Investigation of Thermo-chemical Polishing of CVD Diamond Film

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**Abstract.** ZnO/Diamond structure has attracted a lot of attentions and heavy investment recently just because diamond has the capability of producing very high surface acoustic wave (around 10,000 m/s). In this present study, the microwave chemical vapor deposition (CVD) method was employed to produce diamond films on silicon single crystal. Thermo-chemical polishing experiments were then conducted on the obtained diamond films. The underlying material removal mechanisms, microstructure of the machined surface and related machining conditions were also investigated. Thermo-chemical polishing was proved to be able to remove the diamond film very effectively (4.8 μm deep of diamond film was removed in 30 minutes when polishing at 550°C and 5.7 m/s). The material removal rate was increased with polishing speed and pressure. Higher polishing temperature would improve the chemical reaction and result in better surface finish.

**Introduction**

Owing to the rapid development in the mobile telecommunication and optoelectronic area, surface acoustic wave (SAW) devices such as SAW-resonator, SAW-duplexer and SAW-sensors are in great demand. Crystals such as LiNbO\(_3\), LiTaO\(_3\), and Quartz are frequently used as the substrate material for SAW devices. However, the surface acoustic wave velocity obtainable on these materials is relatively low. Crystals such as LiNbO\(_3\), LiTaO\(_3\), and Quartz are frequently used as the substrate material for SAW devices \([1-2]\). However, the surface acoustic wave velocity obtainable on these materials is relatively low (2,500–4,500 m/s). The frequency of the device is governed by the equation \(F = \frac{v}{\lambda}\), where \(F\) is the frequency, \(v\) is the velocity of the surface acoustic wave and \(\lambda\) is the wavelength which is determined by the line width of the IDT (Interdigital Transducer). This means that higher frequency can be obtained either by choosing a substrate material with a higher surface acoustic wave velocity or by reducing the line width of the IDT. Shown in Table 1 are the line widths of various substrate materials when a 2.5GHz working frequency is required. It is clear that the required line width is much easier to achieve when diamond is used as substrate material.

<table>
<thead>
<tr>
<th>Substrate Materials</th>
<th>Velocity (m/s)</th>
<th>Freq.</th>
<th>IDT linewidth (μm) @2.5GHz</th>
</tr>
</thead>
<tbody>
<tr>
<td>LiTaO(_3)</td>
<td>3,300</td>
<td>800MHz</td>
<td>0.33</td>
</tr>
<tr>
<td>LiNbO(_3)</td>
<td>3,500</td>
<td>900MHz</td>
<td>0.35</td>
</tr>
<tr>
<td>Quartz</td>
<td>3,200</td>
<td>800MHz</td>
<td>0.32</td>
</tr>
<tr>
<td>ZnO/Glass</td>
<td>3,200</td>
<td>800MHz</td>
<td>0.32</td>
</tr>
<tr>
<td>ZnO/Sapphire</td>
<td>5,500</td>
<td>1.4GHz</td>
<td>0.55</td>
</tr>
<tr>
<td>ZnO/Diamond</td>
<td>10,000</td>
<td>2.5GHz</td>
<td>1.0</td>
</tr>
</tbody>
</table>
ZnO/Diamond structure has attracted a lot of attentions and heavy investment recently because diamond has the capability of producing very high surface acoustic wave (around 10,000m/s). However, diamond has to be machined to specified thickness and surface finish before it is to be used as substrate material of a SAW device [3-4]. The trouble is that diamond has the highest hardness of the known materials and is extremely difficult to be machined. Many researches have been conducted, in the past decades, on machining CVD-diamond film by mechanical grinding/lapping/polishing [5-14], by laser ablation [15-18], by laser-assisted etching [19-20], by ion-beam machining [21-22] or by RIE (reactive ion etching) [23]. Apart from material removal rate, the underlay physical/chemical phenomena involved in the wear of diamond and various machining techniques were also studied/reviewed in details by some researchers such as Paul et al [24], Malshe et al [25]. Though some very promising results have been obtained, the problems related to the machining of CVD diamond film are still far from being fully resolved. In the present study, the CVD diamond films were hot-lapped to investigate its machinability.

**Experimental Setup**

Diamond wafers with thickness up to 100μm have been grown on silicon single crystal in this study by MWCVD process. The deposition is carried out in a plasma reactor (Axitron) having an ellipsoid cavity to generate intense, spatially extended plasmas from a CH₄/H₂ gas mixture. The microwave plasma reactor was powered with 6 kW microwave power, operating at 915 MHz. The substrate temperature was monitored by an optical pyrometer and was kept at 1200°C. To ensure the quality of the coated diamond film, the growth rate was kept relatively low at around 1~2μm/hr. Thermo-chemical polishing experiments were then conducted on the obtained diamond films. Shown in Fig.1 is the schematic representation of the setup for hot polishing experiments. The iron/steel bar or plate was heated by either infrared irradiation or inductive heating devices to various skin temperature. In the case of turning experiments, the specimen was mounted on a flexure and was then brought to contact with the heated iron/steel bar. The contact force was monitored through the displacement of the flexure.

![Fig.1 Schematic representation of the setup for thermo-chemical polishing experiments](image)

The relative speeds of polishing, duration of polishing were also recorded. Some minimeters of the iron/steel bar or plate surface was taken off with a turning/dressing tool each time before starting a new set of experiment to ensure the surface conditions maintained as closely the same as possible.

**Results and discussion**

The material removal mechanisms involved in the thermo-chemical polishing process are generally categorized into graphitization (metal catalyzed), diffusion (into metal), oxidation and formation of metal-carbon complex. Cast iron and steel with various carbon contents are used in this study as the catalytic materials to transform diamond into graphite which is subsequently removed by micro-chipping during the polishing process or diffusing into the iron/steel surface. The polishing temperatures, pressure and speeds used in the present study ranged from 50~650°C , 0.5~4.5MPa and
2.0–5.7 m/s, respectively. The iron/steel bar or plate were pre-heated to the designed temperature before the polishing experiments were carried out and the temperature was monitored during the polishing process by both infrared detector and thermal couple. Owing to the heat generated by the friction, the temperature at the contact zone should be higher than the pre-heated temperature. Shown in Fig. 2 is a SEM micrograph of partially polished diamond film with its peaks removed and metal debris trapped inside the valley. The EDAX analysis of the debris showed clearly the peaks of carbon, iron and oxygen which were the results of the diffusion and oxidation during the polishing process. Based on the laws of diffusion, the diffusion flux is a function of the polishing temperature and carbon concentration drops across the metal polishing pad. Higher polishing temperature and lower carbon content inside the metal polishing pad are in favor of stronger diffusion process. Thus carbon atoms are in a better situation to dislodge from diamond grains and diffuse into metal pad. Shown in Fig. 3 are the schematic representation (Fig. 3a) and the SEM micrographs (Fig. 3b–3f) of the morphology changes of the diamond film during polishing against a low carbon steel polishing pad at 550 ºC. The apexes of the diamond grains were rapidly removed to form mesa-facets which gradually expanded to join into one big flat surface. This is the typical mechanism involved in polishing at even higher temperature.

In the case of polishing against high carbon steel and/or low polishing temperature, the diffusion process is not strong enough to fast remove the peak into facet. Instead, the weak bonded atoms at the corners and the sharp edges were the easier targets to be removed first. Shown in Fig. 4 are the schematic representation (Fig. 4a) and the SEM micrographs (Figs. 4b–4f) of the morphology changes of the diamond film during polishing against a high carbon steel polishing pad. The as-grown sharp edged diamond grains were gradually worn out and flattened during the polishing process.

Fig. 2 SEM micrograph of partially polished diamond film with its peaks removed and metal debris trapped inside the valley. The EDAX analysis of the debris showed clearly the peaks of carbon, iron and oxygen which were the results of the diffusion and oxidation during the polishing process.
Fig. 3 Schematic representation (a) and the SEM micrographs (b-f) of the morphology changes of the diamond film during polishing against a low carbon steel polishing pad.

Fig. 4 Schematic representation (a) and the SEM micrographs (b-f) of the morphology changes of the diamond film during polishing against a high carbon steel polishing pad.

Shown in Fig. 5 and Fig. 6 are the SEM micrographs of the CVD diamond film which were hot polished for 30 minutes under various polishing speeds (2.1m/s and 4.27m/s) at a temperature around 550°C (skin temperature of the iron/steel just outside the polishing area). Based on the obtained results, some 1.5 μm and 4.8 μm deep of diamond film was successfully removed in 30 minutes in the case of polishing at a temperature around 550°C and at 2.1 m/s and 5.7 m/s respectively.
Summary

Thermo-chemical polishing was conducted on the CVD diamond films in this research. Thermo-chemical polishing was proved to be able to remove the diamond film very effectively (4.8 μm deep of diamond film was removed in 30 minutes when polishing at 550°C and 5.7 m/s). The removal rate increased with polishing speed and pressure. The polishing temperature and carbon concentration of the polish pad had profound effect on achievable material removal rate and the removal mechanisms involved.

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References