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Preparation and Performance Study of Chemical Mechanical Polishing Slurry for New-type Aluminium Alloy

Yali Zhao, Genjie Yu, Yamin Li, Guixia Wang

Mechanic and Electronic Engineering, Xinlian College, Henan Normal University, Zhengzhou 451400, China

zhaosil@126.com

Aluminium alloys are widely used in various industries because of their lightness and easy processing, leading to higher requirements for aluminium alloys in terms of the flatness and finish of aluminium alloy surface. In this paper, chemical mechanical polishing (CMP) technology, which combines the advantages of chemical and mechanical polishing techniques, was studied to prepare one slurry suitable for new-type aluminium alloy and test its polishing performance. CeO2 powders were obtained as abrasive particles by a series of processes such as firing the mixture of NH4HCO3 and CeCl3 by precipitation method. The optimum ratio of the slurry included: 2.5% cerium oxide abrasive particles, NaOH as a pH adjuster at the optimal pH 10, and 3% sodium polyacrylate as the dispersant, so as to mix the CMP slurry with good dispersibility and stability. Then, the performance evaluation test of polishing was made by using the CMP slurry prepared under the optimal conditions on the aluminium alloy. The results showed that the polishing efficiency of the CMP slurry was higher, and the surface flatness, finish and roughness after polishing were in conformity with the requirements about aluminium alloy surface precision, with good polishing effect.

1. Introduction

The reserves of aluminium in nature are second only to oxygen and silicon. Aluminium and its alloy products are widely used in various sectors and industries such as aerospace, transportation, and structural materials etc. The greatest advantages of aluminium and its alloys are light and easy to process, but it also has defects such as soft texture and low hardness (Mondolfo, 1976; Ma, 2012). From the perspective of the protection and aesthetics of the alloy surface, it is necessary to polish the tool made of the aluminium alloy. Polishing technology can improve the surface smoothness, finish, and certain mechanical properties of aluminium and aluminium alloy products (Urbano et al., 2014; Zhao et al., 1995; Hryniewicz et al., 2007). This is of great significance to improve the added value of aluminium and aluminium alloy products.

The common polishing methods include chemical polishing, electrolytic polishing, and mechanical polishing etc. Chemical polishing is the use of polishing slurry to selectively dissolve the surface of the alloy to achieve a bright and smooth surface, and to enhance the mechanical strength of the alloy by improving the mechanical damage layer of the alloy; mechanical polishing is the process of surface smoothing by mechanically polishing the protruding part of the tool using the polishing machine; electrolytic polishing utilizes the electrolysis of the electrolyte to dissolve the protruding part of the alloy surface. Mechanical polishing is only applicable to alloy products with simple shapes, electrolytic polishing requires the huge equipment and funds, and the chemical polishing is easy to operate with low cost and wide application range, but with high corrosivity (Sun et al., 2009; Sun et al., 2004; Lee et al., 2010; Zhang et al., 1989). In this paper, the chemical mechanical polishing (CMP) technology, which combines the advantages of chemical and mechanical polishing techniques, was studied to prepare a polishing slurry suitable for the new-type aluminium alloy and tested for its polishing performance (Sheu et al., 2012).

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2. Preparation of new-type chemical mechanical polishing slurry

2.1 Composition and function of chemical mechanical polishing slurry

Chemical mechanical polishing (CMP) combines the advantages of chemical polishing and mechanical polishing. It is currently the only surface finishing technology capable of achieving overall planarization (Luo and Dornfeld, 2001; Ahmadi and Xia, 2001; Currie et al., 1998; Park et al., 2000. The single mechanical polishing can obtain a high surface consistency but damage the surface and reduce the surface finish; the single chemical polishing can achieve a higher surface finish despite of the high polishing rate and low surface damage, but the overall surface of the metal after polishing has lower flatness; therefore, the combination of both can improve the surface flatness and have a high surface finish.

The slurry is the core of this technology. Its performance and ratio have a decisive influence on the quality of the alloy surface to be processed. It is mainly composed of three major parts: the abrasive particle, corrosive medium, and additive, which functions are shown in Figure 1 below.

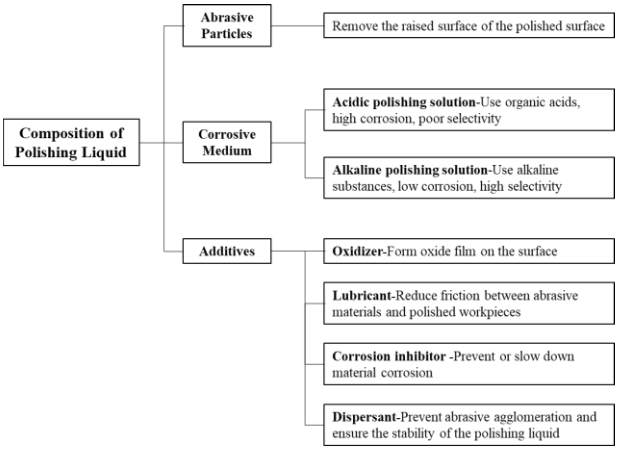


Figure 1: The composition and function of the polishing liquid

2.2 Preparation process of chemical mechanical polishing slurry

2.2.1 Test materials

Table 1 below lists the test materials.

Table 1: Test materials

Reagent	Specification	Function
Ceria	Industry Grade	Raw Material
Ammonium Bicarbonate	Industry Grade	Raw Material
Ammonia	Analytical Purity	PH Regulator
Anhydrous Ethanol	Analytical Purity	Detergent
Organic Base	Analytical Purity	PH Regulator
Sodium Hydroxide	Analytical Purity	PH Regulator
SDBS	Analytical Purity	Dispersant
Sodium Polyacrylate	Analytical Purity	Dispersant
Hexametaphosphate	Analytical Purity	Dispersant
Hydrogen peroxide	Analytical Purity	Oxidizer

2.2.2 Experimental technology and process

The preparation process of this experimental CMP slurry was mainly divided into two steps:

(1) Preparation of cerium oxide abrasives: In this experiment, CeCl3 and NH4HCO3 were used to synthesize precursors through the process of precipitation and aging, and then the precursors were calcined to prepare CeO2 powders. During the preparation process, attention should be paid to the effect of solution concentration, pH, and temperature on the particle size of the abrasive.

(2) Preparation of CMP slurry: The polishing liquid was prepared by using the obtained abrasive material in step 1 that meets the application requirements of the polishing material. The polishing effect of the CMP slurry

was determined by the synergistic effect of the chemical action and the mechanical action, so the composition and content of the polishing slurry played a decisive role in the final polishing effect. During the preparation of the slurry, the stability and dispersibility of the CMP slurry were ensured by adding the dispersant and pH adjuster etc.

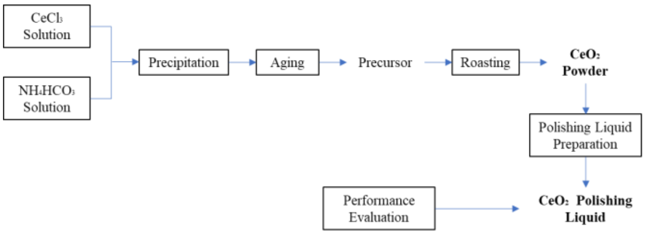


Figure 2: Process flow diagram

3. Chemical mechanical polishing slurry formulation

After repeated research and comparison of the preparation process, the powder of slurry with better crystallization was obtained by controlling the ratio of raw materials, the time and temperature of firing etc. The primary particle size of the powder was less than 100 nm, with the uniform size distribution. So, it can be used for the next-step preparation of the slurry. The ratio of the polishing slurry directly affects the final polishing rate and efficiency. In this chapter, stability, suspension property and polishing efficiency of the polishing slurry were improved by adding other chemical substances.

3.1 pH value determination

The proper pH value can improve the stability of the polishing slurry. Only when the polishing material has related requirement for the pH value, the polishing efficiency of the slurry can be ensured. In the experiment, the stability of the slurry was measured by measuring the Zeta potential. The greater the absolute value of the Zeta potential, the better the stability of the slurry. Therefore, the appropriate pH is determined by measuring the Zeta potential of the polishing slurry at different pH values.

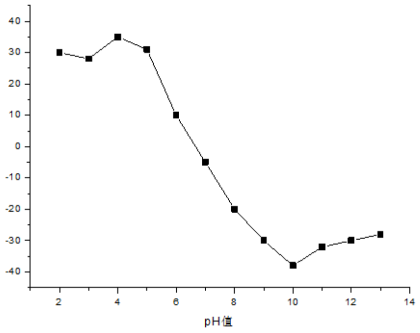


Figure 3: The relationship between Zeta potential and pH value of CeO_2 suspension

The experimental results (Figure 3) above show that the absolute value of the Zeta potential approaches zero when the pH is between 6 and 7, that is, the neutral environment. When the pH is around 4 and 10, the absolute values of the Zeta potential reach maximum value in the acidic and alkaline states respectively, so the overall stability is best when the pH of the entire slurry is around 4 or 10. However, at 4 pH value, the polishing slurry is acidic with too strong corrosive effect. Thus, the alkaline polishing slurry with a pH of 10 is preferred in this paper.

3.2 pH adjuster selection

After determining the alkaline polishing slurry, the NaOH, P3 organic base and ammonia water were selected to conduct experiments. They were diluted and then the slurry was added when adjusting its pH to 10. Finally, the stability of the suspension liquid was observed through the sedimentation rate.

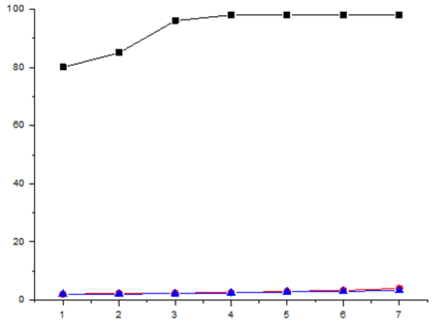


Figure 4: The relationship between sedimentation rate and the time

Figure 4 shows the precipitation of three suspensions with different pH adjusters over time, in which P3 organic base completely settled in a short period of time, while the sedimentation rate of NaOH and ammonia as the adjuster for the polishing liquid remained relatively low; the sedimentation rate after 7 days did not exceed 5%, but the ammonia is volatile, easy to cause environmental pollution, so the pH adjuster NaOH was selected in this paper.

3.3 Selection of dispersant type

The dispersant is added to change the force between the suspended particles in the polishing slurry o ensure the stability of the entire slurry. Five dispersants were selected to be added to the suspension at 10pH value containing NaOH as the pH adjuster. Then, through observing the corresponding sedimentation rate, the most suitable dispersant could be chosen. The lower the sedimentation rate, the more stable the suspension system is.

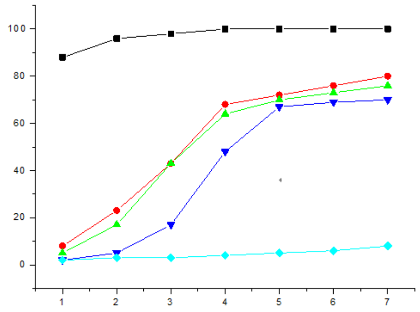


Figure 5: Different type of dispersant suspension stability

The experimental results in Figure 5 show that the sedimentation rate of sodium polyacrylate remains low for a period of time and is a suitable dispersant; that of sodium dodecyl benzene sulfonate at the beginning is already close to 100 %, with poor dispersion effect; the other three PEG4000, PEG6000 and sodium hexametaphosphate all have similar sedimentation patterns, and their sedimentation rate gradually increases to about 80%. Therefore, the effect of sodium polyacrylate on the surface of cerium oxide is better, which can ensure the stability of polishing slurry for a long time. It is a good dispersant.

3.4 Determination of dispersant dosage

Sodium polyacrylate was used as a dispersant and the polishing slurry was mixed with 1 to 9% proportion to observe the sedimentation rate of the suspension system. The results in Figure 6 shows that when the dispersant content was 1%, the sedimentation rate was higher due to the insufficient dispersant dose. When the dispersant dose was added to 3%, the sedimentation decreased and remained at a low level, which has the same effect with more than 3% of the dose of dispersant. So, 3% dispersant content was used in this paper.

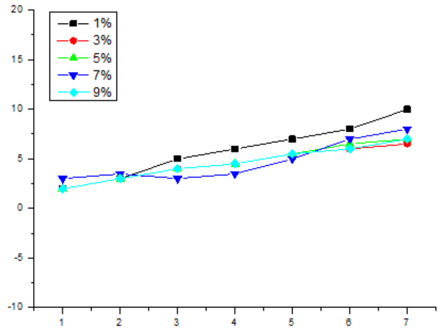
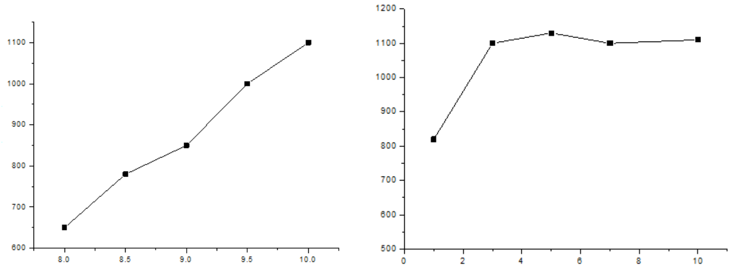


Figure 6: Suspension stability of the different amount of dispersant

4. Performance evaluation of chemical mechanical polishing slurry

4.1 Effect of slurry on polishing removal rate

The effect of pH and abrasive content on the polishing rate was determined by polishing experiments on new type aluminum alloys with different pH and abrasive contents. The experimental results in Figure 7 (a) shows that with the increase of pH, the polishing rate is also in the linear growth, and at the pH of 10, the polishing rate reaches the highest, with the best dispersion performance, so the pH is set to be 10. In Figure 7 (b), the different abrasive contents have little effect on the polishing rate, and the polishing rate can be kept high at 2.5%, which can ensure sufficient chemical mechanical polishing.

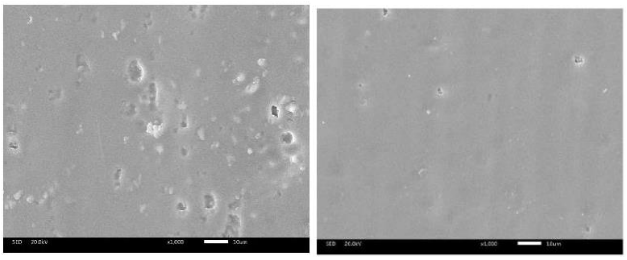


(a) (b)

Figure 7: Removal rate of the polishing solution of different PH (a) and abrasive content (b)

4.2 The effect of the slurry on polished surface

The polishing slurry prepared under the optimal conditions was used to polish the new-type aluminum alloy. Scanning Electron Microscope (SEM) was adopted to take the pictures before and after polishing (Figure 8). It is found that after polishing, the surface of the aluminum plate is smooth with better polishing effect.



(a) (b)

Figure 8: The polishing comparison SEM pictures before (a) and after (b)

Atomic force microscope (AFM) can be used to study the surface results of solid materials. The polishing effect of the polishing slurry was observed and judged by testing the AFM of the new-type aluminum alloy before and after polishing. The data of AFM before and after polishing are shown in Table 2 and Figure 9.

Table 2: The data of AFM pictures

Picture	Rp/nm	Rm/nm	Ry/nm	Ra/nm
a	653.661	-1085.658	1808.209	183.688
b	120.028	-118.534	242.605	20.564

From the figures and data above, it can be found that the roughness of the aluminium alloy surface is greatly reduced after polishing, and the surface roughness is reduced to less than 20% of the original, which greatly lowers the surface roughness of the aluminium alloy and has a good polishing effect.

5. Conclusion

In this paper, the preparation process and polishing performance evaluation were studied for the CMP slurry of new-type aluminium alloy. The specific conclusions are as follows:

Acknowledgement

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