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# LOW VOLTAGE AC ANODIZING ON MAGNESIUM ALLOY IN SILICATE SOLUTION

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**Abstract:** The anodic film formed on magnesium alloy, AZ80 using alternating current (AC) power supply is studied and the effect of different anodizing parameters on the film formed is focused. Anodizing is carried out in different concentration and temperature of sodium silicate, $Na_2SiO_3$ solution for 15 minutes. The effects of concentration and temperature of $Na_2SiO_3$ on surface roughness and morphology of AZ80 are studied by using profilometer and scanning electron microscope (SEM); respectively. The anodizing parameters are focused on concentration and temperature of $Na_2SiO_3$. In order to analyse the phase composition of AZ80 before and after anodizing, x-ray diffraction (XRD) is used. As the concentration of $Na_2SiO_3$ increased, the surface roughness decreased and increased at 0.8 M. The surface roughness increased along with the increased temperature of $Na_2SiO_3$. The oxide film formed on the AZ80 consists of MgO and $Mg_2SiO_4$.

Keywords: AC-anodizing, magnesium AZ80, surface roughness.

## Introduction

Magnesium (Mg) alloys are a combination of Mg with other metals. Aluminum (Al), zinc (Zn), silicon (Si) and manganese (Mn) are metals that commonly used to form Mg alloys. Mg alloys are highly demanded in various industrial fields such as cellphone, automobile, and aerospace due to their advantageous properties (Echeverry-Rendon *et al.,* 2018). The excellent properties of Mg alloys are lightweight, high thermal conductivity, high dimensional stability and good machinability (Birss *et al.*, 2004). However, Mg alloys displayed major disadvantage properties which are poor corrosion resistance and cause a limitation to their widespread usage. One of the reasons for its poor corrosion is the internal galvanic corrosion by impurities which are iron (Fe), nickel (Ni), and copper (Cu) in Mg alloys (Salman *et al.*, 2010). Anodizing is one of the methods to overcome the poor corrosion resistance of Mg alloys.

Anodizing is an electrochemical process or surface treatment to produce a stable oxide film on the surface of the alloy (Blawert *et al.*, 2006). As for Mg anodizing, the oxide film formed has a porous and compact layer with a rough surface structure on the surface. The porous layer keeps growing while the compact layer becomes thinner as the anodizing process occur (Salman & Okido, 2013). Anodizing of Mg alloys being carried out either using alternating current (AC) or direct current (DC) as the power source with high anodizing voltage (Salman, 2015). There are several processes in anodizing of Mg alloys but the two most popular processes are DOW17 and HAE. Both processes produced an intensive spark on the anode forming ceramic-like films. However, the spark produced did not produce the current environmental protection regulations. Besides that, the electrolytes used in HAE and DOW17 processes are harmful chemicals such as fluoride, chromate, and phosphate (Qian *et al.*, 2008).

Within the past few years, researchers are had focused their interest in environmental friendly electrolytes anodizing of Mg alloys and changing various anodizing parameters such as the temperature of the electrolyte, anodizing voltage and anodizing time in order to produce a better corrosion resistance of the Mg alloys.

These parameters are affecting the properties of anodic films formed (Ono *et al*., 2017). This study is focused on low AC anodizing of AZ80 in silicate solution.

## Materials and Methods

### *Material and Chemical Preparation*

The material used is AZ80 Mg alloy (Al 7.5 wt.%, Zn 0.22 wt.%, Mn <0.51 wt.%, Si <0.01 wt.%, others <0.01 wt.% balance Mg). The AZ80 is cut to into rectangular shape with a size of 15 mm × 15 mm × 4 mm. The samples are mounted with epoxy resin and hardener to ensure only one surface exposed during the anodizing process. The samples are ground up to 1200 grit paper and polished with diamond paste. Then, the samples are degreased with acetone and cleaned with distilled water. The electrolyte used which is sodium silicate, $Na_2SiO_3$ is prepared by mixing the sodium silicate powder with deionized water.

### *Anodizing*

The experiment is prepared for a basic anodizing setup complete with an AC power supply and two electrodes. A platinum electrode is set as the cathode while the AZ80 sample functions as the anode. This study is focused on the concentration and temperature of $Na_2SiO_3$. The anodizing parameters details are listed in Table 1. The voltage is fixed to 15 V for 15 minutes and the anodizing current is recorded against each parameter. During the experimental process, $Na_2SiO_3$ is stirred by magnetic stirring to maintain a uniform distribution of solution concentration and temperature. The anodized AZ80 is then cleaned with distilled water.

### *Material Characterization*

The microstructure analysis of the AZ80 surface before and after AC-anodizing process is studied by observing the surface under scanning electron microscopy (SEM). Shimadzu XRD 6000 diffractometer is set at 2θ values of 20 ° to 80 ° with CuKα radiation to analyse the phase present before and after the AC-anodizing process. A 3D profilometer is used to measure the surface roughness of the anodic film formed before and after the anodizing process. The focused area for the surface roughness analysis is at the centre of the anodic film.

## Results and Discussion

### *Effect of Concentration and Temperature of $Na_2SiO_3$ on Current Density*

Figure 1 shows the graph of different anodizing parameters which are the concentration of $Na_2SiO_3$ and the temperature of $Na_2SiO_3$; respectively. From the graphs result, the current increased rapidly to a plateau for both parameters. As the concentration of $Na_2SiO_3$ solution increased, the current density also increased. This is due to the content of reactant which is $Na_2SiO_3$ and the conductivity of concentrated electrolyte (Sharma *et al.,* 1996). With regards to the temperature of $Na_2SiO_3$, the increase of current density as the temperature is increased is due to the heat energy produced during the anodizing process (Aerts *et al.*, 2007).

Table 1 : The anodizing parameter used in the experiment

| **Concentration of $Na_2SiO_3$ (M)** | **Temperature of $Na_2SiO_3$ (ºC)** |
|---|---|
| 0.2 | 5 |
| 0.4 | 15 |
| 0.6 | 25 |
| 0.8 | 35 |
| 1.0 | 45 |

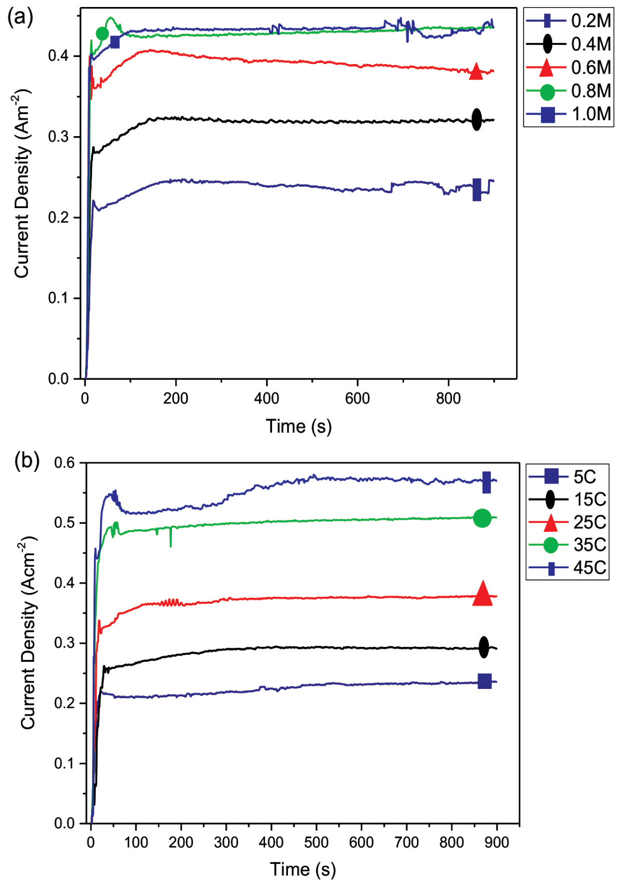


Figure 1 : Current density versus anodizing time different anodizing parameters (a) concentration of $Na_2SiO_3$ and (b) temperature of $Na_2SiO_3$.

***Surface Morphologies of Anodic Film***

The surface morphologies of anodized AZ80 with different anodizing parameters are shown in Figure 2 and Figure 3. For the concentration of $Na_2SiO_3$ parameter, the anodic film formed on the surface of AZ80 at 0.2 M and 0.4 M in Figure 2(a) and Figure 2(b) are lightly scattered with flakes and pores. The pore existence increased as the concentration of $Na_2SiO_3$ increased at 0.6 M, 0.8 M to 1.0 M as shown in Figure 2(c), Figure 2(d) and Figure 2(e). This indicates that the anodic film is started to complete its anodizing coating process (Li *et al.*, 2013).

Figure 3 shows the surface morphologies of anodized AZ80 for the different temperature of $Na_2SiO_3$ parameters. As the temperature increase, the surface of anodic film is rougher and less uniform. From Figure 3(a) and Figure 3(b), which are at 5 ºC and 15 ºC; respectively, the anodic film surface is smooth and pores are barely detected compare to anodic film in Figure 3(c), Figure 3(d) and Figure 3(e) which the temperature are 25 ºC, 35 ºC and 45 ºC; respectively. The reason for this to occur is because the particles in the electrolyte received enough heat energy from the increase of temperature to complete the oxide film formation (Hassanzadeh *et al.*, 2013).

***Phase Composition***

The analysis for phase composition of AZ80 before and after the anodizing process by XRD are shown in Figure 4(a) and Figure 4(b);

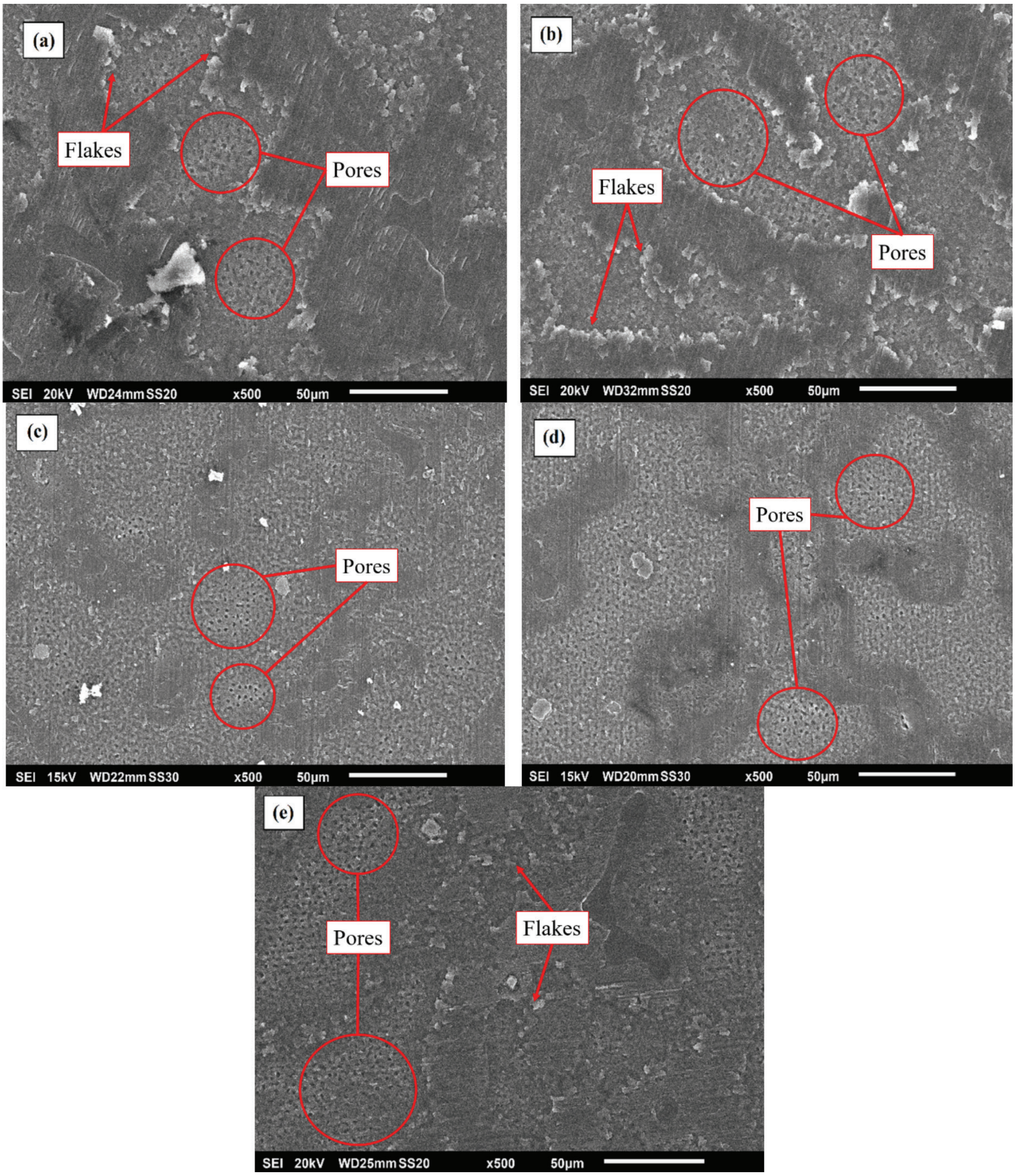


Figure 2 : Surface morphologies of anodic film after anodizing process with concentration $Na_2SiO_3$ of (a) 0.2 M; (b) 0.4 M; (c) 0.6 M; (d) 0.8 M and (e) 1.0 M for 15 minutes.

respectively. The phase composition of AZ80 before anodizing process is mainly composed of Mg and $Al_3Mg_2$. The anodized AZ80 film is composed of Mg, $Al_3Mg_2$, $Mg_2SiO_4$, $SiO_2$, and MgO. The Si element is the component of the electrolyte which is $Na_2SiO_3$ and the Al is the component of the AZ80. This indicated that these components have participated in the anodic film reaction. There are two reactions that occurred at both electrodes which are an anodic reaction (1), and cathodic reaction (3) (Jamil *et al.*, 2018).

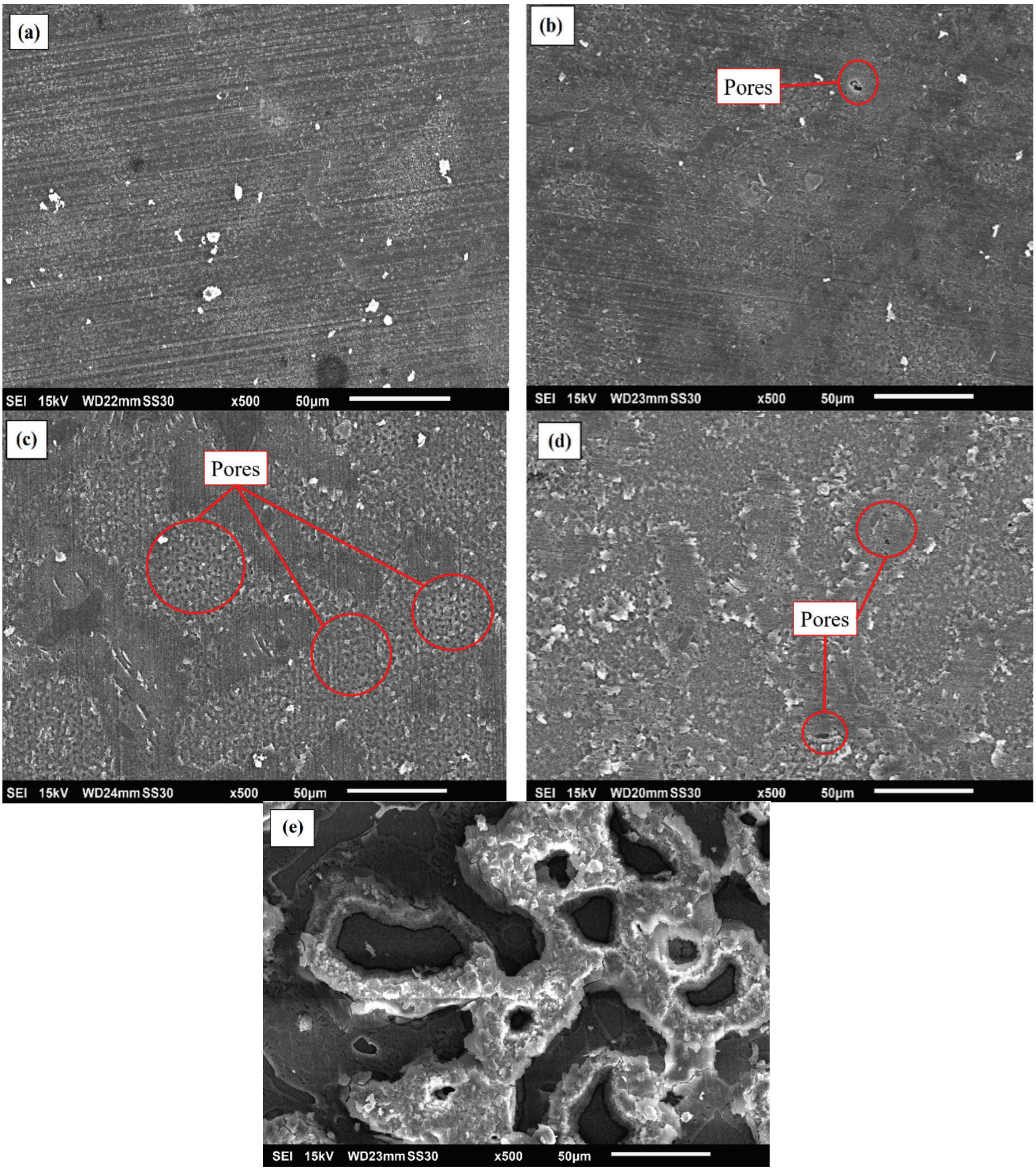


Figure 3 : Surface morphologies of anodic film after anodizing process with $Na_2SiO_3$ temperature of (a) 5 ºC; (b) 15 ºC; (c) 25 ºC; (d) 35 ºC and (e) 45 ºC for 15 minutes.

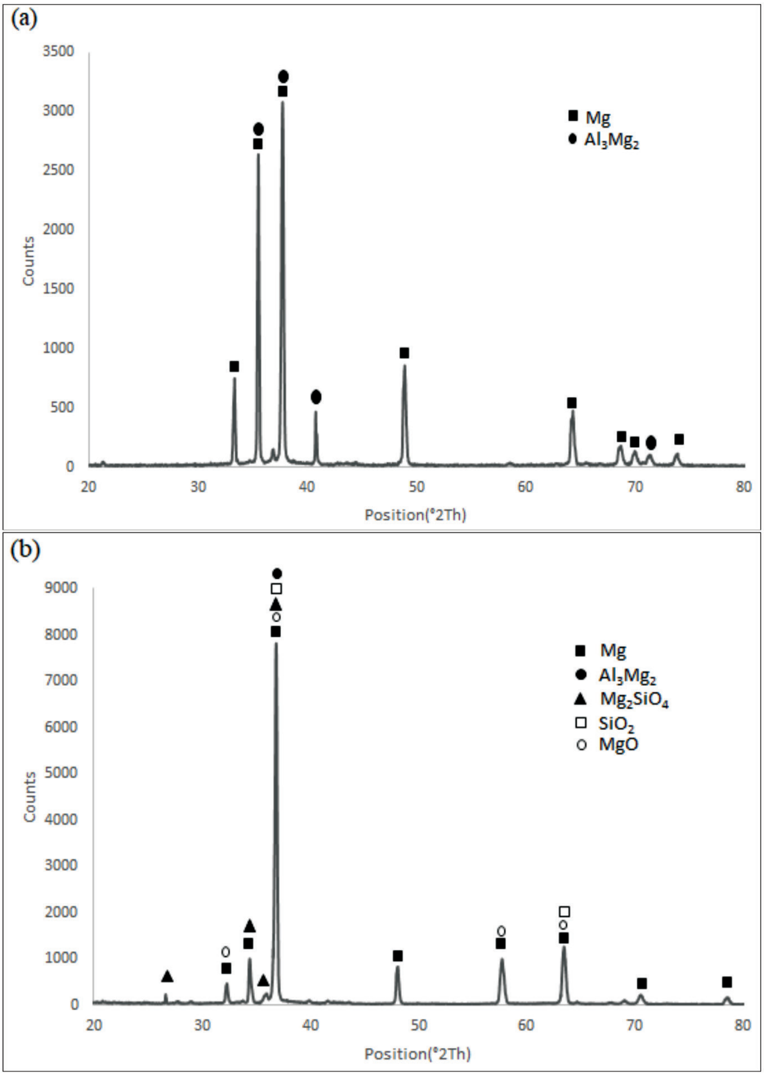


Figure 4 : The XRD composition of AZ80 (a) before the anodizing process and (b) after the anodizing process.

$$Mg = Mg^{2+} + 2e \quad (1)$$

$$Mg^{2+} + 2OH = Mg(OH)_2 = MgO + H_2O. \quad (2)$$

$$4OH= 2H_2O + O_2 + 4e \quad (3)$$

$$2Mg+O_2= 2MgO \quad (4)$$

$$SiO_2 + 2MgO = Mg_2SiO_4 \quad (5)$$

***Surface Roughness***

The roughness of surface film after the anodizing process of different anodizing parameters are determined using 3D profilometer and the result are shown in Table 2 and the data are plotted in Figure 5(a) for the concentration of $Na_2SiO_3$ and Figure 5(b) for the temperature of $Na_2SiO_3$. For the concentration of $Na_2SiO_3$ parameter, the surface roughness decreased as the concentration increased, however, at 1.0 M, the roughness showed some increase. This is due to the flakes formed on the anodic film at 1.0M, the surface becomes porous and this can be supported by the surface morphologies

Table 2 : Surface roughness of AZ80 after the anodizing process with different concentration of $Na_2SiO_3$.

| **Anodizing Parameters** | | **Surface Roughness, Ra (nm)** |
|---|---|---|
| Concentration of $Na_2SiO_3$ (M) | 0.2 | 222.98 |
| | 0.4 | 196.85 |
| | 0.6 | 197.45 |
| | 0.8 | 149.45 |
| | 1.0 | 438.05 |
| Temperature of $Na_2SiO_3$ (ºC) | 5 | 114.49 |
| | 15 | 210.82 |
| | 25 | 385.53 |
| | 35 | 861.39 |
| | 45 | 4743.46 |

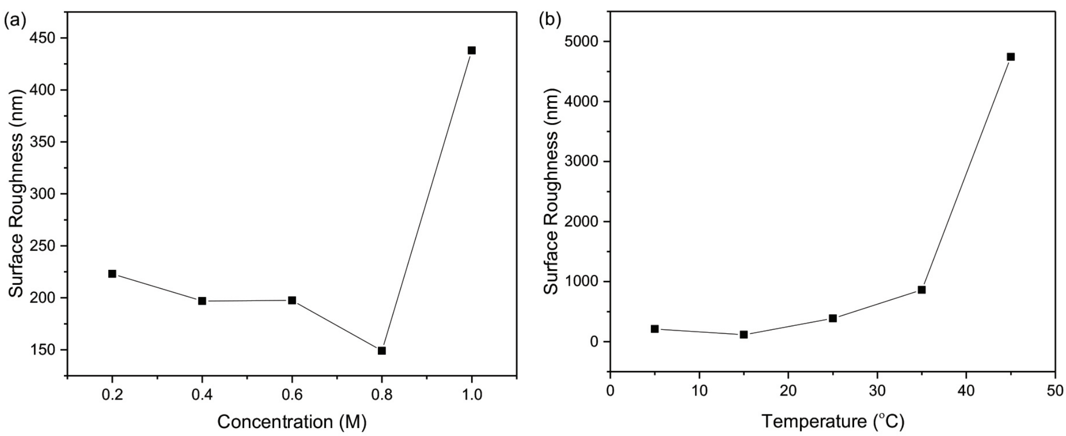


Figure 5 : Surface roughness of AZ80 after the anodizing process against different (a) concentration of $Na_2SiO_3$ and (b) temperature of $Na_2SiO_3$.

in Figure 2. As for the temperature of $Na_2SiO_3$ parameter, the surface roughness increased as the temperature increased. This indicated that the anodic film formed is become more porous as in Figure 3 (Darband *et al.,* 2017).

## Conclusion

The current density graph indicates that as the anodizing time increased, the current density also increased with the increase of concentration and temperature of $Na_2SiO_3$. The surfaces of the anodized AZ80 films are porous in order the complete the anodizing coating process. The anodic film formed after the anodizing process is composed of Mg, $Al_3Mg_2$, $Mg_2SiO_4$, $SiO_2$ and MgO. The surface roughness of AZ80 surface is decreased as the electrolyte $Na_2SiO_3$ become more concentrated but the surface roughness is increased as the temperature of $Na_2SiO_3$ increased.

*Abbreviations: HAE. DOW17, AZ80*

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