THICKNESS AND CORROSION RESISTANCE OPTIMIZATION OF MICRO-ARC OXIDATION COATING ON Mg-Al-Li-Zn ALLOY USING TAGUCHI APPROACH

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Abstract - Microarc oxidation method has been developed in the field of surface protection of magnesium alloys and considered as a simple, highly effective, commercial and environmentally friendly method in industry. MAO coatings are fabricated on novel Mg-Al-Li-Zn alloy to improve the anti-corrosion performance of surface by using friendly alkaline electrolytes under a high electrical potential. The Taguchi method and optimal analysis are used to identify the effects of the three factors including current density, processing time and electrical frequency on coating's characteristics. The results show that the main factor that affects coating thickness and corrosion resistance of coating is the processing time. The results obtained by optimal conditions are consistent with prediction values of Taguchi analysis. The thickness of the coating can help to improve the long-term corrosion protection of a MAO coating in corrosive environments.

Key words - Micro-Arc Oxidation; Mg-Al-Li-Zn alloy; Taguchi method; corrosion resistance

1. Introduction

The weight reduction requirement of aerospace and transportation industries drive the development and application of lightweight materials to an increased demand. Magnesium lithium alloys have always been attractive to designers due to their low density and high specific strength [1, 2]. However, poor corrosion resistance of magnesium lithium alloys restrict their wide application in corrosive environments. The addition of aluminum helps to increase the mechanical and chemical stability of magnesium lithium alloys. Magnesium aluminum alloys are generally perceived to be materials stable in corrosive media and resistant to corrosion under natural conditions. With the difference in minor additives in the magnesium lithium system, the corrosion behaviors are different [3]. Magnesium lithium alloys corrode rapidly in corrosive environments, causing severe attacks on the surface resulting in a decrease in their mechanical integrity and creating an unattractive appearance [4-6]. Therefore, a further requirement for anti-corrosion performance developments has been demanded for magnesium lithium alloys.

The MAO process is a relatively new technique which is basically electrochemical process of oxidation of metal in an aqueous alkaline electrolyte. It has received wide spread acceptance as an eco-friendly process for modifying the surface of valve metals [7-9]. This process is operated at high voltage, which generates plasma and spark discharge accompanied by the release of gas on the metal surface. The structure, element and phase composition of the coating depend on the electrolytic compositions [10-13]. The thickness, surface morphology and anti-corrosion behavior of MAO coatings mainly depend on key process factors: current density, processing duration, frequency, etc. [14-19]. P. Bala Srinivasan et al. [20] presented the idea that the properties of MAO coatings are affected by the applied current density. MAO coatings fabricated by a low current density are thin, smooth and uniform. Yue et al. [19] studied the anti-corrosion behavior of MAO coating on magnesium alloys at various current densities. MAO coating fabricated by higher current density created better corrosion resistance due to it becoming more compact and smoother. Current density and processing time exhibited similar effects on the properties of MAO coatings [21]. At the same current density, prolonged oxidation time will increase the anti-corrosion behavior of the MAO coatings. When the oxidation time exceeds an optimal value the anti-corrosion behavior decreases. With the long duration of the coating process, it is possible that the coating formed on the sample may dissolve and be completely destroyed, and the corrosion resistance of the sample decreased significantly [22]. Applied electrical frequency is a significant factor that affects morphology and the quality of MAO coatings. Lv et al. [23] demonstrated that the surface morphology of coatings produced at a low frequency is rougher and has larger pore sizes than that produced at higher frequencies. However, longer discharge events result in high local temperatures, which creates more molten material in the discharge channels. Hence, the coatings fabricated at a low frequency have a greater growth rate. Su et al. [24] research showed that MAO coatings created at a high frequency had higher corrosion resistance compared with those fabricated at a low frequency.

Quite a few investigations have been conducted to identify the optimal parameters for the creation of MAO coatings. Hence, in this work, the Taguchi design of experimentation is used to optimize the process parameters in producing MAO coatings on Mg-Al-Li-Zn substrate. The corrosion resistance of coatings is primarily determined by several key factors such as current density, processing time and electrical frequency. The statistical analysis software Minitab 17 is used for the design and analysis of the experiments to derive optimal levels. The confirmation runs are carried out to compare with predicted results.

2. Experimental investigation

2.1. Preparation of MAO coatings

In this study, Mg-Al-Li-Zn alloy samples were used as substrates. The samples (33 x 75 x 5 mm) were polished using Silicon Carbide Grinding paper up to 1200 grit; then they were degreased ultrasonically in DI water for 10 min, rinsed and dried.

The MAO treatment was conducted in a stainless-steel water-cooled bath, which served as the cathode. The cooling system was used to keep the temperature of the
MAO bath at ambient temperature. The Mg-Al-Li-Zn alloy specimens were used as anodes. The electrolyte was prepared similar to that of the previous study [25]. Finally, the MAO samples were rinsed with DI water and dried.

2.2. Characterization of MAO coating

In this study, average coating thickness \( t_\text{av} \) and corrosion resistance \( R_p \) were utilized for surface characteristics.

The thicknesses of the samples were measured by Fischer dual-scope MP20 for non-ferrous materials. The average thickness was obtained by averaging the nine measurements on the surface.

The 1285 PotentiostatSolartron was used to perform the potentiodynamic polarization to evaluate the corrosion resistance of MAO coating samples. All measurements were carried out in a 3.5 wt% Sodium chloride, using a conventional three-electrode cell with 1 cm\(^2\) exposed area of substrate act as the working electrode. The silver/silver chloride electrode was used as the reference electrode and Platinum coil was acted as the counter electrode.

The Stern-Geary equation (1) [26] was used to calculate the corrosion resistance of coating.

\[
R_p = \frac{\beta_a \beta_c}{2.3 l_{\text{corr}}(\beta_a + \beta_c)}
\]  

(1)

Where:
- \( R_p \) - corrosion resistance of coating (kΩ cm\(^2\));
- \( l_{\text{corr}} \) - corrosion current density (A/cm\(^2\));
- \( \beta_a \) constants of the anodic Tafel slopes;
- \( \beta_c \) - constants of the cathodic Tafel slopes.

2.3. Taguchi design of experiments

Table 1. Design factors and levels of MAO coating

| Factors          | Unit       | Factor levels |
|------------------|------------|---------------|
| (A) Current density | (A/dm\(^2\)) | 3 4 5        |
| (B) Processing time | (mins)     | 5 10 15      |
| (C) Electrical frequency | (Hz)      | 500 1000 2000 |

| Table 2. Experimental design matrix L9 orthogonal array |
|--------------------------------------------------------|
| Exp no. | Current density (A/dm\(^2\)) | Time (min) | Frequency (Hz) |
|---------|-------------------------------|------------|---------------|
| 1       | 3                             | 5          | 500           |
| 2       | 3                             | 10         | 1000          |
| 3       | 3                             | 15         | 2000          |
| 4       | 4                             | 5          | 1000          |
| 5       | 4                             | 10         | 2000          |
| 6       | 4                             | 15         | 500           |
| 7       | 5                             | 5          | 2000          |
| 8       | 5                             | 10         | 500           |
| 9       | 5                             | 15         | 1000          |

3. Taguchi analysis of the S/N ratio

Table 2 showed the parameters of nine experiments design by Taguchi method. The results of the average coating thickness and corrosion resistance for coatings on Mg-Al-Li-Zn alloy substrate are given in Table 3.

| Table 3. Responses of thickness and corrosion resistance of MAO coatings |
|---------------------------------------------------------------|
| Exp no. | Test 1 | Test 2 | Test 1 | Test 2 |
|---------|--------|--------|--------|--------|
| t (µm)  |        |        |        |        |
| 1       | 6.9 ± 0.5 | 7.1 ± 0.5 | 330.659 | 393.571 |
| 2       | 13.2 ± 0.5 | 13.9 ± 0.5 | 258.191 | 231.468 |
| 3       | 12.8 ± 0.9 | 13.0 ± 0.5 | 94.098  | 104.760 |
| 4       | 10.6 ± 0.5 | 11.3 ± 0.72 | 391.369 | 311.384 |
| 5       | 16.7 ± 0.74 | 17.7 ± 0.91 | 153.314 | 152.988 |
| 6       | 26.2 ± 0.86 | 25.8 ± 1.00 | 115.479 | 137.023 |
| 7       | 10.0 ± 0.55 | 10.7 ± 0.65 | 378.925 | 376.061 |
| 8       | 20.9 ± 1.20 | 22.2 ± 1.30 | 194.063 | 195.448 |
| 9       | 26.4 ± 1.10 | 27.2 ± 0.97 | 192.044 | 138.991 |

3.1. Coating thickness

Based on Equation (2), the average coating thickness in two trials was analyzed by the statistical software package, Minitab and made into a S/N ratio. The mean S/N ratios with the average thickness values and level numbers are compared in Table 4. Figure 1 shows the effect of the three factors, i.e. current density, processing time and electrical frequency, on the mean S/N ratios. It can be observed that the mean S/N ratio goes up as the current density and time increase.

The observation of the response of the S/N ratio to current density was made because of the properties of spark discharge. It was observed that increasing the current density resulted in the creation of more intense sparks during the MAO coating that led to an increase in the coating thickness. Processing time (delta = 7.04) has the most significant effect on coating thickness. The prolonged processing time will increase the MAO coating thickness. Moreover, the electrical frequency also affected the increase of thickness of MAO coating. Decreasing the electrical frequency generates intense sparking and creates thicker MAO coatings. From above results, the optimized MAO coating thickness can be fabricated under 5 A/dm\(^2\) of current density, 15 min of processing time and 500 Hz of electrical frequency.
The S/N ratio encodes the S/N ratio value.

Figure 1. Plot of main effects for signal-to-noise ratios vs. factor levels of coating thickness

3.2. Corrosion behavior

The “larger is better” S/N ratios was selected to find the optimum conditions for corrosion resistance. Hence, the Equation (2) was used for analysis the signal-to-noise ratios in this situation. Table 5 presented the S/N ratio values for all responses. By examining the plot of the main effects for S/N ratios, shown in Figure 2, it can be concluded that the processing time (delta = 9.16) expresses greatest effect on corrosion resistance, followed by electrical frequency (delta = 2.46) and current density (delta = 1.66). The mean S/N ratio decreases at first, as the current density increases from 3A/dm² to 4 A/dm². The S/N ratio then increases further, to a maximum value as the current density reaches 5 A/dm².

Table 4. Response table for S/N ratios for average coating thickness

| Level | Current density | Processing time | Electrical frequency |
|-------|-----------------|-----------------|---------------------|
| 1     | 20.58           | 19.32           | 23.95               |
| 2     | 24.59           | 24.66           | 23.99               |
| 3     | 25.17           | 26.36           | 22.40               |
| Delta | 4.59            | 7.04            | 1.59                |
| Rank  | 2               | 1               | 3                   |

Table 5. Response table for S/N ratios for corrosion resistance

| Level | Current density | Processing time | Electrical frequency |
|-------|-----------------|-----------------|---------------------|
| 1     | 46.24           | 51.12           | 46.27               |
| 2     | 45.46           | 45.74           | 47.51               |
| 3     | 47.12           | 41.96           | 45.05               |
| Delta | 1.66            | 9.16            | 2.46                |
| Rank  | 3               | 1               | 2                   |

The observation on the response of the S/N ratio to corrosion resistance is dependent on the surface morphology, porosity and coating thickness of MAO coatings. Mu and Han [29] researched the effect of the current density on coating thickness and surface morphology. When the applied current density is low, the uniform distribution of spark discharge was obtained on the surface leading to the fabrication of a coating that is thin and smooth. When a current density becomes high, the intense spark discharge appears, leading to the formation of a porous outer layer of coating. The thickness and adhesion of MAO coating improve with high current density, which makes the reactivity between the magnesium alloy substrate and oxygen increase.

Lv et al. [30] discussed that the thickness, morphological features and corrosion resistance of coatings are greatly influenced by the processing time, one of the important factors in MAO coating process. With a short processing time, the weak sparks uniform distribution and move quickly on the substrate. This produces high density and uniform small-scale pores that spread uniformly over the MAO coating surface. When the processing time exceeds the optimal value, the spark discharges become more energetic, more violent and move slowly over the substrate. Thus, resulting in the surface microstructure of the MAO coating is porous, rough and non-uniform.

The S/N ratios value of the electrical frequency increases until it reaches a peak at 1000 Hz of electrical frequency. Thereafter, this value decreases with the electrical frequency increased. A compact and dense MAO coating was fabricated at a suitable electrical frequency. Thus, the anti-corrosion behavior of MAO coating significantly improved. The variation in discharge characteristics is considered responsible for the observed difference in the extent of porosity and cracks as a function of electrical frequency. Moreover, the electrical frequency, which affects coating thickness, plays a significant role in improving anti-corrosion performance of MAO coating. The MAO coating with optimized corrosion resistance can be produced at level 3 of current density (5 A/dm²), level 1 of processing time (5 min) and level 2 of electrical frequency at (1000 Hz).

4. Confirmation tests and discussions

The confirmation tests are the final step in verifying the conclusions from the optimized experimentation. The experimental parameters of the optimal coating thickness and corrosion resistance are shown in Table 6.
and corrosion resistance conditions, respectively. A low coating fabricated under the optimized coating thickness to 4.16×10
7.36×10
coatings is reduced by two to three orders of magnitude be clearly seen that the corrosion current density of MAO from the Stern relevant polar
(Ea), are shown in Figure 3 and Table 8, respectively. The optimal polarization curves of (O) the untreated and MAO treated Mg-Al-Li-Zn alloy at optimized coating thickness and corrosion resistance conditions
4.1. Optimal coating thickness
Table 7 shows the predictions and thicknesses of the coatings fabricated under the optimized conditions of coating thickness and corrosion resistance. The thickness produced on Mg-Al-Li-Zn alloy substrate from the optimized coating thickness parameters is 28.2 ± 1.20 µm. According to the Taguchi prediction method, the optimized average coating thickness was calculated to be 27.2 µm. This result is coincided with the value of confirmation test. The thickest coating can be produced by applied high current density, prolong action of processing time and low electrical frequency. The coating fabricated under optimized corrosion resistance condition has thickness about 14.0 ± 0.80 µm, which is approximately with the result of calculated by Taguchi prediction (13.6 µm). The small difference with the Taguchi prediction method is in confirming agreement with statistical analysis. The results have indicated that processing time probably creates an advantage on the formation of the coating thickness.

Table 7. The coating thickness, and, corrosion resistance of prediction and confirmation runs of MAO treated Mg-Al-Li-Zn alloy under optimal conditions

| Sample | Coating thickness, t (µm) | Corrosion resistance, Rp (kΩ·cm²) |
|--------|--------------------------|----------------------------------|
|        | Prediction | Confirmation | Prediction | Confirmation |
| A      | 27.2       | 28.2 ± 1.20 | 142.94     | 163.05      |
| B      | 13.6       | 14.0 ± 0.80 | 402.40     | 440.68      |

4.2. Electrochemical corrosion behavior of the optimal coatings
The corrosion behavior of the untreated Mg-Al-Li-Zn alloy substrate and the MAO coatings achieved under various treatment conditions was estimated by a potentiodynamic polarization investigation using 3.5% NaCl. The obtained polarization curves with the following parameters electrochemical parameters including the constants of the anodic (βa) and cathodic (βc) Tafel slopes, corrosion current density (i_corr) and corrosion potential (E_corr), are shown in Figure 3 and Table 8, respectively. The relevant polarization resistance (Rp) values calculated from the Stern-Geary equation are listed in Table 8. It can be clearly seen that the corrosion current density of MAO coatings is reduced by two to three orders of magnitude more than the bare Mg-Al-Li-Zn alloy. In detail, it is shown that the corrosion current density decreases from 7.36×10⁻⁵ A/cm² for the bare Mg-Al-Li-Zn alloy substrate to 4.16×10⁻⁷A/cm², and 8.27×10⁻⁸ A/cm² for the MAO coating fabricated under the optimized coating thickness and corrosion resistance conditions, respectively. A low corrosion current density indicates superior corrosion protection properties for the MAO coating.

Table 8. Electrochemical corrosion behavior of bare-substrate and MAO treated Mg-Al-Li-Zn alloy at optimized coating thickness and corrosion resistance conditions

| Samples                  | βa   | βc   | i_corr (A/cm²) | E_corr (kΩ·cm²) | Rp (V) |
|--------------------------|------|------|----------------|-----------------|-------|
| (O) Untreated Mg-Al-Li-Zn alloy | 42.00 | 354.66 | 7.36×10⁻⁵ | -1.58 | 0.22 |
| (A) Optimized coating thickness | 214.91 | 567.58 | 4.16×10⁻⁷ | -1.58 | 163.05 |
| (B) Optimized corrosion resistance | 165.45 | 170.04 | 8.27×10⁻⁸ | -1.60 | 440.68 |

As shown in Table 8, the corrosion resistance of the coating fabricated at 5 A/dm², 5 min and 1000 Hz are 440.68 kΩ·cm², which was the optimized value achieved in the current research and approximately to the value predicted by Taguchi method (402.40 kΩ·cm²). The corrosion resistance of the coating produced at optimized conditions is about 2000 times higher than that of the bare substrate (0.22 kΩ·cm²). It is known that corrosion resistance is related to the structure and morphology of the coating. The improvement of the corrosion resistance of MAO coating results from more perfect, homogeneous morphology and a compact layer.

Figure 3. Polarization curves of (O) the untreated and MAO treated Mg-Al-Li-Zn alloy at optimized (A) coating thickness and (B) corrosion resistance conditions

5. Conclusions
The Taguchi method for the design experiments has been assigned to optimize the coating thickness, thickness coating uniformity and corrosion resistance of the MAO coating process on Mg-Al-Li-Zn alloy. The effects of current density, coating time and electrical frequency have been studied and the results show that the processing time is the major factor affecting MAO coating characteristics.

The optimized MAO coating thickness can be produced under 5 A/dm² of current density, 15 min of processing time and 500 Hz of electrical frequency. The optimal coating thickness is 28.2 ± 1.20 µm, which is approximate
to the value estimated by Taguchi method (27.2 µm). The optimized processing conditions, which is set for fabricating the coating with the best corrosion protection, were set at 5 A/dm² for 5 min at 1000 Hz in order. The thickness of the coating was about 14.0 ± 0.80 µm which is close with the predicted value (13.6 µm).

The anti-corrosion performance of the MAO coating improved greatly compared with the untreated Mg-Al-Li-Zn alloy substrate. The corrosion resistance of the optimized coating is more than 2000 times better than that of the bare Mg-Al-Li-Zn alloy substrate.

The Taguchi design experiment proved to be a robust and effective method to design and control the thickness, thickness coating uniformity and corrosion resistance of the MAO coating process. The results obtained in confirmation runs were consistent with the Taguchi prediction values.

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