Effect of micro-arc oxidation time on coating formation of AZ91 magnesium alloy in silicate electrolyte

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Abstract. The micro-arc oxidation behavior of AZ91 magnesium alloy in silicate alkaline electrolyte was studied. The effects of micro-arc oxidation time on film formation and microstructure were investigated and characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), emission spectrometry (ICP) and salt spray test. The results show that oxidation time has an important effect on the surface morphology, thickness, phase structure and corrosion resistance of micro-arc oxidation film. With the increase of treatment time, the surface roughness and pore size increased. The acid ion (SiO$_3^{2-}$) in electrolyte plays an important role in the formation of phase in the early stage of micro-arc oxidation, and the formation rate of MgO is dominant in the later stage. The corrosion rate decreases first and then increases with the increase of treatment time, and the corrosion rate of the sample processing 30min is lower under our technological conditions.

1. Introduction

The content of magnesium in the earth's crust is about 2.35%, and in the sea water is about 0.13%. It is one of the most abundant light metal elements on the earth, which is the sixth metal element after aluminum, iron, calcium, sodium and potassium. Magnesium alloy is the least density metal structure material used in engineering up to now. It has the characteristics of high specific strength and specific stiffness, good damping and shock absorption, good casting performance and easy to recycle, etc. Magnesium alloy is widely used in electronics, automobiles, aerospace and various components of consumer goods. They are regarded as "green engineering materials with the most potential for development and application in the 21st century" [1-3]. Especially with the world energy crisis, environmental pollution and other problems are becoming more and more serious, energy saving and lightweight is a powerful thrust of magnesium alloy development. However, due to the extremely low potential of Mg standard electrode ($E_0 = -2.37V$), the corrosion resistance of magnesium alloy is poor, which can not meet the requirement of engineering application. In order to improve the corrosion resistance of magnesium alloys, coating treatment is an important technical approach. At present, there are many methods to prepare coatings on magnesium alloys, such as anodizing, electroplating, chemical deposition, conversion films and so on [4-7]. Ceramic coating can be prepared on the surface of magnesium alloy by micro-arc oxidation in situ. Compared with anodic oxidation and other coating treatment technology, the MAO technology has many technical advantages, such as simple process, better coating performance, and alkaline electrolyte which belongs to the environment-friendly type and has lower environmental pressure. At present, the micro-arc oxidation technology of magnesium alloy has been studied extensively and deeply at home and abroad. The research contents include the
mechanism of micro-arc oxidation, electrolyte formula, electric parameters, oxidation process and so on[8-15]. However, so far, the development of technology is not mature, there are only a few industrial applications in the world, some key technical problems still need to be solved[16-18]. Although there are some reports on the effect of micro-arc oxidation treatment time on the film formation, due to the different technology and different research angles and emphases, the results are also very different[19-20]. In this paper, the effects of oxidation treatment time on film formation are discussed from the aspects of ion diffusion, phase structure, epitaxy and internal growth, which provide experimental and theoretical support for this technology.

2. Materials and experimental procedures

The experimental material was AZ91 magnesium alloy, and its chemical composition(wt.%) shows down: Al: 8.56, Mn: 0.82, Zn: 0.41, Mg: Bal. The specimen was rectangular solid(50mm×30mm×4mm). The size of each part of the sample is shown in Fig.1. H and L were measured by Leica DMIRM/DFC 320 metallographic microscopy, h was measured by eddy current coating thickness gauge (which style is phynix made in Germany). The calculation of a, b is as follow: $a=L-H$, $b=h-a$. The specimen treat procedure followed: magnesium ingot→ cutting→ drilling→ rough grinding→ fine grinding→ polishing→ cleaning→ degreasing→ micro-arc oxidizing→ testing. The power supply style of micro-arc oxidizing was MOP-60 which researched by ourselves. Its power was 42KVA. Electrolytic solution was alkaline solution that its major composition was: Na2SiO3: 5-30g/L, NaOH: 1-5g/L, KF: 5-8g/L, Na3C6H5O7: 0.5-2g/L, EDTA: 0.5-2g/L. The whole reaction process was controlled by constant current, current density was 20A/dm², the reaction temperature was 20-40℃, the oxidizing time was 25s-30min.

The surface morphology of the micro-arc oxide film was observed on Leo 1530V with EDS analysis. The phase structures of the film determined with a X-ray diffractometer(XRD, X-pert MPD, Philips company, made in Holland). The film thickness was measured with an eddy current coating thickness meter(phynix, made in Germany). The corrosion resistance of the film was evaluated by neutral salt spray test. The test was carried out according to the national standard (GB/T10125-1997, artificial atmosphere corrosion Test — Salt spray Test), the machine style was FQYO/0A(produced by Shanghai Experimental instrument General Factory in China), the corrosion time was 36 hours. The surface roughness of the micro-arc oxidizing film was measured using surface profilometer(BMT expert3D, BMT Company, made in Germany). The change of Mg and Al element content in electrolyte was measured by ICP-AES Analyzer (Prodigy ICP-AES, LEEMAN-LABS INC, made in America). The measurement of surface porosity and pore size of the coating was analyzed using image analysis processing software ImageJ1.8.

![Figure 1. Schematic diagram showing the coating growth pattern during MAO processes](image)

3. Results and discussion

3.1 Effect of the processing time on morphology of MAO coatings

The effect of oxidation time on the surface morphology of the film was studied in Fig.2. It can be seen from Fig.2a that a large number of micropores appear on the surface of the sample, which is caused by microarcs. At the same time, there are some areas on the surface of the sample that are not
occupied by micropores. According to the characteristics of micro-arc oxidation technology, the prerequisite for the appearance of micro-arc oxidation is to form an anodic passivation film on the surface of the sample\cite{21-22}. The area not occupied by micropores is anodic passivation film, which is easily broken down by microarcs and forms a micro-arc oxide film. The establishment of anodic passivating film corresponds to the incubation period of micro-arc oxidation, which is usually shorter, from a few seconds to tens of seconds, in our technological conditions, this incubation period is about 15s.

After the incubation period, the sample begins to enter the phase of micro-arc oxidation to form film. When the oxidation time reaches 80s (Fig.2b), the surface of the sample has formed a complete oxide film. When the oxidation time is short, such as less than 360s (Fig.2b-d), the film surface is smooth and even. When the treatment time is more than 1800s (Fig.2e-f), the surface roughness of the film increases, and many small particles are adsorbed on the surface of the sample. The appearance of these small particles should be due to the later period of micro-arc oxidation, the thickening of the film layer, the elongated discharge channel, the more oxides ejected each time, some of which have no time to stack and enter the electrolyte. The oxide particles entering the electrolyte are readorsorbed on the surface of the sample under the action of electric field. It is also possible that in the late stage of micro-arc oxidation the film is destroyed due to the appearance of large arc spot and some oxides remelt into the electrolyte and then adsorb on the surface of the sample under the action of electric field. It may also be due to a solution equilibrium between the film and electrolyte under the action of heat and electrochemistry.

Figure 2. Effect of the processing time on morphology of MAO coatings (a)25s; (b)80s; (c)100s; (d)190s; (e)360s; (f)1800s; (g)3600s
There are two types of pore structure on the surface of the micro-arc oxide film, one is a small and compact circular structure (as indicated by arrow A in Fig.2a), the other is a large, pea shaped structure (as indicated by arrow B in Fig.2a). Small circular pores are formed by tiny, fast moving microarcs. The formation of micropores in the petal structure is complex. It used to be thought that such structure was caused by large arcs in the arc discharge stage \cite{19}. Large arc can promote the formation of this structure, but judging from the experimental time node, there should be other factors to promote the formation of the structure. There are various defects and the unbalanced distribution of different phases in the matrix on the surface of the sample makes the charge form charge agglomeration in these places. And the microarcs stay in these places and does not move, thus the oxide in these places is repeatedly broken down, resulting from continuous stacking.

The surface roughness of the film under different time was measured in figure 3. The result shows that the surface roughness increases with the increase of the oxidation processing time. This is in accordance with the SEM observation results in Fig.1. The porosity and micropore size were measured in Figure 4. The porosity decreases first, then rises and falls again. The pore size rises first and then drops. The porosity of processing time for 80s is lower than that of 25s because the micro-arc oxide film formed at 25s is equivalent to the first thin layer, and the micropores are completely bare, however, with the development of micro-arc oxidation, the newly formed oxides will be stacked on this basis to cover some of the micropores. When oxidation time is more than 1800s, porosity and pore size will decrease. On the one hand, is due to the adsorption of a large number of particles on the surface of the film, which have blocked the original pores, resulting in the decrease of porosity and pore size in statistics. On the other hand, is due to the increase of pea petal structure in the late stage of micro-arc oxidation, this will lead to partial aggregation, annexation and even collapse of the pea petal structure and block up some small micropores(as indicated by arrow C in Fig.2f).

![Figure 3. Effect of processing time on roughness](image1)

![Figure 4. Effect of processing time on porosity and micropore size](image2)

3.2 Effect of oxidation time on the phase structure of the film

Fig.5 shows the element distribution in the cross section of the film obtained by oxidizing for 30 minutes. Table 1 shows the element content of the four positions marked on the cross section. The results show that the film is mainly composed of four elements( Mg, O, Al, Si). The Si element mainly distributes in the surface layer of the film (position 1), corresponding to loose layer. The distribution of O and Al in the whole film is basically unchanged. Mg decreased significantly in the surface layer, but changed very little in the dense layer(position 2 and 3). Therefore, the dense layer of micro-arc oxide film is mainly composed of MgO, and the content of MgSiO$_3$ in the surface layer is relatively high.
Figure 5. Element distribution of micro-arc oxide film on cross section  
(a) SEM+EDS pattern; (b) EDS pattern

Table 1. Element distribution in different positions in MAO coatings

| position | Element composition (wt. %) |
|----------|----------------------------|
|          | Mg | O   | Al   | Si  |
| 1        | 44.83 | 37.33 | 2.28 | 15.56 |
| 2        | 59.49 | 37.53 | 2.56 | 0.42  |
| 3        | 60.97 | 36.48 | 2.57 | 0.12  |
| 4        | 91.24 | 2.33  | 6.31 | 0.12  |

Figure 6 studies the changes of film phase with oxidation time in silicate electrolyte. It can be seen from the diagram that the phases in the coating are mainly composed of Mg, MgO, MgSiO$_3$ and MgAl$_2$O$_4$. However, the proportion of each phase is different at different time nodes. In the first 6 minutes, the main diffraction peaks are Mg, which is because the oxidation time is short and the coating is thin, Mg in the matrix is included in the analysis system during X-ray irradiation, so the three strong peaks \{101\}, \{0002\}, \{101\} are very strong. Because d values of \{1011\}$_{Mg}$, \{111\}$_{MgO}$, \{112\}$_{MgSiO_3}$ and \{311\}$_{MgAl_2O_4}$ have very little difference, and the grains formed in the process of micro-arc oxidation are very small$^{23-24}$, resulting in the broadening and overlapping of diffraction peaks, so the relative content of these phases cannot be determined by these peak. For MgO, the relative content can be judged qualitatively using \{200\} peak which belongs to the three strong diffraction peaks. It can be found that the \{200\} peak is weak or even invisible in the first 6 minutes, which indicates that the content of MgO is relatively low in this time period. In comparison, The relative intensity of \{103\} diffraction peak of MgSiO$_3$ and \{400\} diffraction peak of MgAl$_2$O$_4$ is slightly higher in this time period. So the contents of them may be higher than MgO. It can be inferred that these two or one of the main phases should be formed in the early stage of microarc oxidation. After 6 minutes, the \{200\} and \{220\} diffraction peaks of MgO gradually increase, but the diffraction peaks of MgSiO$_3$ and MgAl$_2$O$_4$ do not, which indicate that the formation rate of MgO should be higher than that of MgSiO$_3$ and MgAl$_2$O$_4$. This may be a thermodynamic reason or a kinetic factor. Generally, liquid phase mass transfer velocity is higher than solid phase. At the beginning of oxidation, the film is thin and the reaction occurs mainly on the outermost surface of the film. At this time, under the action of strong electric field, the electromigration of anions (SiO$_3^{2-}$, Al$_2$O$_4^{2-}$) from the solution is adsorbed to the electrode surface at a very high speed, and was attracted to the arc region directly involved in film formation. In the later stage, when the film is thicker, the oxidation takes place inside the film. All kinds of elements want to participate in the formation of film, the elements must diffuse into the oxide film, but the diffusion rate of the acid ions is much slower than that of the O ion. Therefore, MgO is the main phase formed in the later stage.
Fig. 6  Effect of processing time on phase structure of the coating (a) 25s; (b) 80s; (c) 100s; (d) 190s; (e) 360s; (f) 1800s; (g) 3600s
(b) change of a/h, b/h with processing time

3.3 the change of size under different oxidation time

Fig. 7 is the data curve obtained by measuring the actual sample according to the schematic diagram of Fig. 1. As can be seen from Fig. 7a, the sizes of ‘a’, ‘b’ and ‘h’ increase with the increase of time. But the law of change of ‘a’, ‘b’ and ‘h’ is different. In the range of selected oxidation time (60min), ‘b’ and ‘h’ have been rising, but ‘a’ has been linearly increased in the initial 10 minutes, and the size has basically not changed after 10 minutes, even if there is an increase in the size, the increase is not much. Compared with the value of ‘a’, the value of ‘b’ in front 10 minutes increases slowly. Because ‘a’ represents the outward growth of the film, ‘b’ represents the inward growth of the film, so in front 10 minutes of micro-arc oxidation the film mainly grows outward to the matrix. This is consistent with the observed phenomena in the experiment. Under the selected experimental conditions, the electrical sparks on the surface of the samples were strong and the screams during the reaction were relatively loud at the beginning of 10min, and the reaction occurred mainly on the surface of the samples. After 10min, the electric sparks on the surface of the sample became weaker, and the screams in the reaction process were also weak. After 10min, the increase in the value of ‘b’ becomes faster. At this time, the inner growth of the film is dominant.

Fig. 7b is the relationship between a/h, b/h and processing time. It can be seen from the diagram that in silicate electrolyte a/h decreases with the increase of oxidation time, while b/h increases with oxidation time. That is to say, the proportion of the outer growth of the film is smaller and smaller with the increase of time, while the proportion of the inner growth of the film is increasing. Through the analysis of the data, it is found that the coating of inner growth to the matrix can eventually occupy 75%–85% of the whole coating. It can also be qualitatively considered that the loose layer of the micro-arc oxide film is the result of outward growth, while the dense layer is the result of the inward growth.
3.4 Effect of oxidation time on the solute ions in the electrolyte

Figure 8 studies the effect of micro-arc oxidation time on solute ions in electrolyte. It can be seen from the diagram that the content of Mg in electrolyte increases rapidly during the first 10 minutes of micro-arc oxidation, and then increases slowly. And the content of Al also increases rapidly in the first 10 minutes, after 10 minutes, although the rate of increase has slowed down a little bit, but it is also much faster than that of Mg element.

The process of micro-arc oxidation is a result of plasma chemistry, electrochemistry, thermochemistry and so on [25-26]. The formation of film is controlled by oxidation process, precipitation dissolution equilibrium and electrochemical deposition. There is material exchange between magnesium alloy substrate and electrolyte. O ion and acid ion in the electrolyte are adsorbed and diffused to the surface of the substrate, while the elements such as Mg, AL, Zn in magnesium alloy are dissolved and diffused into the electrolyte. At the position 5 in figure 5, the content of Al was only 6.31%, while the content of Al in magnesium alloy measured before oxidation was 8.56%. So a part of Al diffuses outside to take part in film formation or dissolution into electrolyte. At the beginning of micro-arc oxidation, the film layer is thin and the diffusion channel is shorter, which leads to the rapid diffusion of matrix alloy elements into electrolyte. At the same time, the corrosion resistance of the film formed at the initial stage is poor, and it is etched more seriously by the electrolyte under the action of high current and high voltage. These factors lead to the rapid increase of Mg and Al content in the electrolyte at the initial stage of micro-arc oxidation. With the development of micro-arc oxidation, the film become thicker and the diffusion channel is elongated, as a result, the diffusion and dissolution of Mg and Al elements into the electrolyte slow down.

It is also seen from the diagram that the content of the Mg element in the electrolyte is higher than that of the Al element. This is due to the higher content of the Mg element in the matrix and the greater affinity with O. And the main component in the micro-arc oxidation film is MgO, so it has more opportunities to contact the electrolyte, and thus the possibility of dissolving is greater. In relation to the concentration of the alloy elements in the matrix, it should also be related to the affinity of the electrolyte. The metal element easy to dissolve and its oxide transfer into the electrolyte should be faster, but the insoluble one is slower.

3.5 Effect of the processing time on corrosion performance

Fig.9 is a study of the effect of oxidation time on the corrosion resistance of the film. The sample was prepared in a constant current mode with a current density of 20A/dm². The results show that the corrosion rate of the film first decreases and then increases with the increase of the treatment time of micro-arc oxidation, and the better corrosion resistance appears in the oxidation treatment time for about 30min. With the increase of the treatment time of micro-arc oxidation, the film thickness is gradually increased, so the corrosion resistance is also improved. However, the effect of film thickness on corrosion performance is only one factor. In the late stage of micro-arc oxidation, with the increase of processing time, under the action of electrochemistry, plasma chemistry and thermochemistry, many microcracks and even macroscopic cracks will appear on the surface of the film, and stress concentration may occur, which will reduce the corrosion resistance of the film and increases corrosion rate. From the observation of the surface morphology in the front, we can see that when the time of micro-arc oxidation reaches 60min, there are many small particles adsorbed on the surface, which should be the result of the readsorption of corroded substances under the action of electrochemistry, plasma chemistry and thermochemistry. Therefore, the film of 60 min is more easily corroded than 30min.
4. Conclusions

(1) The micro-arc oxidation treatment time has an important effect on the surface morphology and structure of the film. With the increase of the oxidation treatment time, the surface roughness and pore size of the film increase.

(2) The phase composition and distribution in the film are influenced by the treatment time of micro-arc oxidation. The acid ion(SiO$_3^{2-}$) in electrolyte played an important role in the formation of phase in the early stage, and the formation rate of MgO was dominant in the later stage.

(3) In the early stage of micro-arc oxidation, the outward growth of the coating is the main, and in the later stage, the inward growth is the main.

(4) The content of Mg and Al in electrolyte increases with the increase of oxidation treatment time. The increase rate of Mg and Al in the electrolyte is higher in the early stage and slower in the later stage.

(5) The micro-arc oxidation treatment time has an important effect on the corrosion performance. The corrosion rate decreases first and then increases with the prolongation of the treatment time. The corrosion rate of 30min processing time is lower under our technological conditions.

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