Mechanical Machining of Thin Polycrystalline Diamond Films
Hans H. Gatzen, Christos Kourouklis
Institute for Microtechnology, Hanover University, Germany

Key Words: Diamond films, CVD, Nanogrinding

Abstract: Polycrystalline diamond films can be deposited on a variety of materials by chemical vapor deposition (CVD). Due to its rough surface, such a material is unsuited for many applications. This paper describes a mechanical machining process which allows to reduce the average roughness $R_a$ of a polycrystalline diamond film from 350 nm as deposited to a couple of nanometers. This is done with a three step process, starting out with lapping plates made of composite cast iron/copper to remove the protruding grains, followed by machining on a copper and a tin plate.

1. Introduction
Diamond films are used for coating tools, for diaphragm coating, heat sinks for large scale integrated circuits, or as x-ray windows [1, 2]. In micro electro-mechanical systems (MEMS) they are used as coatings to improve the mechanical and tribological properties [3]. A typical deposition technique is the hot filament CVD method which can produce thin diamond films of a few micron thickness. Because of their polycrystalline structure, these films are rather rough, due to crystallites protruding from the surface. However, a smooth surface is mandatory for many applications. One approach to smoothen the surface is to subject it to mechanical machining. Since the diamond films are extremely hard, eliminating the roughness through a machining process is challenging [4]. Several polishing technologies have been investigated for smoothening: mechanical polishing, chemical polishing, chemical-mechanical polishing, high energy beam (ion, electron, laser) polishing, and electrical discharge machining (EDM) [5]. So far, known mechanical polishing techniques achieve a maximal surface roughness $R_{max}$ of as much as 1 µm [5], far to rough for many applications. The work described in this paper is aimed at achieving an average roughness two to three orders of magnitude better, i.e. achieving a roughness in the nanometer range through mechanical machining. The challenges of mechanical machining are an inefficient material removal rate at one end of the machining spectrum, as well as damage and crushing of the film due to its weak adhesion to the substrate on the other. To overcome these challenges, a multi step process using a combination of lapping and nanogrinding processes has been chosen. While lapping uses a loose grain, nanogrinding employs a tool with imbedded grain. The commonality with lapping is the use of a lapping kinematic [6]. Nanogrinding was developed at the Institute for Microtechnology (imt) at the Hanover University in Hanover, Germany and is capable of machining planes on micro parts made of brittle materials with an average surface roughness $R_a$ of less then 1 nm [7].

2. Experimental Setup and Procedure
The polycrystalline diamond films were provided by the Institute of Microstructure Technology and Analytics at the University of Kassel in Kassel, Germany [8]. The diamond films were deposited on 500 µm thick silicon substrates. The deposition was done with the hot filament method at 50 mbar by using a methane hydrogen mixture. By heating up tungsten wires located above the substrate to 2,250°C, diamond was deposited on the silicon surface. The resulting film had a thickness of about 5 µm and a polycrystalline structure. The substrate was subsequently cut in 5 mm x 5 mm pieces for the machining experiments.

To investigate the mechanical machining of the diamond films, Peter Wolters 3R40 polishing machines were used. The plate diameter was 400 mm and the load was applied through weights. A three step process was developed, using three different plates, and a combination of lapping, polishing, and nanogrinding. Table 1 provides an overview of the process steps as well as over the key process parameters.

For the first lapping process, a lapping plate made of composite cast iron/copper was used as depicted in figure 1. This type of plate has been designed specifically for the machining of natural and synthetic diamonds and diamond films. The plate chosen consists to 90% of cast iron and 10% of copper. The purpose of the cast iron is to achieving a high removal rate while the copper is responsible for the surface finish.

For the second process step, a pure copper plate was used. Two machining alternatives were investigated: lapping and nanogrinding.

The third and final step is aimed at achieving the final finish. For it, a tin plate was used. As before, this process was run in two versions: with lapping and with nanogrinding.
As abrasive material in all process steps, diamond grain in different sizes was used. For lapping, the diamond grains were dispersed in either a water or an oil based fluid, while for the nanogrinding, the diamond grain was embedded in the respective plate and the fluid did not contain any cutting grit.

### Table 1

**PROCESS PARAMETERS FOR THE MACHINING OF THE DIAMOND FILMS**

<table>
<thead>
<tr>
<th>Process</th>
<th>Plate</th>
<th>Diamond grain</th>
<th>Fluid</th>
</tr>
</thead>
<tbody>
<tr>
<td>First step: lapping on composite plate</td>
<td>Composite (90% cast iron, 10% copper)</td>
<td>4 – 6 µm, 1.5 – 3 µm, 0.5 – 1 µm</td>
<td>Water- and oil-based</td>
</tr>
<tr>
<td>Second step: lapping</td>
<td>Copper</td>
<td>1.5 – 3 µm</td>
<td>Water- and oil-based</td>
</tr>
<tr>
<td>Alternative second step:</td>
<td>Copper</td>
<td>1.5 – 3 µm</td>
<td>Water-based</td>
</tr>
<tr>
<td>nanogrinding</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Finishing step: lapping</td>
<td>Tin</td>
<td>0.5 – 1 µm</td>
<td>Water-based</td>
</tr>
<tr>
<td>Alternative finishing step:</td>
<td>Tin</td>
<td>0.5 – 1 µm</td>
<td>Water-based</td>
</tr>
<tr>
<td>nanogrinding</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

3. **Process Experiments and Results**

In order to select the influencing process parameters, preliminary investigations were conducted. It was found that a pressure of 3.3 MPa and a cutting speed of 0.5 m/s are appropriate to machine the film on the different plates without creating any optically observable fractures. This pressure was then used in all processing steps. For the determination of the machining result the machined surfaces were analyzed with atomic force microscopy (AFM) and optical microscopy.

![Figure 2: AFM topography analysis of the diamond film (a) as deposited and after lapping with a composite plate (b) for 10 minutes (top right), (c) 40 minutes (bottom left), and (d) 100 minutes (bottom right) ](image)

The challenge of the first process step was to develop a technology allowing to cut the protruding peaks of the single diamond grains of the diamond film at a reasonable rate without jeopardizing the film integrity. As it is shown in figure 2, the deposited film had a rough surface with sharp peaks. Using a soft plate resulted in a grazing of the plate, combined with a fracture or delamination of the film. Such a damage was observed in first investigations with a soft tin plate to machine the films. By using a relatively hard plate, this issue could be resolved. Three sizes of diamond grain were used: 4-6 µm, 1.5-3 µm, and 0.5-1 µm. To ensure that a transition to smaller grain was not compromised by larger grain embedded in the lapping plate, the plate surface was lapped by using pumice grain.

An investigation using 4-6 µm diamond grain for lapping showed no better result regarding the removal rate than for the 1.5-3 µm grain. Figure 2 shows the surface before the test and for three different machining times with 1.5-3 µm diamond grain. The surface roughness of $R_s$ of 337 nm before machining was reduced to a value of 17 nm after 100 minutes of lapping. Longer machining time did not result in any further roughness reduction. The surfaces did show any fractures anywhere at the film surface.
To investigate if an even smaller grain results in a further improved roughness, diamond grain with a size of 0.5-1 µm was used during an additional test run. By using the smaller diamond abrasives, the roughness of the probes did not change at the same speed as it did before with the bigger grain. After a machining time of 120 min with 0.5-1 µm abrasives, the roughness was lowered from a $R_a$ of 17 nm to 10 nm. Also, further machining did not smoothen the surface any further. The use of oil instead of water based fluids resulted in a surface waviness with a wavelength of 60 µm and an amplitude of 40 nm. The reason for the waviness could not yet be determined. This showed that the use of oil on the composite plate will not enhance the machining result as it should because of the higher viscosity of the oil and the resulting lower penetration of the abrasive in the film. The higher viscosity leads to a higher gap between workpiece and plate and this should have led to a smoother removal process. Figure 3 depicts the results of the finer grain and the usage of oil as fluid.

While the first process step accomplishes the brunt of the cutting by removing the protruding grains, step two and three are aiming at further smoothening the diamond film, ultimately aiming at an average roughness $R_a$ in the nanometer range. To do so, for each consecutive step a softer plate than used for the previous one is used. As previously mentioned, process step 2 is executed on a copper plate. Two process versions are investigated: using a water-based and an oil-based lapping slurry. Figure 4 depicts the resulting surface after lapping for 60 minutes with 1.5-3 µm diamond grain. The roughness is further reduced when using a water-based slurry, while an oil-based one doesn’t result in further roughness improvements. What may also be noticed is a fine texture pattern which the authors expect to be caused by the variation in crystal orientation in the polycrystalline diamond film. A similar effect may be observed when surface machining multiphase ceramics where the different orientation of the single crystals leads to different elastically behavior and removal rates [7]. By conditioning a copper plate with diamond grain in a slurry, diamond grains were embedded in the copper. Combining it with a machining process using a water based fluid without diamond grain results in a process similar to nanogrinding. This approach did not yield any improvements over lapping: the machining time was up to two times longer than for lapping, while the surface roughness achieved was always in the same range as the one accomplished with lapping.

In order to reduce the roughness further, a third step using a tin plate which is very soft was executed. When machining ceramics, the use of a tin plate substantially decreases the cut rate. This could also be observed for the diamond film. A reduction of the roughness occurred much slower than when machining the film on a copper plate. Both lapping and nanogrinding using a water based slurry or nanogrinding fluid, respectively. Figure 5 shows a surface of a nanoground diamond film. The roughness could be further reduced to a $R_a$ of 6 nm after 120 minutes of machining. Lapping and nanogrinding achieved similar roughness results. The surface also shows the same texture as it did after machining on the copper plate.

After showing that mechanical machining techniques could be used to smoothen the diamond film to an average roughness $R_a$ in the nanometer range, additional tests were done to investigate the machining of discontinuous diamond surfaces. Such a discontinuous surface was
created by depositing the polycrystalline diamond film on a silicon wafer with surface waffle pattern created by anisotropic etching. As previously, the film thickness was 5 µm. A mechanical machining results in a cutting of the film at the surface while the film on the side walls and bottom of the pits remained. The process sequence developed was applied to this film. Figure 6 shows a series of optical micrographs made at different times while the diamond film was run through the different process steps. Figure 6(a) shows the film as deposited. In figure 6(b) and 6(c) the crystallites protruding from the surface were cut away. In figure 6(d) the diamond film was partially machined off and only at the edges some diamond film remained. By applying nanogrinding, the remainder of the film originally 5 µm thick was totally machined off the wafer surface, with film material remaining only at the pit sidewalls and bottom (Figure 6(e)). As previously in the case of the continuous film, no cracks or delaminations occurred.

![Figure 6: Microscope images of the diamond film removal.](image)

**Figure 6:** Microscope images of the diamond film removal. (a) starting point, (b) lapping on the composite plate, (c) lapping on the copper plate, (d) nanogrinding (e), width of the machined bar: 300 µm

### 4. Conclusion

The investigations showed that mechanical machining of polycrystalline diamond films created by HFCVD yields excellent roughness results. Thin diamond films on silicon substrates are machinable and with the three step process presented a smooth surface with an average roughness $R_a$ of as little as 6 nm can be achieved. A three step process using plates with staggered hardness yielded excellent results. For the first step, a composite plate composed of cast iron and copper is employed. For the second step, a copper plate and for the third a tin plate is used. The experiments further proved the feasibility of machining a polycrystalline diamond film with a diamond grit.

### Acknowledgement

This work was sponsored in part by the Center of Competence for Ultra Precise Surface Treatment in Braunschweig, Germany. The authors would like to thank the Institute for Microstructure Technology and Analysis at the University of Kassel in Kassel, Germany for the fabrication of the polycrystalline diamond films.

### References


