Selection of Abrasive for Chemical Mechanical Polishing of the 304 Stainless Steel

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Abstract. Chemical mechanical polishing (CMP) technology is widely used in ultra-precision machining of various materials, and the abrasive are an important component of CMP slurry. In this paper, the effects of the abrasive type, the abrasive size and its concentration on material removal rate (MRR) and surface quality in CMP 304 stainless steel were studied through a series of experiments. The results show that the MRR in CMP 304 stainless steel using slurry with the white corundum abrasive is the highest, and the MRR in CMP 304 stainless steel using slurry with the silica abrasive is the lowest, but the surface quality in CMP 304 stainless steel using slurry with the silica abrasive is the best. The MRR and surface roughness increase with the increase of abrasive size, but the surface roughness increases slowly with the increase of abrasive size. With the increase of abrasive concentration, the MRR first increases and then decreases, while the surface roughness value changes slightly, indicating that the abrasive concentration in polishing slurry has little effect on the surface quality. These research results can provide a reference for the design of CMP slurry.

1. Introduction

Flexible display has many advantages, such as lightweight, durable, ultra-thin, freely designed, flexible, retractable, impact resistant and so on [1-2]. They will be widely used in mobile phones, personal digital assistants, and notebook computers, e-books, electronic posters, car dashboards, sensors, environmental displays, general lighting, robotic skin [3-4] and other industrial, civil and military industries. Many research institutes and manufacturers in many countries and regions have invested in the research and application of flexible display technology [5-6]. In recent years, many companies have continuously introduced foldable or bendable OLED screens. [7].

The flexible display is based on a flexible material. There are very strict in the surface quality and performance of the flexible substrate. The surface roughness must be less than 5 nm, the thermal stability is high, the light weight is high, and the thickness is ultra-thin. High flexibility and toughness, therefore, the low cost of stainless steel materials will become the main material for flexible large-size display substrates in the future [8-9]. The quality and precision of ultra-thin stainless steel sheets will directly affect the performance of their devices [10]. When the surface of the ultra-thin stainless steel sheet has minute defects, it will be inherited to the epitaxial growth film and become a fatal defect of the device. Therefore, how to efficiently obtain high-quality large-size flexible display substrates to meet the requirements of flexible displays now and in the future is a top priority for the flexible display industry [11].

Many scholars have carried out in-depth and extensive research on the polishing technology of stainless steel surface. But the chemical mechanical polishing (CMP) technology is considered to be the best process to meet the requirements of surface roughness and surface flatness. It will be probably
the most suitable and highly efficient ultra-precision machining technology for large-size ultra-thin stainless steel flexible display substrate to achieve an ultra-smooth, damage-free machined surface.

CMP slurry is one of important parts of CMP system. Its quality determines the efficiency, surface quality and cost of CMP. It is imperative to study the CMP slurry of stainless steel with environment friendly and efficient. Abrasive is an important component in CMP slurry. During CMP process and these have micro-cutting effect on the oxidation corrosion layer on the workpiece surface. The mechanical effect of the abrasive determines material removal rate (MRR) of stainless steel. Therefore, the abrasive is the main source of CMP mechanical action, and the type, the content and the size of the abrasive play a significant role in improving the performance of the CMP slurry. In this paper, through a series of experiments, the effects of abrasive type, abrasive size and concentration on MRR and surface quality were studied, and the optimum abrasive type, abrasive size and content were obtained, which can provide reference for further study of stainless steel CMP slurry.

2. Experimental preparation and experimental parameters
All CMP experiments were carried out in a Class 1000 clean room with an ambient temperature control of 22°C. The deionized water resistivity used in the experiment was 18.24 MΩ•cm. The CMP test samples are a number of 304 stainless steel sheets with a diameter of 50 mm. The roughness Ra after lapping is 40 to 50 nm. The CMP experiment is carried out on a ZYP300 type lapping and polishing machine manufactured in Shenyang. Rode IC1000 pad is used for CMP.

2.1. Selection of polishing parameters
The polishing platen has a rotation speed of 60r/min, the rotation speed of the carrier disk is 60r/min, the polishing pressure P is 2 psi, and the polishing time is 15 min. After each CMP, the CMP pad is conditioned, and the pad is conditioned for 15 min. During the CMP process, the carrier disk reciprocates along the arc, the swing range is 20mm, the swing frequency is 10s, and the center distance between the carrier disk and the polishing platen is set to 80mm.

2.2. Test instruments used in the experiment
The weight of the sample before and after the CMP experiment was measured with a Sartorius CP225D precision balance (accuracy 0.01 mg), and then the material removal rate (MRR) was calculated. The surface roughness and surface topography of the samples before and after CMP were measured using a Contour GT-K 3D surface microscope (Vertical Resolution: 0.01 nm) manufactured by BRUKER, USA. The original image of the 2D surface before and after CMP was detected by Lecia DM2500M upright metallographic microscope, the pH value of the CMP slurry was measured using a pH electronic test pen (accuracy 0.1).

2.3. The basic composition of the CMP slurry
According to the results of the previous orthogonal test of the CMP slurry and the material properties of 304 stainless steel, the composition content is shown in Table 1. A total volume of 250 ml of slurry was prepared for each experiment.

| Factor | pH | Abrasive size(μm) | Dispersant (g) | Oxidant (g) | Abrasive content(g) | Other            |
|--------|----|-------------------|----------------|-------------|--------------------|------------------|
| Value  | 2  | X                 | 3             | 6           | Y                  | Deionized water  |

Note: X and Y are variables.

3. Test results and analysis

3.1. Selection of abrasive types
Four kinds of CMP slurry are prepared named A, B, C and D, which have the same chemical composition and different type of abrasive with the same molar mass. In slurry A, the abrasive content
of boron carbide with the size W28 is 5.0 wt%. In slurry B, the abrasive content of diamond with the size W28 is 1.0 wt%. In slurry C, the abrasive content of white corundum with the size W28 is 3.0 wt%. In slurry D, the abrasive content of silica sol with the size 50nm is 12.7 wt%. The results of CMP test are in the table 2 and Fig.1.

According to Table 2, the MRR of the 304 stainless steel after CMP with white corundum abrasive is the highest, followed by diamond and boron carbide abrasive, and the MRR used the silica abrasive is the lowest.

| Slurry Factor | Slurry | A    | B    | C    | D    |
|---------------|--------|------|------|------|------|
| MRR (nm/min)  |        | 105.619 | 118.987 | 127.051 | 58.207 |
| Ra before CMP (μm) | 0.046 | 0.046 | 0.047 | 0.046 |
| Ra after CMP (μm)  | 0.081 | 0.031 | 0.035 | 0.020 |

**Figure 1.** Surface morphology after CMP with the different kinds of abrasive
(a) Boron carbide, (b) Diamond, (c) White corundum, (d) Silica.

According to Fig. 1, the surface of the 304 stainless steel polished by the slurry with the abrasive of the boron carbide is black and the surface roughness is the largest. Due to the high hardness of the diamond, the surface of 304 stainless steel polished by the slurry with the abrasive of the diamond has deep defects and the scratches are more obvious, and the roughness is larger. In Fig.1c, The pit-like defects on the surface of 304 stainless steel polished by white corundum slurry appear similar to black spots. The pit-like defects are white by electron microscopy observation, which may be the residual
ferrochromium oxide in the pit. The surface quality of polished by silica slurry is the best and the surface roughness is the lowest. Therefore, according to the MRR and the surface quality after CMP with the four kinds of slurry and the economy, the white corundum is more suitable as the abrasive of the slurry for CMP of 304 stainless steel.

3.2. Selection of abrasive size of white corundum

Five different diameters of white corundum abrasive were selected for CMP experiments. The experimental results are shown in Table 3.

| Abrasive size (the abrasive content is 3.0 wt%) | W0.5 | W3.5 | W7  | W14 | W28  |
|---------------------------------------------|------|------|-----|-----|------|
| MRR (nm/min)                                | 40.511 | 60.822 | 95.390 | 116.442 | 127.051 |
| Ra before CMP (µm)                          | 0.045 | 0.046 | 0.047 | 0.049 | 0.044 |
| Ra after CMP (µm)                           | 0.013 | 0.017 | 0.027 | 0.036 | 0.035 |

According to Table 3, with the increase of the abrasive size of the white corundum, the MRR of 304 stainless steel increases, and the surface roughness also increases, but the increase is slower. So, large size abrasive can be used for rough polishing and small size abrasive can be used for fine polishing.

3.3. Selection of the concentration of white corundum abrasive

Take the abrasive size of W3.5 as an example. Five kinds of abrasive concentrations are as follows in table 4. The results of CMP are shown in Table 4.

| Selection of abrasive concentration (W3.5) | Content (wt%) | 0.6 | 1.2 | 1.8 | 2.4 | 3.0 |
|------------------------------------------|---------------|-----|-----|-----|-----|-----|
| MRR (nm/min)                             | 48.277 | 60.550 | 76.209 | 61.392 | 60.822 |
| Ra before CMP (µm)                       | 0.045 | 0.047 | 0.045 | 0.040 | 0.048 |
| Ra after CMP (µm)                        | 0.020 | 0.015 | 0.014 | 0.018 | 0.017 |

According to Table 4, with the increase of the concentration of the abrasive, the MRR first increases and then decreases, and the maximum value is obtained when the abrasive concentration of white corundum is 1.8 wt%. The surface roughness value changes little, and the minimum value is obtained when the abrasive concentration of white corundum also is 1.8 wt%. It shows that the abrasive content in CMP slurry has little effect on the surface quality, but it is not the higher the abrasive content in CMP slurry, the better the CMP effect. On the contrary, the higher the abrasive content is, the thicker the CMP slurry is, the poorer the aggregation and deposition of abrasives, and the poorer dispersion of abrasives is, this will affect the CMP process.

4. Results and conclusions

The MRR of white corundum abrasive is the highest for polishing 304 stainless steel substrates. Silica abrasive has the lowest MRR, but the surface quality after CMP with silica abrasives is the best. Considering the MRR, surface quality and the economy factor, the white corundum will be selected as the abrasive of slurry for CMP 304 stainless steel.

With the increase of size of the white corundum abrasive, the MRR and surface roughness in CMP 304 stainless steel increase, but with the increase of abrasive size, the surface roughness increases slowly.
With the increase of abrasive concentration, the MRR first increases and then decreases, while the surface roughness value changes little. When the abrasive concentration of the white corundum with abrasive size W3.5 is 1.8wt%, the maximum value of MRR is obtained. It is indicated that the abrasive content in the CMP slurry has little effect on the surface quality.

Acknowledgements
The authors acknowledge the financial support of the National Natural Science Foundation of China (No.U1804142), Science and Technology Research Project of Henan Province (No.192102210058).

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