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Characterization of oxide films and conversion layers on aluminium alloys

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ABSTRACT

Corrosion resistance and specific functional properties (dielectrical and decorative properties, adhesion, wear resistance) of aluminium alloys can be improved by surface treatments as electrochemical or chemical conversion reaction.

The purpose of this study is to discuss the applicability of spectroscopic ellipsometry (SE) and electrochemical impedance spectrometry (EIS) for the characterization of the obtained conversion surface layers.

It can be concluded that SE yields an accurate characterization for the thickness and the interfacial properties of both the barrier and porous oxide layer. The EIS allows to measure and to determine the sealing grade of the porous layer. These two complementary techniques can be used to investigate the growth mechanism of phosphate chromate conversion layers.

INTRODUCTION

Corrosion resistance and specific functional properties (dielectric and decorative properties, adhesion, wear resistance) of aluminium alloys can be improved by several surface treatments.

Each of these treatments : roughening, anodizing, sealing, conversion reactions induces chemical and morphological changes at the surface which are strongly correlated with the surfaces properties. To deal with this kind of investigations a number of analytical techniques have been used successfully during the last years [1,2]. Yet some methods are destructive, can not be used in situ and require a long and delicate specimen preparation. The purpose of this study is to discuss the applicability of spectroscopic ellipsometry (SE) and electrochemical impedance spectroscopy (EIS) for the characterization of oxide and conversion layers on aluminium alloys.

EXPERIMENTAL

The principles of SE [3] and EIS [4] were already extensively explained. Smooth, clean and highly reflecting aluminium substrates suited for ellipsometry were prepared by vapor deposition of 2-3 μm thick aluminium films on silicon wafers.

Barrier oxide layer were formed in a 3 % ammoniumtartrate solution of pH 5.5 at 22°C. The porous oxide films were grown in a 20 % sulphuric acid solution at 25°C. Sealing of this porous layer occurred in water at 95°C.

Aluminium surface was chemically converted in a 16 g/l chromic acid / phosphoric acid mixture containing 0.25 g/l HF at 60°C.

RESULTS

In order to show the complementary of the techniques to investigate in situ surface treatments of aluminium alloys SE and EIS (Bode diagrams) spectra of chemical conversion reaction are presented in figures 1 and 2 with as parameters the immersion time.

These results of SE can be interpreted with the SELLMEIER relation [5] (figure 3) and those of EIS with an equivalent network [6] (figure 4). Other results concerning these surface treatments of aluminium alloys can be found in several publications [7,8].

CONCLUSIONS

It can be concluded that SE yields an accurate characterization for the thickness and the interfacial properties of both the barrier and porous oxide layer. The EIS allows to measure the barrier layer thickness and to determine the sealing grade of the porous layer.

These two complementary techniques can be used to investigate the growth mechanism of phosphate chromate conversion layer.

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Figure 1 : Ellipsometric spectra for a conversion in a 0.34 ml HF/I bath solution at 50 °C for different immersion times.

a. $\tan \Psi$

b. $\cos \Delta$

Figure 2 : Impedance for a conversion in a 0.34 ml HF/I bath solution at 50 °C for different immersion times.

a. Bode-amplitude diagram

b. Bode-phase diagram

Figure 3 : Ellipsometric model for converted aluminium.

Φ_0 : angle of incidence

N_0 : refractive index of ambient medium

N_1 : refractive index of conversion layer, modelled by the Sellmeier dispersion relation

N_2 : complex refractive index of the substrate

d_1 : thickness of the conversion film

Figure 4 : Impedance model for converted aluminium immersed in an electrolyte.

R_1 : resistance of the electrolyte

R_2 : resistance of imperfections in the layer

R_3, C_3 : resistance and capacity of the conversion layer

Q_3 : impedance in the pores of the conversion layer