



Effect of positive pulse voltage on color value and corrosion property of magnesium alloy black micro-arc oxidation ceramic coating

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ARTICLE INFO

Keywords:

Magnesium alloy
Micro-arc oxidation
Positive pulse voltage
Copper content
Color value
Corrosion resistance

ABSTRACT

To improve the corrosion resistance of the magnesium alloy and to extend its applications in light absorption field, black anti-corrosion and absorption integrated ceramic coatings were prepared on AZ31 magnesium alloy by asymmetric bipolar pulse micro-arc oxidation (MAO) technique. The effect of applied positive pulse voltage on the color value and corrosion properties of the coatings were investigated. The results showed that the Cu^{2+} ions were dissolved into MgO lattice to form the substitutional solid solution ($\text{Mg}_{1-x}\text{Cu}_x\text{O}$) during MAO of magnesium alloys. The color value was mainly determined by the effect of the content of Cu^{2+} ions on the band gap of MgO. On the premise of constant negative pulse voltage, with the increase of positive pulse voltage, the solid solubility of Cu^{2+} ions in ceramic coating increased first and then remained unchanged, while the color value decreased first and then kept constant. The average absorption rate of black ceramic coating to visible light could be up to 95%. Moreover, the rising positive pulse voltage increased the thickness and decreased the density of the black ceramic coating, resulting the anti-corrosion property increases first and then decreases.

1. Introduction

Magnesium alloys have been attracting particular interest and considered as one of the candidate materials using in the fields of aerospace and optical instruments owing to their excellent properties [1,2], such as low weight, well electromagnetic shielding property, and high specific strength and stiffness. However, the poor corrosion resistance caused by its serious chemical activity has greatly limited their applications. Though some existing methods are used to prepare coatings on magnesium alloys and achieve improved corrosion resistance, among which the micro-arc oxidation (MAO) technique have been studied mostly [3,4], the light absorption property of the prepared coating is insufficient and not qualified for the optical instruments. In general, the black surface has the strongest absorption ability to visible light. Therefore, the protection coatings on magnesium alloys are expected to achieve integrated corrosion resistance and light absorption property.

The MAO technique is to generate an oxide ceramic coating in-situ on the metal surface, and the coating possesses good corrosion resistance and excellent adhesion of film-substrate. During the MAO process, the ceramic coating composed of metal oxide is grown in situ under the action of instantaneous high temperature and high pressure produced by surface plasma discharge. Usually, the ceramic coating

prepared by MAO on magnesium alloys is white, but it's reported that the ceramic coating with different color could be prepared by adding colored salts in the MAO electrolyte. During the electrochemical reaction process, the colored anions are adsorbed to the surface of the sample in electric field, and then, the colored substances melt and enter the ceramic coating in the plasma discharge. Finally, the colored ceramic coating with good corrosion resistance is prepared on magnesium alloys [5–7]. In the electrolyte, with the increase of the electric field intensity between the sample and the electrode, the number of colored anions adsorbed on the surface of the sample increases, the content of colored substances in the ceramic coating increases, and the color change of the ceramic coating becomes more significant [8–11]. However, if the forward oxidation voltage is too high and the anions around the micropore are aggregated too strongly, the arc discharge energy in the oxidation process is too large, and large-size openings can be formed on the ceramic coating [12–14]. Meanwhile, the more heat generated at the moment of discharge, the lower the stability of electrolyte, resulting in salting out effect of electrolyte and affecting the coloring of ceramic coating. Finally, when the oxidation reaction is too fast, there are too many oxygen and molten products, and it is relatively difficult to solidify the ceramic coating. It is easy to produce defects such as holes and hot cracks on ceramic coating, causing the corrosion resistance worsening [15–17]. So, in order to prepare compact ceramic

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<https://doi.org/10.1016/j.surfcoat.2019.05.067>

Received 4 March 2019; Received in revised form 22 May 2019; Accepted 23 May 2019

Available online 30 May 2019

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coating, bi-directional pulsed MAO have been developed to reduce the anion concentration around the sample [18–20] and slow down the burning loss of the ceramic coating caused by single plasma discharge [21,22]. But the negative pulse will affect the growth of the ceramic coating which is dominated by the positive pulse.

It was reported that the ceramic coating prepared by bi-directional pulsed power supply has good corrosion resistance because negative pulse can effectively reduce the anion concentration around the sample [18–20]. In the process of discharge, it can also melt and repair the protruding tip of the ceramic coating surface, making it smoother and smoother. At the same time, it can also reduce the next positive pulse effect. The discharging intensity can slow down the burning loss of the ceramic coating caused by single plasma discharge [21,22], but the negative pulse will affect the growth of the ceramic coating by the positive pulse. Based on this, the appropriate ratio of positive and negative pulses is the key to prepare the colored anti-corrosion and light-absorbing integrated MAO ceramic coating.

In this paper, black ceramic coatings with good corrosion resistance were prepared on AZ31 magnesium alloy by MAO under constant voltage mode at different positive pulse voltage. The effect of positive pulse voltage on the color value, and corrosion resistance of the Ceramic coating was studied, in order to provide a useful exploration for the anti-corrosion and light absorbing surface integral ceramic treatment of magnesium alloy.

2. Experimental

2.1. Preparation of the ceramic coatings

Commercial extruded AZ31 magnesium alloy sheet was used in the experiment, and the sample size was 35 mm × 20 mm × 10 mm. The dynamic impedance adaptive asymmetric bipolar pulse power supply MAO120EHD-III developed by Xi'a University of Technology was adopted. The equipment features that the output pulse can be adjusted in a wide range of multi-degree of freedom. Accurate pulse regulation was achieved by using dynamic impedance adaptive control technology, in which stainless steel was used as cathode and the sample was anode. The mode of power supply was constant voltage mode, and the positive pulse voltage was 400 V, 420 V, 440 V, 460 V and 480 V, respectively. The frequency was 3000 Hz and the duty cycle was 12%. Negative pulse voltage was 50 V, frequency was 2000 Hz, duty cycle was 10%, series was 10:2, MAO treatment time was 10 min; electrolyte was made of silicate system, and coloring salt was CuSO₄ (9–12 g/L).

2.2. Characterization of the ceramic coatings

The thickness and roughness of the ceramic coatings were measured by TT240 eddy current thickness gauge (accuracy 0.1 μm) and DLSURF DR160 roughness gauge, respectively. The color value of the ceramic coating was calculated by using the x-rite 530 type Spectro densitometer. The UV–visible light absorption curve of the ceramic coating was measured by Lambda950 UV–visible-near infrared spectrophotometer. The breakdown voltage and dielectric strength of ceramic coatings were measured by CJ2678 withstand voltage tester (parameter selection: leakage current is 0.5 mA, mode is DC). The phase composition of ceramic coatings was determined by XRD-7000S X-ray diffractometer, where, the X-ray source was K-alpha ray of Cu target, the tube voltage was 40 KV, the tube current was 40 mA, and the scanning speed was 8 degree/min. The morphology of the ceramic coating was analyzed by JSM-6700F field emission scanning electron microscopy (SEM) and LEXT 3D MEASURING LASER MICROSCOPE OLS4000 laser confocal. Element content and distribution were analyzed by Oxford INCA high performance X-ray spectrometer. Kratos AXIS DLD (K-alpha ray source) multifunctional X-ray photoelectron spectroscopy (XPS) was used to analyze the element composition and valence state of the ceramic coating.

2.3. Corrosion behavior of the coatings

The corrosion performance of the ceramic coating was carried out at PARSTAT4000 electrochemical workstation with three electrodes system. Among them, the three electrodes are auxiliary electrode, working electrode and reference electrode (saturated calomel electrode). The corrosion medium is 3.5 wt% NaCl aqueous solution, pH = 7. The electrolyte used for each sample is 400 ml. The corrosion area of the sample is 0.78 cm². The Potentiodynamic polarization test was conducted from −0.5 V to 1 V. The step height is 1 mV and the step time is 0.2 s.

3. Results and discussion

3.1. Surface morphologies and element analysis of MAO coatings

As is well known, the light is divided into three parts: reflected light, transmission light and absorption light, when the incident light project on the material. After selectively absorbed by the material, the wavelength range of reflected light and transmission light will change, comparing with original incident light, and the wavelength range of the reflected light determines the color of the material. Therefore, the color mostly depends on the characteristic of the material. However, for rough and porous materials, the color of the surface is not only depended on the characteristic of the material, but also related to the microstructure of the surface. Because the rough surface will cause the secondary absorption of light, reducing the intensity of reflected light and affecting the color of the material [23]. Fig. 1 shows the surface morphology of the ceramic coatings prepared on AZ31 magnesium alloy under different positive pulse voltages.

From Fig. 1, it can be seen that the surface of ceramic coating prepared by different positive pulse voltage is obviously uneven, and shows typical ceramic sintering morphology. There are holes with different pore sizes among the bumps, and the existence of holes is inevitable. During the growth of the ceramic coating, pores that are the channels for electro-discharge are inevitable, because the negative ions accumulate on the surface of the ceramic coating to form a partial discharge center in the electrolyte, and the high temperature and high pressure of plasma discharge will produce discharge channels in the ceramic coating, causing the formation of pores in the prepared ceramic coating. At the same time, the molten metal and oxides would eject and be oxidized, solidified and quenched through the discharge channels, and the generated gas in the electrochemical reaction would also be released through the channels. As a result, pores, even cracks, are formed and inevitable in the prepared ceramic coatings. With the increase of the thickness of the ceramic coating and the positive pulse voltage required for breakdown, the energy of micro-plasma discharge increases. Correspondingly, the melting area and the ejected material of the ceramic coating increase continuously, resulting in the protrusion of the void size and the aggregation of the burnt-out material on the surface of the ceramic coating. The reflection of incident light will also vary with the surface state of the ceramic coating, and the color value of the ceramic coating will also be different. Table 1 is the composition analysis of black ceramic coatings prepared under different positive pulse voltages.

With increasing the positive pulse voltage, the ratio of Cu/Mg in the ceramic coating increases from 3.94% at 400 V to 5.1% at 440 V, then decreases to 4.76% at 480 V. As mentioned above, the colored complex anion (CuY)[−] in the electrolyte migrates and adsorbs to the surface of the sample under the action of pulsed electric field, and enters the ceramic coating through the discharge channel during the growth process of the ceramic coating. Thus, the intensity of electric field in the electrolyte will affect the content of colored ions in the ceramic coating inevitably. According to the calculation formula of electric field strength $E = U/d$ (where: E is pulse electric field strength; U is pulse voltage; D is the distance between cathode and anode), when the

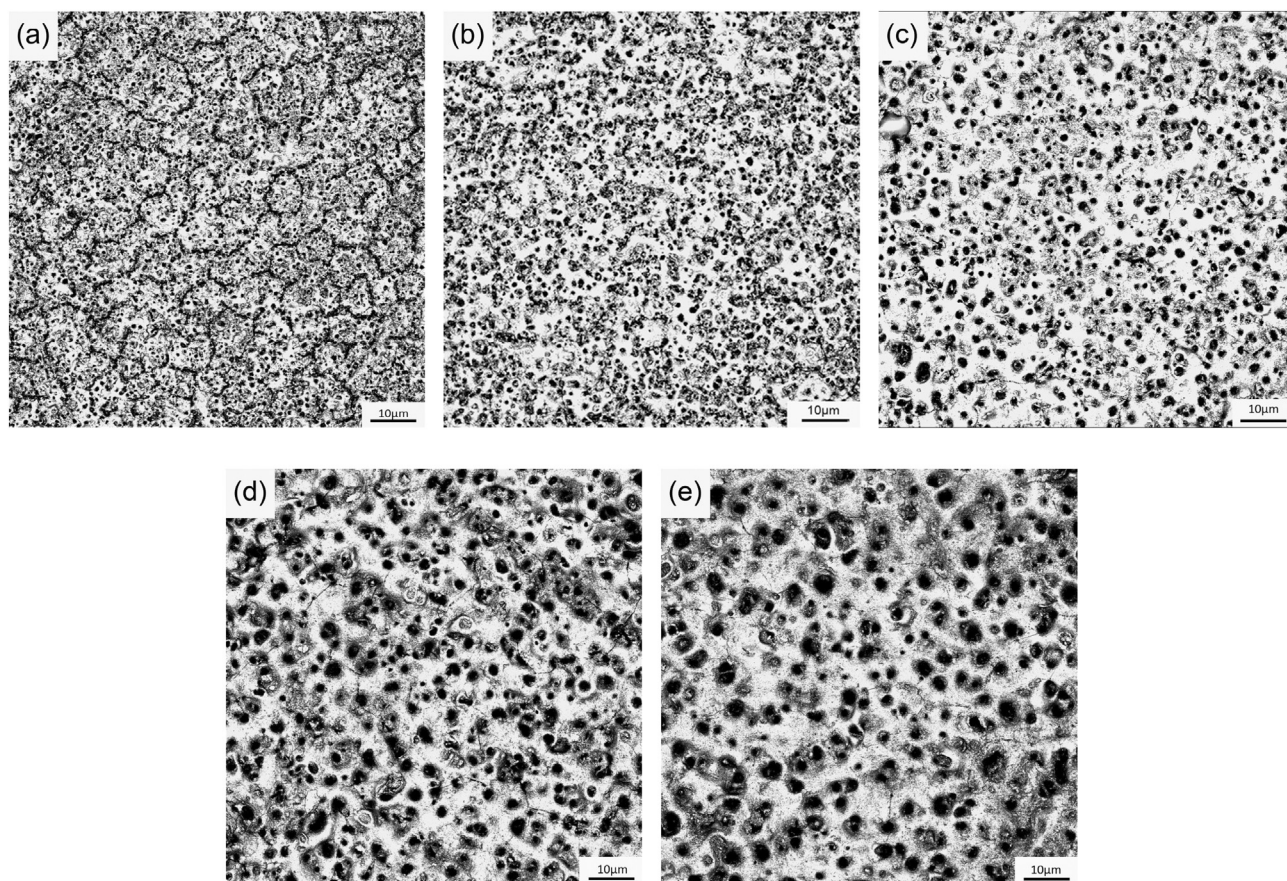


Fig. 1. Surface morphology of ceramic coatings prepared under different positive pulse voltages (a)400 V; (b)420 V; (c)440 V; (d)460 V; (e)480 V.

Table 1

Elemental analysis of black ceramic coatings prepared under different positive pulse voltages.

Positive pulse voltage(V)	O (at. %)	Mg (at. %)	Cu (at. %)	Cu/Mg
400	54.07	44.19	1.74	3.94
420	53.59	44.52	1.89	4.24
440	52.99	44.73	2.28	5.10
460	53.24	44.58	2.18	4.89
480	53.38	44.50	2.12	4.76

distance between sample and electrode is fixed, the positive pulse voltage is positively correlated with the electric field strength between cathode and anode in electrolyzer. The higher the electric field intensity strength, the more $(\text{CuY})^-$ complex ions attracted to the sample, and the more Cu ions will be in ceramic coating.

Moreover, as the voltage continues rising to much high, the exothermic reaction of oxidation would increase extremely, causing the local temperature of the electrolyte rising up and leading to salting-out effect. Then, burning loss appears in the growth process of the ceramic coating, the ablation area reached 5%. As a result, the content of Cu ion in the ceramic coating decreases.

Fig. 2 is the relationship between the ceramic surface color value, roughness and Cu/Mg ratio of different samples.

With increasing the positive pulse voltage, the color value of the ceramic coating decreases first and then remains unchanged, the Cu/Mg ratio of the ceramic coating increases first and then decreases slightly. As shown in Fig. 2b, the change of the surface roughness of different ceramic coatings is less than $0.07 \mu\text{m}$, indicating that the surface roughness has little influence on color value of the ceramic coatings.

However, the variation of the color value is exactly the same as that of the Cu/Mg ratio. When the forward pulse voltage is 440 V, the color value of the ceramic coating is the lowest to 23. Namely, the content of Cu ions in the ceramic coating is the main factor that affect the surface color value of black ceramic coatings prepared by MAO on AZ31 magnesium alloy, while the positive pulse voltage can affect the color value of ceramic coatings by the content of Cu ions in the ceramic coatings.

3.2. Electrochemical corrosion properties

During the MAO process of the formation of the ceramic coating, it's a cyclic process that the oxidation of the magnesium, the discharge breakdown, the melting solidification and ceramic sintering was carried out at the same time. The increased positive pulse voltage could cause the increase of the discharge energy of a single positive pulse and the thickness of the ceramic coating, but it would inevitably reduce the compactness of the ceramic coating and cause holes in the coating. Fig. 3 shows the cross-sections micrographs of ceramic coatings prepared under different positive pulse voltages. Fig. 4 shows the thickness and dielectric strength of the ceramic coating prepared under different positive pulse voltages.

Fig. 3 shows that the thickness of the ceramic coating is positively correlated with the positive pulse voltage. In addition, there are microcracks and holes in the ceramic coating. With the increase of the positive pulse voltage, the number of microcracks and holes in the ceramic coating increases gradually, and the compactness of the ceramic coating decreases. As shown in Fig. 4, the dielectric strength of the ceramic coating increases first and then decreases with the increase of the positive pulse voltage, which is consistent with the change of the compactness of the ceramic coating. For cathodic protection coatings,

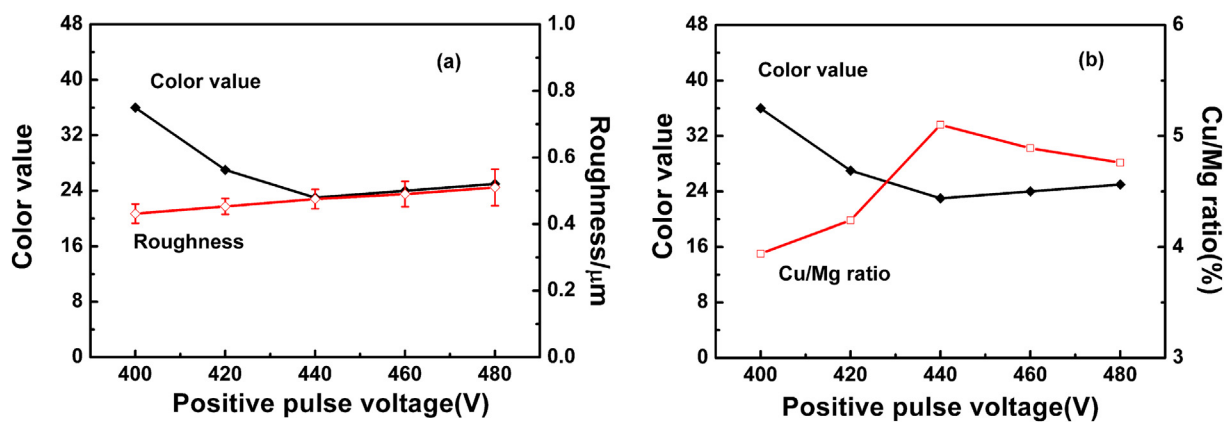


Fig. 2. The relationship of ceramic coating between color value vs. Cu/Mg ratio and roughness (a) The color value vs. the roughness; (b) The color value vs. the Cu/Mg ratio.

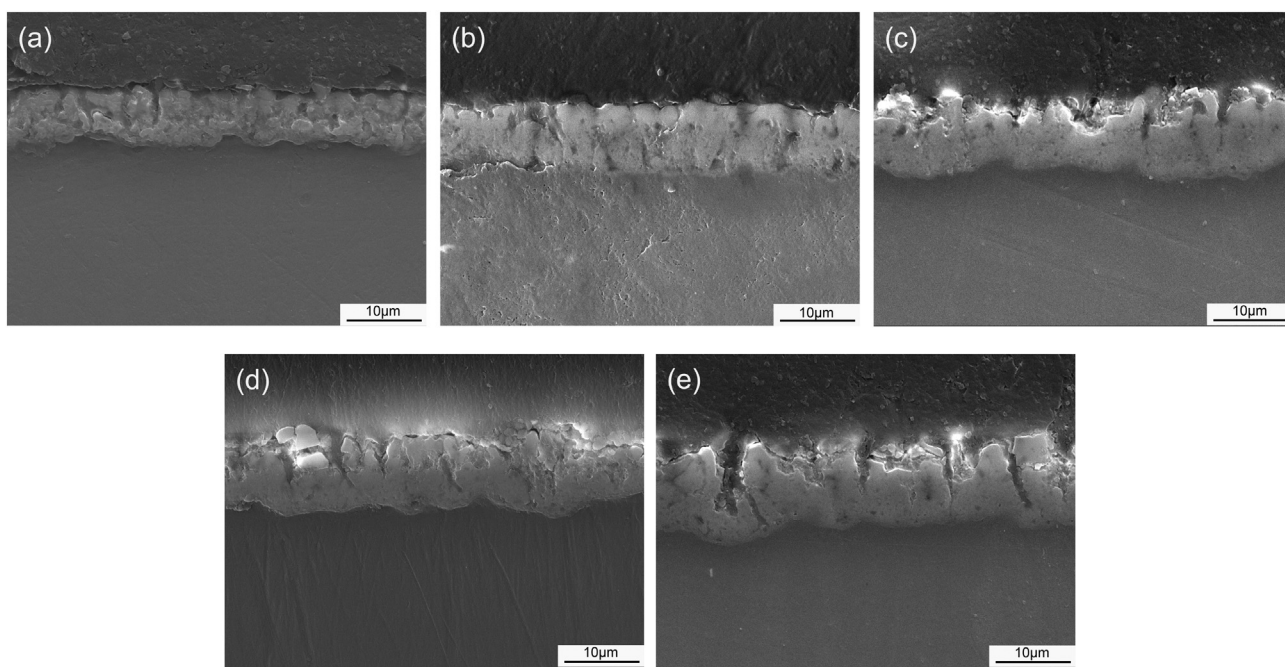


Fig. 3. The cross-section micrographs of coatings prepared under different positive pulse voltages (a) 400 V; (b) 420 V; (c) 440 V; (d) 460 V; (e) 480 V.

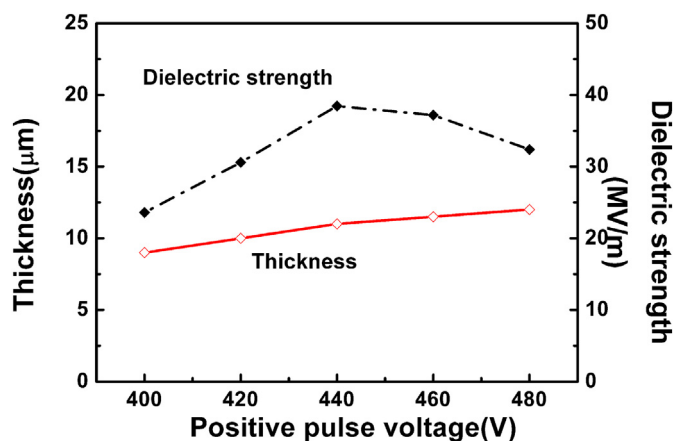


Fig. 4. The thickness and dielectric strength of ceramic coatings vs. applied positive pulse voltage value.

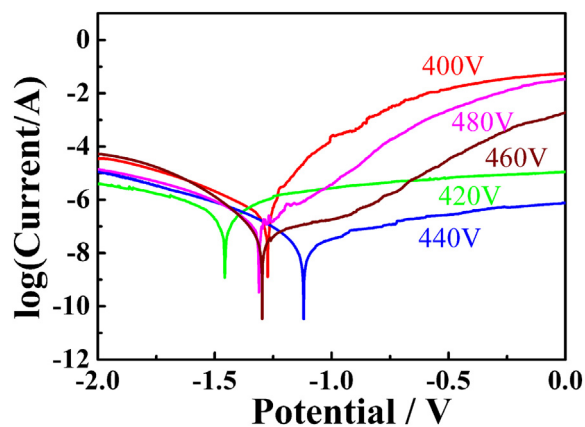


Fig. 5. The Tafel curves of samples prepared under different positive pulse voltages.

Table 2
The fitting results of samples prepared under different positive pulse voltages in 3.5 wt% NaCl aqueous solution.

Positive pulse voltage/V	400	420	440	460	480
Ecorr(V)	−1.274	−1.457	−1.297	−1.12	−1.311
Rp(Ω/cm ²)	2.272 × 10 ⁸	2.330 × 10 ⁸	8 × 10 ⁸	1.902 × 10 ⁹	3 × 10 ⁸
Icorr(A/cm ²)	2.409 × 10 ^{−7}	1.861 × 10 ^{−7}	4.427 × 10 ^{−8}	1.637 × 10 ^{−8}	1.32 × 10 ^{−7}
Corrosion rate/(mm/a)	0.00311	0.00240	0.000571	0.000211	0.00170

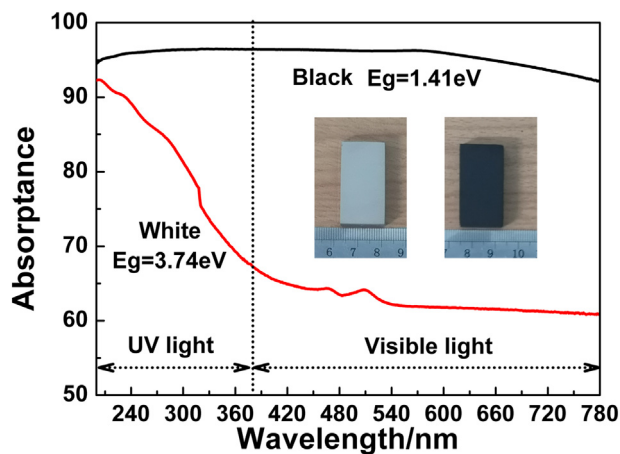


Fig. 6. UV–visible light absorption curve of different color ceramic coatings.

the thickness and compactness determine the corrosion performance of ceramic coatings.

Fig. 5 shows the Tafel curves of samples prepared under different

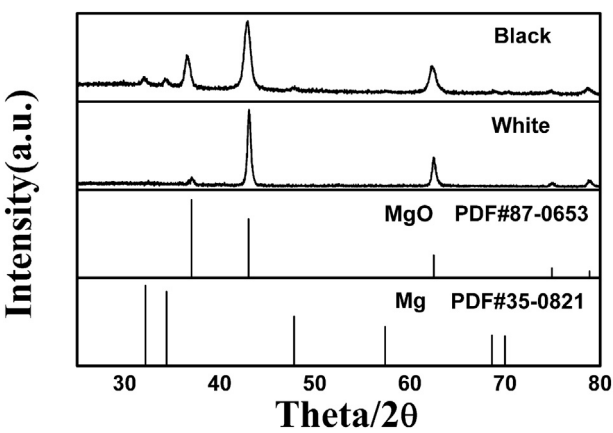


Fig. 8. XRD patterns of ceramic coating with different colors.

positive pulse voltages. Table 2 is the fitting results of samples prepared under different positive pulse voltages in 3.5 wt% NaCl aqueous solution.

With the increase of positive pulse voltage, the thickness of MAO

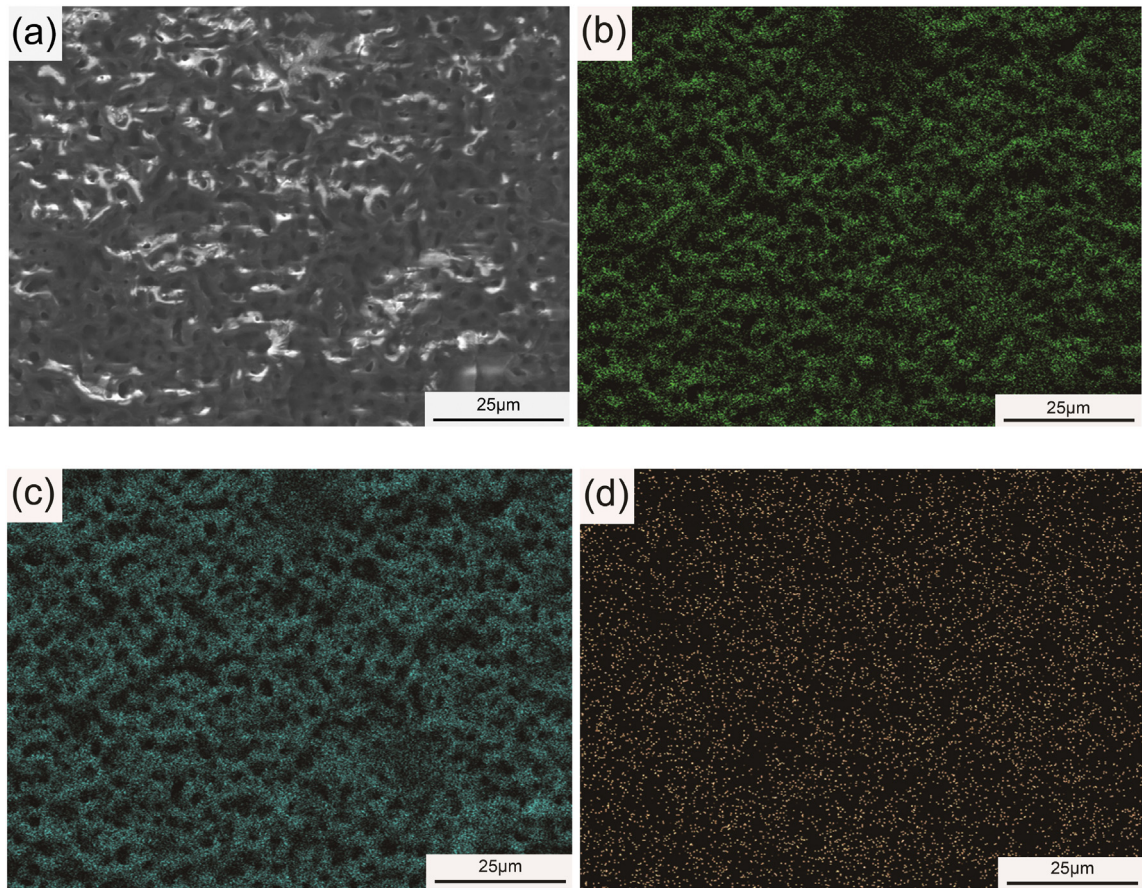


Fig. 7. Element distribution maps on the black ceramic coating (a) Scanning area; (b) O; (c) Mg; (d) Cu.

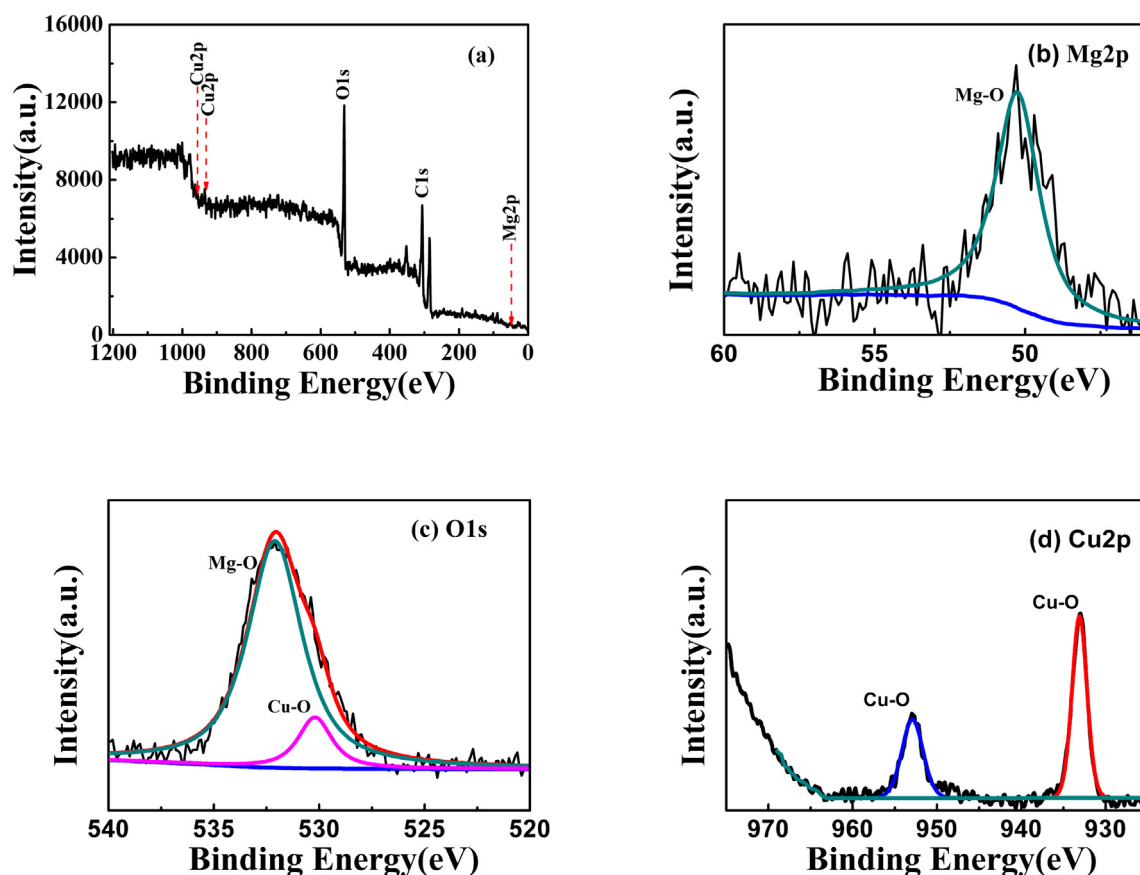


Fig. 9. XPS analysis spectrum of black ceramic coating (a)XPS spectrum; (b)Mg₂p; (c)O1s; (d)Cu₂p.

samples increases, but the polarization resistance R_p of samples increases first and then decreases. Corrosion current density and corrosion rate show opposite trends. When the positive pulse voltage is 460 V, the corrosion current density of the sample is the smallest, about 1.637×10^{-8} A/cm², the polarization resistance is the largest, and the corrosion rate is the lowest, 0.000211 mm/a. As well known, the corrosion resistance of the specimen depends on both its thickness and compactness. With the rise of positive pulse voltage, the thickness of ceramic coating increases, and its role as cathodic protection coating raises gradually. The polarization resistance of the sample decreases when the positive pulse voltage exceeds 460 V. This is due to the decrease of the compactness of the ceramic coating, which hinders the ability of the corrosive medium to pass through the ceramic coating and consequently reduces the corrosion performance. It can be seen that the effect of positive pulse voltage on the corrosion properties of MAO specimens increases first and then decreases. On the premise of the negative pulse given in this paper, the corrosion current density and rate of the sample are in the same order of magnitude when the positive pulse voltage is between 440–460 V, and the corrosion performance is optimal.

3.3. Discussion

Previous analysis shows that the solid solubility of Cu in the ceramic coating is vital to its apparent color. This is because the crystal structure, ionic valence and radius of CuO is similar to that of MgO, and the solid solution of CuO in MgO crystal structure could change the band gap and light absorption range of MgO according to the isomorphism principle of ceramics. As a result, the composite ceramic coating could show different colors.

During the MAO process, when the surface of the magnesium alloy substrate was being oxidized, the complex ions of $(CuY)^-$ in the

electrolyte was adsorbed to the surface of the sample under the effect of the applied electric field. At the same time, CuO formed on the surface of the sample. In an instant of the discharge, the temperature of the micro-plasma is as high as 8000 K–20000 K [24], and liquid phase of MgO and CuO could exist in the ceramic coating. So, the environment is satisfied for the MgO and CuO to form solid solution in theory. In addition, due to the effect of the negative pulse on the re-melting and sintering of ceramic coating, Cu ions could distribute in the ceramic coating uniformly using bipolar pulse power supply mode [25,26].

In electrolyte without Cu salt, white ceramic coating was prepared with the same process parameters. Fig. 6 is the UV-visible light absorption curve of ceramic coatings with different colors.

Fig. 6 shows that in the visible range of 380–780 nm, the light absorption ratio of black ceramic coating is up to 95%, which is much higher than that of white ceramic coating. According to Tauc-plot formula [27], the band gap of white ceramic coating, which is mainly composed of MgO, is 4.87 eV, while that of black ceramic coating is only 1.51 eV, which is far less than the band gap of 1.7 eV to absorb visible light totally. So, it indicates that the solid solution of Cu in MgO ceramic can reduce the band gap of MgO, and the color of MgO ceramic coating can also be changed. Moreover, the larger the solid solution content in the MgO is, the more the band gap of MgO decreases and the wider the ceramic coating absorb the visible light. Therefore, the ceramic coating with appropriate amount of Cu ion will show black color.

Figs. 7 and 8 are the elemental distribution maps of the MAO black ceramic coatings and XRD patterns of different color ceramic coatings, respectively.

Fig. 7 illustrate that the ceramic coating is composed of Cu, O and Mg, and they distribute in the black ceramic coating uniformly. Fig. 8 indicates that the phase composition of the ceramic the white and the black ceramic coating are all composed of MgO, but the peaks of MgO

in the black ceramic coatings are shifted to the left. The shift of the peak indicates that the lattice of MgO happens positive distortion. According to the homogeneity principle of ceramic materials, Cu^{2+} ions can replace Mg^{2+} ions in the cationic coordination octahedron of MgO crystals, thus forming the solid solution of $\text{Mg}_{1-x}\text{Cu}_x\text{O}$. However, the ionic radius of Cu^{2+} (0.073 nm) is larger than that of Mg^{2+} (0.072 nm) and makes the lattice constant of MgO increase and the peak of MgO shifts to the left inevitably. As a result, the ceramic coating with the solid solution of Cu shows black.

Fig. 9 shows the XPS analysis spectrum of the black ceramic coating.

According to the full spectrum analysis of elements in black ceramic coating, the main elements are Mg and O, and the content of Cu is very small. The Mg2p spectrum analysis of Fig. 9 (b) shows that Mg element exists in the form of MgO, which is consistent with XRD results. The two peaks of Fig. 9 (c) O1s appear at the standard binding energies 532.1 eV and 530.2 eV, respectively, corresponding to the Mg–O and Cu–O bonds. The Cu element of Fig. 9 (d) has two peaks at the standard binding energies 932.9 eV and 952.7 eV, and the difference between the two peaks is 19.8 eV, corresponding to the +2 valent Cu–O bond. The results show that the black ceramic coating by MAO is a $\text{Mg}_{1-x}\text{Cu}_x\text{O}$ substitution solid solution structure formed by the solid solution of Cu ions in MgO.

4. Conclusions

Black ceramic coating was prepared on AZ31 magnesium alloy successfully by MAO technique in this work. During the MAO process, Cu^{2+} ions were dissolved into MgO lattice to form the solid solution of $\text{Mg}_{1-x}\text{Cu}_x\text{O}$, and the color of the MgO ceramic coating could be changed by adjusting the band gap of MgO by the content of Cu^{2+} . With increasing the positive pulse voltage, the solid solubility of Cu^{2+} ions in ceramic coating increased first and then remained unchanged, and the color value decreased first and then kept constant at the same time. Among them, the minimum color value of ceramic coating was 23 when the positive pulse voltage was 440 V, and the average absorptivity of visible light was up to 95%. In addition, the too high positive pulse voltage value would decrease the compactness and corrosion resistance of the ceramic coating due to the burning loss of ceramic coating and the increased holes, although the thickness of ceramic coating was increased slightly. Therefore, for the requirement of anti-corrosion and absorption integration of magnesium alloy, the appropriate range of positive pulse voltage for micro-arc oxidation of AZ31 magnesium alloy was 440–460 V. Based on the composition and coloring mechanism of the ceramic coating, the anti-corrosion and light absorption properties of the magnesium alloy micro-arc oxidation black ceramic coating are stable.

Acknowledgements

This research was financially supported by the Key Project of Science and Technology of Shaanxi Province (Grant no: 2016JZ018), the Key laboratory project of Shaanxi Education Department (Grant no. 17JS083), and Science and Technology Innovation Project for College Students of the Ministry of Education (Grant no. 201810700066).

References

- [1] C.E. Castano, M.J. O'Keefe, W.G. Fahrenholtz, Microstructural evolution of cerium-based coatings on AZ31 magnesium alloys, *Surf. Coat. Technol.* 246 (2014) 77–84.
- [2] Z. Song, G. Yu, Z. Xie, B. Hu, X. He, X. Zhang, Performance of composite coating on AZ31B magnesium alloy prepared by anodic polarization and electroless

- electrophoresis coating, *Surf. Coat. Technol.* 242 (2014) 83–91.
- [3] Z. Song, Z. Xie, G. Yu, B. Hu, X. He, X. Zhang, A novel palladium-free surface activation process for electroless nickel deposition on micro-arc oxidation film of AZ91D Mg alloy, *J. Alloys Compd.* 623 (2015) 274–281.
- [4] Y. Gu, S. Bandopadhyay, C. Chen, Y. Guo, C. Ning, Effect of oxidation time on the corrosion behavior of micro-arc oxidation produced AZ31 magnesium alloys in simulated body fluid, *J. Alloys Compd.* 543 (2012) 109–117.
- [5] J. Han, Y. Cheng, W. Tu, T. Zhan, Y. Cheng, The black and white coatings on Ti-6Al-4V alloy or pure titanium by plasma electrolytic oxidation in concentrated silicate electrolyte, *Appl. Surf. Sci.* 428 (2018) 684–697.
- [6] G. Kou, L. Bai, P. Wang, W. Wang, Y. Wang, Color, tribological and corrosion properties of black micro-arc oxidation coating on AZ41 magnesium alloy, *J. Chin. Ceram. Soc.* 43 (2015) 991–996.
- [7] T. Arunnellaippan, L. Rama Krishna, S. Anoop, R. Uma Rani, N. Rameshbabu, Fabrication of multifunctional black PEO coatings on AA7075 for spacecraft applications, *Surf. Coat. Technol.* 307 (2016) 735–746.
- [8] L. Feng, L. Zhang, S. Li, D. Zheng, C. Lin, S. Dong, Effect of ferric citrate on microstructure and corrosion resistance of micro-arc oxidation black film on Mg-alloy AZ40M, *J. Chin. Soc. Corros. Rot.* 37 (2017) 360–365.
- [9] K. Li, W. Li, G. Zhang, P. Guo, Preparation of black PEO layers on Al–Si alloy and the coloring analysis, *Vacuum* 111 (2015) 131–136.
- [10] Z. Yao, B. Hu, Q. Shen, A. Niu, Z. Jiang, P. Su, P. Ju, Preparation of black high absorbance and high emissivity thermal control coating on Ti alloy by plasma electrolytic oxidation, *Surf. Coat. Technol.* 253 (2014) 166–170.
- [11] J. Bai, S. Li, D. Zheng, L. Zhang, L. Feng, Q. Cui, J. Wang, W. Jiang, C. Lin, Preparation and characterization of black micro-arc oxidation films, *Acta Phys.-Chim. Sin.* 32 (2016) 2271–2279.
- [12] J. Martin, A. Nominé, F. Brochard, J.-L. Brinçon, C. Noël, T. Belmonte, T. Czerwicz, G. Henrion, Delay in micro-discharges appearance during PEO of Al: evidence of a mechanism of charge accumulation at the electrolyte/oxide interface, *Appl. Surf. Sci.* 410 (2017) 29–41.
- [13] H.F. Guo, M.Z. An, Growth of ceramic coatings on AZ91D magnesium alloys by micro-arc oxidation in aluminate-fluoride solutions and evaluation of corrosion resistance, *Appl. Surf. Sci.* 246 (2005) 229–238.
- [14] Y. Zou, Y. Wang, Z. Sun, Y. Cui, T. Jin, D. Wei, J. Ouyang, D. Jia, Y. Zhou, Plasma electrolytic oxidation induced 'local over-growth' characteristic across substrate/coating interface: effects and tailoring strategy of individual pulse energy, *Surf. Coat. Technol.* 342 (2018) 198–208.
- [15] Y. Liu, S. Liu, L. Yu, J. Liu, W. Jiang, Summary on corrosion behavior and micro-arc oxidation for magnesium alloys, *J. Chin. Soc. Corros. Rot.* 35 (2015) 99–105.
- [16] C. Du, J. Chen, L. Chen, S. Lu, X. Lu, L. Xu, Effect of positive voltage on micro-arc oxidation process and coating of ZK60 Mg alloy, *Chin. J. Nonferrous Met.* 24 (2014) 1118–1126.
- [17] P. Zhang, J. Yan, W. Chen, Anode voltage on hydrogen permeation barrier performance of zirconium hydride with micro-arc oxidation, *J. Inorg. Mater.* 33 (2018) 793–797.
- [18] R.O. Hussein, D.O. Northwood, X. Nie, Processing-microstructure relationships in the plasma electrolytic oxidation (PEO) coating of a magnesium alloy, *Mater. Sci. Appl.* 5 (2014) 124–139.
- [19] R.O. Hussein, X. Nie, D.O. Northwood, An investigation of ceramic coating growth mechanisms in plasma electrolytic oxidation (PEO) processing, *Electrochim. Acta* 112 (2013) 111–119.
- [20] F. Jaspard-Mécuson, T. Czerwicz, G. Henrion, T. Belmonte, L. Dujardin, A. Viola, J. Beauvill, Tailored aluminium oxide layers by bipolar current adjustment in the plasma electrolytic oxidation (PEO) process, *Surf. Coat. Technol.* 201 (2007) 8677–8682.
- [21] Z. Yao, Y. Xu, Z. Jiang, F. Wang, Effects of cathode pulse at low frequency on the structure and composition of plasma electrolytic oxidation ceramic coatings, *J. Alloys Compd.* 488 (2009) 273–278.
- [22] R. Arrabal, E. Matykina, T. Hashimoto, P. Skeldon, G.E. Thompson, Characterization of AC PEO coatings on magnesium alloys, *Surf. Coat. Technol.* 203 (2009) 2220–2220.
- [23] Y. Shi, H. Na, Design, Preparation and Evaluation of Solar Selective Absorbing Coatings, first ed, Tsinghua University, Beijing, 2009.
- [24] B. Kasalica, J. Radić-Perić, M. Perić, M. Petković-Benazzouz, I. Belča, M. Sarvan, The mechanism of evolution of microdischarges at the beginning of the PEO process on aluminum, *Surf. Coat. Technol.* 298 (2016) 24–32.
- [25] A. Nominé, J. Martin, G. Henrion, T. Belmonte, Effect of cathodic micro-discharges on oxide growth during plasma electrolytic oxidation (PEO), *Surf. Coat. Technol.* 269 (2015) 131–137.
- [26] Y. Cheng, T. Wang, S. Li, Y. Cheng, J. Cao, H. Xie, The effects of anion deposition and negative pulse on the behaviours of plasma electrolytic oxidation (PEO)—a systematic study of the PEO of a Zirlo alloy in aluminate electrolytes, *Electrochim. Acta* 225 (2017) 47–68.
- [27] J. Kulkarni, R. Ravishankar, H. Nagabhushana, K.S. Anantharaju, R.B. Basavaraj, M. Sangeeta, H.P. Nagaswarupa, L. Renuka, Structural, optical and photocatalytic properties of MgO/CuO nanocomposite prepared by a solution combustion method, *Mater. Today* 4 (2017) 11756–11763.