

Effect of Heat Treatment on the Electrochemical Behavior of Anodized Aluminum Oxide Formed in Ammonium Adipate Solution

Jeng-Kuei Chang, Chia-Mei Lin, Chi-Min Liao*,
Chih-Hsiung Chen*, Wen-Ta Tsai

Department of Materials Science and Engineering
National Cheng Kung University
Tainan, Taiwan

*New Materials Research and Development Department
China Steel Corporation
Kaohsiung, Taiwan

Effect of heat treatment (before and after the anodization) on the microstructure and electrochemical characteristics of the anodized aluminum oxide films formed in 85°C aqueous ammonium adipate electrolyte were investigated in this study. The morphology and crystal structure of the anodized oxide were examined by TEM. The relative dielectric constant, electrochemical impedance spectroscopy and breakdown voltage of the oxide film were also determined.

The conditions for the preparations of anodized aluminum oxide film are listed in Table 1. Both pre or post heat treatment of 500°C could induce the formation of crystalline γ' -Al₂O₃ within the anodized oxide and consequently increase the relative dielectric constant of the film. Examples of TEM images for the oxide anodized in ammonium adipate solution with and without heat treatment are shown in Fig. 1. The breakdown voltage of the dielectric layer was also raised when the crystalline oxide presented. The difference in the morphology and crystalline characteristics between the anodized oxides, with pre or post heat treatments, led to its variation on the electrochemical properties. Thermal induced phase transformation from amorphous to crystalline of the anodized oxide film due to the post heat treatment might leave some defects and cause a substantial decrease of the resistance. The re-anodization would further develop the crystalline oxide and in turn cause an increase of the relative dielectric constant of the oxide film. Additionally, the thickness and resistance of the anodized oxide film were increased by the re-anodization. The film thickness, capacitance and relative dielectric constant of various anodized aluminum oxides are listed in Table 2.

Table 1. Conditions for anodization and heat treatment for the preparations of aluminum oxide in ammonium adipate solution.

Specimen	Pre heat treatment	Anodization	Post heat treatment	Re-anodization
A	---	85°C, 10 min	---	---
B	---	85°C, 10 min	---	85°C, 2 min
C	500°C, 2 min	85°C, 10 min	---	---
D	500°C, 2 min	85°C, 10 min	---	85°C, 2 min
E	---	85°C, 10 min	500°C, 2 min	---
F	---	85°C, 10 min	500°C, 2 min	85°C, 2 min

Table 2. The thickness, capacitance and relative dielectric constant of the anodized oxide films formed under various conditions.

	d (nm)	C (nF/cm ²)	Relative dielectric constant
Specimen A	165	626	11.7
Specimen B	170	610	11.7
Specimen C	180	661	13.4
Specimen D	185	695	14.5
Specimen E	175	625	12.3
Specimen F	180	620	12.6

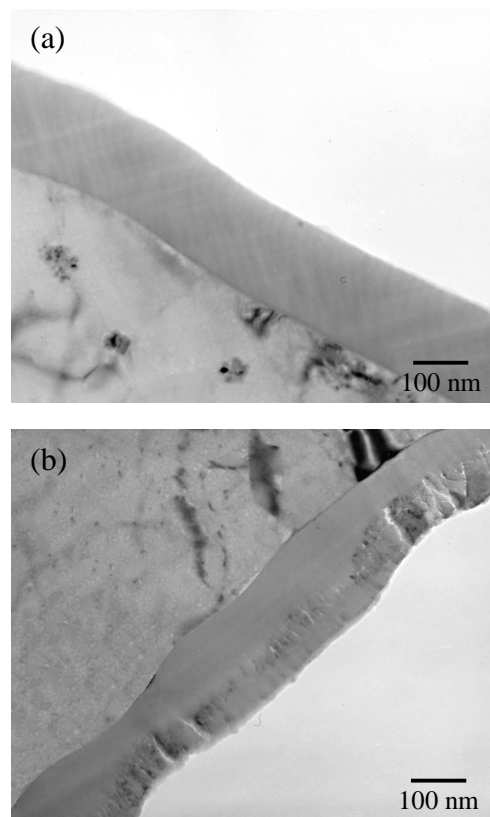


Figure 1. TEM cross-sectional image of the anodized aluminum (a) without and (b) with pre heat treatment.