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# NANO/MICRO SMOOTHING AND REFLECTIVITY ENHANCEMENT FOR PREPARATION OF ALUMINUM SURFACE MIRROR

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**ABSTRACT**

Optical characteristics of aluminum surface are vital for several applications and high reflectivity is required for applications of solar devices and optical microscopes. Electrochemical polishing (EP) was used for enhancement of leveling of the surface and elimination of nano/micro asperities, cavities and scratches. Electrolytic solution was prepared using mixture $H_3PO_4$, $H_2SO_4$ and $H_2O$, with concentrations 55%, 14%, 31%, respectively. Ethylene glycol (EG) was added with different concentration 10, 20, 30 and 40 ml/l. After EP process, the samples were washed well and dried at 110 ˚C. Then, the surface was investigated by SEM, AFM to evaluate the roughness of the surface. Gloss value and reflectance of the surface were obviously increased after EP treatment. Hardness and thickness of the surface were measured after EP treatment, where hardness was increased from 49 to 53 Brennel HB and thickness was increased from 1 to 3 μm after EP with adition of 20ml EG/l. Addition of 10ml EG/l showed the increase of corrosion resistance of Al surface to 36.707 KΩ and decrease of corrosion rate to 0.01890 mm/year after EP more than other concentrations.

**Keywords:** reflectivity, aluminum surface, electrochemical polishing, electrolyte, ethylene glycol.

## 1. INTRODUCTION

Aluminum metal is recommended for several applications, due to its characteristics like, workability, lightness, luster, conductivity, corrosion resistance and environmental friendliness. When the surface is professionally modified to reduce roughness as nano/micro burrs, imperfections or scratches; highly smooth and reflectance aluminum surface mirrors could be produced. So, aluminum surface is promised to be used as a vital component in different applications such as concentrated solar power (CSP) systems and reflective optical microscope objectives [1-3].

Different methods such as mechanical polishing (buffing) and chemical polishing could be used to achieve surface brightness. On the other hand, electrochemical polishing (EP) is highly selective to eliminate ultra-fine humps over the level of the surface and fill micro recesses down the level of the surface. It is considered the best choice due to low time, low cost, energy saving, simplicity and nano/micro size accuracy [4-6].

EP is efficiently enough for brightening and nano/micro smoothing of metals and alloys such as aluminum, copper, stainless steel, bronze and brass. Engineering and practical interests paid attention to this technique due to its precise treatment without surface deformation [7-10].

In recent decades, several efforts were exerted to improve smoothness, luster and reflectivity of metal surfaces. The effectiveness of an acid solution of phosphoric acid and ionic liquid medium of ethylene glycol and choline chloride was examined to enhance the efficiency of EP process of aluminum surface [11]. In this study, it was ascertained that pitting is relatively inevitable, less surface area was achieved and bubbles was flowing over low surface area as the sample polished [12, 13].

EP of aluminum was carried out in ethanol–perchloric acid electrolytes and ultrasonic was used for agitation. The addition of ethanol to perchloric acid influenced the plateau wideness, height of polarization curve and hence uniformity of the surface [14].

Electrolytic solution could be supported with some additives to reduce pitting and defects on the sticky surface may form during EP process due to oxygen bubbles evolution. Some workers compared the addition of soluble starch, methanol and ethylene glycol to improve the surface smoothness and they deduced that samples after EP using ethylene glycol showed better gloss values and lower roughness more than samples after EP using either soluble starch or methanol. Others added ethanol to prevent the destroying of the film and formation cavities [15, 16].

Electrolytic solution composed mainly of thiourea and additives such as polysaccharides as reducing materials and mineral acids as conductivity improvers were used by Edson [17] for brightening of copper, silver and gold. Also, Mayer [18] separated between anode and cathode using non-conductive slit to inhibit bubbles transfer to the surface of anode and enhance the ubiquitous distribution of electric current.

In the patent US3970529, Sylvia Martin invented a process for reduction of pitting, polishing and brightening of aluminum and aluminum alloys. In this process, a bath of 30-95% $H_3PO_4$, poly alkylene ether and a minor amount wetting agent. When lower amount of $H_3PO_4$ (< 60%), relative long time, high electric potential and higher operating temperature should be used [19].

Some workers examined electrolytic polishing solution for aluminum and aluminum alloy using a mixture of $H_3PO_4$, $H_2SO_4$, poly ethylene glycol and potassium sodium tartrate, and others avoided addition of

hazards solutions like chromic acid due to environmental threatening [20, 21].

In the present study, EP of aluminum was carried out using bath composition of mixture of phosphoric acid $H_3PO_4$ and sulfuric acid $H_2SO_4$ and water $H_2O$. The purpose of this work is to enhance the surface smoothness and produce highly reflective and super reflective aluminum surface mirrors. So, ethylene glycol (EG) was added to reduce pitting occurred and defects formed during EP process. Different concentrations of EG were added to maintain the best smoothness and reflectivity. In this work, the assessment of samples after EP was evaluated by measurements of Field emission SEM, AFM, surface hardness, surface thickness, gloss value, reflectance and corrosion resistance.

## 2. EXPERIMENTAL PART

### 2.1 Materials and Apparatus

Aluminum 99.5% and lead plates were used as anode and cathode in the electrolytic cell. Phosphoric acid 85%, Sulfuric acid 98%, deionized water and ethylene glycol were used for preparation of electrolytic solution.

DC Power supply GW Lab DC Power Supply (30V/3A) GPR-3030, EG&G Potentiostat / Galvanostat (Applied Princeton Research) Model 273A, driven by software M352/252 Corrosion and Magnetic stirrer (PMC-BARNSTEAD/THERMOLYNE-USA).

### 2.2 Procedures

Aluminum sheet of thickness 0.5 ml was cut into samples 10 x 2.5 cm for bench scale and 60x50 cm for pilot scale. The experimental work was carried out by using two electrodes system, where sample was fixed as working electrode and lead plate (10 x 2.5 cm) as counter electrode into a glass cell 15 × 7.5 × 8 cm using GW Lab DC Power Supply GPR-3030.

Three electrodes system comprised aluminum working electrode, platinum auxiliary electrode and saturated calomel reference electrode (SCE) was used. Each electrode was fixed well into the same glass cell and connected to EG&G Potentiostat / Galvanostat (Applied Princeton Research) Model 273A, driven by software M352/252 Corrosion.

Electrolytic solution was prepared using mixture $H_3PO_4$, $H_2SO_4$ and $H_2O$, with concentrations 55%, 14%, 31%, respectively. A magnetic stirrer (PMC-BARNSTEAD/THERMOLYNE-USA) was used for agitation during preparation of the electrolytic solution, where the electrolytic solution was mixed well for 5 min to have a homogenous solution, and then agitation was stopped before starting EP. Then, the sample was taken out, and rinsed with tap water and distilled water. All the samples were dried at 105 °C for 15 min, and then the average gloss value (G) of the surface (five points) was measured after 10 min ($\Sigma G/i$, i=5). All samples were stored well in a desiccator before the surface investigation.

### 2.3 Measurements

- Gloss value (G) was measured as indication of brightness of the surface by using ERICHSEN PICOGLOSS 503 Gloss-meter.
- SEM images were measured by using JEOL, JXA-840 A: ELECTRON PROB MICROANALYZER Dimension 3100 Scanning Probe Microscope (SPM) to detect the morphology of aluminum surface before and after electrochemical polishing technique.
- Atomic Force Microscope (AFM), Shimadzu SPM-9600 Dynamic Mode (Japan) was used to measure the surface roughness in micro meter of Al surface.
- Thickness meter of passive thin film was measured using Elecometer (Elcome ter Instruments Ltd, England) Br. Pat. No 624572 US Pat. No 2469476 (with sensitivity of +3%).
- Hardness meter of the Al surface was measured using Braive Instruments.
- Spectrophotometer V-570 UV/VIS/NIR serial number CO296357 was used for the measurements of the surface reflection.

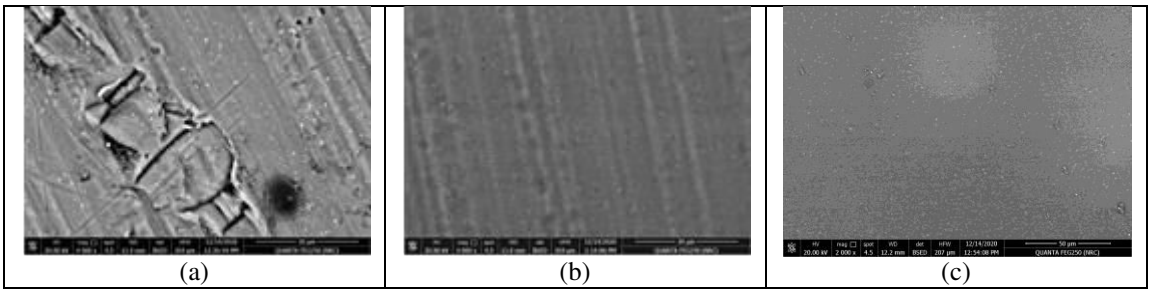
## 3. RESULTS AND DISCUSSIONS

It is essential to avoid roughness and nano/micro scratches may present over aluminum surfaces, when highly smoothness, brightness and reflectivity are required for applications in vital devices. EP technique was successfully used for alleviation nano/micro roughness, and removal of fine burrs and depressions. So, EG solution was added with different amounts 10, 20, 30 and 40 ml/l. It was observed that aluminum surface was turned to aesthetical appearance, highly smooth, shiny and seems like mirrors as shown in Figure-1.

**Figure-1.** Aluminum samples (60 x 50 cm) after EP treatment in pilot scale.

### 3.1 Morphology of Al Surface

The surface of Al samples was investigated to explore the variation of morphological structure of the surface after EP treatment. Figure-2 revealed the arrangement of crystal structure of the outer surface after EP, where it turned from rough and non regular to smooth and regular patterns. While more uniformal smooth surface was appeared and leveling was achieve after elimination of micro asperities and sharp roughness of the surface using EP electrolytic solution supported with 20 ml/l EG added.



(a) (b) (c)

**Figure-2.** Scanning electron microscope (SEM) images of aluminum substrate (a), electro polished sample (b) and electro polished sample by addition of 5 ml EG solution (c).

### 3.2 Topography of Al Surface

Inspection of topography of Al samples using AFM (Figure-3), the resules indicated that peaks and cavities were ceased after EP treatment compared with aluminum substrate surface. Moreover, enhancement of leveling and smoothness was achieved, where plane images af AFM results ascertained the surface leveling and smoothness after addition of 20 ml EG/l to the electrolytic solution (Figure-4a-c).

**Table-1.** Measurements of Al surface roughness.

| Sample | Ra, nm | Rz, nm | Rzjis, nm | Gloss value (G) |
|---|---|---|---|---|
| Al substrate | 77.28 | 1047 | 510.91 | 911 |
| After EP | 21.76 | 454.09 | 208.94 | 1804 |
| After EP addition of 20mlEG/l | 3.52 | 108.62 | 44.91 | 2968 |

The results of surface roughness measurements using AFM showed that the date of $R_a$, $R_z$, $R_{zjis}$ were obviously decreased after EP process (Table-1). Al substrate showed high values 77.28, 1047 and 510.91 of $R_a$, $R_z$ and $R_{zjis}$, respectively, while these values were decreased to 21.76, 454.09 and 208.94, respectively after EP treatment of the surface. More enhancement of smoothness and reflectivity was achieved after addition of 20ml Eg/l during EP treatment, where roughness was highly decreased as the values of $R_a$, $R_z$ and $R_{zjis}$ were decreased to 3.52, 108.62 and 44.91, respectively.

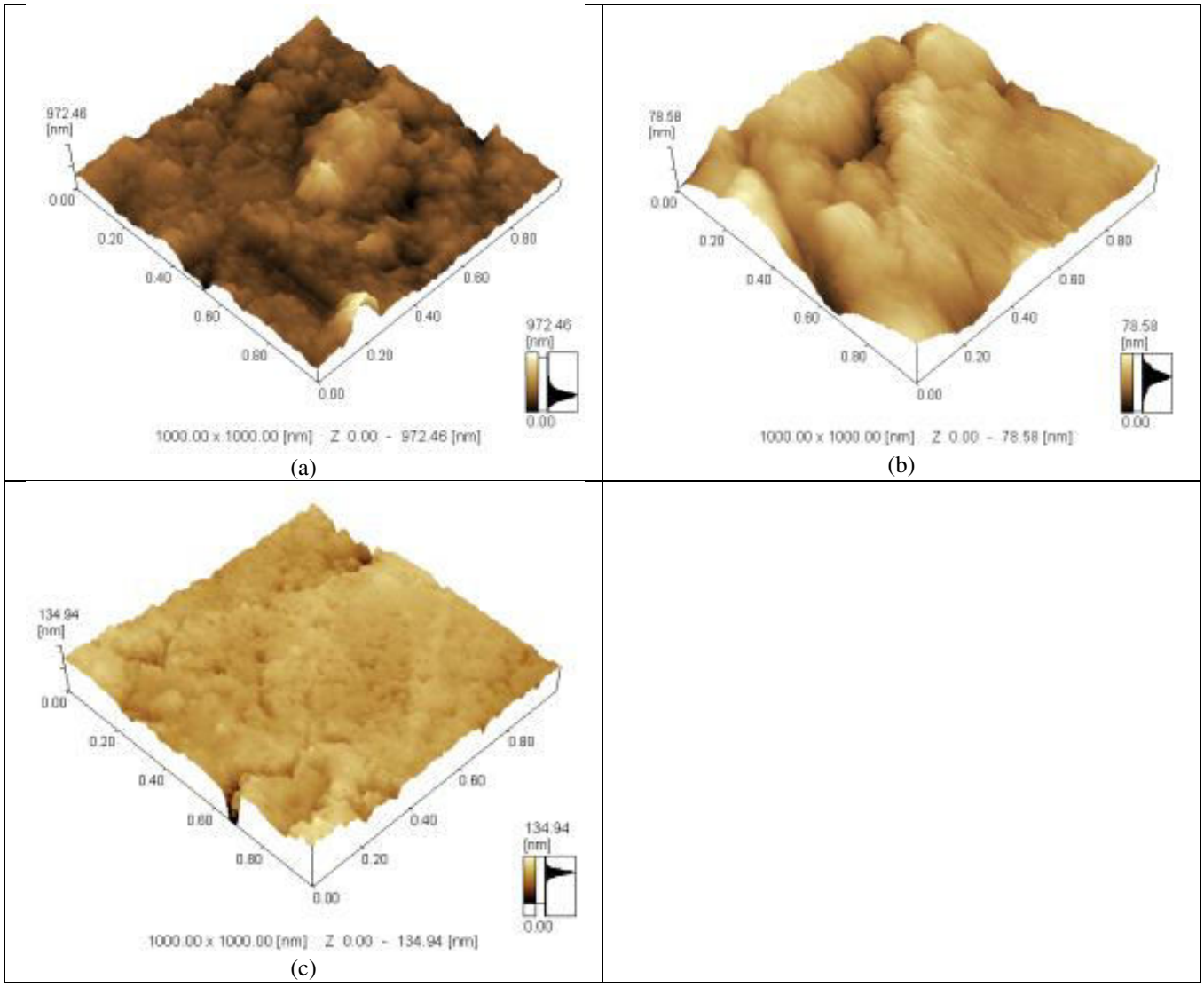



**Figure-3.** Atomic Force Microscope (AFM) images of Al substrate 1µm x 1µm (a) Al substrate, (b) Al sample after EP and (c) Al sample after EP by addition of 20 ml/l EG.

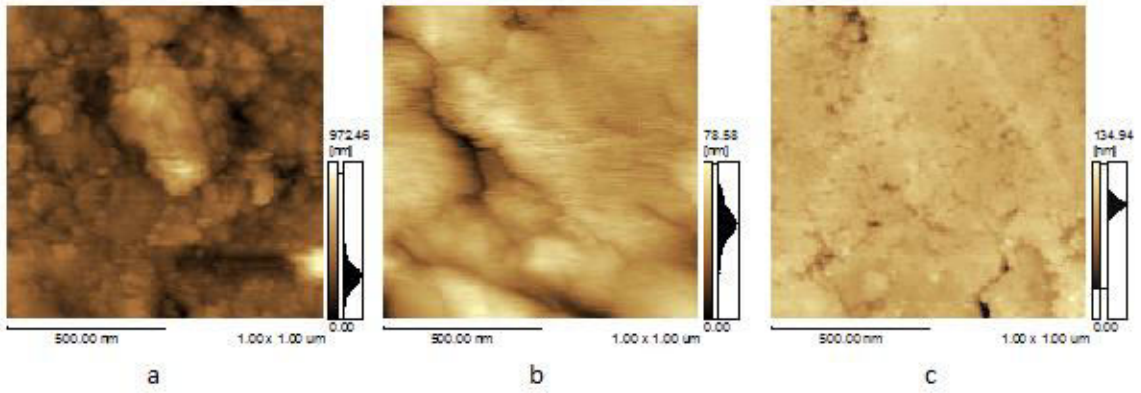



**Figure-4.** Atomic Force Microscope (AFM) plane images (a) Al substrate, (b) Al sample after EP and (c) Al sample after EP by addition of 20 ml/l EG.

### 3.3 Measurements of Gloss Value of Al Surface

The measurements of gloss value ( G ) between diferent Al samples confirmed the above results, where the surface after EP process was clearly enhanced after addition of EG as supprting electrolyte. The results revealed that EP treatment of Al surface after addition of

20 ml EG/l showed vastly variation of gloss value and the highest brightness (G = 2968) compared with Al substrate (G = 911), while it showed barely variation of gloss value after EP without addition of EG (G = 1804) as shown in Table-1.

### 3.4 Measurements of Hardness and Thickness of Al Surface

After EP process of Al samples, the measurements showed the increase of both hardness and thickness (Figures 5 and 6), where a new compact thin passive layer was formed due to anodic oxidation during EP process according the following equations:

$$Al \rightarrow Al^{3+} + 3e^-$$

$$6H_2O \rightarrow 4H_3O^+ + O_2 \uparrow + 4e^-$$

$$Al^{3+} + OH^- \rightarrow Al(OH)_3$$

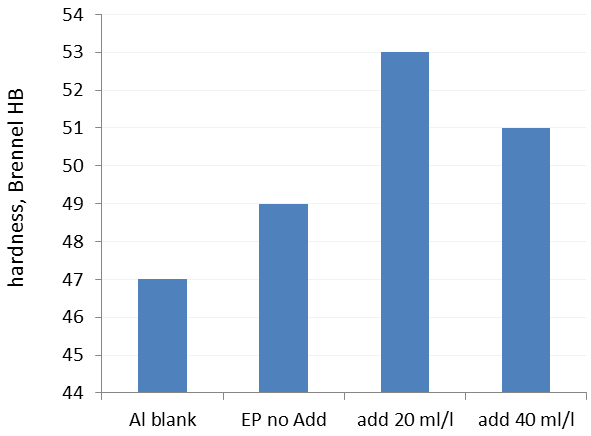



**Figure-5.** Hardness measurements of aluminum after EP and addition of EG comparing with Al substrate.

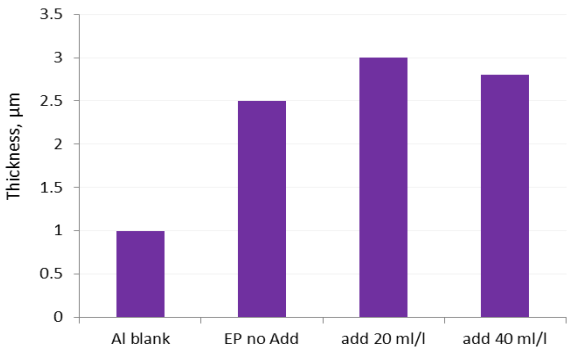



**Figure-6.** Thickness measurements of aluminum after EP and addition of EG comparing with Al substrate.

The surface hardness measurements displayed the increase of hardness after EP process from 47 to 49 Brennel HB and thickness from 1 to 2.5 µm. Also, hardness and thickness were vastly increased to 53 Brennel HB and 3 µm, respectively after adition of 20ml EG/l to the electrolytic solution. This indicates that the addition of EG motivate the reactions of anodic oxidation and decrease the defects of the formed passive film due to the effect of oxygen bubbles evolution. While, it was found that more addition of EG (40 ml/l) showed lower hardness and thickness 51 Brennel HB and 2.75 µm than addition of 20ml/l, this may occurred after the increase of viscosity and low mass transfer and ohmic resistance through the electrolytic solution.

### 3.5 Measurements of Optical Characteristic of Al Surface

Optical characteristics of Al surface was also investigated by measuring reflectance as indication of brightness ensuing from enhancement of nano/micro smoothness and avoiding fine asperities and scratches. Fig.7 shows that the reflectance of Al surface was clearly increased through a wide range of wavelength including regions of UV, visible and IR (λ= 250-2500 nm). In the visible region (λ= 350-850 nm), the reflectance of Al substrate (R= 5-18 %) was increased to (R= 19-40 %) after EP treatment. In contrast, the reflectance was obviously improved after EP and addition of 20 ml EG/l, where the reflectance of Al surface measured in visible region increased to (R= 20-60 %). This indicates the success enhancement of brightness and reflectivity of Al surface and the optical surface characteristics is promised and qualified for using in advanced applications.

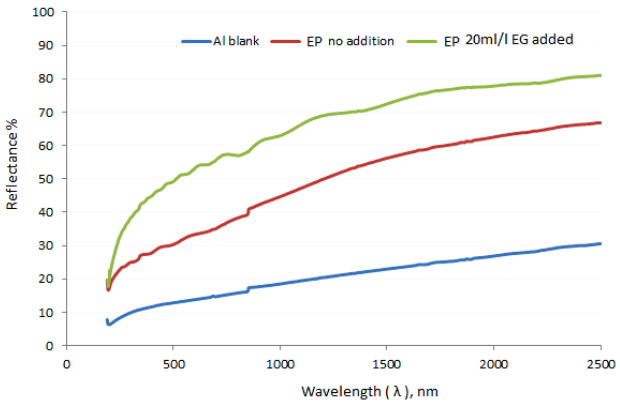



**Figure-7.** Reflectance measurements of aluminum after EP and addition of EG comparing with Al substrate.

### 3.6 Linear Polarization of Al Surface

The investigation of linear polarization of Al substrate, Al surface after EP and Al surfaces after EP and addition of EG in the same aggressive corrosion conditions (3.5% NaCl) is shown in Figure-8.

As shown in Fig.8, the surface of Al which exposed to EP with addition of lowest dosage of EG (10ml/l) showed the highest corrosion resistance where the corrosion potential shifted to the anodic direction more

than other samples with other doses of EG. Also, it showed the lowest current density comparing with other samples.

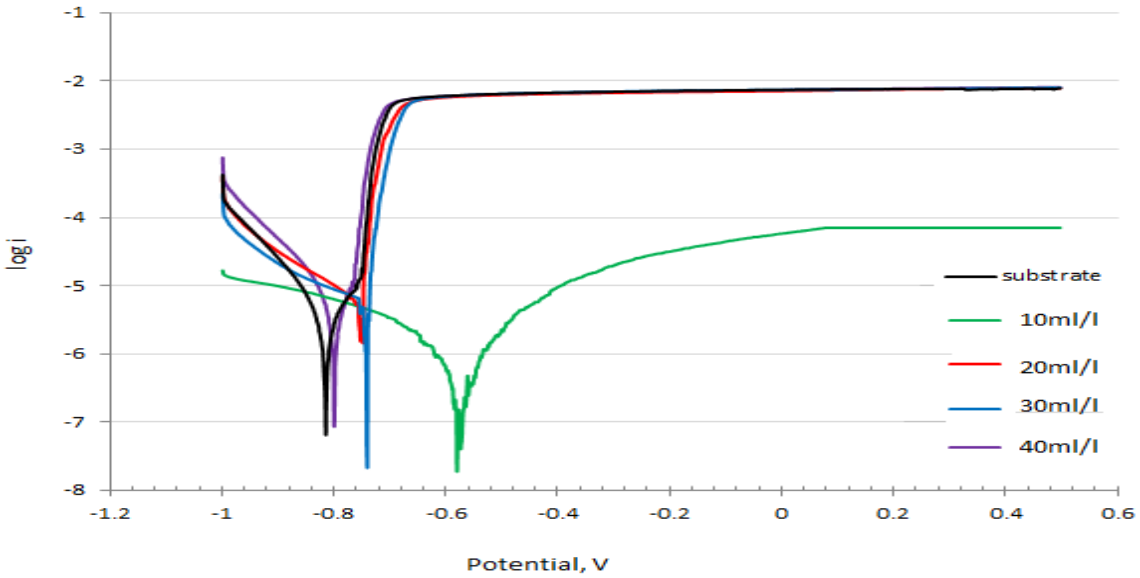



**Figure-8.** Linear polarization and corrosion resistance measurements of aluminum in 3.5 % NaCl solution after EP and addition of EG comparing with Al substrate.

The results of Tafel correlation are shown in Table-2, where the highest polarization resistance was achieved (36.707 KΩ) and lowest corrosion rate (0.01890 mm/year) and lowest current density ($J_{corr.}$ = 1.6266 µA/cm²).

So, the addition of EG with lowest dose 10ml/l enhanced the corrosion resistance and decreased the corrosion rate along time, so it could be recommended for increasing the life time of these components when they exposed to moisture, aggressive corrosion mediums or mechanical stress.

**Table-2.** Tafel parameters of EP of Al samples in 3.5 % NaCl at 25˚C.

| ml added of EG/l | $\beta_a$ (mV/dec) | $\beta_c$ (mV/dec) | $E_{corr.}$ (mV) | $J_{corr.}$ (µA/cm²) | Corrosion rate (mm/year) | Polarization resistance (KΩ ) |
|---|---|---|---|---|---|---|
| 10 | 367.67 | 219.6 | -572.3 | 1.6266 | 0.01890 | 36.7070 |
| 20 | 23.419 | 329.43 | - 742.33 | 3.7035 | 0.04303 | 5.5096 |
| 30 | 18.2060 | 212.060 | - 744.22 | 4.2134 | 0.04896 | 2.5640 |
| 40 | 117.82 | 165.410 | -815.96 | 5.4239 | 0.06302 | 1.7282 |

The mechanism of EP process illustrates that the protruded sharply micro humps are firstly decomposed during EP treatment than the recesses regions and cavities on the surface due to the gradient of electric potential and ionic migration from the bulk electrolytic solution to the surface of aluminum [22]. Accordingly, the surface is turned from sharply rough to swimming wavy surface.

So, post treatment is recommended by EP process by addition of EG to alleviate the surface roughness, where it slower the velocity of mass transfer of ions towards Al surface during EP process. This is essential to reduce pitting, where oxygen bubbles evolution is inevitably and this phenomenon could be avoided by addition of EG serving as an excellent coolant, where the viscosity was slightly increased and the exothermic heat formation was reduced. The presence of excess $OH^-$ groups of EG with negative charges in the electrolytic solution may reduce the evolution of oxygen adjacent to the surface of aluminum during EP treatment which leads to defects and pitting over the surface.

## CONCLUSIONS

Roughness of aluminum surface was alleviated after EP process in the electrolytic solution $H_3PO_4$, $H_2SO_4$ and $H_2O$ and more success treatment was achieved after addition of EG solution. The values of roughness $R_a$, $R_z$ and $R_{zjis}$ were decreased from 77.28, 1047 and 510.91 to 3.52, 108.62 and 44.91, respectively. SEM and AFM revealed the cease of nano/micro burrs and cavities and the

surface turned to highly smooth and bright, where gloss value (G) increased from 911 to 2968.

Also, aluminum surface showed enhancement of hardness from 49 to 53 Brennel HB and thickness were increased from 1 to 3 μm after EP and adition of 20ml EG/l. Optical characteristics of the surface showed the increase of reflectance after EP from R= 5-18% to R= 19-40% and more enhancement was achieved after addition of EG to R=20-60%.

Linear polarization of the surface revealed the increase of corrosion resistance to (36.707 KΩ) and decrease of corrosion rate and lowest current density 0.01890 mm/year and $J_{corr.}$= 1.6266 μA/cm², respectively after EP and addition of 10 EG ml/l. the results indicate the vital addition of EG during EP process.

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**REFERENCE**

[1] Wang Z., Qu R.T., Scudino S., Sun B.A., Prashanth K.G., Louzguine-Luzgin D.V., Chen M.W., Zhang Z.F., Eckert J. 2015. Hybrid nanostructured aluminum alloy with super-high strength. NPG Asia Mater. 7, e299.

[2] Li X., Li B., Zhang Q., Shi T., Yu J., Tang M., Huang X. 2016. Fabrication of micro- and nano-scale hierarchical structures on Al surface with enhanced wettability, anticorrosion and wear resistance. Mater. Express. 6, 10-18.

[3] Nascimento F.C., Paresque M.C.C., de Castro J.A., Jacome P.A.D., Garcia A., Ferreira I.L. 2015. Application of computational thermodynamics to the determination of thermophysical properties as a function of temperature for multicomponent Al based alloys. Thermochim. Acta. 619, 1-7.

[4] Loftis J.D., Abdel-Fattah T.M. 2016. Nanoscale electropolishing of high-purity silver with a deep eutectic solvent. Colloids Surf. A Physicochem. Eng. Asp. 511, 113-119.

[5] Abdel-Fattah T.M., Loftis D., Mahapatro A. 2015. Nanoscale Electrochemical Polishing and Preconditioning of Biometallic Nickel-Titanium Alloys. Nanosci. Nanotechnol. 5, 36-44.

[6] Taylor E.J., McCrabb H., Garich H., Hall T. and Inman M. 2011. A Pulse/Pulse Reverse Electrolytic Approach to Electropolishing and Through-Mask Electroetching. Faraday Technology.

[7] Bing Dua, Ian Ivar Suni. 2004. J. Electrochem. Soc. 151, 6.

[8] Vignal V., Roux J.C., Flandrois S., Fevrier A. 2000. Corros. Sci. 42, 1041.

[9] Sazou D. 1997. Electrochim. Acta. 42(4): 621.

[10] Wynick G.L., Boehlert C.J. 2005. Mater. Characterization. 55, 190.

[11] Abdel-Fattah Tarek M. and Loftis Derek J. 2020. Comparison of Electropolishing of Aluminum in a Deep Eutectic Medium and Acidic Electrolyte. Molecules, 25, 5712; doi: 10.3390/molecules25235712.

[12] Hou Y., Li R., Liang J., Su P., Ju P. 2018. Electropolishing of Al and Al alloys in AlCl3/ trimethylamine hydrochloride ionic liquid. Surf. Coat. Technol. 335, 72-79.

[13] Kityk A.A., Protsenko V.S., Danilov F.I., Kun O.V., Korniy S.A. 2019. Electropolishing of aluminum in a deep eutectic solvent. Surf. Coat. Technol. 375, 143-149.

[14] Di Maa, Shuying Li, Chenghao Liang. 2009. Electropolishing of high-purity aluminium in perchloric acid and ethanol solutions. Corrosion Science. 51, 713-718.

[15] Awad Abouelata A.M., Abdel Ghany N.A., Dahy T.M. 2010. Removal of tarnishing and roughness of copper surface by electropolishing treatment. Applied Surface Science. 256, 4370-4375.

[16] Ehsan Saeb Noori, Ali Navidinejad, Amin Rabiei Baboukani. 2016. Mechanism Study and Parameter Optimization of A356 Aluminum Alloy Electrochemical Polishing. Proceedings of Iran International Aluminum Conference (IIAC2016) May 11-12.

[17] Edson G.I. 1987. US 4,663,005.

[18] Mayer S.T., Contolini R.J., Bernhardt A.F. 1992. US 5, 096, 550.

[19] 1976. United States Patent. US3970529.

[20] 1987. European Patent. EP0249650A1.

[21] 2015. Chinese Patent. CN102747411B.

[22] Protsenko V.S., Butyrina T.E., Bobrova L.S., Korniy S.A., Danilov F.I. 2020. Enhancing corrosion resistance of nickel surface by electropolishing in a deep eutectic solvent. Mater. Lett., 270, 127719.