P-35

Electrochemical Analysis of Silicon Oxide Coated on Anodized Substrate by Sol-Gel Method

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1. Introduction

Al_2O_3 layer has good adhesive strength to graphite/epoxy complex matrices and SiO_2 layer is strongly protective against moisture penetration. Therefore, SiO_2 coating on Al_2O_3 undercoating layer is known effective for good oxidation resistance at high temperature for complex materials of polyamide-matrix/carbon-fiber. Also, oxygen diffusion rate of Al_2O_3 is lower than oxygen diffusion rate of SiO_2, SiO_2 coating can be used for decreasing surface defects such as voids, pin hole etc. Another merit of SiO_2 coating on anodized aluminum substrate is omissible steam sealing process as a final anodizing process by penetrating SiO_2 sol solution into voids. A third merit of SiO_2 coating on anodized aluminum substrate is effective for alkaline resistance. This study is aimed to analysis of synthesis and characterization of SiO_2 layer coated on anodized foils fabricated for 400V high voltage resistant capacitor. SiO_2 coatings were obtained by Sol-Gel process on anodized aluminum foils. According to Sol composition and heat treatment conditions, penetration of SiO_2 Sol into anodized voids andimpedance measurement for alkaline resistance of SiO_2 coated anodized Al foils were observed. SEM micrographs, Impedance measurement, Tafel plots and DT-TGA etc analysis were evaluated.

2. Experimental procedure

To obtain a adhesive SiO_2films on anodized Al foil, composition of Sol solution with $HCl/Si(OC_2H_5)_4$=0.01 air hydrolyzed at 80℃were indicated in Fig 1.Schematic diagram of experimental apparatus and parameters were showed in Fig.2 From the equation of $h = 0.94(\eta U_o)^{2/3}\gamma_V^{1/6}(\rho g)^{1/2}$,here h = thickness,η= viscosity, Uo =dipping spped ,γ_V =surface tension, ρ= density and g=gravity, we can calculate coated thickness from viscosity and surface tension values. We used pyrex flask for condensation under the constant heating condtion at 70℃. To obtain a desirable thickness several times dipping and drying processes were repeated.

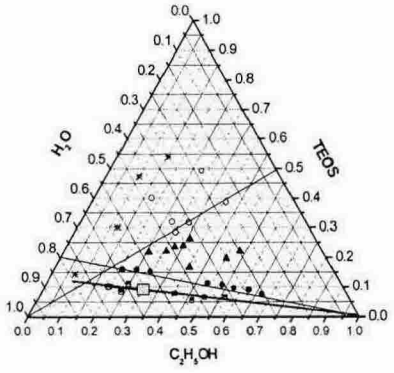

Fig.1 Relationship between Coating layer and composition of Sol solution with HCl/Si(OC2H5)4=0.01 hydrolyzed at 80℃

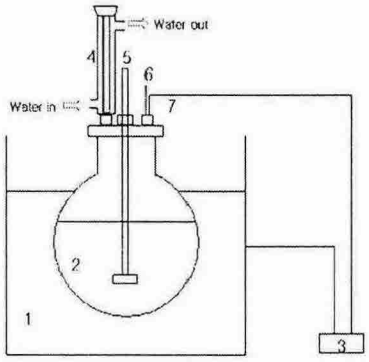

Fig.2 Schematic diagram of experimental apparatus and parameters

3. Results and discussion

Change of intrinsic viscosity with relative timet/tg for different mole ratio of Water/TEOS at mole ratio Ethanol/TEOS=2 showed in Fig.3. Water to TEOS ratio at 4.0 showed more larger viscosity value than other values of ratio which means more water content is favorable for rapid hydration process. Fig.4 showed FT-IR analyses according to heat treatment temperatures .According to FT-IR spectrum analyzing results, a little variation appeared from 450℃ and showed distinct shape changing at 500℃. From this result for Gel crystallizationheat treatment at 500℃ is optimum. Fig.5 shows FT-IR analyses according to heating durations at 500℃. Heating duration of 15 minute at 500℃ is enough for Gel crystallization. Also, according to TG measurements, heat treatment at 150℃showed rapid weight loss by dehydration and organic volatilization.

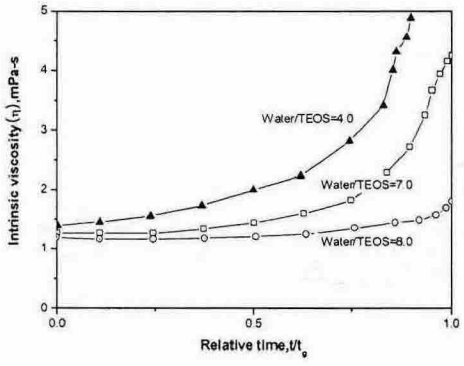

Fig.3 Change of intrinsic viscosity with relative time t/tg for different mole ratio of Water/TEOS at mole ratio Ethanol/TEOS=2

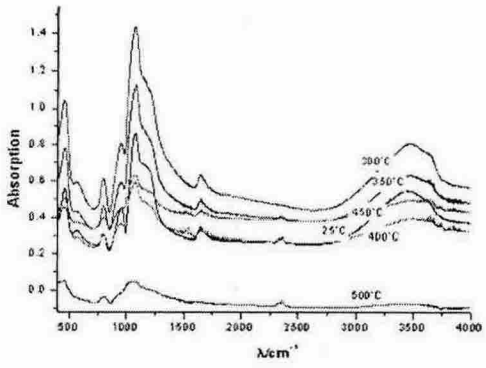

Fig.4 FT-IR analyses according to heat treatment temperaturte

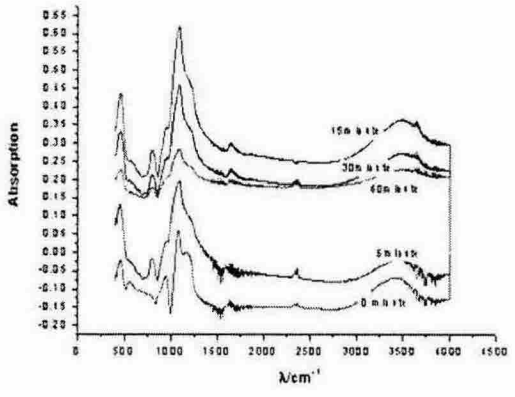

Fig.5 FT-IR analyses according to heating durations at 500℃

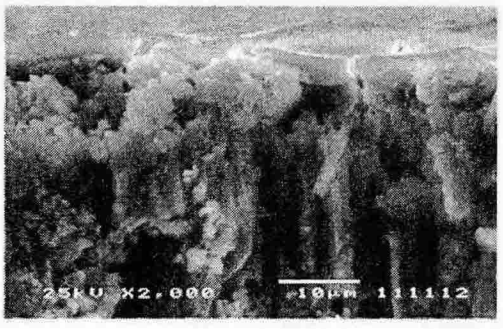

Photo.1 shows cross-sectional micrographs of SiO_2 coated layers after heat treatment at 500℃

After gelation cracks appeared on the surface in the case of having over viscosity value of 2.5mP-s. By Sol-Gel process SiO2 coating layers attached into the inner hole of anodized voids which showed well adhesion phenomena.

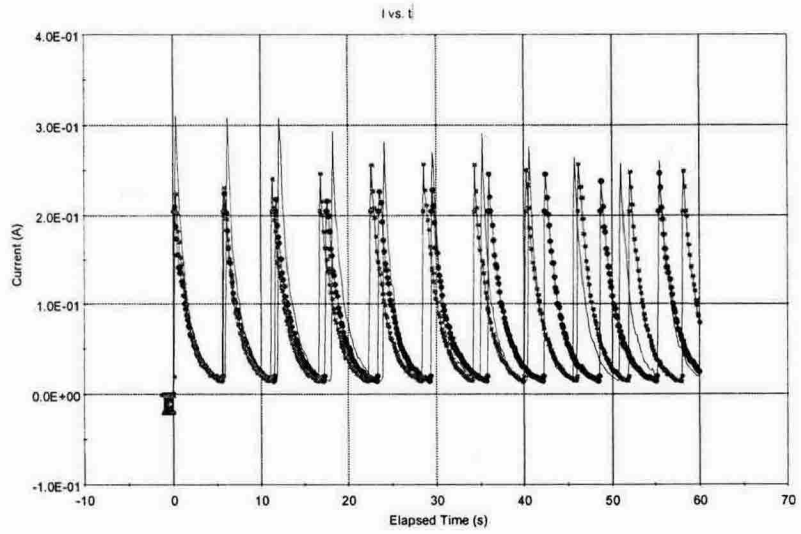

Fig.6 Chronoamperometry curves of SiO_2 coated on anodized foils at constant 1Volt condition

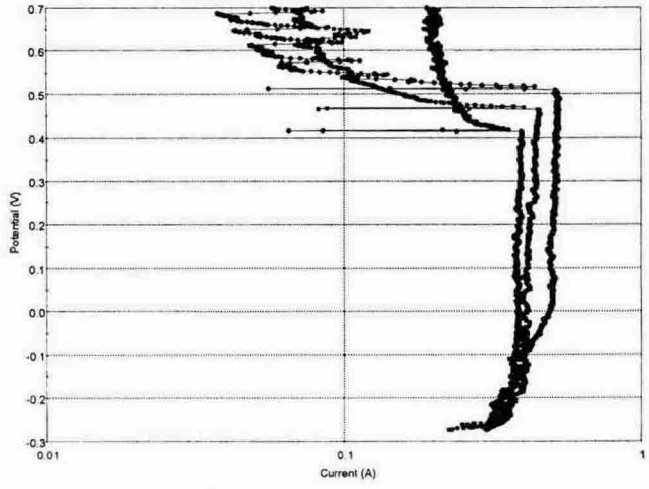

Fig.7 Potentiodynamic curves of SiO_2 coated on anodized for high voltage

Reference

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