Novel Polishing Process to Fabricate Ultra Low Thickness Variation Diamond Substrates For Next Generation Beam Tracking Detectors

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Outline

- Introduction- Sinmat
  - Sinmat- overview
  - Sinmat Technology & Products

- SBIR Project
  - Objectives
  - Results

- Conclusions and Future Directions
Overview: Sinmat Inc.

- University of Florida Spin-off. Developing planarization technologies for semiconductor industry
- Employees and consultants: 30
- **Global leader in SiC polishing slurries** (> 60% of global market): electronics for inverters, hybrid cars and SSL
- Approx 65% revenue from commercial products: Growth rate > 50%/year.
- Developing several CMP centric technologies – LEDs; Power/RF devices; Ultra large wafer polishing
- 30 patents (Issued and Pending)

President Obama congratulates Sinmat at White House for transforming R&D into clean energy jobs (March 2009)
Ultra-hard substrates for electronic & optics

- Silicon Carbide (SiC)
- Gallium Nitride (GaN)
- Sapphire (Al₂O₃)
- Diamond Substrates

- Among the hardest known materials
- Of Immense importance in electronic and photonic applications
Wide Band Gap Materials

Applications of (SiC, GaN, Sapphire & Diamond)

Power Devices

- AC-DC Converter
- DC to DC Converter
- Inverters

Light Emitting Devices (LEDs)

- Applications of Wide Band Gap Materials (SiC, GaN, Sapphire & Diamond)
- Power Devices: AC-DC Converter, DC to DC Converter, Inverters
- Light Emitting Devices (LEDs)
Sinmat develops products via planarization-enabled technologies that can be used in semiconductor manufacturing for computer chips, solid state lighting, and power devices.

Please visit www.sinmat.com
Polishing slurries:
- Diamond
- Silicon Carbide
- Nitrides
- Sapphire
- Patterned Sapphire Substrates
- Metals and Dielectrics
- Other Customized Slurries

Sinmat has over 20 different slurry products
For more info please visit www.sinmat.com
Polishing Services

Sinmat offers custom and standard polishing services for:

Silicon Carbide
Gallium Nitride
Diamond
Sapphire
Metal
Dielectric
Device Polish

Epiready Polish
Improving Flatness
Thinning
Specific Device Polish
Regular wafer Polish and reclaim

Sinmat has over 20 different slurry products
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Diamond Applications in Nuclear Physics

• High Thermal Conductivity
• Extreme Radiation Stability
• High Transparency (Optical/High Freq.)
• Excellent Electronics Properties

Ideal material of choice for wide range of applications in nuclear Physics!!!
Diamond Applications in Nuclear Physics

• Beam tracking detectors
  – National Superconducting Cyclotron Lab, Michigan State (US), GSI Darmstadt Germany

• Coherent bremsstrahlung radiators for high energy polarized photon beams
  – Nuclear experiments at JLAB and elsewhere

• Neutron detectors
  – Nuclear Power Industry, Homeland Security

• Dosimetry for protons, electrons and neutrons

• Detectors for high luminosity experiments –CERN

• X-ray monochromators, Optics and X-FEL-ANL,PETRA
# Ultra-Hard Materials: Polishing Challenges

<table>
<thead>
<tr>
<th>Materials</th>
<th>Hardness Knoop (Kg/mm²)</th>
<th>Chemical Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon Carbide</td>
<td>2150 - 2900</td>
<td>Inert</td>
</tr>
<tr>
<td>Gallium Nitride</td>
<td>1580 - 1640</td>
<td>Inert</td>
</tr>
<tr>
<td>Sapphire (Al₂O₃)</td>
<td>2000-2050</td>
<td>Inert</td>
</tr>
<tr>
<td>Diamond</td>
<td>8000 - 10000</td>
<td>Inert</td>
</tr>
</tbody>
</table>

- Polishing rate is slow
- Surface/Sub-surface Damage
Roughness Reduction of Micro Crystalline samples with RCMP

• Before Polishing

- Img. Rms (Rq) 81.127 nm
- Img. Ra 64.822 nm

• After Polishing

- Img. Rms (Rq) 0.335 nm
- Img. Ra 0.222 nm

Prior to Project
Silicon on Diamond Substrates

Diamond - Reactive chemical mechanical polishing process

- Ultra Smooth Diamond films (<0.3 nm rms roughness)
- Rapid, reliable, scalable polishing technology

Prior to Project
Timeline of Diamond Growth & Polishing

1952
First Synthetic Diamond methods (Eversole, Union Carbide)

1954
High pressure, high temperature diamond growth demonstration (GE)

1968
Low pressure vapor solid liquid technology for diamond growth (Derjayuin, Russia)

1981
Consistent demonstration of low pressure CVD growth of diamond (1µm/hr) Setaka NRIM, Japan

1989
Large area CVD growth of diamond company established (Diamonix)

2000
Ultra Nano Crystalline Diamond (UNCD) low-roughness diamond (~100 Å roughness (Krauss Argonne Lab)

2002
High rate single crystal diamond growth ability (Carnegie Institute, Washington)

2005
Super-smooth 1Å finished 100mm diamond substrates commercialized Sinmat

2007
Sales of CVD diamond plate products (Element 6, a De Beers Company)

1989
Large area CVD growth of diamond company established (Diamonix)
Sinmat’s Diamond Strategy

• Leverage its novel diamond polishing technology to fabricate high performance diamond based devices for Nuclear Physics Applications
  – Diamond Detectors
  – Ultra-Thin (<50 microns) Diamond radiator crystals
  – Diamond X-ray Optics
  – High thermal conductivity substrates

• Work collaboratively with diamond technology providers (e.g. Element Six) and National facilities to integrate diamond based products
SBIR Phase II Project Objective

Use Sinmat’s RCMP process to fabricate and evaluate diamond based detectors for high energy beam tracking application

- Optimize RCMP process
- Test & evaluate RCMP process for Detector fabrication

Phase II Plan

- Process Optimization
- Joint Detector Testing/Evaluation
- Large Area Diamond Detectors
- Flatness Optimization LTV
- Process Scalability
**Problem**

**Surface Polishing**

- X-ray topograph of single crystal diamond showing scratches
- Cathodoluminescence image of subsurface damage caused due to diamond based polishing
- AFM Picture shows surfaces scratch on diamond

a) Xiang Rong Huang, Albert T. Macrander, 10 International Conferences on Synchrotron Radiation Instrumentation
Example of energy straggling in detectors (a) showing poor energy resolution due to energy straggling in the detector and (b) Showing better energy resolution with lesser energy straggling [Muller]
Reactive CMP (RCMP): Soft layer Polish

- Chemically convert hard Diamond into a soft-layer
- Use nanoparticles
- Remove Soft layer
  - Achieve High Removal Rate
  - No Scratches
- Single Component Slurry
Reactive CMP (RCMP)

Coated Abrasive Particles → Diamond surface → Pressure

Surfactant → Tribo-Chemical mechanical action

Chemistry

Soft surface formation

Rapid-removal, Planarization

Double-sided polishing

No damage, Å level smooth

Low Thickness Variation
TEM pictures of ceria coated hard base particles
Coated Particle Enhances Chemical Reaction Under Pressure Locally for achieving higher material cut rate
Low Surface Damage
Material Removal Rate with three different RCMP Process

High Removal Rate RCMP Slurry

Medium Removal Rate RCMP Slurry

High Finish RCMP Slurry
**p-Crystalline Diamond Grain Flattening using RCMP**

(a) AFM of as received samples (RMS 1.2 micron)  
(b) After Polish RMS 0.6 micron  
(c) Peak to valley roughness before polish 5 micron  
(d) Peak to valley roughness 1.6 micron  
(e) As received sample showing sharp grains  
(f) Polished sample showing flattened grains  

(note porous structure between grains prevented achieving low RMS or PV).
p-Crystalline Diamond Polishing

<25 micron grains

As-received Lapped Sample

RMS ~ 59.4 nm
(5x5 sq.µm)

AFM Image

RMS ~ 4.6 nm
(5x5 sq.µm)

Wyko Image

Ra ~ 2.2 nm
(600 µm x 450 µm)

<25 micron grains
Large Grain Flattened by RCMP

> 100 micron grains

The initial surface and progressive changes over time during polishing
**Single Crystalline Diamond Polishing**

As-received Lapped Sample

RMS 119.6 nm (5x5 sq.µm)

AFM Image

RMS ~ 1.3 Å (1x1 sq.µm)

Wyko Image

Ra ~ 5.3 Å (300 µm x 235 µm)
X-ray Rocking Curve Studies

RCMP Process – reduced X-ray rocking curve width
Optical Microscope Images

- Surface morphology of HPHT diamond before and after RCMP

Mechanical polishing

- Scratch lines
- Fracture points
- Striations aligning to <110> direction

Reactive CMP

- Scratch lines absent
- Traces of fracture points
- Completely devoid of striation marks
SEM & Panchromatic CL (HPHT)

- Mechanical polishing
  - Dark spots indicating the fracture defects as non-emission points
  - Multiple V-fracture

- Reactive CMP
  - No dark spots
  - Completely free of fracture defects

Arul Arjunan, Jinhyung Lee, R.K. Singh, MSEB
CL Spectra (Spot Area Mode)

Mechanical Polished (5kV, 4000X)

(a) → Scratch line defect

(b) → V-fracture defect

(c) → Polishing defect free region

Arul Arjunan, Jinhyung Lee, R.K. Singh, MSEB
CL spectra (Mechanical vs. RCMP)

CL spectra of HPHT diamond (at 5kV)

CL spectra of HPHT diamond (at 10kV)

Arul Arjunan, Jinhyung Lee, R.K.Singh, MSEB
Cross-sectional TEM

- Depth of polishing damage ~ 150nm
- Fractures penetrated with 54.7° direction of polished plane
  - Lower strength and energy for fracture on \{111\} planes

Arul Arjunan, Jinhyung Lee, R.K. Singh, MSEB
RCMP Scale-up

Large Area Substrate Polishing

4 inch sample polishing

Multiple Sample Polishing

Five 3” inch samples polishing
Solution 1: Ultra Flat Holder

Proposed RCMP Processes using specialized holder

Damage-free smooth Diamond-High TTV >10µm

Low TTV holder

Damage Free/ Smooth/ Low TTV Diamond

Thickness Variation <0.5 µm

100µm
Solution 2: Double-sided polishing

Schematic of (a) double sided polishing (b) Gear sample fixture (c) unpolished samples (d) double sided polished sample with ultra low TTV
Custom Double-sided polishing

(a) Rotation of carrier and plate in the same direction & Plate will wear concave
(b) Carrier and plate rotation in opposite direction & Plate will wear convex
Custom Double-sided polisher

Samples Polished
Ultra Flat Polishing

Peak to Valley Roughness
2.9 micron

Peak to Valley Roughness
0.55 micron

Single crystalline sample (a) before polishing showing non-flat surface (PV~2.9) (b) flat surface after polishing (PV~ 0.55)
Ultra smooth Surface Finish

RMS roughness reduction by 1 order and optical flatness reduction from 1.6 micron to 0.5 micron with Sinmat’s Reactive CMP (RCMP) polishing, (b) AFM picture of standard polish showing fractured surface (RMS 20A, (Peak to Valley) PV 90A) and (c) AFM picture of RCMP surface shows atomic smooth surface (RMS 1A, PV 10A).
Stress Free Polishing - Reduced Bow

- Mechanical Polishing
- RCMP
- As is
Detector response for U232 Alpha source at 100V bias (a) vendor polished sample – showing pulse height of 20-30 mV (b) RCMP polished diamond sample showing pulse height of 80mV. Both the plates were approximately 100µm thick (Courtesy: Dr. Stolz, at NSCL)

Polishing did not degrade the detector performance
Detector Testing-OSU

90Sr source

Set-up to Measure Charge Collection Distance

RCMP Polished Sample showing Higher Charge Collection Distance: Courtesy: Dr. Harris Kagan OSU
Detector Testing-OSU

- RCMP Improves Detector Performance Significantly
- Mechanical Polishing Degrades Detector Performance

Courtesy: Dr. Harris Kagan OSU
Enhanced Charge Collection Distance after removing surface defects

Courtesy: Dr. Harris Kagan OSU
Conclusion

RCMP Process helps achieving surface which are:

- Ultra smooth ~ 1-2 A
- Damage Free surface
- Ultra Flat (Peak valley roughness <1 micron)

Detectors fabricated using RCMP process have 30 to 40% higher charge collection distance

RCMP process is scalable to large size diamond crystals

Future Work:

Reduce thickness of as received diamond plates from 500 to 100 micron with two step process & test the process.

Scale-up & Integration of RCMP for diamond that can be used higher energy beam tracking applications.
Thank you for Collaboration

Dr. Andreas Stolz MSU
Dr. Harris Kagan MSU