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A study of the Morphology and Optical Properties of Electropolished Aluminum in the Vis-IR region

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In this paper, the commercial pure aluminum specimens (Al 99.3 %) are electropolished in an acidic electrolyte. The morphology, reflection, and roughness of the specimens are investigated. Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX), X-ray Fluorescence (XRF), Vis-IR Spectroscopy, and Dek-Tak method are used to characterize of specimens. The results show that by increasing current density from 5 A/dm^2 to 25 A/dm^2 the roughness and reflectance of Al specimens decrease/increase respectively. The opposite trend is seen when the current density increases more than 25 A/dm^2. The roughness of Al specimen before electropolishing is 3.45 μm and it will decrease to 0.45 m by electropolishing at optimum conditions. A similar trend is also seen in reflection; it increases from 10% to 93% for the optimized electropolishing specimen. The SEM study confirms that the electropolishing could decrease the surface roughness, and it can form a smooth surface in micro/nano meter scale. According to the results, the metal mirror that is prepared by Physical Vapor Deposition (PVD) method has higher reflectance in comparison with the optimized electropolishing specimen. It shows that the purity of Al also has an essential effect on the reflection spectra.

Keywords: Electropolishing, Aluminum, Metal mirror, Reflection, Roughness

1. INTRODUCTION

The combined properties of high reflectivity and low absorption give rise to the use of aluminum as a mirror in a variety of optical devices, such as lasers, solar cells, interferometers, and astronomer's instruments. The Physical Vapor Deposition (PVD) technique is usually used to create the metal mirrors. In this technique, the metal mirror is created by vaporizing the metal (aluminum) in a vacuum and allowing it to re-condense on to substrate. High cost and limited substrate area are the main disadvantages of the PVD technique [1-10].

The electropolishing (EP) process is a surface treatment technology. When performed properly, it produces a surface finish that can be used as a metal mirror. In EP process, electrochemical anodic dissolution is carried out on the surface of anode according to equations 1 and 2 (for aluminum) [1, 11-12].

$$Al \rightarrow Al^{3+} + 3e^{-} \quad (1)$$
$$2Al + 6OH^{-} \rightarrow Al_2O_3 + 3H_2O + 6e^{-} \quad (2)$$

During the anodic dissolution, the dissolution rate at the anode is slowest and is the controlling factor. Therefore, the electrochemical reaction is under diffusive mechanism. Due to the diffusive mechanism, a viscous layer will be formed on the anode (figure 1). With respect to the bulk of the electrolyte, this layer has higher viscosity and greater electrical resistivity. The thickness of the insulating layer is greater in crevices (β) than on projections (α). The current density on projections is higher than in crevices. For this reason, projections dissolve more rapidly than crevices, and this produces a surface-leveling effect [1, 12-14].

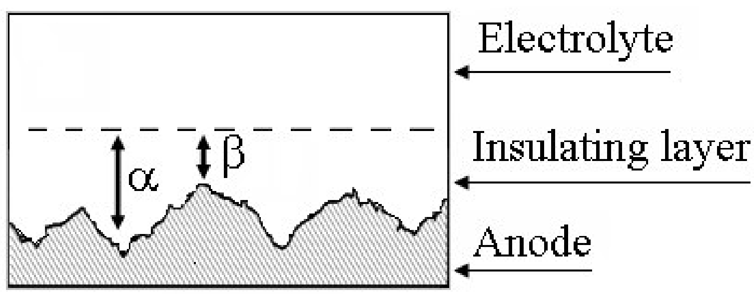

Figure 1. Schematic of typical formation insulating layer on the surface of anode.

In recent years, the EP process has become an important technology for precision engineering and high-tech industries. It is widely used for components industries where the demand for surface qualities such as smoothness, brightness, and cleanliness is high. The EP is employed in the medical, petrochemical, pharmaceutical, semi-conductor, biomedical, electronic, and optic industries. In addition, electropolishing has been employed to prepare surfaces for further treatment in the PVD technique [15-17].

In this paper, we present studies of Al electropolishing in acidic electrolyte, and the effects of current density, electrolyte temperature, and EP time on the morphology and optical properties of specimens investigated.

2. EXPERIMENTAL PART

The electrolyte employed was a mixture of 58% phosphoric acid, 14% sulfuric acid, and 8% chromic acid in distilled water [18]. Specimens of sheet aluminum (3×9 cm) were initially degreased in trichloroethylene and then cleaned in 40% HF-60% HNO_3 solutions. In EP process, the range of current density was from 5 to 30 A/dm^2, the electrolyte temperature range was 80 to 100 °C (±2 °C), and the electropolishing was carried out for 2 to 15 minutes. The morphology of the specimens was characterized by Scanning Electron Microscopy (SEM). Surface roughness measurements were made using a profilometer (Sloan DekTak auto-leveling). Measurements were repeated three times for each specimen. A Hitach U-3410 spectrophotometer was used to obtain reflection spectra of specimens in the range of 300-1500 nm. Elemental analysis of Al specimen was carried out by X-ray fluorescence (XRF). In situ Energy Dispersive X-ray (EDX) spectrophotometer was also done to determine the element compositions. A specimen is prepared by Physical Vapor Deposition (PVD) method and it nominated PVD-Al.

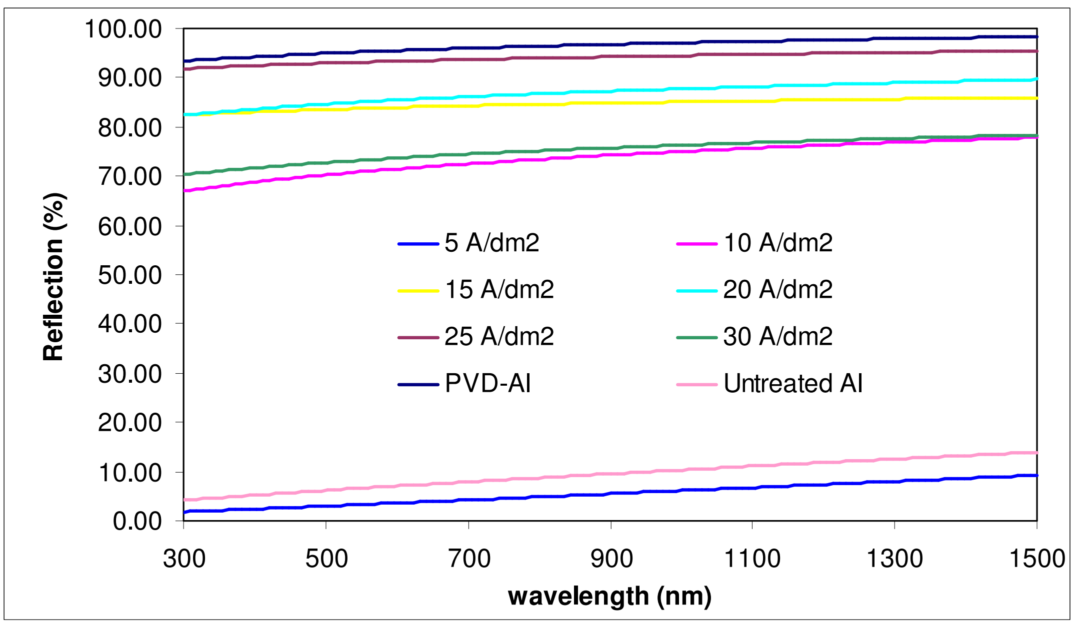

Figure 2. The Vis-IR spectra of electropolished specimens (at different current density), untreated and PVD specimens.

3. RESULTS AND DISCUSSION

The Vis-IR spectra of untreated specimen, PVD-Al, and the specimens that electropolished in different current density is shown in figure 2. It is seen that the reflectance of electropolished specimens are strongly affected by varying current density from 5 to 30 A/dm^2. It is clear that the current density is an important indicator for the formation of the insulating layer on the anode and according to applied current density, there are three zones in electropolishing process: etching zone, polishing zone, and gassing zone [13]. In low current density and in the etching zone, the thickness of insulating layer is low and weak corrosion carries out on the surface of anode. So the surface is unsmoothed by electropolishing. Therefore, the reflectance of the specimen that electropolished in 5 A/dm^2 is low even in comparison with untreated specimen. By increasing current density in the range of 10 to 25 A/dm^2, the reflectance of the specimens will be enhanced and approached it's maximum value of 25 A/dm^2. In other words, in all current densities higher than 5 A/dm^2, electropolishing takes place in the polishing zone; 25 A/dm^2 is the optimum current density in which to achieve higher reflectance. When the current density falls below 25 A/dm^2, the formation rate of the insulating layer- and thus thickness- is low and not appropriate for producing a high reflection metal mirror. At the optimum current density, the formation and dissolution rates of the insulating layer are balanced, obtaining the best result. By increasing the current density from 25 A/dm^2 to 30 A/dm^2, the electropolishing of occurs in the gassing zone. In this zone, the anodic dissolution is accompanied by the evolution of oxygen (equations 3) [1, 11-12].

$$4OH^- \rightarrow O_2 + 2H_2O + 4e^- \tag{3}$$

The faster formation of the insulating layer and evolution of oxygen will generate more heat on the aluminum surface, which could lead to the deformation of the insulating layer and a non-uniform dissolution, consequently producing a rough surface. Thus, increasing the current density from 25 A/dm^2 to 30 A/dm^2 causes a decrease in the reflectance from 93% to 77 %.

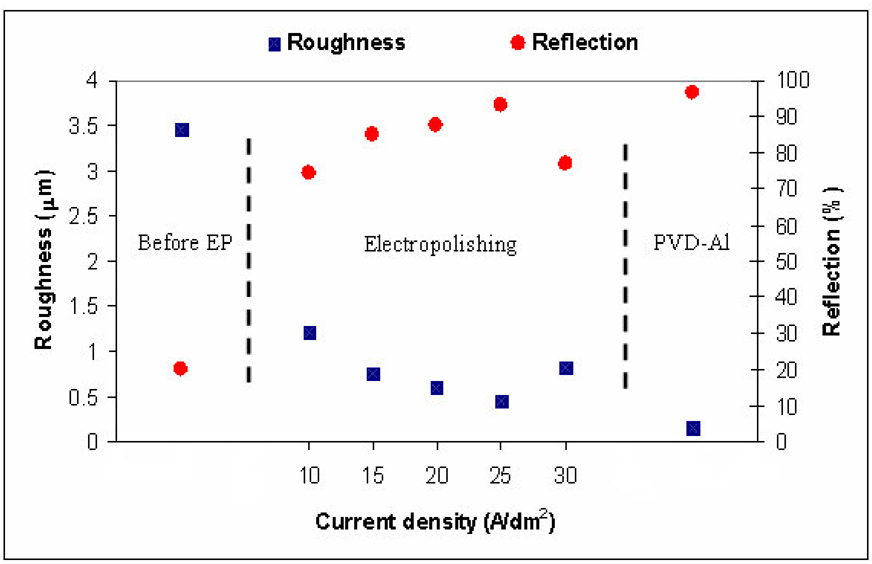

Figure 3. Surface roughness and Reflection values before and after electropolishing (EP) and theirs variety with current density.

The surface roughness of electropolished specimens is believed to be changeable and controllable by varying the electropolishing conditions [17, 19]. The reflection of the specimens can be ascribed to their surface roughness. In Figure 3, the surface roughness values and their reflectance of 900 nm show for untreated specimen, PVD-Al specimen, and specimens that were electropolished at a different current density. Significant differences are found for the surface roughness values before and after the electropolishing. The surface roughness was 3.45 μm for untreated-Al and then decreased to 0.45 μm after electropolishing at 25 A/dm^2. Further, as shown in Figure 3, the current density will affect the surface roughness. The lowest degree of roughness is obtained with 25 A/dm^2 as the optimum current density. The same effect was also observed for the surface reflection. Therefore, keeping an average current density of 25 A/dm^2 is a reasonable choice.

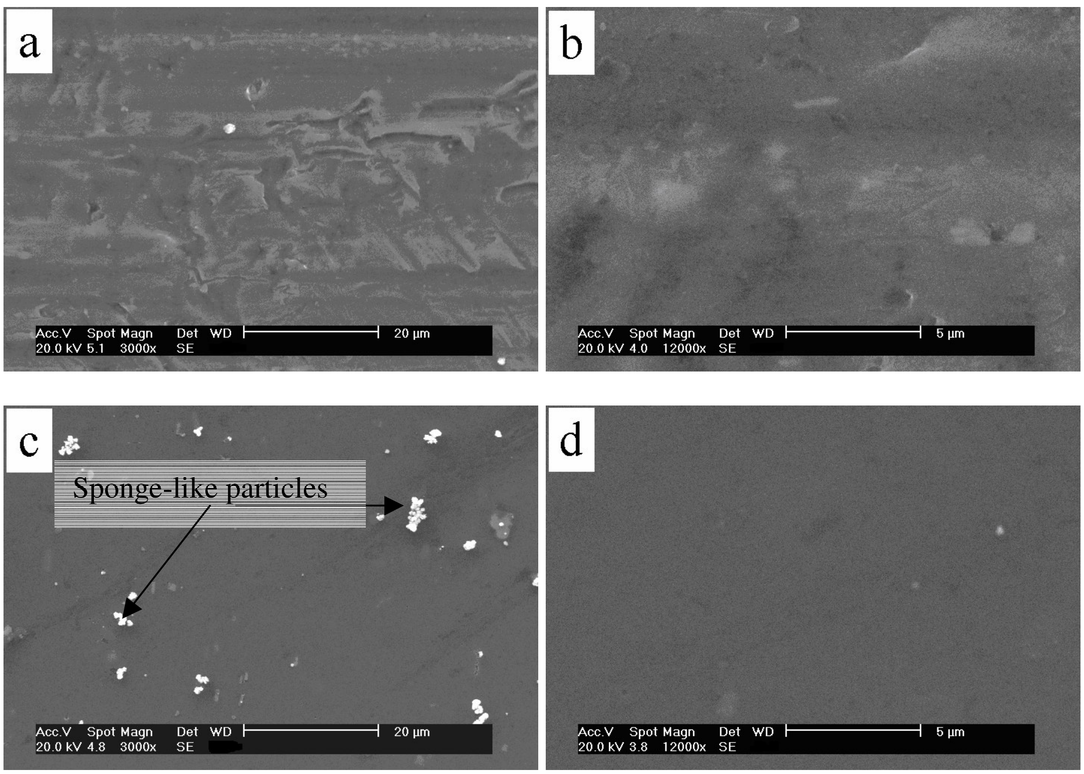

Figure 4. SEM micrographs of the surfaces of Al specimens: (a), (b) before and (c), (d) after the electropolishing

With respect to the effect of morphology on refection, the typical SEM micrographs of surfaces before and after the electropolishing of Al specimens are shown in Figure 4. The Al specimen before EP shows an uneven and concave surface with considerable streaks viewed under both low and high magnification (Figures 4-a, 4-b, respectively). The electropolished surface is also smooth under low magnification (Fig. 4-c) and completely featureless under high magnification (Fig. 4-d).

Many amounts of fine dispersed sponge-like particles, which are less than 500 nm in size, are survived on the electropolished surfaces (Fig. 4-c). A large amount of Al and traces of Fe, Mg, and Si are detected by EDX analysis and its spectrum showed in figure 5. With respect to EDX analysis, it seems that an accumulation of impurity occur during EP process. By accumulation the impurity, the sponge-like particles are formed on the surface of anode. These particles may be absorbed light and affected the reflection of specimens.

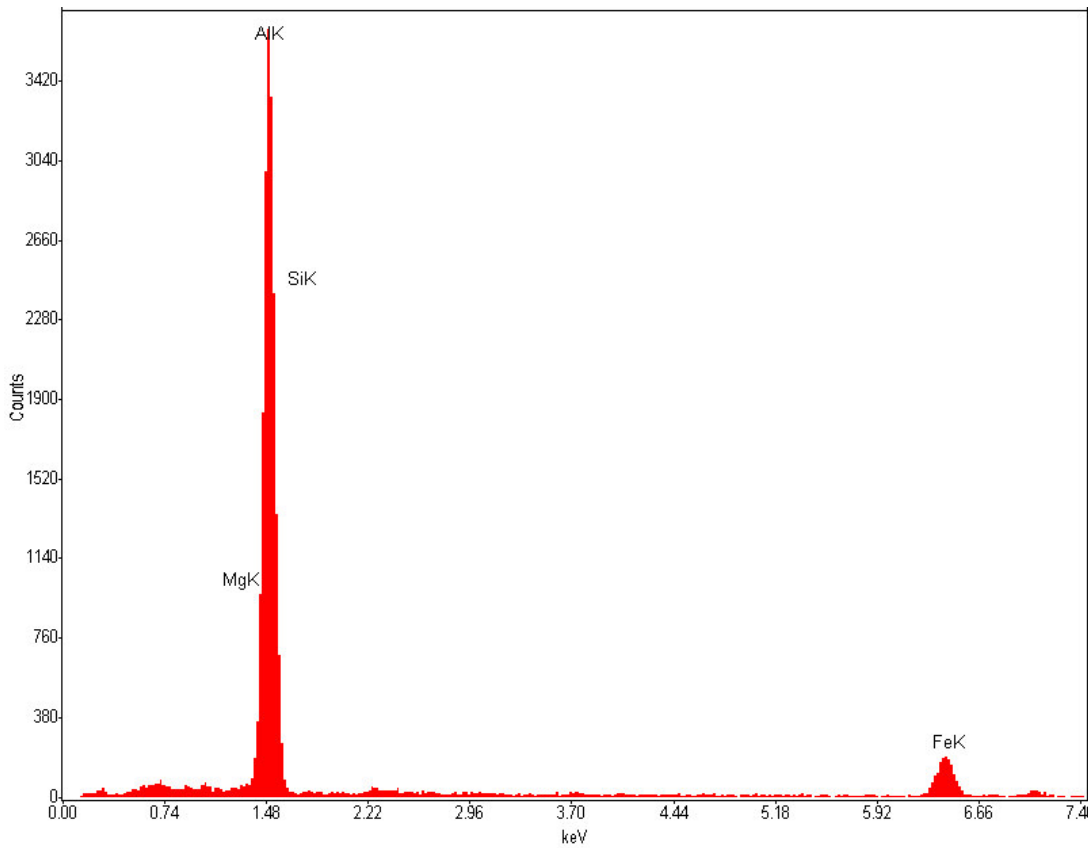

Figure 5. EDX spectrum of the sponge-like particles on the electropolished surface in figure 4-c.

Table 1. Elemental analysis of the aluminums that used for preparation EP and PVD specimens

	Al	**Si**	**Fe**	**Mg**
EP specimens*	99.3%	0.4%	0.2%	750 ppm
PVD-Al specimen**	≥99.99%	% trace metals basis		

*obtained by XRF. **from Balzers co.

According to Figures 2 and 3, the reflectance of PVD-Al specimen is higher than the reflectance of the best specimen that was electropolished. There are two reasons for this difference: roughness and purity. The surface roughness of PVD-Al is lower than other specimens. Therefore, it is expected that the PVD-Al has higher reflectance than the best EP specimen. Furthermore, the purity has an effect on reflection so that whatever the purity of Al increases to the reflection will be enhanced [2]. Table 1 provides elemental analysis of aluminum that they are using for electropolishing and PVD-Al. The purity of Al is 99.3 % in EP and 99.99% in the PVD.

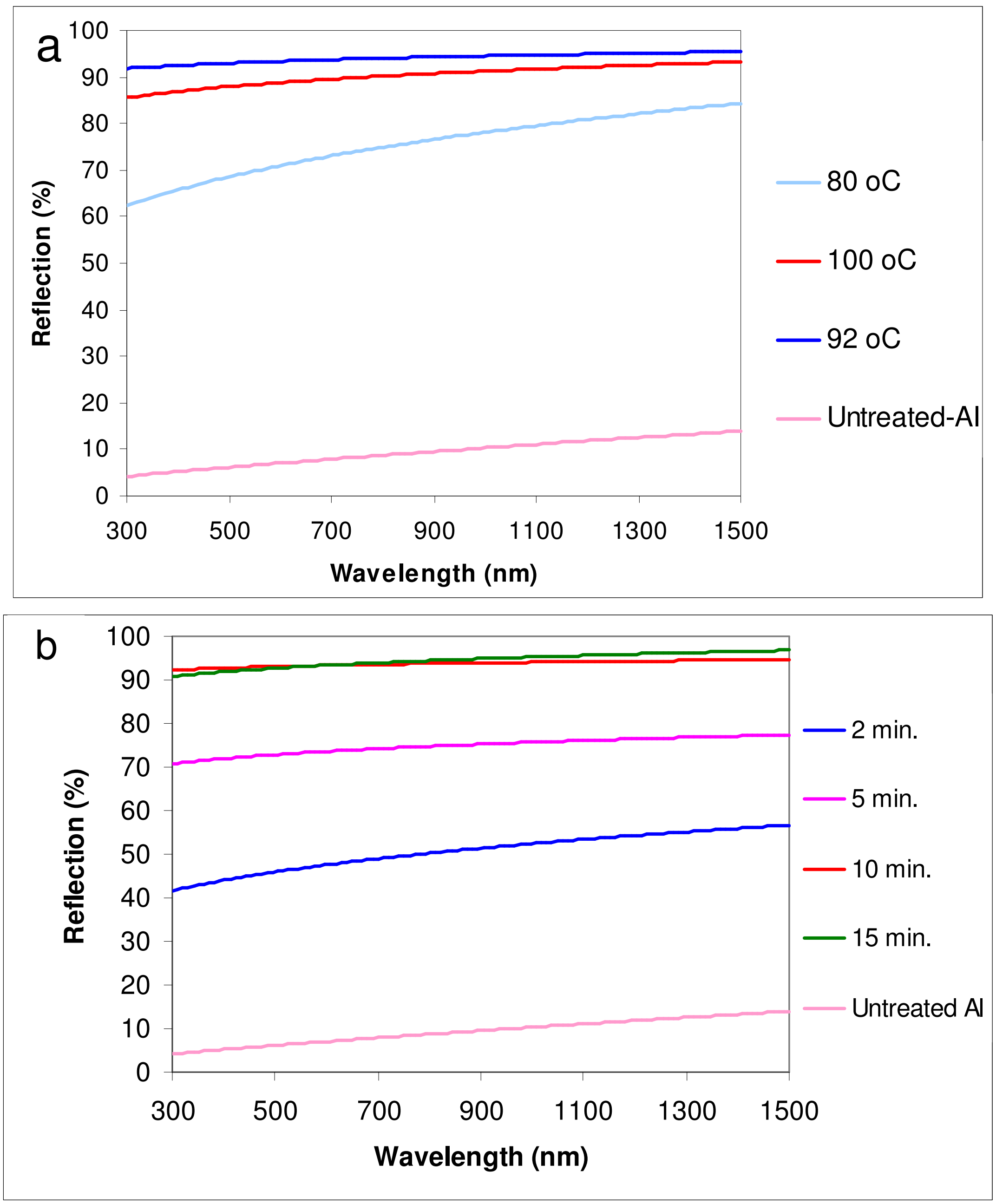

Figure 6. Effects of (a) electrolyte temperature and (b) electropolishing time on reflection of Al.

Figure 6-a shows the reflection spectra obtained from electropolishing electrolytes at temperatures from 80°C to 100°C. The current density is fixed on 25 A/dm^2. High reflection property is obtained when the electrolyte temperature adjusted to 92 °C. In lower temperatures (80°C), the formation rate of the insulating layer is high (it depends to current density) and the dissolution rate is low. Consequently, the thickness and resistance of insulating layer increase. By increasing temperature to 92°C, we can achieve optimum thickness and resistance. If the temperature is increases too much, the insulating layer will reduce in thickness due to the higher dissolving power of the electrolyte. Moreover, the diffusive mechanism of the electrochemical reaction will be eliminated, so the insulating layer will not form completely on the anode surface. In these conditions, the differences between the current density passing through projections and crevices decrease. Therefore, projections and crevices dissolve at the same rate, and this does not produce a surface-leveling effect. Hence, the optimum electrolyte temperature is proposed as 92°C.

A similar effect of electropolishing time can be seen in the reflection of specimens. The spectra shown in figure 6-b is obtained by electropolishing at 92°C and with a current density of 25 A/dm^2. It is clear that the optimum time for electropolishing is 10 minutes. In this time, the projections are eliminated and leveled out equal to crevices. Continuing the electropolishing after this time has no further effect on the reflection of specimens. Therefore, the reflections of specimens electropolished at 10 and 15 minutes are the same.

4. CONCLUSIONS

In this work, electropolishing was used to provide smooth and highly reflective aluminum sheets with commercial purity (99.3% Al). The specimens were electropolished in an electrolyte that is a mixture of phosphoric/sulfuric/chromic acids. When a current density of 25 A/dm^2 was applied to Al specimens, with the electrolyte temperature maintained at 92 ^{0}C and the electropolishing carried out for 10 minutes, an apparently mirror-like surface with high reflective properties was achieved. The results show that electropolishing could be used as a method for preparation of metal mirror, like the PVD technique.

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