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Change of Crystallographic Orientation Characteristics of (001)-Oriented Electrodeposited Silver Films during Self-Annealing at Room Temperature

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Abstract

Self-annealing behaviors of the electrodeposited silver films which preferentially orient in the (001) direction were investigated by in situ EBSD analysis. Self-annealing started in storage for a few hours at room temperature (R. T.) and was almost complete after storage for 6 h at R. T. In the initial stage of self-annealing, recrystallization of (001)-oriented grains progressed with the formation of twins with (212) orientation. Membrane stress in the as-electrodeposited film was compression stress of approximately 20 MPa. The compression membrane stress seems to become driving force for self-annealing of the (001)-oriented electrodeposited silver film. Membrane stress in the film changed from compression stress to tensile stress by recrystallization in self-annealing.

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Keywords: Electrodeposited silver film; Self-annealing; Electron back scatter diffraction patterns; In situ observation; Crystal orientation

1. Introduction

Good electrical contact resistance of the surface, good bend formability, good wear resistance and so forth are required to an electrodeposited silver film used for connectors and switches in automobiles. It is well known that self-annealing which is recrystallization at room temperature (R. T.) occurs immediately after electrodepositing in

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electrodeposited silver films [1-3]. Self-annealing occurs within a very short time such as one day and crystal grains orient in a certain direction by it. Since the crystal orientation strongly effects on physical and mechanical properties such as electrical contact resistance, wear resistance and corrosion resistance, elucidation of the self-annealing behaviors is very important. Miyazawa et al. have developed a new high-cyanide silver plating solution by optimizing the Se concentration and successfully formed a {200}-oriented electrodeposited silver film by self-annealing [3]. The silver film had good electrical contact resistance of the surface and good bend formability. Conventionally, the crystal orientation induced by self-annealing has been investigated by the X-ray diffraction (XRD) method [2, 4]. In the method, although the crystallite size, the orientation density and so forth can be investigated, the changes in crystallographic texture in individual grains can not be clarified. Recently, with the increasing use of the electron backscatter diffraction pattern (EBSD) method [5], the change in crystallographic texture during self-annealing can be investigated by in situ observation [6]. In the previous study, we have investigated self-annealing behaviors of an electrodeposited silver film preferentially oriented in the (001) direction by the EBSD method and clarified the change in crystallographic texture during self-annealing [7]. The aim of this study is to investigate the change of several characteristics of a (001)-oriented electrodeposited silver film in self-annealing process. In particular, the ratio of coincidence grain boundaries in the recrystallized area was investigated. Furthermore, the change in membrane stress of the film in self-annealing was also examined.

2. Experimental procedure

An electrodeposited silver film preferentially oriented in the (001) direction was fabricated on a Cu plate with 67 x 50 x 0.3 mm^3 size. The Cu plate was processed by electrolytic degreasing and cleaning with sulfuric acid before electrodeposition. Subsequently, a silver strike plated layer was deposited on the Cu plate to ensure adhesion between the Cu plate and the electrodeposited silver film. The solution used for silver strike plating contained 5.5 g/L $KAg(CN)_2$ and 90 g/L KCN, and the solution temperature and current density were 18 °C and 2 A/dm^2, respectively. A stainless-steel plate was used for the anode for silver strike plating. For (001)-oriented electrodeposition, a cyanide based plating solution containing 148 g/L $KAg(CN)_2$, 140 g/L KCN and 11 mg/L KSeCN was used. The solution temperature and current density were 18 °C and 5 A/dm^2, respectively. A pure silver plate was used for the anode for electrodeposition of silver. The electrodeposited silver film with 5 μm thickness was fabricated. Immediately after electrodeposition, each film was kept in a freezer at -20 °C to inhibit the progress of self-annealing.

Self-annealing behaviors were investigated by in situ observation using a field-emission scanning electron microscope (FE-SEM, Hitachi S-4300SE/N) with an EBSD analysis system (TSL Solutions OIM-Analysis 5) at R. T. For the observation, the electrodeposited silver film was cut into specimens with 10 x 10 x 0.3 mm^3 size. The normal direction of the surface of the film was the normal direction (ND) in the EBSD analysis. The analysis was conducted for the 200 x 400 μm^2 area with a step size of 1.5 μm and for the 5 x 5 μm^2 area with a step size of 25 nm. Membrane stress of the film was also examined in self-annealing process by XRD (Rigaku SmartLab) with Co-Kα radiation by iso-inclination method. Membrane stress is defined as the force per unit area of the vertical section in a film. The anisotropy of membrane stress depending on the section was not seen in the films investigated in this study. The XRD analysis was conducted at voltage of 40 kV and current of 40 mA. Furthermore, the cross section of the film which was stored at R. T. for six months was also investigated with the EBSD analysis. The cross section of the film was treated by Ar milling (Hitachi IM4000 ION Milling System). Ar milling was conducted for 2 h with 6 kV acceleration voltage, 1.5 kV discharge voltage and 0.08 ml/min Ar flow rate. The EBSD analysis was conducted with a step size of 60 nm.

3. Results and discussion

3.1. Inverse pole figure map

Fig. 1 shows inverse pole figure (IPF) maps of a (001)-oriented film obtained by the EBSD analysis with a step size of 1.5 μm. In the as-electrodeposited state, the microstructure consists of very fine grains that are too small to be analyzed by EBSD and there are few grains with a size of μm order.