

Research Article

Enhancing The Wear Resistance of Pure Mg Coated by Plasma Electrolytic Oxidation by Adding Silver

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Abstract : In this study, the tribological properties of pure Mg coated in different concentrations of silicate-based electrolytes containing with/without AgNO₃ and at different coating times at each concentration by Plasma Electrolytic Oxidation (PEO) were investigated. The parameters examined whose effect on tribology for silicate-based electrolytes were the 5, 8, and 10 g/L Na₂SiO₃ concentrations and 5, 10, and 15-minute coating times, while the parameters for silicate-based electrolytes containing AgNO₃ were 0.1, 0.2, 0.4 g/l AgNO₃ concentrations and 5, 10, and 15-minute coating times. The coatings were characterized by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD). The mechanical properties were determined by Vickers microhardness measurements and dry sliding linear wear tests performed by a linear ball-on-disc tribometer. Increasing the silicate concentration increased the average pore size, porosity percentage, and surface roughness. On the other hand, with increasing the coating time, the surface morphology gradually became flatter, and the coatings gained a more compact and homogeneous appearance. Long-time coating significantly improved the wear resistance of Mg. Smoother and denser PEO coatings were produced by adding AgNO₃ to the electrolyte. The best hardness value and wear resistance were obtained from coatings manufactured by adding AgNO₃ to the silicate-based electrolyte.

Keywords : Plasma Electrolytic Oxidation, Pure Mg, Silver, Wear resistance.

1 Introduction

Magnesium (Mg) and magnesium alloys are important materials used in many industrial fields. Their low density, high specific strength, high modulus, excellent castability, and high recyclable ability make them popular. However, their poor wear resistance and corrosion properties have restricted their wide range of applications [1, 2, 3, 4, 5]. The methods such as thermal spray, physical vapor deposition, anodizing, plasma electrolytic oxidation can be used to protect the surfaces of Mg and Mg alloys. Plasma electrolytic oxidation from these methods is also called as micro-arc oxidation in the literature [3, 4, 5, 6]. Plasma electrolytic oxidation (PEO) is a very economical, environmentally friendly, and effective method used to increase the surface properties of magnesium (Mg) and magnesium alloys [4, 7, 8]. A hard and stable ceramic oxide layer grows on the substrate surface by PEO. The adhesion strength of the hard and stable ceramic oxide layer formed by PEO improves the mechanical properties [4, 8, 9].

The structure coated on the substrate by PEO contains forms like volcanic micro pores and pancakes. Unfortunately, due to these forms, the structure sometimes includes cracks and micro pores in the oxide coating, which can cause problems in corrosion and wear resistance, and the coating structure loses its protective effect on the substrate. Many parameters, such as electrical parameters (frequency, voltage, current) and electrolytes (phosphate-based, silicate-based, boride-based, and aluminate-based, etc.), are very functional in improving the protective effect of PEO coating formed on the substrate [9, 10, 11]. Therefore, the surface morphology of the coating formed on the substrate changes depending on process parameters during treatment. For this reason, optimum parameters must be determined accurately through experimental studies.

There are many studies made by many researchers on the effect of coating parameters. For example, Küçükosman et al. [12] have studied the effect of graphite on AZ91-Mg coating properties. It is expressed that the direct current was more effective on surface properties in another experimental work reported by Zhao et al. [13]. Khorasanian et al. [14] indicated that the addition of starch into electrolyte during PEO coating of titanium alloy developed to form a denser layer with better wear properties as compared to the counterpart that occurred from electrolytes without starch. Liu et al. [15] have studied different electrolytes consisting of silicate and phosphate-based and examined the growth mechanism of PEO coating on 2A70 aluminium alloy.

Table 1: Samples and PEO parameters

Sample codes	Na ₂ SiO ₃ (g/l)	NaOH (g/l)	Na ₃ PO ₄ (g/l)	AgNO ₃ (g/l)	Voltage (V)	Oxidation Time (min.)	pH
(M-Si-1)-5	5	3	1	-	310	5	11.5
(M-Si-2)-5	8	3	1	-	310	5	12.0
(M-Si-3)-5	10	3	1	-	310	5	12.5
(M-Si-1)-10	5	3	1	-	310	10	11.5
(M-Si-1)-15	5	3	1	-	310	15	11.5
(M-Si-1-Ag)-1	5	3	1	0.1	330	5	11.5
(M-Si-1-Ag)-2	5	3	1	0.2	330	5	11.5
(M-Si-1-Ag)-4	5	3	1	0.4	330	5	11.5

A thick, hard, and dense ceramic coating with oxides-based occurs after Plasma Electrolytic Oxidation. After the oxide coating, the substrate becomes a valuable engineering material due to its properties. However, the oxide coating formed has high porosity. Because different applied parameters generate a high-intensity micro-arc discharge, leading to high porosity in the oxide coating. If we want to improve the coating properties, the porosity of the layer must be decreased. Therefore, incorporating additives into an electrolyte during the PEO treatment will significantly improve surface properties such as wear and corrosion resistance [4, 9, 16]. In this study, some additives were added to the electrolyte to reduce porosity and prevent wear.

In this study, the mechanical behavior, such as hardness and wear resistance of the coatings formed on the commercial pure magnesium by PEO, were investigated in detail for process parameters, such as different solutions with/without silver additives and coating time. In addition, the correlations between the microstructure and properties of PEO coatings depending on process parameters were discussed.

2 Experimental Methods

Pure Mg samples with dimensions 30x10x5 mm² were used as a substrate in the experimental studies. Before coating, all samples were ground with SiC sandpaper, polished, washed, and dried. The PEO process was performed in a unipolar power source (10 kW). While a pure Mg specimen was an anode of the system, and a stainless steel sheet was a cathode. Electrolytes with and without AgNO₃ were prepared using Na₂SiO₃, NaOH, Na₃PO₄, and distilled water. The temperature of the electrolyte was kept at 30 °C ± 2 °C. Table 1 includes sample codes and process parameters of pure Mg samples.

3 Results and Discussion

Figure 1 shows the SEM morphologies of coatings with different Na₂SiO₃ concentrations and process times. The structures' common features are normal PEO surfaces, pores of various sizes, and pancake structures [2, 8, 16].

According to Figure 1, increasing Na₂SiO₃ concentration from 5 g/l to 10 g/l increased the average pore size, porosity percentage, and surface roughness. In addition, increasing the concentration of Na₂SiO₃ to 10 g/l caused a negative effect on the coating, and it was seen that some micro cracks occurred. This is likely due to the significant increase in electrical conductivity, which creates a powerful spark discharge and produces a micro-cracked PEO coating with large pores [16, 17, 18]. It is observed that as the coating time increases, the surface morphology gradually becomes smoother, the micro pores diameters decrease, and the coating density increases due to the electrolyte deposited into the pores. This may be due to the intensification of the reaction with the rise of conductive particles and more molten material ejected from the discharge channels [19, 20]. The pancake-like stacking increases thickness and repairs some defect locations, which makes the surface stronger.

Figure 2 shows the SEM morphologies of the coatings produced with solutions containing different concentrations of AgNO₃. Increasing solution concentration is an essential parameter regarding coating quality and discharge characteristics. The morphology, average pore size, and porosity percentage are a function of the solution's concentration and electrical conductivity. As seen in Figure 2, the micropores on the surface decreased with the increased silver's concentration (0.1%, 0.2%, and 0.4% g/l), and the microporosity diameters were approximately measured as 2±1 μm on the surface. In addition, surface roughness also decreased with increasing solution concentration. The solution conductivity has increased with the increase in Ag concentration in the solution due to its high conductivity of Ag, which has increased the coating growth rate. With the rise in molten oxide, the micropores filled with ion flow have become denser. It is an expected result, according to the literature, that a denser layer was obtained with the decrease of micropores and smaller diameters with increasing the solution conductivity [18, 21].

The concentration change was also examined for Na₂SiO₃, and surface roughness (Ra) values of magnesium substrate coated with PEO process in different solutions containing Na₂SiO₃ were measured. The surface roughness values of (M-Si-1)-5, (M-Si-2)-5, and (M-Si-3)-5 coded samples were determined as 4.149, 4.226, and 4.907 μm, respectively. While surface roughness increased with increasing Na₂SiO₃ concentration in the solution, it was seen that surface roughness decreased with increasing AgNO₃ concentration. In coatings made with solutions with the same content, a decrease in roughness values was noted as the coating time increased. The surface roughness values measured 3.598 μm in (M-Si-1)-10 and 3.586 μm in (M-Si-1)-10. A similar change related to the increased coating time was observed in the solution using AgNO₃ (nearly 3.500 μm).

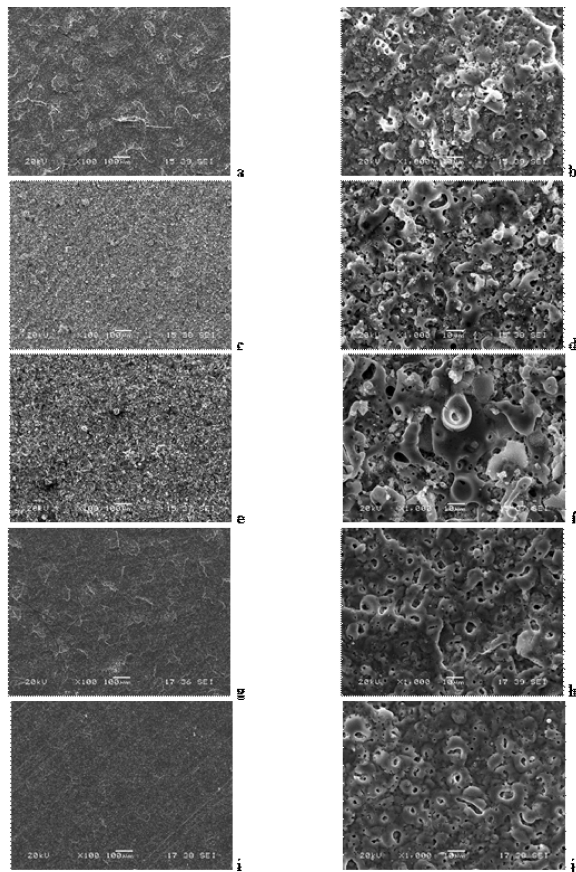


Figure 1: Surface SEM micrographs of (a, b) (M-Si-1)-5, (c, d) (M-Si-2)-5, (e, f) (M-Si-3)-5 (g, h) (M-Si-1)-10, (i, j) (M-Si-1)-15 samples

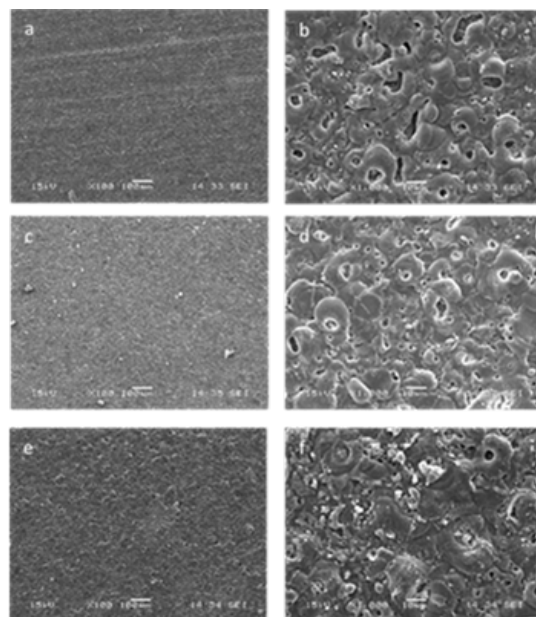


Figure 2: Surface SEM micrographs of (a, b) (M-Si-1-Ag)-1, (c, d) (M-Si-1-Ag)-2, (e, f) (M-Si-1-Ag)-4 samples

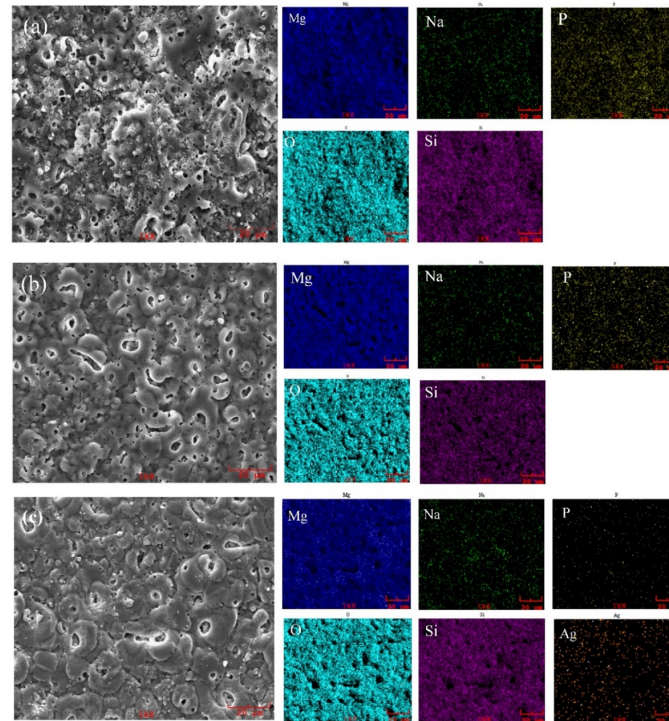


Figure 3: SEM morphologies and elemental mapping of (a) (M-Si-1)-5 (b) (M-Si-1)-15 (c) (M-Si-1-Ag)-1 samples

Figure 3 shows the SEM morphologies and EDS maps of (M-Si-1)-5, (M-Si-1)-15, and (M-Si-1-Ag)-1 samples produced in different electrolytic solutions and different coating times. (M-Si-1)-5 and (M-Si-1)-15 samples contain Mg, O, Na, Si, and P elements uniformly distributed on the surface layer. Si, Na, and P come from the electrolyte, and the maps show that the surface of the coating may consist mainly of MgO or Mg₂SiO₄ phases. XRD analysis will be performed to determine the phases formed on the surface. With increasing the coating time, the coverage of PEO coating rises gradually, and the micro pores on the surface are filled with the particles. The presence of Na, P, and Si elements expresses that the ions in the electrolyte enter the PEO coating through the discharge channels during the PEO process. Figure 3c shows that silver is also distributed homogeneously on the surface of the sample coated in the solution prepared using AgNO₃.

The hardness values of the coatings produced using different electrolyte compositions and different coating times are presented in Figure 4. It is seen that the microhardness of pure Mg is about 60 HV_{0.1}. All PEO coatings have a higher hardness value than pure Mg. The hardness values of the (M-Si-1)-5, (M-Si-3)-5, and (M-Si-1)-15 samples were 134, 188, and 193 HV_{0.1}, respectively. (M-Si-1)-15 sample shows the highest hardness because of has the lowest micro-pore and crack amount, in addition to the densest structure due to the coating time. It indicates that the microhardness of pure Mg can be increased by at least three times with PEO method and significantly improve the surface hardness. The hardness values of the coatings produced using electrolytes containing AgNO₃ were also measured. For example, the hardness value of (M-Si-1-Ag)-4 sample was determined as 246 HV_{0.1}. The change in surface morphology with silver reinforcement was explained in the previous section. The increase in hardness with the addition of Ag can be attributed to the homogeneous coating of the surface and, at the same time, to the decreased amount of porosity.

Naturally, one of the essential factors that determine the hardness of the ceramic coating is the phase content as well as the microstructure. The XRD patterns of the layers containing Si produced with different parameters are presented in Figure 5. Mg, and MgO were determined on the surfaces of all coatings by XRD analysis. When the coating time increased, Mg₂SiO₄ (Forsterite) peaks were also observed on the surface.

Figure 6 presented the XRD patterns of PEO-coated samples containing Ag. Mg, MgO, and Ag phases were determined in the surfaces of these samples. However, it was difficult to identify Ag or Ag-containing phases due to the low concentration of Ag that remained in the (M-Si-1-Ag)-1 and (M-Si-1-Ag)-2 coatings. The (M-Si-1-Ag)-4 sample appeared to have an Ag-rich particle distribution on the surface. That is to say, As the silver concentration of the electrolyte increased, the peak intensity of Ag or Ag-containing phases increased.

Figure 7 shows the wear rates of different coating in dry sliding conditions. The wear rates of pure Mg, (M-Si-1)-5, (M-Si-2)-5, (M-Si-3)-5, (M-Si-1)-10, (M-Si-1)-15 under dry conditions were determined 14.87×10^{-4} , 9.65×10^{-4} , 16.02×10^{-4} , 18.69×10^{-4} , 9.56×10^{-4} , 9.47×10^{-4} mm³/N.m, respectively. (M-Si-1)-5 coated samples showed a lower wear rate than pure Mg, which can be explained by the high surface hardness after coating. However, the wear rate increased with increasing

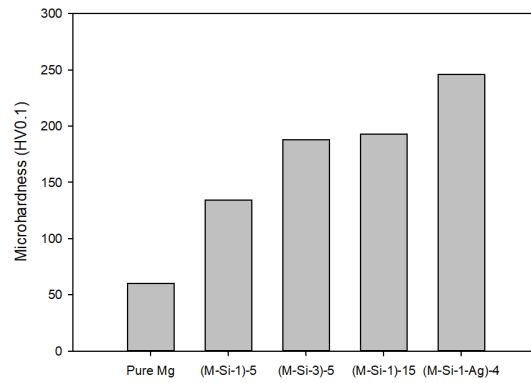


Figure 4: Microhardness values of pure Mg and PEO coatings produced with different parameters

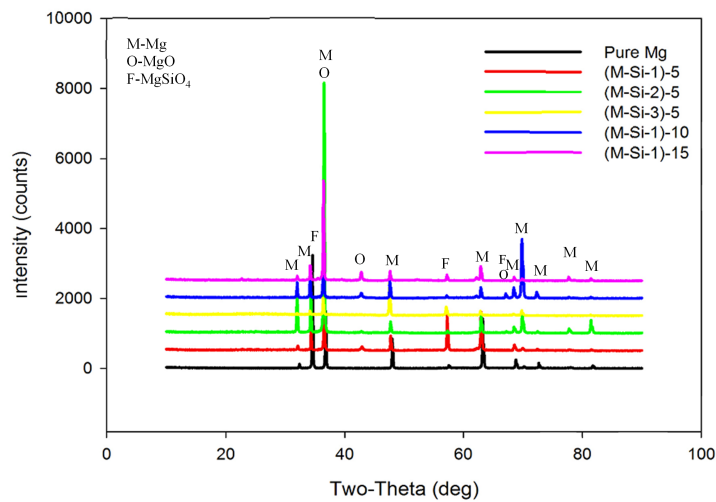


Figure 5: XRD patterns of pure Mg and PEO-coated samples containing Si

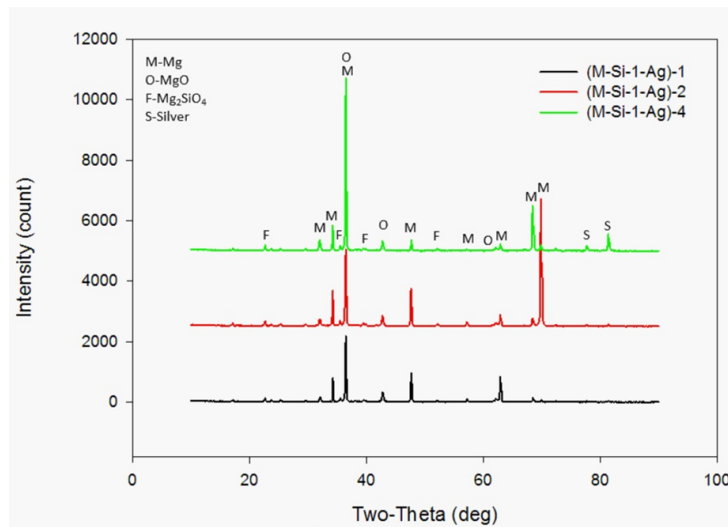


Figure 6: XRD patterns of pure Mg and PEO-coated samples containing Ag

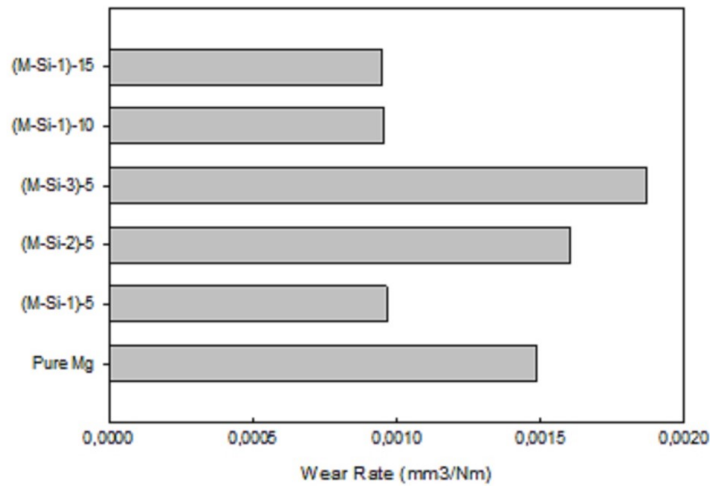


Figure 7: Wear rates of the pure Mg and PEO-coated samples containing Si

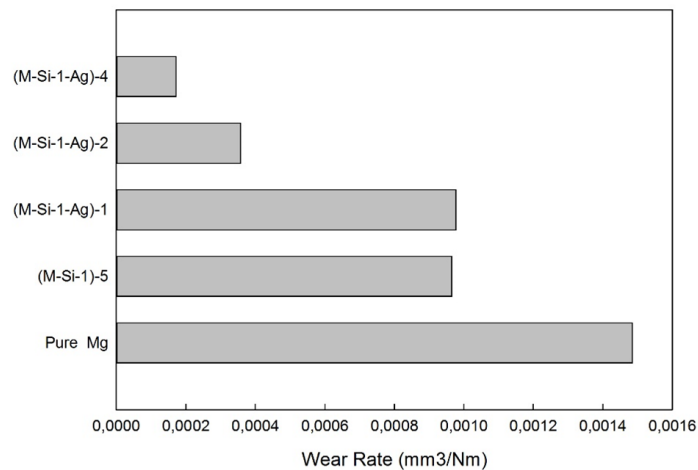


Figure 8: Wear rates of the pure Mg, (M-Si-1)-5, and PEO-coated samples containing Ag

Na₂SiO₃ concentration on (M-Si-2)-5, and (M-Si-3)-5 samples. Here, the reason for the increase in wear rate can be explained by the surface morphology. With increasing silicate concentration from 5 g/l to 10 g/l, the surface’s porosity, microcracks, and roughness increase. Coating particles remove from the surface, which can affect wear and cause strong abrasion on the surface. It is also well known that the PEO coating consists of an outer porous layer and an inner barrier layer. The outer layer of the PEO coating is not only rough but also brittle [22, 23, 24]. On the other hand, raising the coating time positively affected the wear rate, and the wear rate decreased. The best result was detected on (M-Si-1)-10, (M-Si-1)-15 samples. These coatings have the lowest wear resistance compared to pure magnesium because of their higher hardness. Additionally, surface morphology also has a significant effect on the wear mechanism. With increased time, the surface morphology gradually became flattened.

Figure 8 shows the wear rates of PEO-coated samples containing Ag in dry sliding conditions. Additionally, the wear rates of Ag-free pure magnesium and base coating samples are also added to the figure. Comparison of pure Mg and (M-Si-1)-5 to PEO-coated samples containing Ag can be seen that the wear rate of PEO-coated samples containing Ag decreases by nearly 148%. This vast difference between the wear resistance of the samples can be explained by the higher hardness, reduced micropores, and decreased surface roughness of the samples coated using AgNO₃.

Figure 9 and Figure 10 show the wear surface SEM images of pure Mg, (M-Si-1)-5, (M-Si-2)-5, (M-Si-3)-5, (M-Si-1)-10, (M-Si-1)-15 samples. Figure 9 (a, c, e, g) presents the wear tracks of pure Mg and PEO-coated samples containing Si under an applied load of 3 N. It can be seen that the width of the wear track on pure Mg and (M-Si-1)-5 are more extensive than (M-Si-2)-5, (M-Si-3)-5 PEO coatings. But despite this, these samples see less abrasive wear surface. The wear scar depth is deeper on (M-Si-2)-5 and (M-Si-3)-5, which is calculated with a profilometer. It is clearly visible in Figure 9 (f, h) that these samples have high surface roughness after coating by PEO, the brittle and stiff parts that broke off from the surface and acted as abrasive. This situation also caused an increase in abrasion scar depth. Figure 10 presents the SEM images of the wear surface

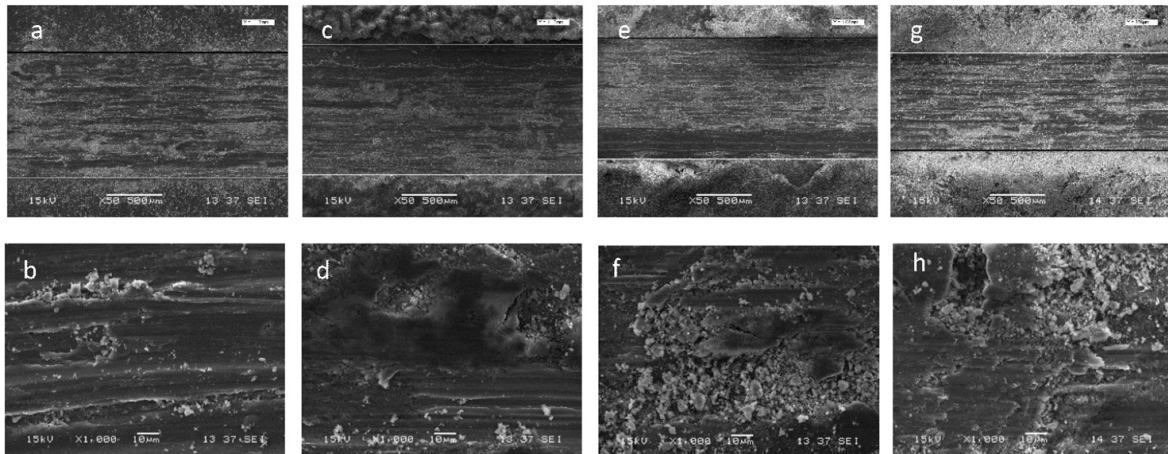


Figure 9: The SEM images of the wear track morphology of (a-b) pure Mg, (c-d) PEO-coated (M-Si-1)-5, (e-f) PEO-coated (M-Si-2)-5, (g-h) PEO-coated (M-Si-3)-5

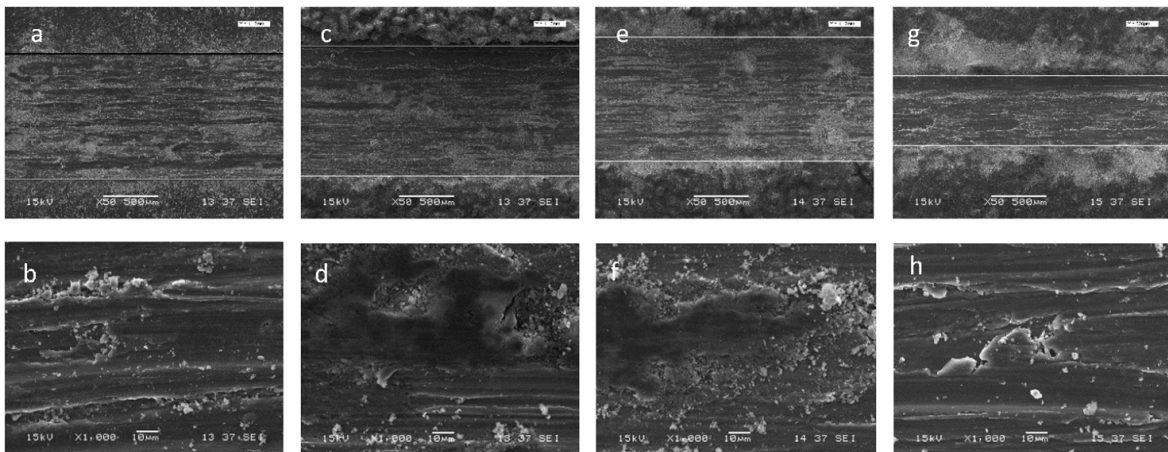


Figure 10: The SEM images of the wear track morphology of (a-b) pure Mg, (c-d) PEO-coated (M-Si-1)-5, (e-f) PEO-coated (M-Si-1)-10, (g-h) PEO-coated (M-Si-1)-15

to show how PEO coating time affects wear behavior. It is seen from Figure 10 (a) that the worn track of pure magnesium is maximal than the coated samples. It has also attracted attention that the abrasion resistance improves with increasing coating time. The increase in the coating time caused a decrease in the wear intensity, and the groove depths decreased on the coated samples. It is mentioned earlier that a denser layer is formed with increased coating time, which makes the coating layer stronger and improves wear resistance.

Figure 11 gives the SEM images of the wear surface of PEO-coated samples containing Ag. The width of the wear track decreased with increasing silver supplementation. It is known that silver affects the surface morphology, and the surface roughness decreases with increasing silver. It can be seen that there are mainly two kinds of morphology in the micro domain; one is a continuous compact and flat sheet-like region corresponding to the blocky black areas in Figure 11 (a); the other is a rough and uneven region corresponding to the white strip in Figure 11 (b, d, f). Among all the PEO-coated samples, the coatings containing Ag show almost no wear loss of the coating, and the (M-Si-1-Ag)-4 sample has the narrowest wear track.

4 Conclusions

In this study, coatings were carried out with the PEO process in silicate-based electrolytes containing with/without AgNO_3 and at different coating times to improve the tribological properties of pure Mg. The obtained results are summarized below; The coatings exhibited a porous, rough, and micro-cracked surface morphology due to the nature of the PEO coating. All samples contained MgO . PEO coating time affected the surface morphology. The coatings became flatter with increasing coating time, and a more homogeneous surface morphology formed. No cracks appeared on the surface during the 15-minute coating period. The samples mainly contained MgO and also Mg_2SiO_4 . It was determined that the micropores on the surface of coatings produced in silicate-based electrolytes containing AgNO_3 were less, and their diameters were reduced. The microhardness of

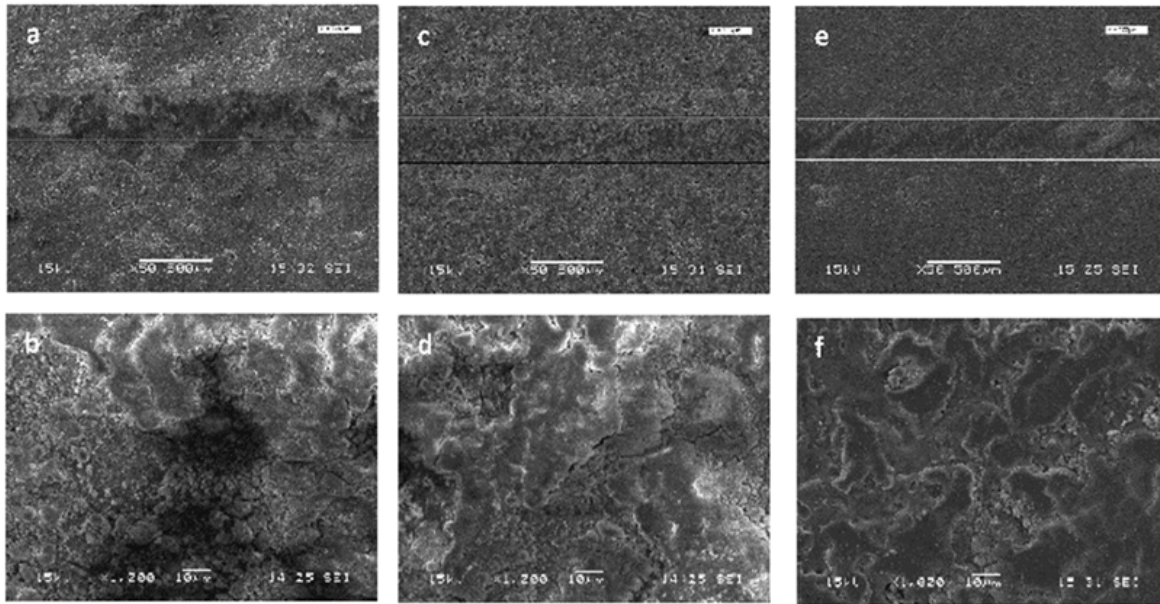


Figure 11: The SEM images of the wear track morphology of (a-b) PEO-coated (M-Si-1-Ag)-1, (c-d) PEO-coated (M-Si-1-Ag)-2, (e-f) PEO-coated (M-Si-1-Ag)-4

the PEO layers produced in silicate-based electrolytes was max. 193 HV_{0.1}, while the microhardness of PEO coatings obtained in silicate-based electrolytes containing AgNO₃ was 246 HV_{0.1}. There was a 3-fold increase in the hardness of the coated surface obtained in silicate-based electrolytes compared to the uncoated Mg substrate and a 4-fold increase in the hardness of the coated surface obtained in silicate-based electrolytes containing AgNO₃. PEO coatings with silver added show much better wear resistance than uncoated Mg and coated in a high-concentration silicate-based solution. The width and depth of wear tracks of PEO coatings decrease with Ag addition.

Authors' Contributions

The supervisor of this project is AA (Aysun Ayday). The experimental studies designed by MS (Merve Söğüt) and AA were carried out by MS. MS made the theoretical calculations in collaboration with AA and wrote the article. Both authors read and approved the final manuscript.

Competing Interests

The authors declares that they have no competing interests.

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