Selection of Surfactants for Hydrogen peroxide-oxalic acid Polishing Slurry in Chemical Mechanical Polishing of 304 Stainless Steel

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Abstract. Stainless steel material will become one of the main materials for flexible large-size display substrates. Chemical mechanical polishing (CMP) will be one of the most practical processing technologies to achieve super smooth, non-damaged stainless steel surface. In this paper, the effects of different surfactants on material removal rate and surface roughness were studied. The results show that when adding the surfactant of the sodium hexadecyl sulfate in CMP slurry, the MRR was significantly higher than the other two kinds of surfactants. When the content of the sodium hexadecyl sulfate was 0.04 wt%, the MRR reached the Maximum. When the content of the sodium hexadecyl sulfate was 0.2 wt%, the surface roughness reached the Minimum. So, the sodium hexadecyl sulfate is selected as the surfactant of the hydrogen peroxide-oxalic acid type CMP slurry.

1. Introduction
Flexible display has excellent performance, such as ultra-thin, flexible, retractable, light weight, etc. [1-2], which has become an important development direction in display technology. It will be widely used in industry, civil and military industries [3-4].

Flexible display must be based on flexible material. As flexible substrate, it must have ultra-thin, high flexibility and toughness, its surface roughness should be less than 5nm. Stainless steel material has the physical properties above mentioned and low cost, which may become one of the main substrate materials for large-scale flexible display in the future [5-6]. However, as the substrate of the flexible display, the biggest problem of stainless steel is that the surface roughness is too large, and it needs to be ultra precision polished [7].

According to the literature, there have been some scholars who have conducted in-depth research on the polishing technology of stainless steel surface, mainly including mechanical polishing, chemical polishing, electrochemical polishing and electrochemical mechanical polishing [8]. Chemical mechanical polishing (CMP) has been considered to be the best ultra-precision machining method and widely used in the ULSI manufacturing process of hard and brittle crystal materials, such as monocrystalline silicon [9]. Therefore, the CMP technology may be the most suitable for high-efficiency ultra-precision machining of large-scale ultra-thin stainless steel flexible display substrate surface to obtain ultra-smooth and damage free machining surface [10].

CMP slurry is an important part in CMP system, but the surfactant is an important chemical component in CMP slurry. It can enhance the activity of surface materials, improve the stability of CMP slurry, and promote the chemical reaction of surface materials, so as to improve material
removal and reduce surface roughness. Therefore, it is an urgent task to study the environment-friendly and efficient CMP slurry for stainless steel [11-12].

In this paper, through a series of experiments, the material removal rate (MRR) and surface roughness polished with the hydrogen peroxide oxidant slurry under different surfactants are studied, and the best surfactant is obtained, which provides a reference for further study the CMP slurry of stainless steel.

2. Experimental preparation and parameters

The CMP experiment was carried out in a clean laboratory with the cleanliness class 1000. The temperature of the clean laboratory was set at 22 °C. in the experiment, the water used was deionized water with a resistivity of 18.24m Ω· cm. Before the experiment, a number of 304 stainless steel pieces with a diameter φ50mm were prepared as the experimental samples. Samples were lapped and their surface roughness Ra was between 40nm to 50nm. The CMP experiment was carried out on the polishing machine with type zyp300. As shown in Figure1 and Figure 2. The type of CMP pad used in the experiment is Rodel IC1000.

![Figure 1. Photo of chemical mechanical polishing machine.](image1)

![Figure 2. Schematics of traditional chemical mechanical polishing.](image2)

In the experiment, the rotational speed of the polishing platen is 60 r/min, the rotational speed of the carrier is also 60 r/min, the polishing pressure P is 2psi, and the polishing time is set to 15min each time. After each CMP, the CMP pad should be conditioned with a diamond conditioner, and the condition time is set to 15min. In the CMP process, the carrier swings back and forth along the arc.
with the range 20 mm and the swing frequency 10 s. The center distance between the polishing platen and the carrier is set to 80 mm.

Before and after CMP experiment, the Sartorius CP225D precision balance (precision 0.01mg) was used to detect the weight of the sample, and then the material removal rate was calculated by calculation. The surface roughness and surface morphology of the samples before and after CMP were measured by contour GT-K 3D surface microscope (vertical resolution 0.01nm) produced by Bruker company in the United States. The pH value of the CMP slurry was detected by pH electronic testing pen (precision 0.1).

3. Experiment

3.1. The basic components of CMP slurry

According to the results of previous research and orthogonal test of CMP slurry, the white corundum is selected as the abrasive, the hydrogen peroxide is selected as oxidant, the oxalic acid is selected as pH regulator, glycerine is selected as dispersant. The CMP slurry of hydrogen peroxide-oxalic acid is prepared for experiment. Its basic composition and content are shown in Table 1. The volume of CMP slurry each experiment is 250ml.

3.2. Selection of surfactants

According to the literature, three kinds of chemical substances, such as sodium hexadecyl sulfate, nonylphenoxyethylene ether and OP-10 emulsifier, were selected as surfactants to study the effects on the MRR and surface roughness of 304 stainless steel. See Table 2 for the three surfactants.

### Table 1. Composition and content of basic CMP slurry.

| Ingredients       | pH | abrasive size (μm) | Dispersant (g) | Oxidant (g) | Abrasive content (g) | Other          |
|-------------------|----|-------------------|---------------|-------------|----------------------|---------------|
| Value             | 4  | 3.5               | 3             | 25          | 4.5                  | Deionized water|

### Table 2. Properties of surfactants.

| Name                 | Molecular formula | Molecular weight | State               | HLB | pH   |
|----------------------|-------------------|------------------|---------------------|-----|------|
| Sodium hexadecyl sulfate | C16H33NaO4S     | 344.49           | White particles     | 38.1| 6.0-7.0 |
| Nonylphenoxyvinyl ether | C33H60O10       | 616.82           | Light viscous liquid| 13.3| 5.5-7.0 |
| OP-10 emulsifier      | -                 | -                | Colorless           | 14.5| 6.5-7.0 |

3.3. Experimental results

Figure 3 and Figure 4 are the experimental results after adding three surfactants into the hydrogen peroxide oxalate CMP slurry. According to Figure 3, in the three surfactants, the maximum of the MRR and the minimum of surface roughness were obtained after CMP using the slurry with the surfactant of the sodium hexadecyl sulfate. By Figure 4, When the content of the sodium hexadecyl sulfate in CMP slurry is 0.2wt%, the minimum of surface roughness were obtained. Therefore,
sodium hexadecyl sulfate is selected as the surfactant of the hydrogen peroxide-oxalic acid type CMP slurry.

![Graph 3](image3.png)

**Figure 3.** The influence of CMP using the hydrogen peroxide-oxalic acid type slurry with the different surfactant on material removal rate.

![Graph 4](image4.png)

**Figure 4.** The influence of CMP using the hydrogen peroxide-oxalic acid type slurry with the different surfactant on surface roughness.

4. Conclusions
In summary, through a series of experiments and results analysis, the following conclusions are drawn.

When adding the surfactant of the sodium hexadecyl sulfate in CMP slurry, the MRR was significantly higher than the other two kinds of surfactants. When the content of the sodium hexadecyl sulfate was 0.04wt%, the MRR reached the Maximum. When the content of the sodium hexadecyl sulfate was 0.2 wt%, the surface roughness reached the Minimum. So, the sodium hexadecyl sulfate is selected as the surfactant of the hydrogen peroxide-oxalic acid type CMP slurry.

The results will provide an important reference for the further study of the CMP slurry of 304 stainless steel.
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