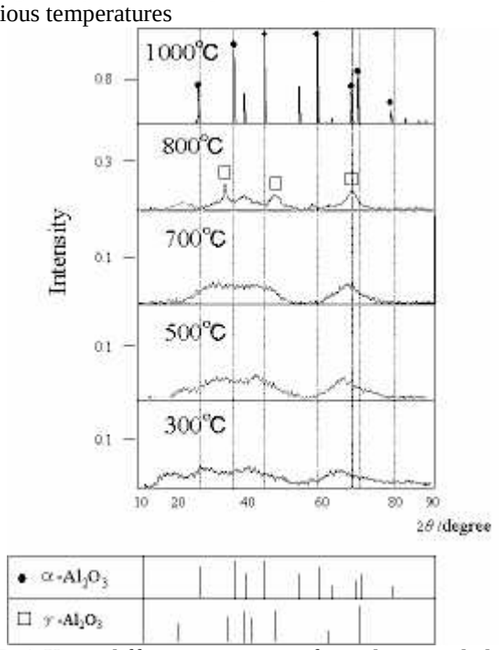

Fig2 X-ray diffraction patterns of powder annealed at various temperatures (Co doped)

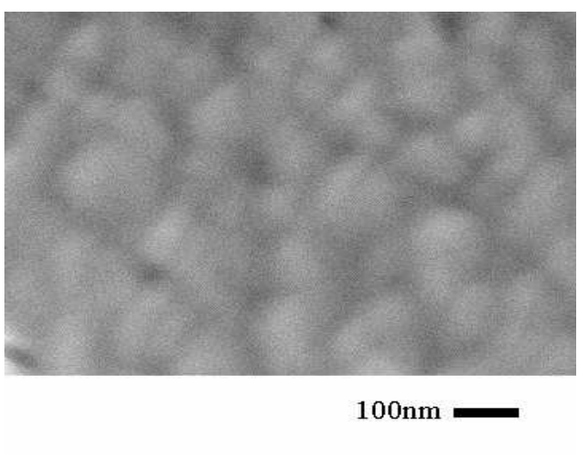

Fig.3. SEM image of Al_2O_3 thin film.