Formation of Porous Aluminum Films by Sputter Deposition for Capacitor Application: Effects of Ar Pressure and Substrate Morphology

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Introduction
Aluminum electrolytic capacitors are widely used in electronic industry. Electrochemical etching in strong acid and alkaline solutions is currently used to produce aluminum electrode with high surface area. However, due to increased environmental regulations, dry process has attracted increasing attention as a more environmentally friendly alternative of wet electrochemical process. Morphology of metallic films prepared by dry process can be controlled by temperature and surface morphology of substrate as well as gas pressure during deposition. In this study, we have tried to prepare aluminum films with isolated columnar structure by sputter deposition by controlling the argon pressure and by using substrate with regular concave cell structure. Slight alkaline etching has been also applied to increase the pore size that is not filled with dielectric oxide even after anodizing of the aluminum electrode to high formation voltages.

Experimental
Porous aluminum films were deposited on flat and textured aluminum substrates by DC magnetron sputtering. The textured aluminum substrate, with regular concave cell structure, was prepared by anodizing aluminum in phosphoric or citric acids and subsequently dissolving the oxide layer in a chromic acid-phosphoric acid mixed solution. The sputtering deposition was done at argon pressures of 0.33 and 3.3 Pa. Then, some specimens thus prepared were chemically etched in 0.1 mol dm⁻³ KOH. The deposited films were observed by SEM, and surface roughness was evaluated from voltage-time responses during anodizing at 10 A m⁻² in 0.1 mol dm⁻³ ammonium pentaborate electrolyte and from AC impedance measurements of the anodized specimens.

Results and Discussion
Non-porous aluminum films were obtained on the flat aluminum substrates, regardless of argon pressure during deposition. Porous films with isolated columnar structure were formed on the textured substrates at argon pressures of both 0.33 and 3.3 Pa, but at the latter pressure, higher surface area was obtained. Random angle of incidence for aluminum atoms due to short mean free path at the high argon pressure as well as the enhanced shadowing effect on rough substrate enhanced the formation of the porous film. Slight alkaline etching of the deposited film effectively enlarged gaps between columns (Fig. 1). Such columnar films were suitable to form thick dielectric alumina films (~360 nm) by anodizing; the gaps were not filled with oxide even though the Pilling-Bedworth ratio for Al/Al₂O₃ was as high as 1.61.

Fig. 1 Cross-section SEM image of the Al film deposited at an Ar pressure of 3.3 Pa on the textured substrate and subsequently chemically etched.
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