LIQUID PHASE INFILTRATION (LPI) PROCESS FOR THE FABRICATION OF HIGHLY NANO ORDERED MATERIALS

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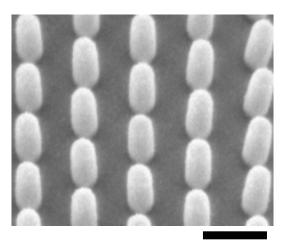
Numerous studies for the materials having periodic structure in submicron or nanometer-scale have been carried out with much attention as photonic crystals. For such materials, various fabrication methods were employed using various chemical and physical techniques. Base on these techniques, well-ordered structure were also prepared by templating techniques using these periodic structures as a mold [1]. A facile technique is thus desirable for the preparation of the higher-ordered architectures. We have previously developed the Liquid Phase Deposition (LPD) method as a new fabrication process for preparing metal oxide thin films has been developed. In this process, metal oxide thin films can be deposited on immersed substrates through the chemical equilibrium reaction between a metal fluoro-complex and a metal oxide in aqueous solution. It is easy to form uniform TiO2 film on substrates with complex morphologies such as glass wool at room temperature without special equipments [2]. In this study, we developed the Liquid Phase Infiltration (LPI) method for the preparation of the metal oxide thin film with higher-ordered periodic structure using a soft solution process as an extended technique of LPD method.

Si wafers with holed with periodic interval (165 -1060 nm) as a template. For the deposition of TiO₂ films with higher-ordered architectures, the substrates first immersed into (NH₄)₂TiF₆ solution containing H₃BO₃. After immersion for several hours, the substrates with deposited films were removed from the solution, washed with distilled water and dried at room temperature, resulting in the formation of TiO2 films on the mold. TiO₂ films were peeled off from the mold, and finally, TiO2 films with higher-ordered architectures were obtained as shown in Fig.1. TiO2 has a structure transferred from the template. For all the films, no crack was observed. Using the LPI the transcription of higher-ordered architectures was achieved.

In order to investigate the growth of the deposited films, FE-SEM observation was performed using the cross-sectional images for films deposited at various reaction time. For the film obtained after 7.5 hours in reaction time, the TiO_2 film was precisely formed on along the nanoscale channels. It can be seen that the TiO_2 were completely infiltrated for the film obtained after 20 hours. From the result of FE-SEM observation, it is easy to form uniform TiO_2 film on substrates with complex morphologies. This procedure provides a direct fabrication of the metal oxide film with the highly ordered structure, and consequently the optical properties of photonic crystals can be modified on the nanometer scale. The present technique may be extended to prepare other oxide including TiO_2 /dye or graded oxide film.

References

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- 2) H. Kishimoto, K. Takahama, N. Hashimoto, Y. Aoi, S. Deki, *J. Mater. Chem.*, **8**, 2019 (1998).



1 μm

Figure 1. TiO₂ film with periodic structure prepared by the LPI process.