Pollution-Free Approaches for Highly Efficient Sapphire Substrate Processing by Mechanical Chemical Polishing

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Abstract: In this study, two pollution-free approaches were used to improve the processing efficiency of mechanical chemical polishing (MCP) for sapphire substrates. The first was dedicated to polishing using synthetic silica abrasives with high reactivity as soft abrasives, and the second was dedicated to polishing using organic acids and bases as catalysts on the polishing pad and coolant, including citric acid, theophylline, green tea, and coffee. The results showed that the rate of material removal of MCP with highly reactive silica abrasives was more than 187.5% larger than that of pristine silica abrasives with low reactivity, and surface roughness was 21% smaller than that of pristine silica abrasives. Meanwhile, in case of citric acid, the removal mass of sapphire was more that 29.2% larger than that of an original polishing method, and surface roughness was about 15.2% smaller than that of original polishing method. However, theophylline, green tea, and coffee affected processing efficiency negligibly or negatively, due to a limited effect on increasing the pH level of the coolant, and the powder residue in the coolant obstructing the contact between the nano-silica abrasive particles and sapphire surface. These results indicate that highly reactive nano-silica abrasives and citric acid show good catalytic activity towards the sapphire processing efficiency of MCP. The promoting mechanism of highly active silica abrasives and citric acid for the processing efficiency of MCP is also discussed in detail in this study.

Keywords: sapphire; mechanical chemical polishing; reactivity; organic acids and bases; removal mechanism

1. Introduction

As a result of energy conversation urgency and the development of optoelectronics devices, sapphire has attracted increasing attention. Sapphire, which is a single crystal form of α-alumina, has excellent mechanical and optical properties and is widely used in a range of applications, such as optics, electronics, and temperature sensing [1,2]. As is well known, sapphire has become the most widely applied substrate material for manufacturing GaN-based light emitting diode (LED) devices [3–5]. In all of these applications, high surface quality of sapphire substrates is critical, as well as in relation to subsurface damage. Currently, chemical mechanical polishing (CMP) with the help of an alkaline slurry is still the main planarization machining method for realizing the precision requirement of sapphire substrates [6–8]. Owing to its intrinsic nature such as great hardness, high mechanical strength, and chemical resistance, sapphire substrates are extremely difficult to polish with regard to simultaneously obtaining both a high material remove rate (MRR) and a perfect polished surface. Moreover, CMP always results a severe waste of slurry, which is extremely difficult to degrade, resulting in serious environmental pollution [9,10].
Mechanical chemical polishing (MCP), regarded as an effective processing method for hard–brittle materials, has been explored in many experimental and theoretical investigations [11–13]. Used towards the surface finishing of sapphire substrates, this process is an efficient and environmentally friendly approach to acquire high-quality surfaces by removing the reactive layer on the surface of sapphire [12,13]. Figure 1 illustrates MCP. It is reported that during MCP, the surface of sapphire comes in contact with active abrasive particles under downward force. The increasing heat and pressure at the local contact region, which is caused by friction, increases the reactivity between the abrasive particles and micro-convex on the sapphire surface. Then, a chemical reaction between the soft abrasive particles and micro-convex occurs, and a passivation layer is formed under the effects of heat and pressure. Finally, the passivation layer can be easily removed by abrasives due to the passivation layer being very thin, and its hardness being lower than that of sapphire. This results in the fresh surface of sapphire being exposed to generate another corrosive thin layer.

![Diagram of mechanical chemical polishing (MCP)](image)

**Figure 1.** Illustration of mechanical chemical polishing (MCP).

In order to enhance the machining efficiency and surface quality for sapphire substrates during MCP, many possible approaches have been carried out. The MCP of sapphire substrates with soft–hard mixed abrasives has been investigated in our previous work, and it was demonstrated to be an effective processing method for sapphire substrates [14]. Soft abrasives are mainly used for the effect of chemical corrosion to generate the passivation layer on the sapphire surface, while hard abrasives are mainly used for the mechanical removal of the passivation layer. The processability of MCP depends on the dynamic equilibrium between the mechanical removal of hard abrasives and chemical corrosion of soft abrasives. However, in fact, considering the weak chemical reaction of sapphire, the processability of MCP is determined critically by the chemical reaction rate between abrasive and sapphire surface.

Based on the principle of sol–gel, a semi-fixed flexible polishing tool called the SG polishing pad has been reported as an ideal tool to satisfy the processing demands of various kinds of hard and brittle materials [15–18]. SG polishing pads are made from sodium alginate (AGS), and the fabrication process mainly includes mixing, screeding, gelling, and drying. This study aims at investigating a pollution-free way to improve the polishing efficiency of MCP in the case of sapphire substrates. Sapphire polishing has been performed using an SG polishing pad with mixed abrasives. Two approaches were performed to improve the chemical reaction rate between active abrasives and sapphire surface during MCP. The first was dedicated to polishing using synthetic silica abrasives with high reactivity, while the second was dedicated to polishing using organic acids and bases, including citric acid, theophylline, green tea, and coffee. This planarization processing is environmentally friendly as deionized water is applied as the coolant without any corrosive liquid during polishing. The MRR of the MCP process was measured, and the effect of different promotion mode on MRR is discussed in detail. The polishing residues that emerged in the coolant during polishing were analyzed via TEM, selected area electron diffraction (SAED), and energy dispersive spectrometry (EDS) to reveal the MCP mechanism with different catalyzing modes.
2. Results and discussion

2.1. Characterization of Polishing Pad with Different Catalysts

In order to explore the influences of different catalysts on mechanical properties of the SG polishing pad, the bonding structure of the matrix of catalyst-based and catalyst-free-based polishing pad was investigated by Fourier transform infrared (FT-IR) spectroscopy in the range of 4000–400 cm$^{-1}$, as shown in Figure 2. In the FT-IR spectra of original polishing pad with alumina and commercial silica abrasives (Figure 2a), the absorption peak at 541.45 cm$^{-1}$ is attributed to the Si–O symmetric stretching vibration mode of silica. Other absorption bands are associated with the matrix of the polishing pad. Compared with the FT-IR spectra of the pristine polishing pad with mixed abrasives, neither a new absorption band nor a new organic function regiment appeared after adding citric acid (Figure 2b) and theophylline (Figure 2c) onto the polishing pad. As seen in Figure 2d, significant changes in spectra occur near 3332.93, 1319.44, 1244.30, 1054.24, 791.40, 763.77, 744.60, 613.50, and 498.20 cm$^{-1}$. These new absorption peaks are all attributed to the theophylline. A weak absorption band attributed to green tea powder is generated at around 2852.54 cm$^{-1}$ in Figure 2e. Meanwhile, the absorption bands around 2923.64, 2853.44, and 1742.18 cm$^{-1}$ correspond to coffee powder. From the results of FT-IR spectroscopy, there is no new chemical bond when a different catalyst was added into the matrix of gels. Thus, this indicates that the mechanical properties of the matrix of the SG polishing pad were not affected by the catalyst.

![Figure 2. FT-IR spectra of the polishing pad with different catalyst: (a) pristine pad, (b) synthetic soft abrasives, (c) citric acid, (d) theophylline, (e) green tea powder, and (f) coffee powder.](image-url)
2.2. The Polishing Performance on Sapphire Substrate under Different Catalyzing Modes

For the planarization performances of MCP with different catalyst testing, the polishing experiments were carried out. Figure 3 exhibits the MRR comparisons of MCP under different catalyzing modes. From Figure 3, it can be seen that the removal mass of sapphire increases with the addition of synthetic silica and citric acid during the polishing process. When synthetic silica abrasives with high reactivity are used, the MRR of sapphire in experiment B (catalyzing mode: synthetic soft abrasives) was 2.07 nm/min, more than 187.5% larger than that of catalyst-free-based polishing process. Meanwhile, when the polishing pad and coolant contain citric acid, the removal mass of sapphire is 0.93 nm/min, more than 29.2% larger than that of original mixed abrasives polishing without a catalyst in the polishing pad and coolant. On the contrary, when theophylline, green tea, and coffee powder were used as catalyst during the polishing process, the removal mass of sapphire was 0.72, 0.00, and 0.41 nm/min, respectively. Under the same processing conditions with others, they have no obvious promoting effect on the MRR for the polishing process, and even had a negative effect, especially when green tea was used as a catalyst during polishing and removed sapphire at a negligible rate.

![Material removal rate of sapphire under different catalyzing modes](image_url)

**Figure 3.** Material removal rate of sapphire under different catalyzing modes: (A) pristine pad, (B) synthetic soft abrasives, (C) citric acid, (D) theophylline, (E) green tea powder, and (F) coffee powder.

Surface roughness of sapphire substrates polished with different catalysts was measured, as shown in Figure 4. It is clear from Figure 4 that after polishing with the SG pad containing synthetic silica abrasives, the mean value of surface roughness measured was 0.83 nm, which is 21% smaller than the 1.05 nm of the catalyst-free-based polishing process in experiment A (pristine pad). Moreover, the roughness acquired from the substrate surface polished using the SG pad with citric acid had a lower mean value (Ra: 0.89 nm) than that of catalyst-free-based polishing process, but the standard deviation of Ra was still large. Furthermore, the sapphire substrates in experiment D, E, and F were still rough, such that the roughness in these cases was approximately equal to the original surface.

![Surface roughness of sapphire substrates after polishing under different catalyzing modes](image_url)

**Figure 4.** Surface roughness of sapphire substrates after polishing under different catalyzing modes: (A) pristine pad, (B) synthetic soft abrasives, (C) citric acid, (D) theophylline, (E) green tea powder, and (F) coffee powder.
On the whole, higher MRR generally corresponds to lower surface roughness. The synthetic silica abrasives with high reactivity, as well as the citric acid, were useful in maintaining a relatively high MRR with a significant reduction in surface roughness.

2.3. The Sapphire Substrate Removal Mechanism

Up until now, studies have shown that the interaction between sapphire and silica involves interdependent chemical and mechanical functions. Although not confirmed by experimental data regarding the composition of the chemical reaction product, it is generally accepted that the chemical reaction between silica particles and sapphire surface during polishing can be described as below [19–21]:

\[
\text{Al}_2\text{O}_3 + 2\text{SiO}_2 + 2\text{H}_2\text{O} = \text{Al}_2\text{Si}_2\text{O}_7\cdot2\text{H}_2\text{O},
\]

where aluminum silicate hydrate (\(\text{Al}_2\text{Si}_2\text{O}_7\cdot2\text{H}_2\text{O}\)) possesses a structure similar to natural kaolin, which has lower hardness than sapphire and can be easily removed from sapphire surface.

Some references confirm that aluminum silicate with the composition \(\text{Al}_2\text{SiO}_5\) is formed on polished sapphire surfaces during CMP [22]. It is found that in the CMP environment, the amorphous silica first dissolves in water to produce a \(\text{Si(OH)}_4\) compound by the following chemical reaction:

\[
(\text{SiO}_2)_x + 2\text{H}_2\text{O} = (\text{SiO}_2)_{x-1} + \text{Si(OH)}_4
\]

Then, the chemical reaction between silica particles and protrusions on sapphire surface is as follows:

\[
\text{Si(OH)}_4 + 2\text{Al}^{3+}_{\text{sur}} + \text{H}_2\text{O} = \text{Al}_2\text{SiO}_5 + 6\text{H}^+
\]

\[
\text{Si(OH)}_4 + 2\text{AlO}^-_{2\text{sur}} = \text{Al}_2\text{SiO}_5 + 2\text{OH}^- + \text{H}_2\text{O}
\]

where \(\text{Al}^{3+}_{\text{sur}}\) and \(\text{AlO}^-_{2\text{sur}}\) are the concentrations of aluminum and aluminum oxide active ions on the sapphire surface, and \(\text{Al}_2\text{SiO}_5\) aluminum silicate is the reaction product generated on the sapphire surface.

To investigate the material removal mechanism of the sapphire substrates under different catalyzing modes, the wear debris extracted from the corresponding coolant was investigated via high resolution transmission electron microscopy (HRTEM), SAED, and EDS to reveal the process that occurs on polished surfaces, and for determination of the material remove mechanism of sapphire. The TEM images, SAED patterns, and EDS spectra of the polishing residue in experiments A, B, and C are shown in Figure 5. The SAED patterns and EDS spectra correspond to the relevant area of the TEM images. From Figure 5, no obvious abrasive particles or impurities existed in all HRTEM results. The elements of carbon and copper in EDS spectra originated from the micrograte. A small amount of calcium (Ca) and iron (Fe) in EDS spectra were obtained from the tool substrate. As shown in Figure 5, the halos in the SAED patterns suggest that the sapphire wear debris was in the amorphous phase. It can be concluded that the amorphous transformation of surface material occurs before they are removed. The elements in the polishing residue are similar to each other, mainly including aluminum, silicon, and oxygen. These results demonstrate that the material removed during MCP with different catalyzing modes is no longer \(\text{Al}_2\text{O}_3\), but is most likely aluminum silicate. Combined with the SAED patterns, it seems this chemical can only be derived from the chemical reaction between the sapphire surface and silica particles. Moreover, regarding the \(\text{Al}/\text{Si}/\text{O}\) ratio, the dependence between the atomic concentrations of all polishing residues with different catalyzing modes almost coincides at 2:1:5. This suggests that silica abrasive particles play a key role during the MCP process regarding the passivation layers (\(\text{Al}_2\text{SiO}_5\) aluminum silicate) that are generated on the surface of sapphire substrates. These results conform to that of aluminum silicate, to a certain degree.
In the synthetic silica abrasive-assisted MCP, the increased number of −OH on the particles surface led to a stronger chemical reactivity of synthetic silica than commercial silica. The amorphous structure and more −OH on the silica abrasive particle surface helped promote the chemical reaction that occurred on the sapphire surface in response to the destruction of interatomic bonds that resulted from the mechanical treatment. Furthermore, in the case of citric acid-assisted MCP, the higher environmental pH level (pH = 4) caused an increase in the solubility of silica abrasives. In the meantime, as pH levels of suspension increase, more hydrogen ions are adsorbed on the polished surface, leading to reaction of aluminum (in the form of Al\(^{3+}\)) according to Equation (3), and passivation layers being generated on the surface of sapphire, facilitating the MRR. Compared with citric acid, on the one hand, theophylline, green tea, and coffee powder have only a very limited effect on increasing the pH level of the coolant (the pH value of their saturated solution was 5.42, 5.40, and 5.14, respectively) and play a negligible role during the MCP process. On the other hand, due to the fact that they are very difficult to dissolve in water, powder residue in the coolant will obstruct the contact between the nano-silica abrasive particles and sapphire surface, leading to the decline in the chemical reaction rate as well as material removal.

3. Experimental Section

3.1. Synthesis of Soft Abrasives with High Reactivity

Compared with commercial silica, silica abrasives with regular spherical shape and high reactivity were prepared through hydrolysis–precipitation. According to the preparation method of nano-silica
abrasives described by Xu et al. [14], a certain amount of sodium silicate (\(\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}\)) was dissolved in ethanol aqueous (1:8 volume mixture of alcohol and deionized water). The sodium silicate solution was slowly dripped into the ammonium chloride solution until the pH value of the solution was 8.5. The solution was heated at 40 °C and was stirred by a thermostatic mixer for 1 h to obtain silica abrasives. Due to more \(-\text{OH}\) on the nano-sphere surface, the synthetic silica had a stronger chemical reactivity than commercial silica. In order to keep the number of organic functional groups on the surface of abrasives, the obtained silica abrasives were directly used for the preparation of the polishing pad without thermal treatment. In this case, deionized water was applied as coolant during polishing.

3.2. Organic Acid- and Base-Assisted Polishing

Environmentally friendly chemical reagents, including citric acid and theophylline, as well as biological reagents, including green tea and coffee, were used to create an acid or alkaline environment for the polishing process. Due to the fact that citric acid is easily soluble in water, the SG polishing pad and coolant were made by mixing citric acid with deionized water, and the pH of citric acid solution was 4.0. For theophylline, green tea, and coffee, 1 wt % of these insoluble substance powders was added to deionized water. After shear and ultrasonic dispersion, homogeneously dispersed suspensions containing different chemical and biological reagents, were separately used to fabricate the polishing pad. Meanwhile, the saturated solution of these insoluble substances was used as a coolant during polishing.

3.3. Polishing Setup and Process Control

A sapphire substrate-oriented (0 0 0 1) plane with a two-inch diameter after grinding was used. The original surface roughness (Ra) of the wafer after lapping was about 1.2 nm. Table 1 lists the experimental design scheme. A rotary-type polishing machine (AUTOPOL-1200S, Kejing, Shenyang, China) with a semi-fixed abrasive polishing pad was used in the sapphire polishing experiments. The schematic diagram of the sapphire substrate polishing process is shown in Figure 6. The mixed abrasives, which contain alumina as hard abrasives and silica as soft abrasives, were used to make the polishing pad. The semi-fixed polishing pad was fabricated based on sol–gel technology, and the abrasives with fine particle sizes were dispersed uniformly in the catalyst-free-based matrix of gels. The abrasives in the semi-fixed polishing pad exhibited effects during polishing that were favorable for achieving a smooth surface. The catalyst-based polishing pads were made by mixing the catalyst-free-based matrix of gels with different catalyst contents. The pristine SG polishing pad with mixed abrasives was fabricated for the comparative study. The SG polishing pads with different catalysts are illustrated in Figure 7. The pressure between the workpiece carrier and the polishing pad was 5 kg. The speed of workpiece/pad rotation was 60/120 rpm, and the treatment time was 120 min. After processing, the substrates were cleaned immediately with ethanol and deionized water under sonication. The weight of the wafers before and after polishing was measured by a precision electronic balance with 0.1 mg precision (BSA224S, Sartorius, Germany) to calculate the MRR according to the following formula:

\[
\text{MRR} = \frac{10^7 \times \Delta m}{\rho \times 2.54^2 \times \pi \times t}
\]

where \(\Delta m\) (mg) is the mass loss of sapphire wafer before and after polishing, \(t\) (min) is polishing time, \(\rho\) is sapphire density (3.98 g/cm\(^3\)), and MRR (nm/min) is the corresponding removal rate.

| Experiment | Catalyzing Mode   | Experiment | Catalyzing Mode   |
|------------|-------------------|------------|-------------------|
| A          | Nothing           | B          | Synthetic silica  |
| C          | Citric acid       | D          | Theophylline      |
| E          | Green tea         | F          | Coffee            |
3.4. Characterization Methods

FT-IR measurements of catalyst-based and catalyst-free-based polishing pads were conducted using a FT-IR spectrometer (Thermo Scientific Nicolet iS10, Thermo Fisher Scientific, Massachusetts, USA). The surface morphology and roughness (Sa) of processed substrates were evaluated by a 3D optical interferometry profiler (New View™ 7300, Zygo, Connecticut, USA). Surface roughness (Ra) was the average value of the 10 areas that evenly dispersed on the wafer surface. To verify the material removal mechanism of sapphire substrate, the surface morphologies of wear debris extracted from the corresponding coolant were characterized by TEM (Tecnai F30, Philips-FEI, Amsterdam, Netherlands) with an accelerating voltage of 300 kV. This process was employed to characterize the chemical products formed on the sapphire surface during polishing. The present study discussed the effect of different promotion mode on processing performance of MCP with mixed abrasives.

![Schematic diagram of the rotary-type polishing experimental setup.](image)

**Figure 6.** Schematic diagram of the rotary-type polishing experimental setup.

![Illustration of SG polishing pad with different catalysts: (a) pristine pad, (b) synthetic soft abrasives, (c) citric acid, (d) theophylline, (e) green tea, and (f) coffee.](image)

**Figure 7.** Illustration of SG polishing pad with different catalysts: (a) pristine pad, (b) synthetic soft abrasives, (c) citric acid, (d) theophylline, (e) green tea, and (f) coffee.

4. Conclusion

This study introduced two pollution-free approaches to improve the processing efficiency of MCP for sapphire substrate. The first was dedicated to polishing using synthetic silica abrasives with high reactivity as soft abrasives, and the second was dedicated to polishing using organic acids and bases, including citric acid, theophylline, green tea, and coffee as additives to the polishing pad and coolant. The highly reactive nano-silica and citric acid showed remarkable catalytic activity towards sapphire processing efficiency of MCP, resulting from more –OH on the synthetic nano-silica particles surface, and the high environmental pH level. Theophylline, green tea, and coffee affect processing efficiency...
negligibly or negatively, due to the limited effect of increasing the pH level of the coolant, and the powder residue in the coolant obstructing the contact between the nano-silica abrasive particles and sapphire surface. Moreover, wear debris analysis revealed that aluminum silicate with the composition of Al$_2$O$_5$ was generated on the polished sapphire substrate surface during MCP.

**Author Contributions:** J.L. designed the experiments; Y.X. performed the experiments; Y.X. analyzed the data; X.X. contributed materials and analysis tools; J.L. and Y.X. wrote the paper.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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