E3S Web of Conferences **385**, 04015 (2023)
*ISESCE 2023*
https://doi.org/10.1051/e3sconf/202338504015

# Investigation of Chemical Mechanical Polishing Slurry of magnesium alloy

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**Abstract.** To improve the surface roughness of magnesium alloy, we researched and developed a chemical mechanical polishing (CMP) solution for magnesium alloy, and the effect of sodium tartrate on the surface quality of magnesium alloy and the mechanism of action were investigated. Through orthogonal experiments, the composition of the chemical mechanical polishing solution for magnesium alloy was determined as 50 nm particle size, 6 wt.% $Al_2O_3$ abrasive, malic acid adjusted pH=5, 1 wt.% sodium tartrate and deionized water. The polished magnesium alloy flakes were characterized by 3D white light interference profilometer and the corrosion inhibition mechanism of sodium tartrate was analysed by X-ray photoelectron spectroscopy (XPS). The surface roughness of magnesium alloy was reduced to 4.626 nm after chemical mechanical polishing in the scan range of 70 μm × 50 μm. By evaluating the valence changes of Mg and O on the surface of magnesium alloy wafers after the original surface, malic acid and sodium tartrate soaking, the reaction equation of magnesium alloy flakes in polishing solution was summarised.

## 1. Introduction

Magnesium alloy is one of the lightest metal alloys, with low density, high specific stiffness, strong resistance to electromagnetic interference, in the field of aerospace and rail transportation has been widely used, such as engine block, steering wheel bracket, seat skeleton, etc [1-2]. Magnesium alloy material performance will also exist in certain weaknesses, including poor corrosion resistance is a fatal defect of magnesium alloy material performance. Due to the magnesium alloy substrate surface usually exist inclusions porosity and other defects and uneven surface, so the surface polishing treatment before the magnesium alloy film and protection will have a great impact on the final appearance of the magnesium alloy surface after the film and protection effect, in order to obtain a high standard and high protection of the magnesium alloy surface film or coating, it is necessary to achieve a mirror surface of magnesium alloy surface through the pre-treatment high flattening effect, and chemical mechanical polishing as the best flattening effect of a surface precision processing technology can well achieve the requirements of the surface pre-treatment before the magnesium alloy film[3-4].

Chemical mechanical polishing (CMP) technology can be used to process metallic materials such as aluminium, copper, platinum, gold, tungsten, tantalum, titanium and their alloys, as well as semiconductor materials and ceramic materials. In additional, CMP technology has been widely used in the manufacturing of ultra-precise surfaces of key components and devices in the fields of aviation, aerospace, military defence, new energy vehicles, communication engineering, Internet of Things, artificial intelligence, etc. in recent years by virtue of its excellent high quality and efficiency, ultra-low damage, and stable effect. Song [5] and Zhou [6] et al. developed chemical polishing solutions for magnesium alloys containing phosphoric acid, and Fazal [7] et al. developed a chemical polishing solution for magnesium alloys consisting of nitric acid and acetic acid. Chemical polishing often adds some environmentally polluting reagents to obtain high material removal rate and surface quality, such as strong acids, strong bases, chemical reagents containing sulfur and phosphorus. These chemicals can affect the health of operators, increase equipment instability, and even have a negative impact on the ecosystem.

In this paper, based on the concept of green environment protection, AZ61M magnesium alloy is used as the research object, and the orthogonal experiment method is used to investigate the effect law of $Al_2O_3$ concentration, pH value of $Al_2O_3$ particle size polishing solution and pH adjuster type on the surface roughness (Ra) of magnesium alloy after CMP. And using the single cause control variable method, the effect law of sodium tartrate concentration on Ra after CMP of magnesium alloy was investigated, and the green CMP polishing solution of AZ61M magnesium alloy was developed. The corrosion inhibition effect of sodium tartrate on magnesium alloy was studied by X-ray photoelectron spectroscopy (XPS).

## 2. Experimental

The experimental material was AZ61M magnesium alloy with dimensions of 10 mm × 10 mm × 3 mm. The

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abrasives were $Al_2O_3$ abrasive grains (particle size 10 nm, 20 nm, 50 nm, 100 nm), pH regulators were niacin, citric acid, malic acid, oxalic acid, and corrosion inhibitors were sodium tartrate.

Polishing of magnesium alloy flakes consists of two parts: mechanical grinding and chemical mechanical polishing. Mechanical grinding can remove the damaged corrosion layer of magnesium alloy flakes in a short time and reduce the processing time of CMP process. Three magnesium alloy flakes which have been wire cut are passed through molten paraffin and fixed uniformly at three equal points of the carrier disc. The magnetic resin diamond grinding disc was used on the UNIPOL-802 polishing machine and polished at a polishing speed of 90 rpm for 5 minutes. After grinding, the magnesium alloy sheet was cleaned with alcohol and blown dry with compressed air.

After grinding, polishing was carried out using abrasive leather polishing pads. $Al_2O_3$ abrasive grains and deionized water were configured into a certain mass fraction of polishing solution. pH adjuster was used to adjust the polishing solution to the desired pH value, and finally a certain amount of sodium tartrate was added and fully ultrasonicated for 1 h to obtain a green and environmentally friendly polishing solution. After the CMP test, the magnesium alloy sheet was repeatedly rinsed with a large amount of deionized water and anhydrous ethanol, and finally blown dry with compressed air. The material removal rate is calculated using Equation 1.

$$\delta_{MRR} = \frac{\Delta m \times 10^4}{\rho S t} \quad (1)$$

$\delta_{MRR}$ is the material removal rate (μm/min); Δm is the mass difference of magnesium alloy flake before and after polishing (g); S is the area of magnesium alloy flake ($cm^2$); ρ is the density of magnesium alloy flake (about 1.8 $g/cm^3$); t is the polishing time (min).

To investigate the corrosion inhibition of sodium tartrate, a solution was prepared with the addition of malic acid and sodium tartrate. 5 mm × 5 mm × 3 mm magnesium alloy sheets were placed in the solution and soaked, and after 24 hours, the magnesium alloy was removed and blown dry with compressed air. An XPS instrument (Thermo Scientific K-Alpha) was used to excite the atomic or molecular inner layers or valence electrons of the magnesium alloy flakes using Al Kα rays as the excitation source, and the excited photoelectrons were detected to analyse the composition of the film formed on the surface of the magnesium alloy and thus to analyse the corrosion inhibition mechanism of sodium tartrate.

## 3. Results and Discussions

### 3.1 Orthogonal test

There are many factors affecting the results of chemical mechanical polishing, we selected four main factors of abrasive particle size (A), abrasive concentration (B), pH adjuster type (C) and polishing solution pH (D) as the research objects; according to the number of factors and levels of the experiment, the orthogonal table $L16(4^4)$ (Table 1) for orthogonal test to study the effect of process parameters on surface roughness. Where, L denotes the orthogonal table; 16 denotes that 16 orthogonal experiments are required; 4 denotes that each factor contains 4 levels; 4 denotes that up to 4 levels can be arranged in the orthogonal table. The flow rate of polishing solution was 10mL/min and the polishing time was 0.5 h. The surface roughness and material removal rate after polishing were chosen as evaluation indexes, and the results of surface roughness and material removal rate of magnesium alloy after chemical mechanical polishing were shown in Table 1. The three-dimensional morphology of magnesium alloy surface after polishing in each group of orthogonal experiments is shown in Figure 1. The mean values of roughness and material removal rate $K_{ij}$ can simply visualize the trend of the influence of each experimental level on the experimental results under different factors. The size of the extreme difference $R_j$ can be used to measure the size of the corresponding factors in the experiment on the degree of the effect of chemical mechanical polishing of magnesium alloy, the factor with a large extreme difference means that its four levels of change on the results of polishing magnesium alloy caused by a relatively large impact, usually an important factor; and the factor with a small extreme difference means that its various levels of impact on the experimental results is relatively small, usually a non-important factor. According to the experimental results of roughness and material removal rate in Table1, the mean value and extreme difference of each factor were calculated, and the calculation results are shown in Table 2.

Table 1. Ra and $\delta_{MRR}$ of each orthogonal CMP experiment for magnesium alloy.

| Number | Size (nm) | $Al_2O_3$ (wt.%) | Type | pH | Ra (nm) | $\delta_{\Delta MRR}$(μm/min) |
|---|---|---|---|---|---|---|
| 1 | A1（10） | B1（1） | C1(Nicotinic acid) | D1(6) | 5.201 | 1.73 |
| 2 | A1 | B2（3） | C2(Citric acid) | D2(5) | 4.88 | 1.92 |
| 3 | A1 | B3（6） | C3(Malic acid) | D3(4) | 4.747 | 2.18 |
| 4 | A1 | B4（9） | C4(Oxalic acid) | D4(3) | 6.869 | 2.82 |
| 5 | A2（20） | B1 | C2 | D3 | 5.432 | 1.68 |
| 6 | A2 | B2 | C1 | D4 | 6.522 | 2.27 |
| 7 | A2 | B3 | C4 | D1 | 5.641 | 2.45 |
| 8 | A2 | B4 | C3 | D2 | 4.47 | 2.46 |
| 9 | A3（50） | B1 | C3 | D4 | 5.352 | 2.13 |
| 10 | A3 | B2 | C4 | D3 | 6.75 | 2.53 |
| 11 | A3 | B3 | C1 | D2 | 5.363 | 2.43 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 12 | A3 | B4 | C2 | D1 | 5.389 | 1.74 |
| 13 | A4（100） | B1 | C4 | D2 | 6.563 | 2.67 |
| 14 | A4 | B2 | C3 | D1 | 5.471 | 2.04 |
| 15 | A4 | B3 | C2 | D4 | 5.512 | 2.14 |
| 16 | A4 | B4 | C1 | D3 | 6.528 | 2.42 |

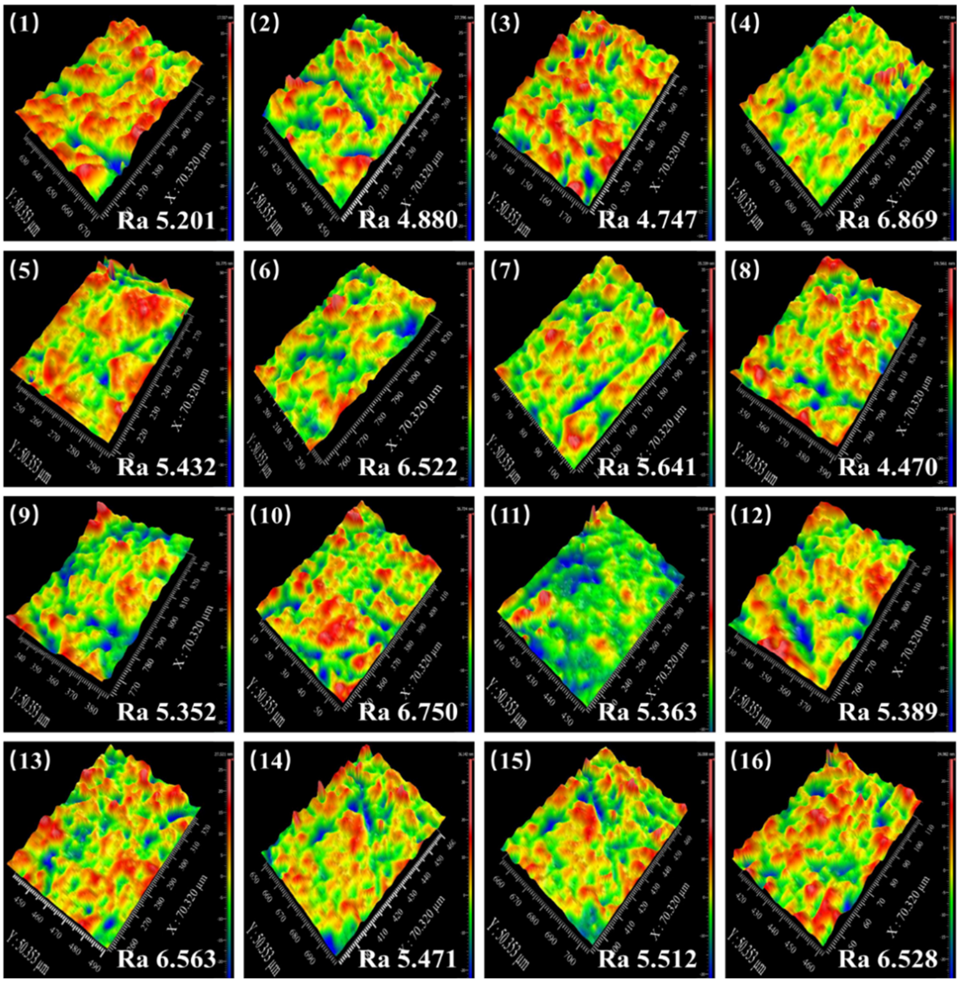


Figure 1. Surface roughness and topographies after orthogonal CMP experiments on the surface of magnesium alloy.

Table 2. Range, average Ra and $\delta_{MRR}$ of each factors of orthogonal experiments

| | Ra (nm) | | | | $\delta_{MRR}$(μm/min) | | | |
|---|---|---|---|---|---|---|---|---|
| | A | B | C | D | A | B | C | D |
| $K_{1j}$ | 5.42425 | 5.637 | 5.9035 | 5.4255 | 2.1625 | 2.0525 | 2.2125 | 1.99 |
| $K_{2j}$ | 5.51625 | 5.90575 | 5.30325 | 5.319 | 2.215 | 2.19 | 1.87 | 2.37 |
| $K_{3j}$ | 5.7135 | 5.31575 | 5.01 | 5.86425 | 2.2075 | 2.3 | 2.2025 | 2.2025 |
| $K_{4j}$ | 6.0185 | 5.814 | 6.45575 | 6.06375 | 2.3175 | 2.36 | 2.6175 | 2.34 |
| $R_j$ | 0.59425 | 0.59 | 1.44575 | 0.74475 | 0.155 | 0.3075 | 0.7475 | 0.38 |

For visualization, the trend graph between factors and indicators was plotted with the factor level as the horizontal coordinate and the mean values of the indicator roughness and material removal rate as the vertical coordinates, as shown in Figure 2.

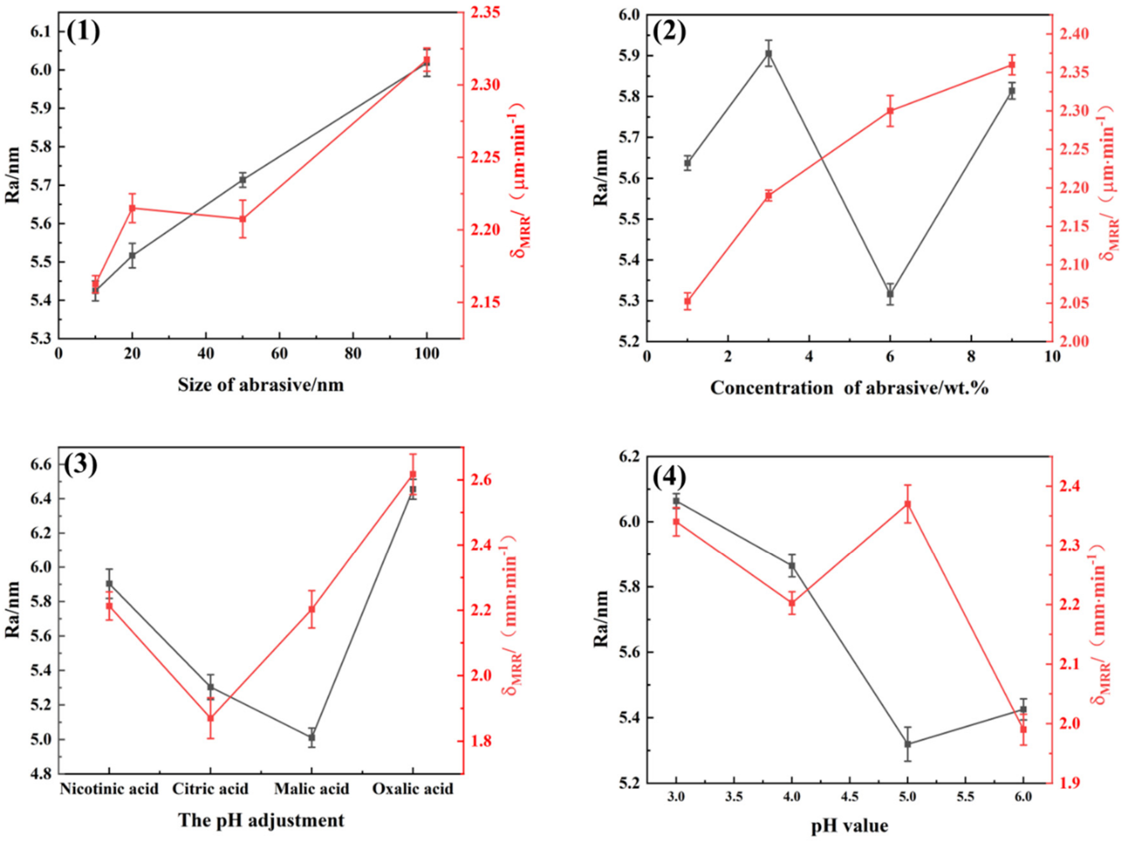


Figure 2. Effect of four factors on the mean value of roughness and mean value of material removal rate of magnesium alloy

As shown in Figure. 2 (1) ~ (4) the mechanical effect was enhanced with the increase of $Al_2O_3$ abrasive particle size, resulting in an increasing trend of Ra and $\delta_{MRR}$. With the increase of $Al_2O_3$ concentration, Ra showed a trend of decreasing and then increasing, and $\delta_{MRR}$ showed an increasing trend of mechanical action, leading to the lowest value of Ra when the $Al_2O_3$ concentration was 6 wt.%. When the $Al_2O_3$ concentration was low, the chemical action was stronger than the mechanical action, and $\delta_{MRR}$ was low but Ra was high, and with the increase of $Al_2O_3$ concentration, the mechanical action was enhanced, $\delta_{MRR}$ increased and Ra decreased, but when the $Al_2O_3$ concentration was too high, the mechanical action was stronger than the chemical one, resulting in high $\delta_{MRR}$ but elevated Ra. With the increase of pH, Ra showed a trend of decreasing and then increasing, and $\delta_{MRR}$ showed a trend of decreasing. Ra was lowest when the pH regulator was malic acid. When the pH was 5, Ra was the lowest and cut $\delta_{MRR}$ was higher. Considering the influence of four factors on the surface quality and material removal rate of magnesium alloy after polishing, the main reference factor should be to reduce the surface roughness, so the magnesium alloy polishing solution is pH regulator for malic acid, polishing solution pH is 5, abrasive particle size is 20nm, concentration is 6wt.%.

## 3.2 XPS analysis

In the CMP process, the mechanical grinding effect of $Al_2O_3$ abrasive grains and the oxidative corrosion effect of chemical reagents are mainly included. In order to investigate the corrosion inhibition effect of sodium tartrate, the soaked treated magnesium alloy flakes were subjected to XPS analysis, and their full and fine spectra are shown in Figure 3.

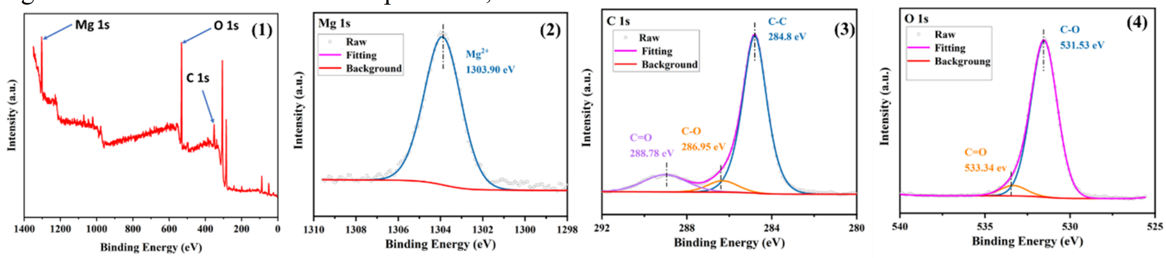


Figure 3. The XPS spectra of magnesium alloy in slurry with sodium tartaric

From Figure 3 to see, the surface film formed by the solution containing sodium tartrate soaked mainly contains Mg, O, C and other elements. Sodium tartrate is hydrolyzed into tartrate ions and sodium ions in the solution, and the tartrate ions have excellent chelating properties, which can chelate the free magnesium ions in the solution and make them form an oxide film on the surface of magnesium alloy. The chemical reaction formula is shown in equations (2)-(4).

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

$$C_4H_4O_6Na_2 \rightarrow {C_4H_4O_6}^{2-} + 2Na^+ \quad (3)$$

$${C_4H_4O_6}^{2-} + Mg^{2+} \rightarrow C_4H_4O_6Mg \quad (4)$$

## 4. Conclusion

In this paper, a green environmentally friendly chemical mechanical polishing solution consisting of deionized water, 6 wt.% $Al_2O_3$, 1 wt.% sodium tartrate and malic acid adjusted pH=5 with polishing pressure of 22 kPa and polishing disc speed of 60 r/min was proposed by orthogonal experiments. after CMP of magnesium alloy, Ra was reduced to 4.626 nm in the scanning range of 70 μm×50 μm. By magnesium the valence changes of Mg and O on the surface of magnesium alloy wafers after the original surface, malic acid adjusted pH=5 and 1 wt.% sodium tartrate immersion, the reaction equation of magnesium alloy wafers in the polishing solution in CMP was summarized, and the process mechanism of material removal from the surface of magnesium alloy wafers achieved by the dual action of chemical corrosion and mechanical grinding was explained.

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