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

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Gaithersburg, MD
Outline

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

- SBIR Project
  - Objectives
  - Proposed Work
  - Results

- Commercialization
  - Customized solutions for broader acceptable markets
  - Accomplishments
Introduction - Sinmat
Overview: Sinmat Inc.

- Global market leader in polishing of ultra hard materials (e.g. Silicon Carbide, GaN, Diamond, Sapphire)
- 80% of SiC global polishing market; (All Major Substrate Manufacturing Companies)
- > 1000 MT/month slurry manufacturing capability.
- Employees and consultants: 30 (6 PhDs, 5 Masters)
- 20,000 sq.ft R&D and manufacturing space
- Approx 75% revenue from commercial products: Growth rate > 50%/year.
- Developing CMP centric technologies: 30 patents
Sinmat’s Core Competency

Develop A High Quality, Affordable Supply Of Innovative Surface Preparation - Promoting Products To Serve A Variety Of Markets

- **Chip Mfg Markets**
  - Intel
  - Samsung

- **Power Device Markets (SiC)**
  - Cree
  - RF Micro
  - Raytheon
  - Rohm

- **LED Market**
  - Sapphire
  - AlN, GaN

- **Emerging Markets (Polishing of Hard Materials)**
  - Smart Phones
  - Wearable Electronics
  - PCD tools for Drilling
  - Diamond/SiC seals (automobiles etc)
  - DOE & DoD Applications
Chemical Mechanical Polishing (CMP) Process

Key Characteristics:

- Slurry consisting of chemicals and abrasive particles
- Platen with a plate or pad and a wafer head holding the wafer
- Planarization with simultaneous application of Chemistry (Chemical) and Force/ Friction (Mechanical).

• Key Differences with Diamond Lapping
  - Use of Plate/Pad
  - Chemical & Mechanical Action in Polishing Process
Sinmat develops products via planarization-enabled technologies for semiconductor manufacturing for computer chips, solid state lighting, power devices, and wearable electronics.

Please visit www.sinmat.com
Sinmat
Innovative CMP solutions

Slurry Products & Polishing Services

Polishing slurries & services:
- Diamond
- Silicon Carbide
- Nitrides
- Sapphire
- Patterned Sapphire Substrates
- Metals and Dielectrics & Device
- Other Customized Slurries

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

Sinmat has over 20 different slurry products
For more info please visit www.sinmat.com
Phase II Project
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!!!*
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)

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

2002
Ultra Nano Crystalline Diamond (UNCD) low-roughness diamond (~100 Å roughness (Krauss Argonne Lab)

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

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

• 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
Sinmat’s Diamond Strategy

• Leverage novel diamond polishing technology to fabricate high performance diamond based devices for NP and HEP Applications
  – Diamond Detectors (NSCL)
  – 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
Roughness Reduction of Micro Crystalline samples with RCMP

- Before Polishing
- After Polishing

Prior to Project

<table>
<thead>
<tr>
<th>Image</th>
<th>Rms  (Rq)</th>
<th>Ra</th>
</tr>
</thead>
<tbody>
<tr>
<td>Img.</td>
<td>81.127 nm</td>
<td>64.822 nm</td>
</tr>
</tbody>
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<thead>
<tr>
<th>Image</th>
<th>Rms  (Rq)</th>
<th>Ra</th>
</tr>
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<tbody>
<tr>
<td>Img.</td>
<td>0.335 nm</td>
<td>0.222 nm</td>
</tr>
</tbody>
</table>
SBIR Phase II Project Objective

Use RCMP process to fabricate and evaluate diamond based detectors for high energy beam tracking applications

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

Flatness and Thickness Tolerance Variation

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

Tribo-Chemical mechanical action

Double-sided polishing

No damage, Å level smooth

Low Thickness Variation

Rapid-removal, Planarization
RCMP slurry - Coated Particles

- 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)
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

AFM Image

Wyko Image

RMS 119.6 nm
(5x5 sq.µm)

RMS ~ 1.5 Å
(1x1 sq.µm)

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)

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

Mechanical polishing

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)

- Scratch line defect
- V-fracture defect
- 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 (Phase I)

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 Phase II

Schematic of (a) double sided polishing (b) Gear sample fixture (c) unpolished samples (d) double sided polished sample with ultra low TTV
Solution 3: Dry Etch + DS-RCMP Phase II Extension

Process flow schematic to show fabrication of ultra-low TTV electronic-grade diamond crystals for detectors using (1) Electronic grade selection (2) vapor phase etching followed by (3) Double side RCMP process
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)
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 (Solution 2)

$^{90}\text{Sr}$ source

Set-up to Measure Charge Collection Distance

RCMP Polished Sample showing Higher Charge Collection Distance:
Courtesy: Dr. Harris Kagan OSU
Detector Testing-OSU (Solution 2)

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

3 micron diamond Polishing

1/4 micron diamond Polishing

Courtesy: Dr. Harris Kagan OSU
Detector Testing-OSU (Solution 2)

Enhanced Charge Collection Distance after removing surface defects

Courtesy: Dr. Harris Kagan OSU
Charge Collection Distance vs High Voltage
As received 530 um sample shows 200 um ccd
Processed sample thickness 230um shows ccd 150um

Dr. Kagan OSU
Charge Collection Distance vs E-field

- As received 530 um sample shows breakdown at 1.75 Volts/um
- Processed sample thickness 230um shows breakdown at 3.1 Volts/um

Dr. Kagan OSU
Detector Testing-OSU (Solution 3)

Charge Collection Distance/thickness vs E-Field (Volts/um)

- Shows > 50% more charge collection and break down

Dr. Kagan OSU
Commercialization Plan
Custom Solution 1: Optical Sapphire/SiC Finishing Technologies

I. Grinding 30-40 micron hard carbide
II. Lapping – 3-5 microns diamond slurries
III. Final Polishing – Silica based slurries

- Finishing more intensive operation
  - Both side polishing
  - 3 dimensional surfaces/multiple edges for wearable electronics
## Nano-Diamond Lapping Dilemma

<table>
<thead>
<tr>
<th>Issues</th>
<th>Mechanical Polishing (MP)</th>
<th>Nano diamond Polishing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Core particle Size</td>
<td>3-5 µm diamond</td>
<td>Nano diamond 5 to 500 nm particles</td>
</tr>
<tr>
<td></td>
<td>High sub-surface damage</td>
<td>Reduced sub-surface damage</td>
</tr>
</tbody>
</table>

- Small diamond polishing significantly reduces size of defects
- Low polishing rates observed

![Graph showing removal rates](image-url)

**Graph:**
- Large Diamond Polish: > 10 - 30 µm/hr
- Nano Diamond Polish: < 1 µm/hr
Nanodiamond CMP can have similar polishing rates as 3-5 micron poly diamond (40-50 microns/hr)

20 to 40 times higher than particles alone
Comparison Surface Finish: Diamond Lapping and Nanodiamond CMP

Diamond Lapping

- Ra ~ 8-10 nm
- Damage 10μm+

Nanodiamond CMP

- Ra ~ 0.7 nm
- Damage 0.25μm

Silica CMP

- Ra ~ 0.4 nm
- No Damage

• Nanodiamond CMP has surface quality close of standard CMP process but with 10-15 X higher rates
  • Significantly smaller flaw size (25X low subsurface damage
  • Reduce Polish Times by 5X
Customized Solution 2: A-Face Sapphire Polishing for Wearable Electronics Applications

Reactive Chemical Mechanical Polishing (RCMP) of Sapphire

- High Polishing Rate, High Surface Finish Process
- Working together with end users
- Process Qualified For Wearable electronics with fortune 100 companies
- Expected High ROI
Customized Solution #2: Ultra-Rapid Polishing of SiC & GaN

- Ultra–Rapid Polishing Rates (SiC, GaN)
- Ultra–smooth Flawless Surfaces (SiC and GaN)
- Low stress/ Robust Polishing Process
- >80% of worldwide CMP market (~1 million wafers/year)
Customized Solution # 3: Polishing of Diamond Films for Thermal Conductivity Applications

Reactive chemical mechanical polishing (RCMP) process
- Ultra Smooth Diamond films (<0.3 nm rms roughness)
- Rapid, damage-free, scalable polishing technology

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

TEM of Fabricated Si on Diamond
TEM of Fabricated GaN on Diamond
Customized Solution Example # 4: Scalable CMP Polishing Poly diamond Composite & Seals

- First time demonstration of smooth (Ra = 4-5 nm) PCD materials using CMP
- Scalable, Low pressure, short time, low velocity, low temperature process
- Can be adapted to non-flat 3D surfaces, nitride materials
- Low consumable cost
- Process qualified with leading manufacturers of PDC

http://www.eagleburgmannnow.com/
Summary Of Accomplishments:

Developed RCMP Process help to achieve:

- Faster removal rate 10X
- Ultra smooth (~ 1-2 A) & Damage Free surface on diamond
- Ultra Flat and Low TTV (Peak valley roughness <0.5 micron) diamond
- Scalable to large size and multiple crystal polishing
- 30 - 50% higher charge collection distance
- Patent filed for the technology

Commercialization:

- DOE & DOD Applications
- Wearable and other electronic applications: Nano diamond reactive mechanical polishing for Sapphire, SiC and other hard materials
- Poly diamond compacts for Oil and Gas drilling applications & cutting polishing
- Silicon on diamond and GaN on diamond for thermal management in high power and future Integrated circuits
Thank you for Collaboration

Harris Kagan Ohio State University
Andreas Stolz NSCL Michigan State University