

Structure and Property Changes of Anodic Alumina Membrane during Crystallization by Heat Treatment

Tatsuya Masuda, Hidetaka Asoh and Sachiko Ono

Department of Applied Chemistry,
Faculty of Engineering, Kogakuin University
2665-1 Nakano, Hachioji, Tokyo 192-0015, Japan

Anodic porous alumina film formed by anodizing of aluminum can be applied to a filtration membrane after detachment from the substrate and through-hole. Previously, we evaluated the fabricated α -alumina membrane by pore size distribution and chemical resistance [1]. In this study, we investigated the changes in pore structure induced by sintering and crystalline structure of heat-treated porous alumina at different temperature. The crystallites size and hardness of the porous alumina membrane were also evaluated by scanning electron microscopy, X-ray diffraction and micro Vickers test.

Electropolished high-purity Al substrate was anodized in 0.3 mol dm⁻³ oxalic acid at 40 V for 1 h as the first anodizing. Porous alumina film formed by the first anodizing was dissolved in mixture solution of phosphoric acid and chromic acid, and subsequently, the second anodizing was conducted for 3 h under the same condition as the first anodizing. The film was detached by anodic polarization in ethanol including perchloric acid [2].

After through-hole treatment in phosphoric acid to dissolve the barrier layer chemically, alumina membrane was heated for 4 h at different temperature under restricted condition with load using ceramic plates. Figure 1 shows the XRD patterns of heat-treated anodic porous alumina for 4 h above 800 °C. The alumina membrane transferred to single α phase completely by heat treatment at 1250 °C for 4 h. Figure 2 shows SEM images of rear surface of anodic porous alumina membrane. The pore bottom was partly covered by residual barrier layer even after detachment from Al substrate as shown in Fig. 2a. The change in pore morphology of alumina membrane occurred specifically at temperature higher than 1200 °C. In particular, the pore morphology changed notably during transition to α -alumina as observed in Fig. 2d. Therefore, the pore diameter of rear surface enlarged to 43 nm finally, because the residual barrier layer was adsorbed into porous layer by sintering with increasing temperature. Figure 3 shows the results of Vickers hardness test of alumina membranes. Although Vickers hardness of heat-treated alumina membrane increased with increasing temperature, the value of alumina membrane with single α phase decreased 20 % as compared with heat-treated membrane at 1200 °C. The decrease in hardness after transition to single α phase is assumed to be the growth of crystal grains by increasing temperature.

References

- [1] H. Asoh and S. Ono, 61st Meeting of ISE, Abstracts No.Ise102299 (2010).
[2] J. H. Yuan, W. Chen, R. J. Hui, X. H. Xia; *Electrochimica Acta*, **51**, 4589 (2006).

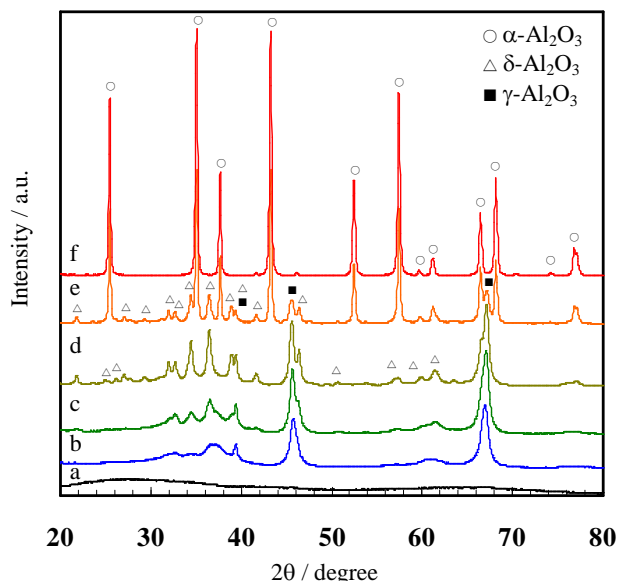


Fig. 1 XRD patterns of heat-treated anodic porous alumina for 4 h at (a) 800 °C, (b) 900 °C, (c) 1000 °C, (d) 1100 °C, (e) 1200 °C, (f) 1250 °C.

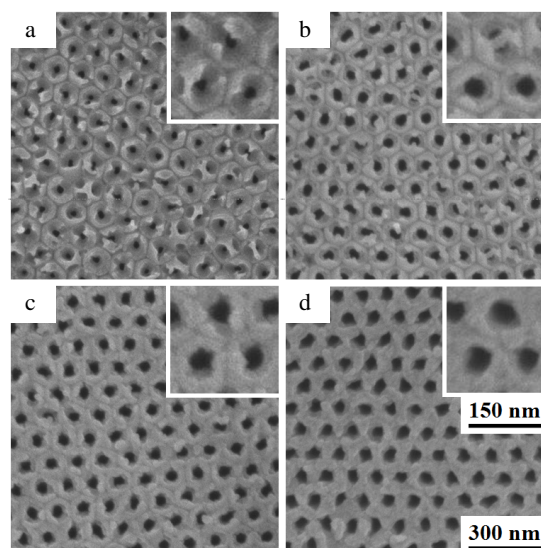


Fig. 2 SEM images of rear surfaces of anodic porous alumina membranes (a) before and after heat treatment for 4 h at (b) 1100 °C, (c) 1200 °C, (d) 1250 °C. Insets indicate high magnification images.

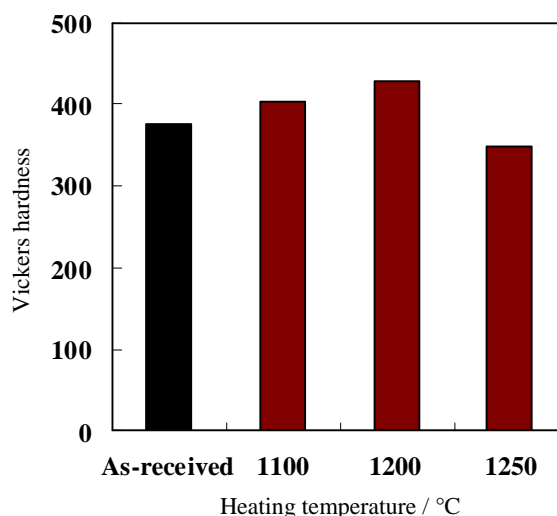


Fig. 3 Change in Vickers hardness of alumina membrane with heating temperature. Micro Vickers test was conducted at 100 gf load with a dwell time of 15 s.