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# Chemical action in CMP 304 stainless steel based on hydrogen peroxide slurry[1]

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**Abstract.** The 304 stainless steel material has become one of the flexible display substrates. Chemical mechanical polishing (CMP) will be one of the most practical machining technologies to achieve super-smooth and damage free on the material surface of stainless steel. The CMP slurry is one of the most important parts in CMP. The oxidant and pH regulator are the most important substances in polishing slurry. So, in this paper, the influence of the content of oxidant the hydrogen peroxide, the pH value and the pH regulator on the material removal rate (MRR) and surface roughness had been studied, respectively, by experiment and analysis. The results show, that the material removal rate and surface quality obtained in the acidic environment were higher than that of in the alkaline environment in CMP 304 stainless steel. The nitric acid and the sodium hydroxide as pH regulator, when used alone in slurry had strong oxidizing, and could increase the material removal rate and improved surface quality. The hydrogen peroxide had a strong oxidation resistance and the action of electrochemical corrosion, and also can produce Fenton type Haber-Weiss reaction with the stainless steel base material, so it could achieve the removal of materials. In the six kinds of pH regulators, the material removal rate reaches maximum value when using the oxalic acid as the pH regulators at pH=2. These research results in this paper can provide a reference for further research the polishing slurry on CMP stainless steel.

**Key words.** Chemical mechanical polishing, 304 stainless steel, slurry, hydrogen peroxide, material removal rate, surface roughness..

## 1. Introduction

Flexible display have the many good performances with ultra-thin, lightweight, durable, large storage capacity, design freedom, flexible, rolling and impact resistant,

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and so on [1]–[2], and it widely used in industrial, civil and military industries in the mobile phones, the personal digital assistant PDA, the notebook computer, the trademarks, the secure identity documents, the e-books, the electronic posters, the car dashboard, the sensor, the environment display, the general lighting, the palpable skin of industrial robot, and so on [3]–[4]. Many research institutions and manufacturers in many countries and regions had joined into the research and application of flexible display technology, because of its huge market prospects [5]–[6]. In recent years, many companies had produced out the foldable or flexible OLED screen continuously [7].

The substrate of flexible display must be a flexible material. Requirements for the flexible substrate on the surface quality and performance are very strict. The surface roughness $R_\mathrm{a}$ of the flexible substrate must be less than 5nm and its waviness must be less than $0.1\,\mu$m. The material of the flexible substrate should have the high thermal stability, high strength with lightweight, thin, high flexibility, toughness and so on. Because the 304 stainless steel materials have the performance abovementioned and the low cost, therefore, it will become the main substrate material in large size flexible display in the future [8]–[9]. But the machining quality and precision of the ultra-thin stainless steel sheet will directly affect the performance of the flexible display device [10]. When the surface of the thin stainless steel sheet has tiny defects, it will be genetic onto the film in the epitaxial growth and lead to the fatal defects on the device. Therefore, how to effectively obtain the large size flexible display substrate with the high quality and the high precision and meet the requirements for flexible displays in the present and future, this is a first priority in the flexible display industry at present [11].

Nowadays, many domestic and foreign scholars have studied largely and deeply on the polishing technology of the stainless steel material. The main polishing methods are the mechanical polishing, the chemical polishing, the electrochemical polishing and the electrochemical mechanical polishing and so on [12]. Not only the surface roughness after polishing with the mechanical polishing, the chemical polishing and the electrochemical polishing was often not to meet the requirements, but also the depth of the damage layer was lager [13]–[14]. The device of electrochemical mechanical polishing of stainless steel is very complex, and the surface quality is very difficult to control because of affecting on the current flow characteristics [15].

Chemical mechanical polishing (CMP) technology has been considered as the best method to meet both the surface roughness and surface flatness and has become one of the most useful processing techniques to achieve hard brittle crystal materials with the surface with ultra smooth and damage free. It has been widely used in large scale integrated circuit, semiconductor lighting and other fields [16]. The CMP technology may be the most suitable method to use in the high efficiency and the ultra precision machining for large-size ultra-thin stainless steel flexible display substrate, and to obtain the surface with ultra smooth and damage free.

Referring to the most Chinese and foreign literatures, there are a few literatures about the study of chemical mechanical polishing stainless steel. The literature [17] had studied the influence of the temperature on the polishing surface quality in chemical mechanical polishing of stainless steel. Our research group [18]–[20] were studied the chemical mechanical polishing of stainless steel in CMP slurry and CMP process, respectively. There is no other literature to be found to report for machining the stainless steel using the chemical mechanical polishing method.

CMP slurry is one of the most important components in the chemical mechanical polishing. The slurry cost is 60 % $\sim$ 70 % accounting for the total cost of chemical mechanical polishing. The ingredients of CMP slurry will determine the efficiency, the quality and the cost of chemical mechanical polishing. Therefore, it is urgent to study CMP slurry of stainless steel with the environmental protection and high efficiency. The oxidant and pH regulator are the most important chemical composition in chemical mechanical polishing slurry. It will react with the surface material of the workpiece and generate a layer of chemical reaction film with a lower binding energy on the workpiece surface, and then removed by the mechanical action in CMP. The CMP process will alternate and cycle to realize the planarization of the workpiece surface.

In order to study the CMP slurry with the environmental protection and the high efficiency, in this paper, a series of experiments on the MRR and surface roughness had been done using the oxidant hydrogen peroxide under the different content, the different pH value in CMP stainless steel, respectively. The best pH regulator and the best pH value in CMP slurry have been obtained in CMP stainless steel. This will provide the reference for the further study on the slurry of CMP stainless steel.

## 2. Experimental preparation and experimental parameters

### *2.1. Experimental preparation*

All the chemical mechanical polishing experiments were carried out in the clean room with grade 1000, and the environmental temperature was controlled at 22 °C, and the water used in the all experiments was DI water with the electrical resistivity 18.24 MΩ · cm. Many of experimental samples for 304 stainless steel sheet with 3 mm in thickness and 50 mm in diameter was used in chemical mechanical polishing experiments. Before the experiment, these samples were fine lapped and the surface roughness $R_a$ is in 40 nm to 50 nm after lapping. The CMP experiments were carried out on the polishing machine ZYP300 type produced by Shenyang. The type of CMP pad used is Rode IC1000.

### *2.2. Selection of CMP parameters*

The rotational speed of the polishing platen and the carrier is set 60 rpm, the CMP pressure $P$ is set to 2 psi, and the CMP time is set to 15 min. The polishing pad must be conditioned every time after CMP and conditioning time is set to 15 min. In the CMP process, the carrier oscillates reciprocating along the arc with the oscillation frequency 10 s and the oscillation amplitude 20 mm. The center distance between the carrier and polishing platen is set to 80 mm.

### *2.3. Testing instruments*

Testing instruments used in the experiment. The precision balance (accuracy 0.01 mg) of Sartorius CP225D was used to test the weight of the sample before and after CMP, and then the material removal rate can be calculated by the weight removed. The 3D microscope Contour GT-K (vertical resolution 0.01 nm) produced by BRUKER corporation of the United States was used to measure the surface roughness and surface morphology of the samples before and after CMP. The metallographic microscope with Lecia DM2500M was used to detect the 2D surface original image before and after CMP. The laser particle size distribution instrument with JNGX JL-1197 was used to test the distribution of abrasive in polishing slurry. The pH electronic test pen (precision 0.1) was used to test the pH value of slurry.

## 3. Experimental results and analysis

### *3.1. Influence of pH value on MRR and surface roughness*

According to the characteristics of the 304 stainless steel material and the orthogonal test results of polishing slurry former researched, the content of each ingredient in CMP slurry sees Table 1. The white corundum is selected as the experimental abrasive, the nitric acid and the sodium hydroxide was selected to adjust pH value of slurry on the level of 2, 4, 6, 9, 11 and 13, the glycerol was selected as the dispersant. A kind of slurry with different content in the total volume 250 ml was confected before each experiment. By Table 1, the testes have divided into 5 groups, and each group has 6 kinds of slurries by different pH value.

Table 1. The basic ingredient and the content of polishing slurry

| Group number | pH value | Abrasive diameter ($\mu$m) | Dispersant (g) | Oxidant (g) | Content of abrasive (g) | Deionized water |
|---|---|---|---|---|---|---|
| 1 | | | | 5 | | |
| 2 | | | | 10 | | |
| 3 | X | 3.5 | 3 | 15 | 4.5 | Others |
| 4 | | | | 20 | | |
| 5 | | | | 25 | | |

**Note.** In the table, X represents 2,4,6,9,11,13.

Figure 1 shows these results of CMP 304 stainless steel using polishing slurry with oxidant hydrogen peroxide. By Fig. 1a, under the same pH value, the MRR increases with the increase of the content of the hydrogen peroxide in polishing slurry. When the content of the hydrogen peroxide is 40 g/l, the MRR reaches the maximum value. After that, the MRR decreases with the increase of the content of the hydrogen peroxide in polishing slurry. Under the same content of the hydrogen peroxide, the

MRR increases with the decrease of the pH value in CMP slurry. When pH = 2 and the content of hydrogen peroxide is 40 g/l, there are the maximum MRR and the minimum surface roughness, these are 107 nm/min and $R_{\mathrm{a}}$ 12 nm respectively. Figure 2 shows the surface morphology of 304 stainless steel after CMP with polishing slurry at pH = 2 and the content of oxidant hydrogen peroxide being 40 g/l. By Fig. 2, there is no spots exist on the surface, but there are many scratches.

The hydrogen peroxide is a weak acid with the strong oxidation ability. When pH $> 4$, the hydrogen peroxide has the poor stability and volatile. When pH $< 3.5$, the hydrogen peroxide has the good stability and not easy to decompose into water and oxygen [21]. Therefore, in the strong acidic environment, it is conducive to the use of oxygen in the CMP process. Therefore, the smaller the pH of the polishing slurry is, the more stable the oxidant hydrogen peroxide is, the larger MRR and the lower surface roughness can be obtained. So the MRR reaches the maximum at pH = 2. From Fig. 1b, the surface roughness increases with the increase of the pH value of the polishing slurry and the surface roughness reaches the minimum when pH = 2 and under different content of hydrogen peroxide in polishing slurry.

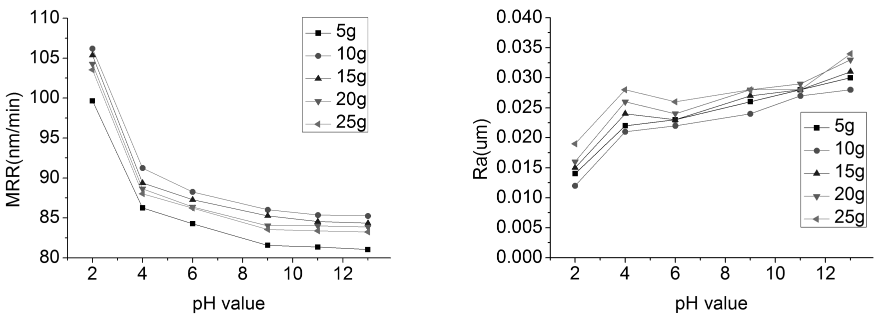


Fig. 1. Experimental results after CMP using polishing slurry with oxidant hydrogen peroxide: Material removal rate (left), Surface roughness $R_{\mathrm{a}}$ (right).

When adding the pH regulator $HNO_3$ into the slurry, because the nitric acid is a kind of inorganic acid with the strong acid and the strong oxidizing, it has the strong oxidation action, corrosion action and passivation action on stainless steel in low concentration. Research shows that the nitric acid in polishing slurry can first dissolve the iron and iron oxides on the surface of stainless steel than that of chromium and chromium oxides [22]. The chemical reaction [23] is shown in the formula (1). At the same time, the nitric acid also can react with these metallic elements in stainless steel surface and generate the metal oxides and the gas NOx in the CMP process. These metal oxides will adsorb on the surface of stainless steel and form a layer of solid passivation film on the surface of stainless steel to hinder the further reaction with the surface material. The composition of the solid passivation film is $Cr_2O_3$, FeO, NiO and etc [24]–[25]. The reaction formula sees (3) and (**??**) [26]–[27]. In formula (3) and (**??**), the Me stand for Cr, Fe and Ni. The layer of the oxide film is very thin [28] and easily to be removed by the abrasive of slurry in the

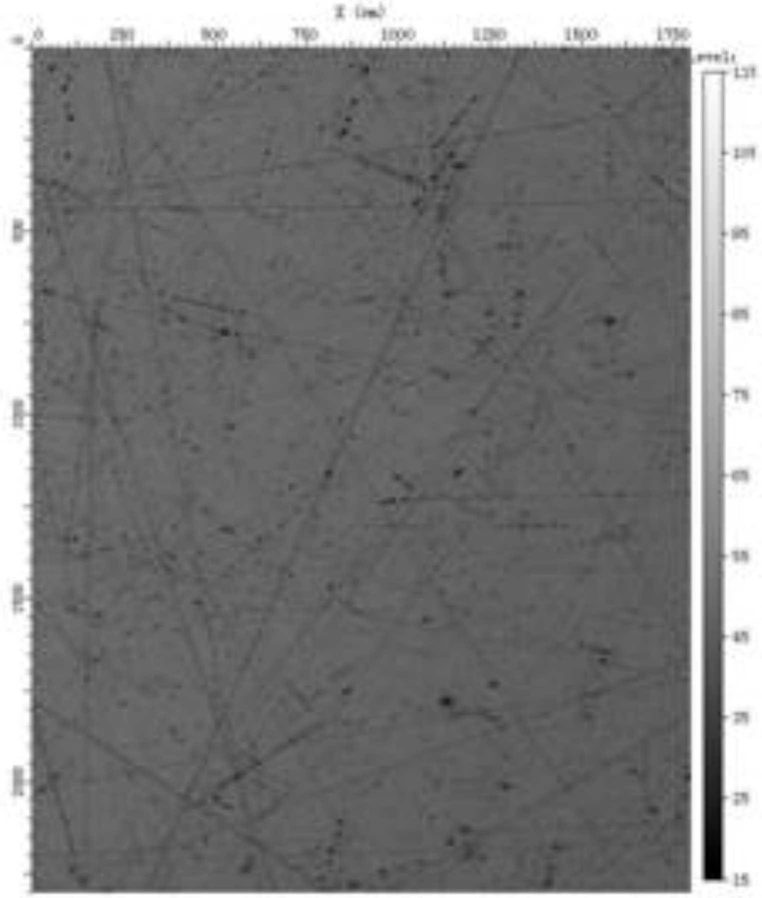

Fig. 2. The surface morphology of 304 stainless steel after CMP with slurry at pH = 2.

chemical mechanical polishing.

$$Fe + 4HNO_3 \rightarrow Fe(NO_3)_2 + 2NO_2 + 2H_2O\,, \tag{1}$$

$$2Me + 6HNO_3 \rightarrow Me_2O_3 + 6NO_2 + 3H_2O\,, \tag{2}$$

$$Me + 2HNO_3 \rightarrow MeO + 2NO_2 + H_2O\,. \tag{3}$$

In alkaline solution, due to being many $OH^-$ ions, the reaction of the ferrous hydroxide is beneficial to occur and can form a layer of ferrous hydroxide film on the surface of stainless steel, and then removed by the mechanical action of the abrasive [29].

### *3.2. Influence of pH regulator on MRR and surface roughness*

According to the above research results, it can be seen that the pH value of the slurry CMP 304 stainless steel should be less than or equal to 2. A lot of acid can be used as a pH value regulator. These commonly used are the nitric acid, phosphoric acid, acetic acid, oxalic acid, citric acid, methane dicarboxylic acid, and so on. Which is the most appropriate pH regulator, it needs to carry out the experimental study.

From the Table 1, taking the content of the hydrogen peroxide, 10 g and other ingredients unchanged, a number of the 250 ml polishing slurry were prepared only changing the pH value. According to the different six pH regulators, the test was divided six groups and only one sort of pH regulator was used in each group. The content of each ingredient in CMP slurry sees Table 2. The experimental results were shown in Fig. 3.

Table 2. The basic ingredient and the content of polishing slurry

| Group number | pH value | Abrasive diameter ($\mu$m) | Dispersant (g) | Oxidant (g) | Content of abrasive (g) | Deionized water |
|---|---|---|---|---|---|---|
| 1 | 2 | | | | | |
| 2 | 3 | | | | | |
| 3 | 4 | 3.5 | 3 | 10 | 4.5 | Others |
| 4 | 5 | | | | | |
| 5 | 6 | | | | | |

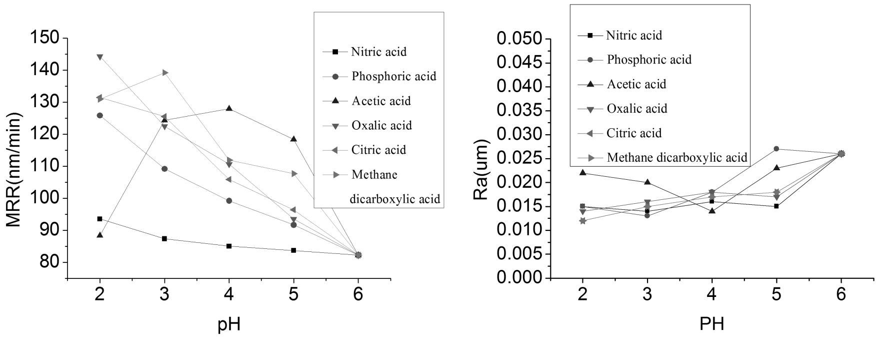


Fig. 3. Experimental results of pH regulator for the hydrogen peroxide based polishing slurry: (a) Material removal rate, (b) Surface roughness $R_{\mathrm{a}}$.

By Fig. 3a, except the acetic acid, when taking the nitric acid, phosphoric acid, oxalic acid, citric acid, methane dicarboxylic acid as the pH regulator, it can be seen that the material removal rate increases obviously with the decrease of the pH value in the polishing slurry and the surface roughness $R_{\mathrm{a}}$ changes between 10 nm and

25 nm slightly. When taking the acetic acid as the pH regulator, at the beginning, the material removal rate increases with the decrease of the pH value in the polishing slurry. When pH = 4, the material removal rate reaches the maximum. After that, the material removal rate decreased with the increase of acetic acid content, this is the decrease of the pH value. By Fig. 3b, at pH = 4, it can be obtained the lowest surface roughness. In the same condition, the material removal rate is the lowest when taking nitric acid as the pH regulator in CMP slurry. The material removal rate when taking the phosphoric acid as the pH regulator in CMP slurry is larger obviously than that of the nitric acid. Because the phosphoric acid is more viscous and has a certain action of complexation, so it is favorable for the surface quality of CMP. From the above six pH regulators, when pH = 2 and taking the oxalic acid as the pH regulator, the material removal rate reaches the maximum value and the surface roughness $R_a$ is lower. Therefore, the oxalic acid is the best pH regulator for the hydrogen peroxide based polishing slurry.

### *3.3. Influence of content of oxidant hydrogen peroxide on MRR and surface roughness*

By Table 1, Fig. 4 shows the influence of content of oxidant hydrogen peroxide on MRR and surface roughness under different pH regulator. By Fig. 4a, under the same pH value, the material removal rate increases with the increase of the hydrogen peroxide content, but when the content of the hydrogen peroxide reaches 40 g/l, the material removal rate reaches the maximum value, after that, the material removal rate decreases with the increase of the hydrogen peroxide content. When the content of the hydrogen peroxide is 40 g/l and pH = 2, the material removal rate reaches the maximum value, 107 nm/min. Here, the surface roughness reaches the minimum, $R_a$ 12 nm.

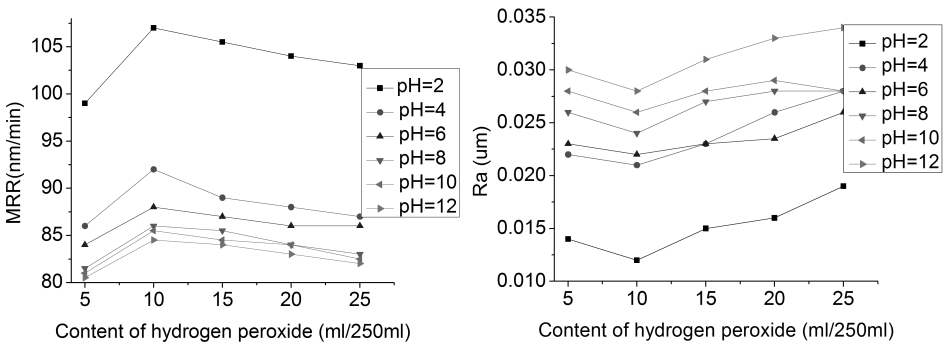


Fig. 4. Experimental results after CMP using polishing slurry with oxidant hydrogen peroxide: (a) Material removal rate, (b) Surface roughness $R_a$.

By these results of experiments, the oxidant hydrogen peroxide can improve the MRR. The possible reasons are as follows.

Due to the friction action in CMP process, the polishing interface can produce high temperature, and it will increase the hydrophilic property on the surface of stainless steel. Under the action of hydrogen peroxide in the slurry, the hydrophilic property will increase the number of oxygen groups on the stainless steel surface. A layer of the dense oxide film can be formed on the surface of stainless steel under the action of the hydrogen peroxide [30]. Then, it will be removed by the mechanical action of CMP. On the other hand, due to being many oxygen groups on the stainless steel surface, it will cause the stainless steel material to produce the electrochemical corrosion with dissolved oxygen [31], the chemical reaction is as follows.

$$\mathrm{Fe} + 1/2\mathrm{O_2} + \mathrm{H_2O} \rightarrow \mathrm{Fe(OH)_2}\,. \tag{4}$$

Under acidic conditions, there are many $H^+$ in the polishing slurry, on the one hand, the $H^+$ can reduce the stability of oxide film on the surface of stainless steel and promote the diffusion of oxygen into the metal interface. On the other hand, it will increase the hydrogen reduction reaction and make the metal more easily dissolve [32]. The hydrogen reduction reaction is as follows.

$$2\mathrm{H_3O^+} + 2\mathrm{e} \rightarrow \mathrm{H_2} \uparrow + 2\mathrm{H_2O}\,. \tag{5}$$

The hydrogen peroxide can produce Fenton type Haber–Weiss reaction on the fresh surface of stainless steel [33]. This reaction will accelerate the decomposition of hydrogen peroxide and produce the iron oxide on the surface of stainless steel material. The main reaction mechanism is as follows.

$$\mathrm{Fe^{2+}} + \mathrm{H_2O_2} \rightarrow \mathrm{Fe^{3+}} + \mathrm{HO}\cdot + \mathrm{OH^+}\,, \tag{6}$$

$$\mathrm{Fe^{2+}} + \mathrm{HO}\cdot \rightarrow \mathrm{Fe^{3+}} + \mathrm{HO^-}\,, \tag{7}$$

$$\mathrm{H_2O_2} + \mathrm{HO}\cdot \rightarrow \mathrm{HO_2}\cdot + \mathrm{H_2O}\,, \tag{8}$$

$$\mathrm{Fe^{2+}} + \mathrm{HO_2}\cdot \rightarrow \mathrm{Fe^{3+}} + \mathrm{HO_2^-}\,, \tag{9}$$

$$\mathrm{Fe^{3+}} + \mathrm{HO_2}\cdot \rightarrow \mathrm{Fe^{2+}} + \mathrm{H^+O_2}\,, \tag{10}$$

$$\mathrm{Fe^{3+}} + \mathrm{HO_2^-} \rightarrow \mathrm{Fe^{2+}} + \mathrm{HO_2}\,. \tag{11}$$

The adhesion force of the iron oxide formed by Haber-Weiss reaction above-mentioned on the surface of the metal substrate is low and easily removed by the abrasive in polishing slurry, then the base metal is re-exposed, the above process is repeated.

## 4. Conclusions

In summary, according to a series of experiments and results analysis, the following conclusions are as follows.

Under the same pH value, when the hydrogen peroxide is 40 g/l, the MRR will reach maximum. The MRR and the surface quality of CMP in acidic environment are higher than that of the alkaline environment. The MRR increases with the decrease of the pH value of polishing slurry. Therefore, when designing the CMP slurry of 304 stainless steel, it should choice acidic polishing slurry.

The optimum pH regulator of hydrogen peroxide based polishing slurry is oxalic acid, which can achieve the highest material removal rate and lower surface roughness.

The oxidant hydrogen peroxide has strong oxidation and the action of electrochemical corrosion, and it can produce type Fenton Haber–Weiss reaction with the stainless steel base material, so it can oxidize and corrode the stainless steel materials.

Because the hydrogen peroxide can increase the hydrophilicity of stainless steel surface and the content of oxygen bearing groups on stainless steel surface, it will oxidize the stainless steel to form a layer of dense oxide film on the surface. This will promote the removal of materials.

Under acidic conditions, it will decrease the stability of oxide film on the surface of stainless steel, promote the diffusion of oxygen into the metal interface and improve the oxygen reduction reaction. Due to the presence of $H^+$, it will increase the hydrogen reduction reaction to dissolve the metal more easily and to promote the material removal.

The research results in this paper will provide an important reference for the future to study the CMP slurry of 304 stainless steel.

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