Fabrication of Barium Titanate using Precursor Solution Prepared by Aqueous Halogen-Free Titanate Solution

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Introduction

Water-based ceramic processing has been receiving increasing attention due to the reduced health and environmental hazards coupled with a low cost compared to the organic solvent-based one. Aqueous titanate solution used in this study was prepared by mixing titanium tetra-*n*-butoxide (TNB), lactic acid (Lac) and water¹). The solution seems to be very valuable as the starting material for another titanium compounds including titanium oxide because they would have higher titanium content and lower carbon dioxide emission on firing process. In addition, the titanate solution is very stable for a long time in air and free from hazardous halogen, nitrogen, sulfur, and metal ions other than titanium. In this study, BaTiO₃ precursor solution was prepared by mixing of aqueous TNB-Lac solution with barium acetate and thin BaTiO₃ films on ITO glass substrates were formed by the spin-coating method using the BaTiO₃ precursor solution, and then BaTiO₃ crystalline films were obtained by heat-treating.

Methods

Aqueous titanate solution was prepared by directly mixing TNB with Lac and water at room temperature. And then exothermic reaction instantly occurred to give a white solid mass. The solid product was gradually dissolved under stirring and a clear solution for a few days. The molar ration of Lac to TNB was 1. This aqueous titanate solution is designated as TNB-Lac. To prepare BaTiO₃ precursor solution, TNB-Lac and aqueous barium acetate $(Ba(OAc)_2)$ solution were mixed in molar ratio Ba : Ti = 1 : 1. The thin films were obtained from the BaTiO₃ precursor solution *via* spin-coating method. The films were dried at 180 °C for 10 min and then heated at various temperatures for 30 min. Thicker films were prepared by repeating the deposition cycle. The thin BaTiO₃ films were characterized by XRD, SEM observation and dielectric measurements.



Fig. 1 XRD patterns of BaTiO₃ powders heated at various temperatures



Fig. 2 SEM image of $BaTiO_3$ thin film deposited on ITO substrate

Results and Discussion

Figure 1 shows that the XRD patterns for the $BaTiO_3$ precursor solutions were heated at various temperatures. The results of XRD pattern showed that $BaTiO_3$ phase was obtained after heating above 650°C in air and the crystalline size became larger with increasing heating temperature.

The scanning electron micrograph of the cross-section of a BaTiO₃ thin film deposited on ITO glass substrate is shown in Fig. 2. This layer was prepared by repeating 5-times deposition cycle. The SEM image of the BaTiO₃ thin film showed that the thin film was multilayered structure. The grain size of multilayered thin films were 40~60 nm. At a frequency of 1 kHz the dielectric constant of thin films heated at 700°C was 261 and the loss tangent was 0.0342.

1) T. Ohya, et al., J. Sol-Gel Sci. Tech., 30, 71-81 (2004).

About Myself

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