

## Fabrication of Porous Niobia by Anodizing of Niobium

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Recently, anodic oxide film formed on niobium has attracted significant attention because of its exploitation in capacitor industry as an alternative material to tantalum anodic oxide. We investigated the anodic behavior of niobium with focusing on the dielectric properties [1,2]. On the other hand, fabrication of mesoporous oxides of niobium is especially important in the field of catalyst in addition to a novel device application. Therefore, an attempt has been done to fabricate porous niobia having high specific surface area by anodizing of niobium.

When pure niobium was anodized in  $0.1\text{mol dm}^{-3}$   $\text{H}_2\text{SO}_4$  at  $10\text{Am}^{-2}$  up to the breakdown voltage, many cracks caused by breakdown events appeared and the number of cracks increased with increasing breakdown time as shown in Fig.1. While, many pores in the size of approximately  $1\mu\text{m}$  appeared when anodizing was performed in  $0.1\text{mol dm}^{-3}$  NaOH (Fig.2). The porosity of the film was in the order of 0.04 after 20min and increased with anodizing time. XRD analysis indicated that the both films formed in  $\text{H}_2\text{SO}_4$  and NaOH were composed of crystalline  $\text{Nb}_2\text{O}_5$ .

For the fabrication of porous niobia having high surface area, HF was added to  $1\text{mol dm}^{-3}$   $\text{H}_2\text{SO}_4$  to enhance the dissolution ability of the electrolyte against the oxide film. Current density – time transient indicates the similar curve to that associates with a porous film formation of aluminum, though the current was very low (Fig.3). As shown in Fig.4, a porous niobia with the pore size of approximately 10nm to 20nm was obtained. The oxide shows a hexagonal cylindrical cell structure just like a porous film formed on aluminum. Thus, porous niobia having high surface area could be fabricated. Further attempt to improve the specific surface area was under investigation.

[1] S Ono, M Baba, M Shimoyama and H Asoh, 204<sup>th</sup> Meeting of the Electrochemical Society, Abstr. 1308 (2003)

[2] H, Odate, H, Asoh and S. Ono, 204<sup>th</sup> Meeting of the Electrochemical Society, Abstr. 34 (2003)

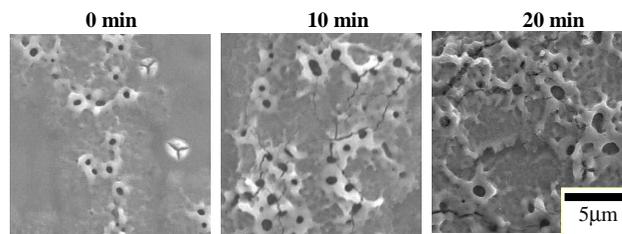


Fig.2 SEM images of anodic films formed in NaOH at  $10\text{Am}^{-2}$  with the change in breakdown time.

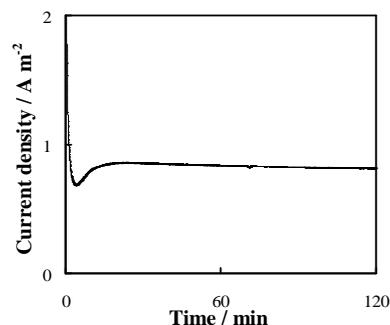


Fig.3 Current – time transient of anodizing of niobium at 20V in HF- $\text{H}_2\text{SO}_4$  solution.

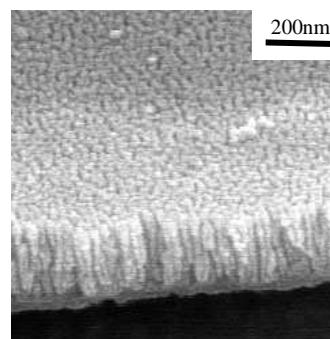


Fig.4 SEM image of a fracture section of porous niobia formed on niobium at 20V in HF- $\text{H}_2\text{SO}_4$  solution.

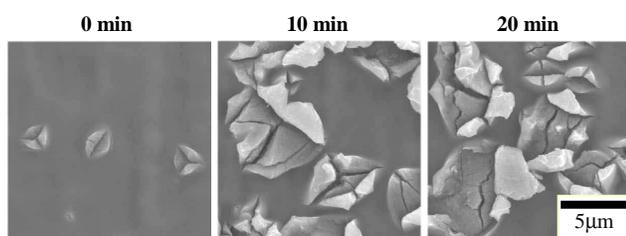


Fig.1 SEM images of anodic films formed in  $\text{H}_2\text{SO}_4$  at  $10\text{Am}^{-2}$  with the change in breakdown time.