The homo-epitaxial growth of thick single crystal CVD diamond for detector applications

N. Tranchant*, P. Bergonzo & M. Nesladek, Darmstadt 2005
Introduction

Substrate pre-treatment

MW PECVD growth parameters

Time Of Flight

Raman

Conclusions
Pre-treatments

Substrate:
High Pressure High Temperature
Size: 3 x 3 x 0.5 mm
Sizes available up to 10 x 10 mm
Orientation (100)

Stripping:
$\text{H}_2\text{SO}_4 + \text{KNO}_3 + \text{Heating}$
(surface oxidation)

Ultrasonic bath DI $\text{H}_2\text{O}$
Microscope

HPHT substrates Sumitomo
Rms ~ 2-3 nm (AFM)
Growth

Plasma enhanced CVD AsTex PDS 17 MW reactor

Aim:
optimization of growth conditions
to obtain high quality layers

$\text{CH}_4$ concentration
Temperature
Pressure
MW Power
Plasma etching

- 4% O$_2$/H$_2$ plasma etching pretreatment for 90 min
  - P: 1000 W
  - p: 60-80 Torr
  - T: 800 °C
  - O$_2$/H$_2$: 4%

Surface quality important to reduce the occurrence of hillocks and unepitaxial crystallites

Anisotropic Plasma etching removes the surface defects (polishing etc.) and leaves smooth (100) surfaces with atomic steps, **Rms : 3 nm - 0.6 nm**

Etching:
- Power = 1000-3W
- Pressure = 70Torr
- Gas : H$_2$ = 96%, O$_2$ = 4%
- Time = 60-90 minutes
- Temperature = 930°C

See for details Growth and characterization of near-atomically flat, thick homoepitaxial CVD diamond films
G. Bogdan et al, Physica Status Solidi, 202 (2005), 2066
Growth parameters

Optimization of growth parameters to obtain right alpha parameter for growth of (100) films

High growth rate

<table>
<thead>
<tr>
<th>Rate (sccm)</th>
<th>H₂</th>
<th>CH₄</th>
</tr>
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<tbody>
<tr>
<td>460</td>
<td></td>
<td>30</td>
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<table>
<thead>
<tr>
<th>Percentage (%)</th>
<th>H₂</th>
<th>CH₄</th>
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<tbody>
<tr>
<td>93</td>
<td></td>
<td>7</td>
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<thead>
<tr>
<th>Time (Hours)</th>
<th>100</th>
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</thead>
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<tr>
<td>Velocity (µm/H)</td>
<td>11</td>
</tr>
<tr>
<td>Thickness (mm)</td>
<td>1-1.5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Power (W)</th>
<th>Pressure (Torr)</th>
<th>Temp (°C)</th>
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<tbody>
<tr>
<td>560</td>
<td>180</td>
<td>860 – 900</td>
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Sample

Result of step flow after 1 mm film growth

⇒ Steps height ≈ 20µm
⇒ No pyramidal hillocks

Growth of 1-1.5 mm thick, flat CVD diamond films
Fabrication of free standing films

Laser cutting (WTOCD Belgium)

Polishing

Free standing CVD diamond plates 5X5 mm²

Characterization: TOF, Spectroscopy
Time Of Flight (TOF)

Information on:

- Drift mobility
- Internal electric field
- Transit time of charge cloud induced by alpha particles
Time Of Flight (TOF)

Plating

Electron Beam Bombardment (oxidized surface)

Nickel \ Gold

Rate : 0.12nm/s

Thickness : 20 nm
Time Of Flight (TOF)

Experiment

Vacuum vessel ➔ Sample ➔ Pipe ➔ Pico second pulse generator ➔ Pre-amplifier ➔ Oscilloscope

Alpha source $^{241}$Am
TOF for electrons
TOF for Holes
Time Of Flight (TOF)

Results

Transit time for electrons and holes (100V)
\[ t_C = 2. - 2.7 \text{ ns} \]

\[ \Rightarrow \text{Drift velocity} : v_{\text{drift}} = 85 \mu\text{m/ns} \]

\[ \Rightarrow \text{Mobility} : \mu = 1800 - 2500 \text{ cm}^2/\text{V.s} \]
Raman spectroscopy

Homo-epitaxial layer

sp$^3$ signature only: cubic diamond
Raman spectroscopy

Peak: 1332 cm\(^{-1}\)
Bandwidth: 1.6 cm\(^{-1}\)

=> No stress
Next planning

- Raman spectrometry on the substrate layer
- Infrared spectrometry on homo-epitaxial layer (FTIR)
- Optimization of the growth conditions
- Studies on the growth parameter ($\alpha$)