# 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
- Winner of four R&D 100 Awards 2004 & 2005, 2008, 2009
- 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)

**2004, 2005, 2008, 2009**

R&D 100 | R&D 100 | R&D 100 | R&D 100

**President Obama congratulates Sinmat at White House for transforming R&D into clean energy jobs (March 2009)**

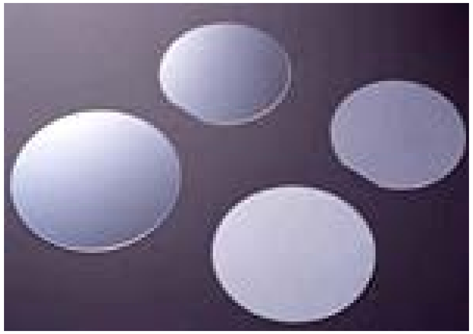
# Ultra-hard substrates for electronic & optics

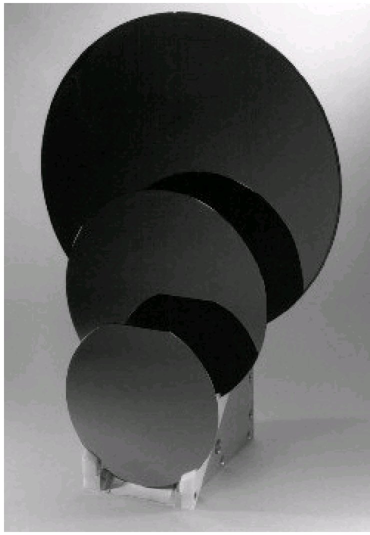
Silicon Carbide (SiC)

Gallium Nitride (GaN)

Sapphire ($Al_2O_3$)

Diamond Substrates





- Among the hardest known materials
- Of Immense importance in electronic and photonic applications

# Wide Band Gap Materials

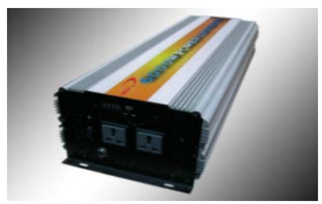
## Applications of (SiC, GaN, Sapphire & Diamond)

### *Power Devices*

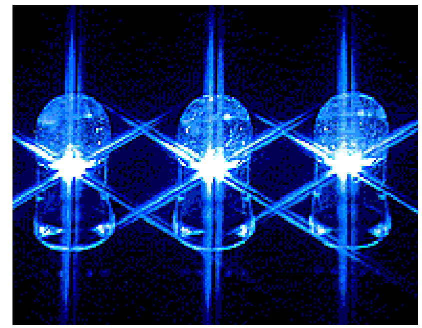
*AC-DC Converter*

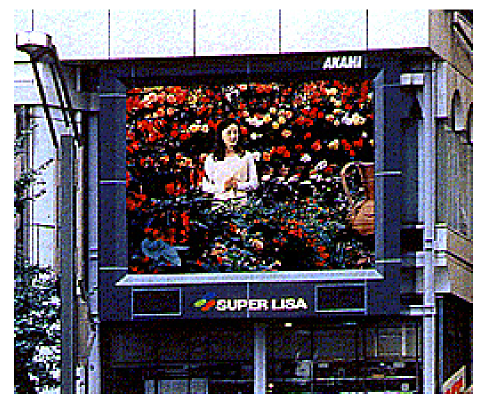
*DC to DC Converter*


*Inverters*

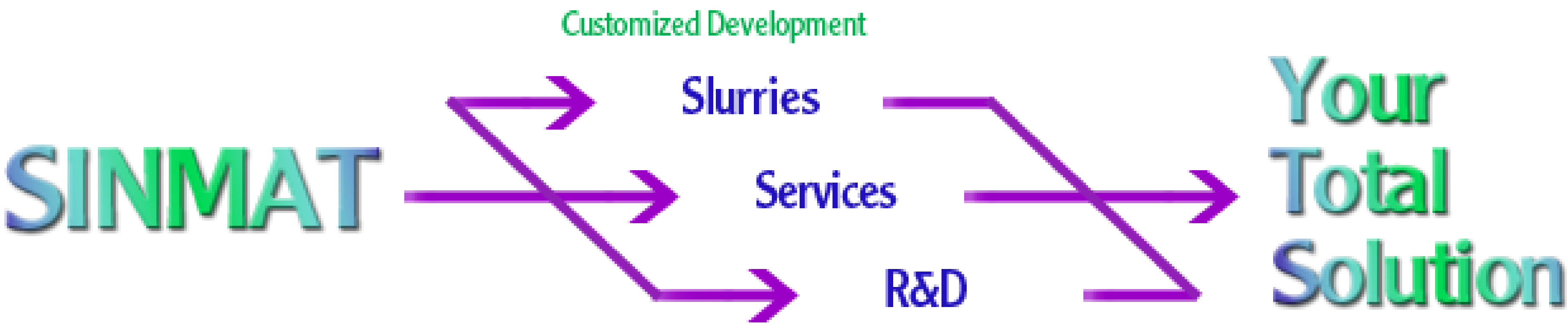


### *Light Emitting Devices (LEDs)*





# Sinmat Inc




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

# Slurry Products


Atomically terraced surface
After SC final CMP (AFM)

**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](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*
*For more info please visit [www.sinmat.com](http://www.sinmat.com)*

# 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

| Materials | Hardness Knoop (Kg/mm$^2$) | Chemical Action |
|---|---|---|
| Silicon Carbide | 2150 - 2900 | Inert |
| Gallium Nitride | 1580 - 1640 | Inert |
| Sapphire ($Al_2O_3$) | 2000-2050 | Inert |
| Diamond | 8000 - 10000 | Inert |

- **Polishing rate is slow**
- **Surface/Sub-surface Damage**

# Roughness Reduction of Micro Crystalline samples with RCMP

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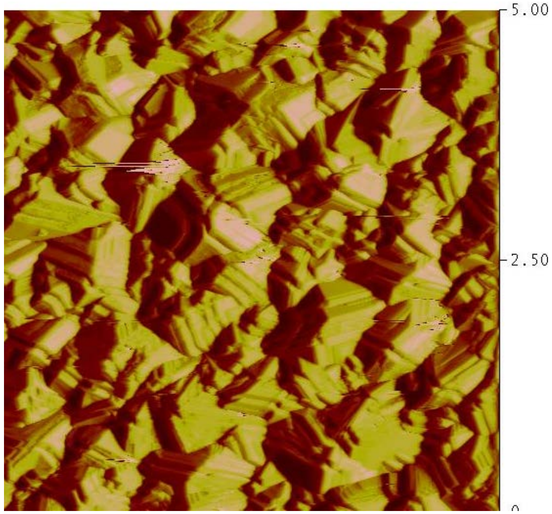
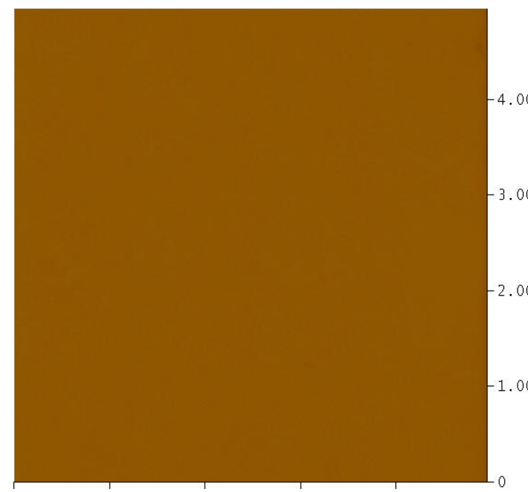
- Before Polishing
- After Polishing




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




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

***Prior to Project***

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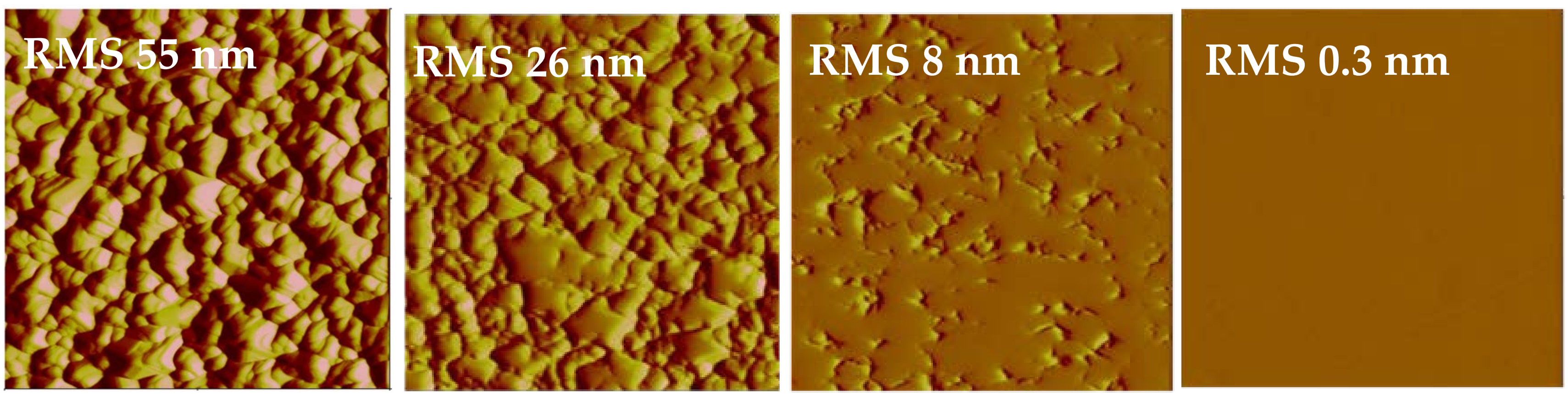
# Silicon on Diamond Substrates

Diamond - Reactive chemical mechanical polishing process

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




***Prior to Project***

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# Timeline of Diamond Growth & Polishing

**1952**
**First Synthetic Diamond methods (Eversole, Union Carbide)**

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

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

**2005**
**High rate single crystal diamond growth ability (Carnegie Institute, Washington)**

**2009**
**Super-smooth 1Å finished 100mm diamond substrates commercialized Sinmat**

**1950 1960 1970 1980 1990 2000 2010**

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

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

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

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

# Sinmat's Diamond Strategy

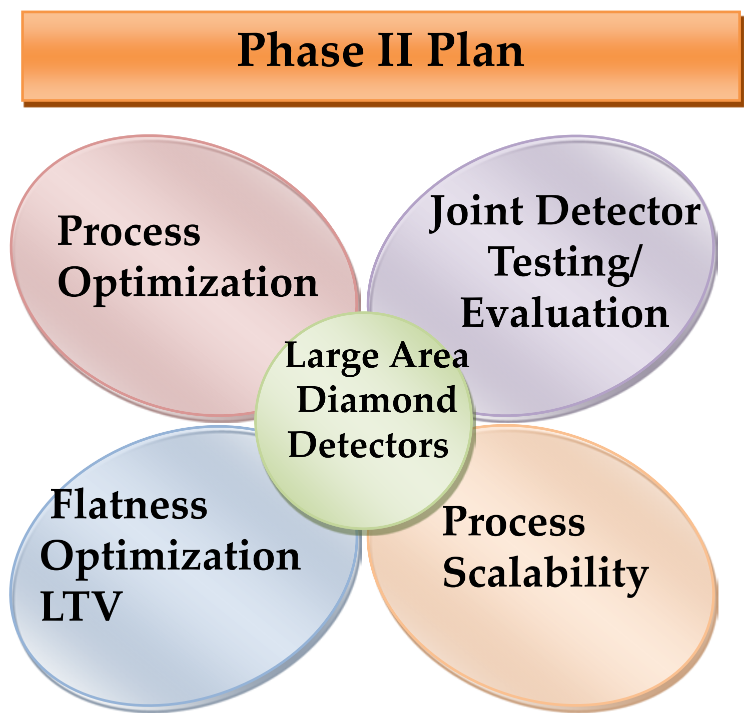
- Leverage its novel diamond polishing technology to fabricate high performance diamond based devices for Nuclear Physics Applications
  - Diamond Detectors
  - Ultra-Thin (< 50 microns) Diamond radiator crystals
  - Diamond X-ray Optics
  - High thermal conductivity substrates

- Work collaboratively with diamond technology providers ( e.g Element Six) and National facilities to integrate diamond based products

# SBIR Phase II Project Objective

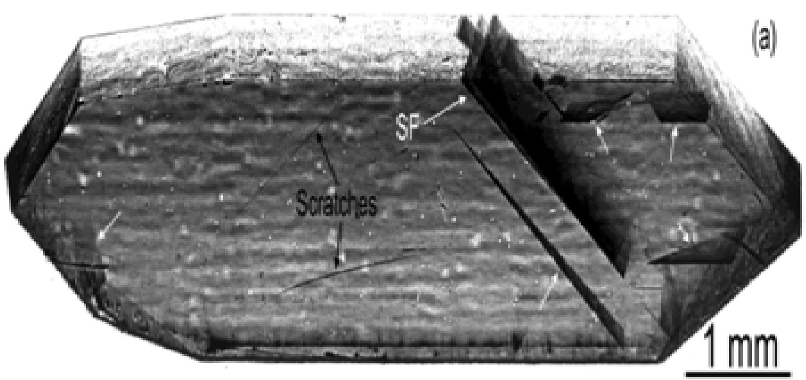
Use Sinmat's RCMP process to fabricate and evaluate diamond based detectors for high energy beam tracking application

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

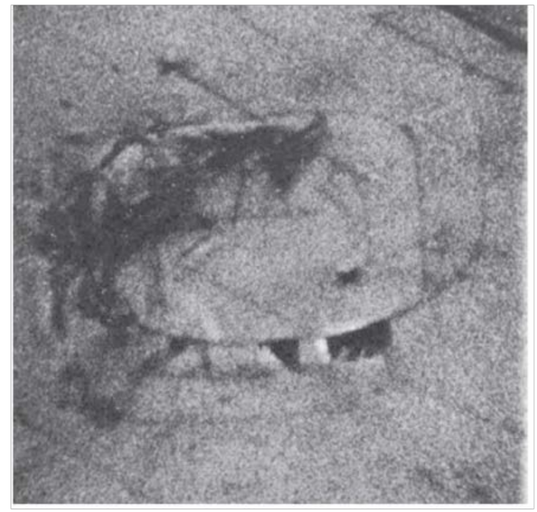


# Problem

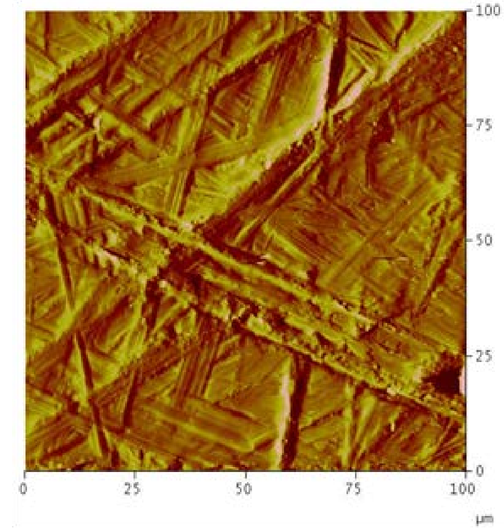
## Surface Polishing




X-ray topograph of single crystal diamond showing scratches



Cathodoluminescence image of subsurface damage caused due to diamond based polishing

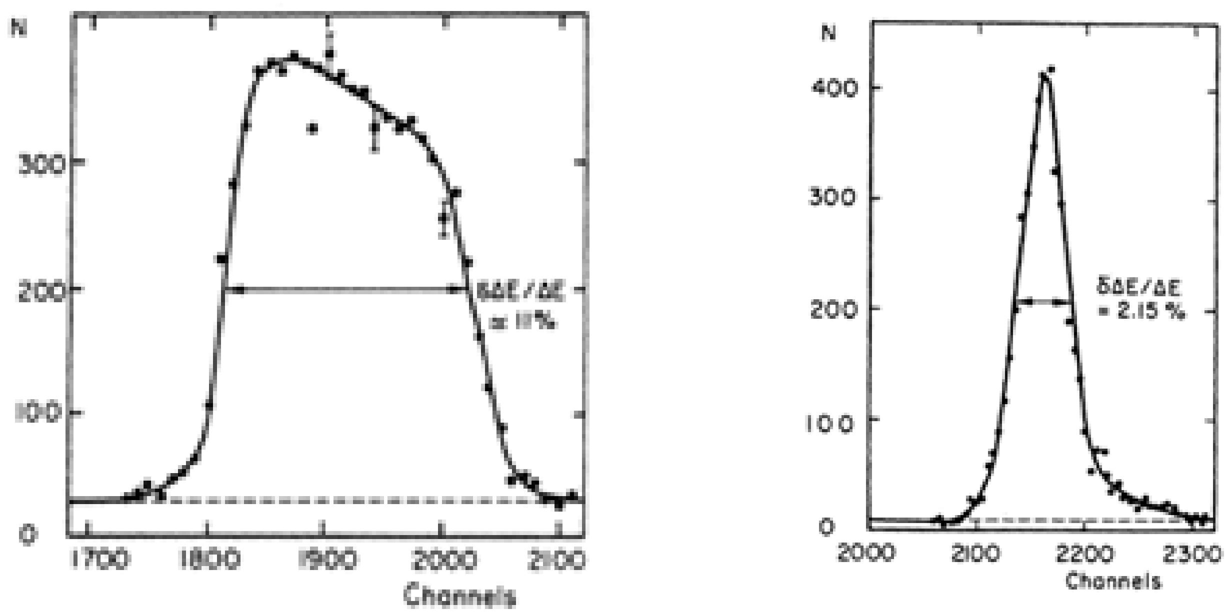



AFM Picture shows surfaces scratch on diamond

a) Xiang Rong Huang, Albert T. Macrander, 10 International Conferences on Synchrotron Radiation Instrumentation
b) Nature Letters M.Casy, Wilks 1973 vol.239 Page 394

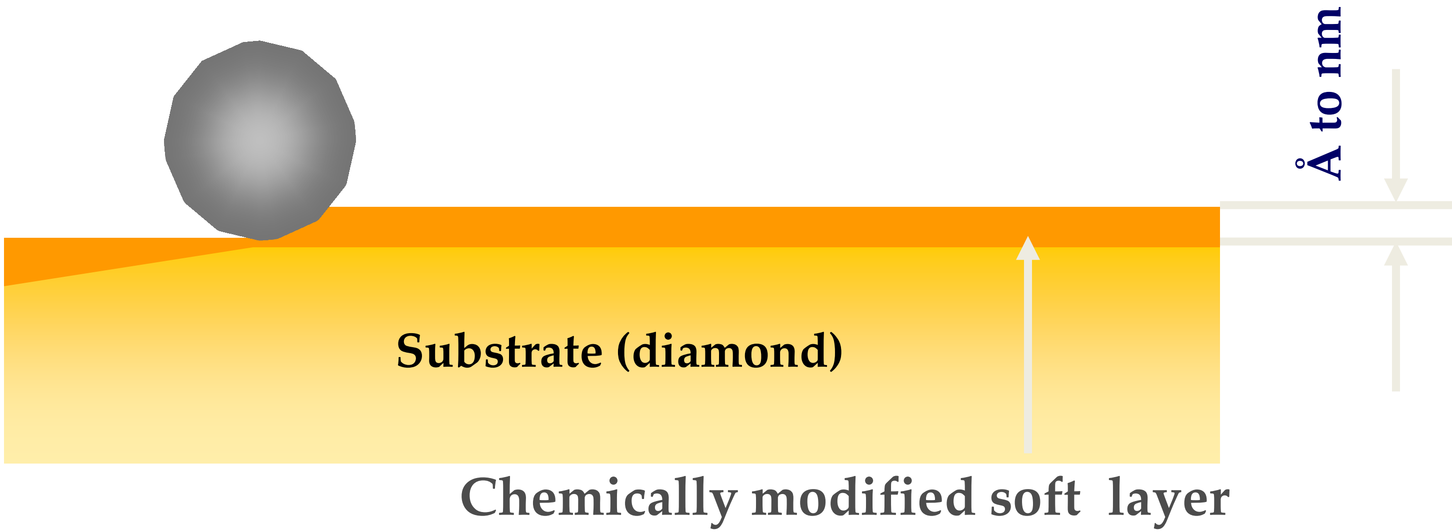
# Problem

## Flatness and Thickness Tolerance Variation



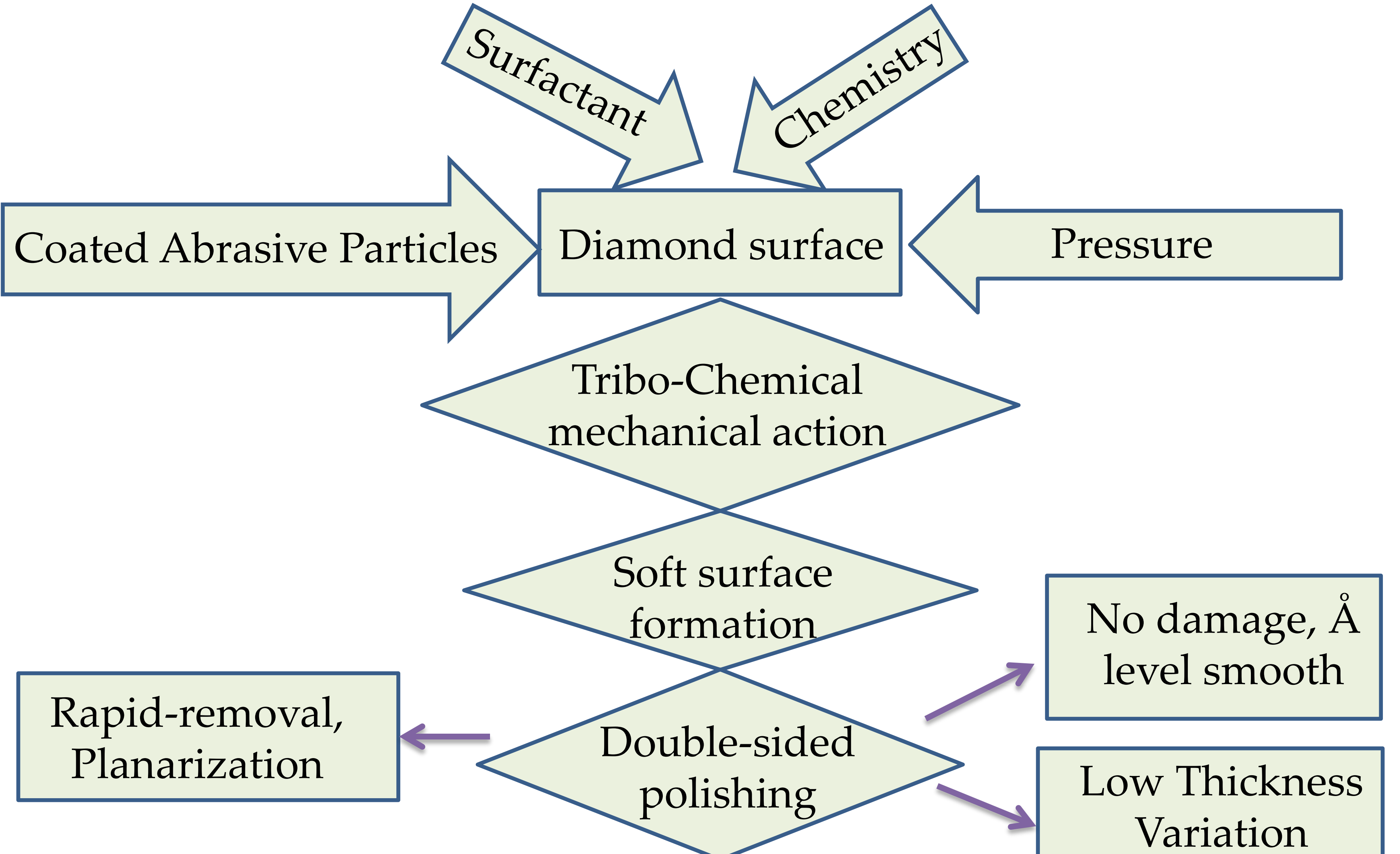

Example of energy straggling in detectors (a) showing poor energy resolution due to energy straggling in the detector and (b) Showing better energy resolution with lesser energy straggling [Muller]

# Reactive CMP (RCMP): Soft layer Polish

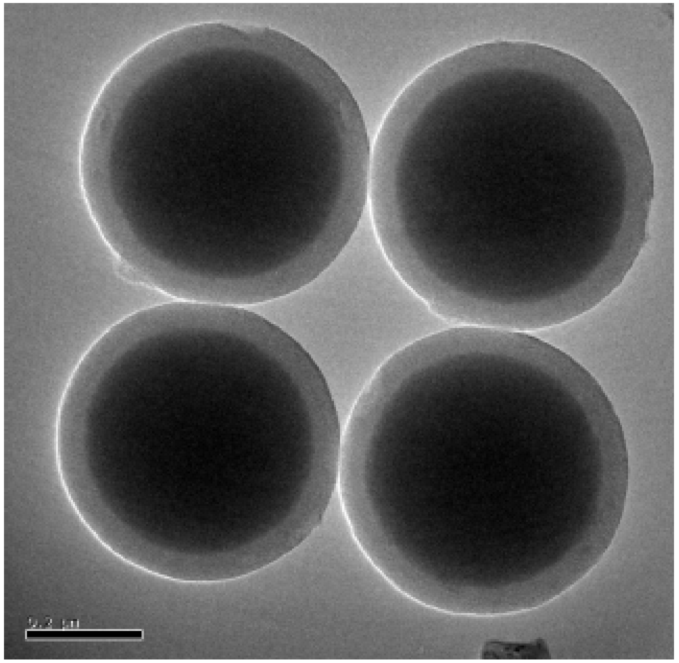



- Chemically convert hard Diamond into a soft-layer
- Use nanoparticles
- Remove Soft layer
  - Achieve High Removal Rate
  - No Scratches
- Single Component Slurry

# Reactive CMP (RCMP)



# 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

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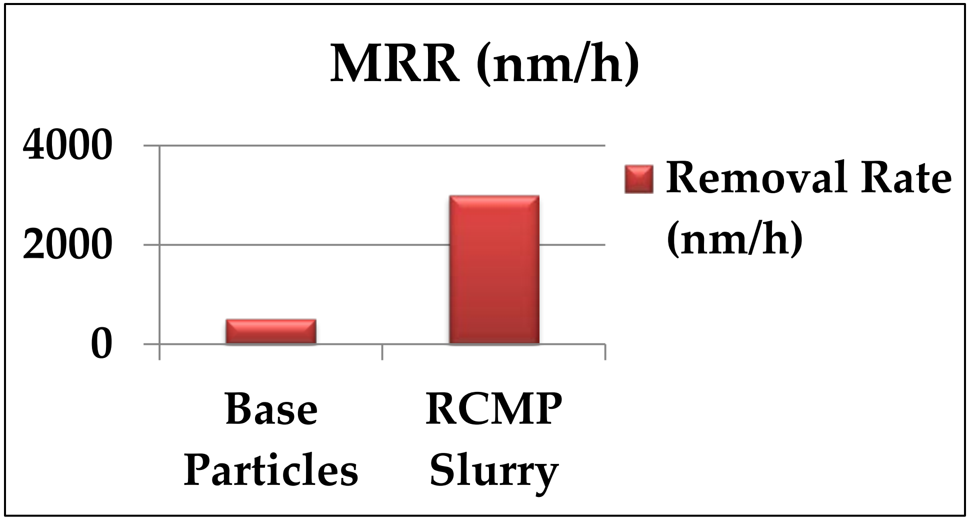
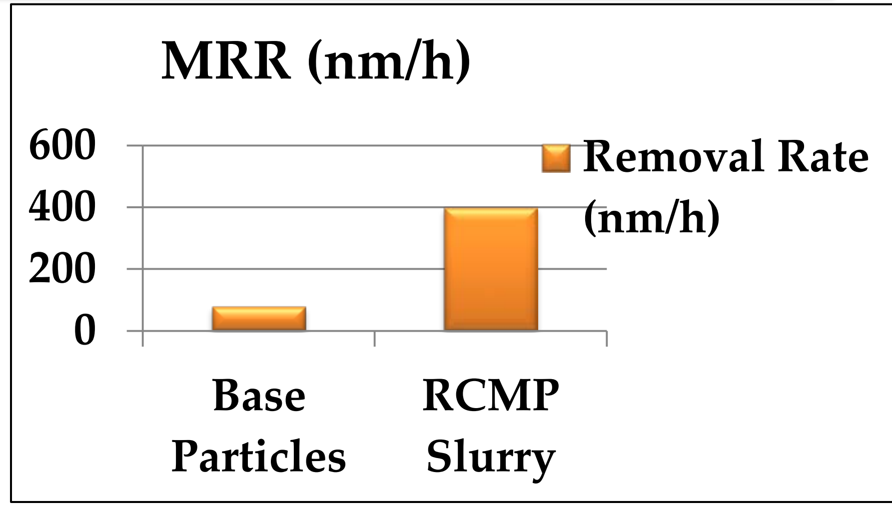
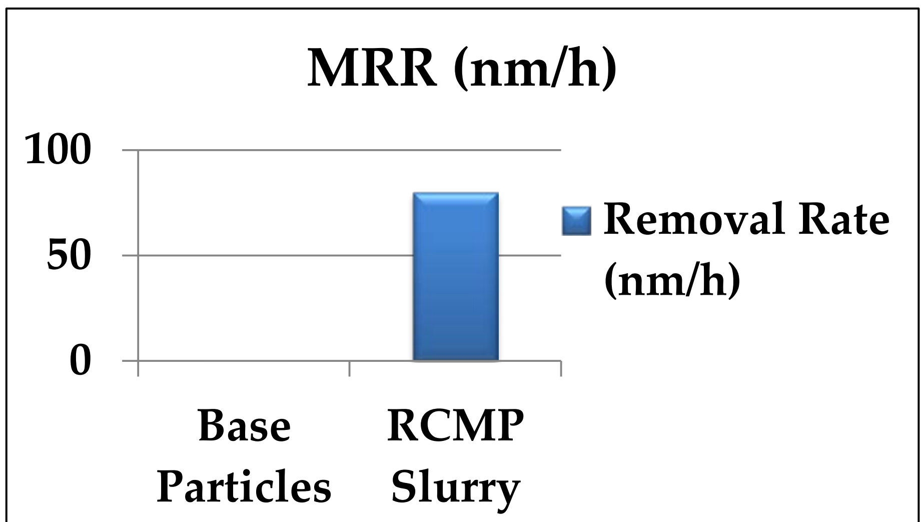



High Removal Rate RCMP Slurry



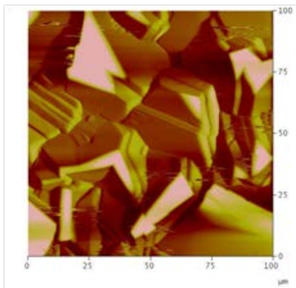

Medium Removal Rate RCMP Slurry



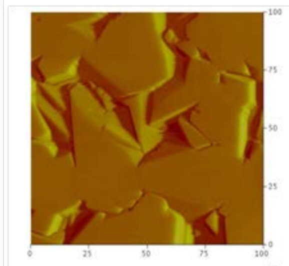

High Finish RCMP Slurry

# p-Crystalline Diamond Grain Flattening using RCMP

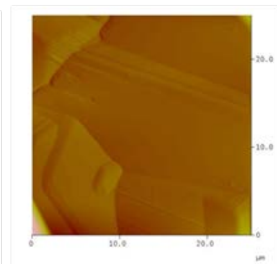




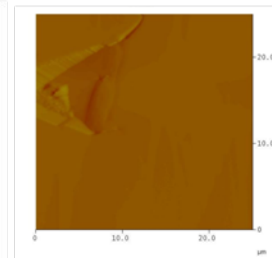
RMS 1.2 micron



RMS 0.6 micron



Thickness variation on PV 5 micron



Thickness variation on PV 1.5 micron

(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).

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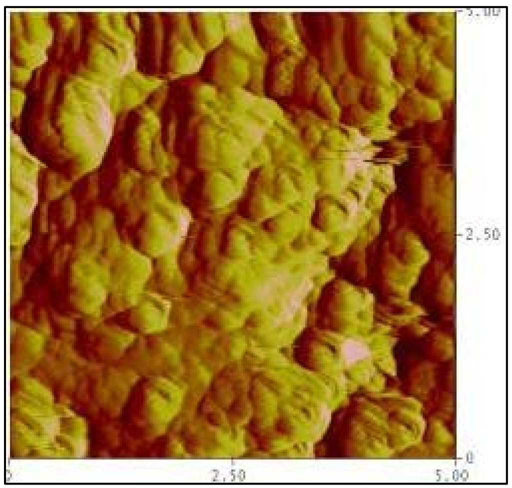
# p-Crystalline Diamond Polishing

## <25 micron grains

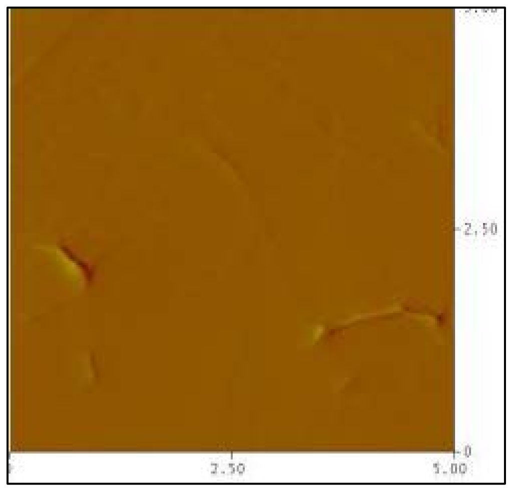
As-received
Lapped Sample




RMS ~ 59.4 nm

**(5x5 sq.µm)**

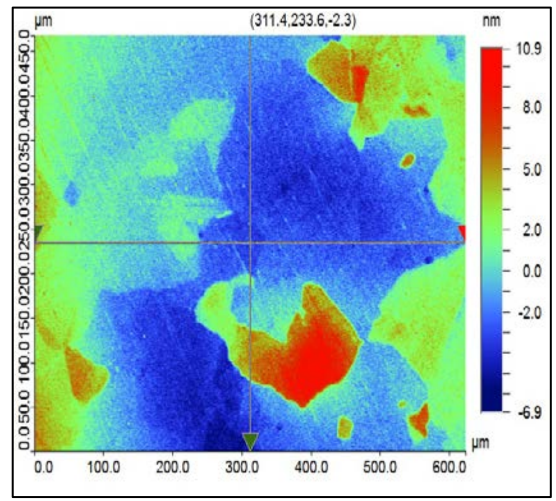
AFM Image




RMS ~ 4.6 nm

**(5x5 sq.µm)**

Wyko Image




| Label | Value | Units |
|---|---|---|
| Average | 0 | nm |
| Ra | 2.263 | nm |
| Rp | 10.889 | nm |
| Rq | 2.837 | nm |
| Rt | 17.745 | nm |
| Rv | -6.856 | nm |

Ra ~ 2.2 nm

**(600 µm x 450 µm)**

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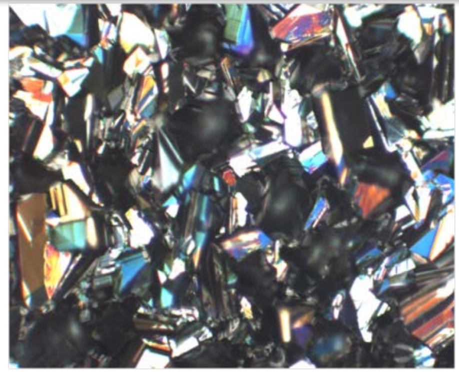
# Large Grain Flattened by RCMP

> 100 micron grains



**The initial surface and progressive changes over time during polishing**

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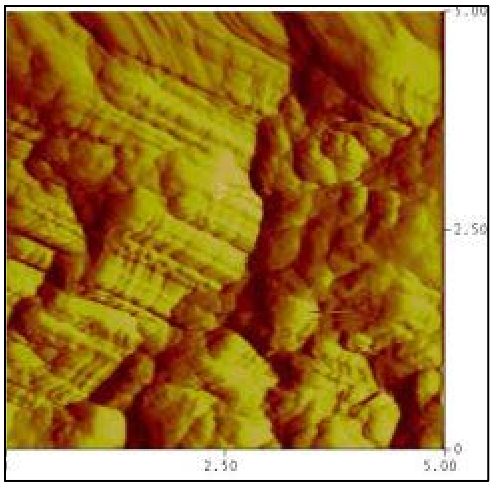
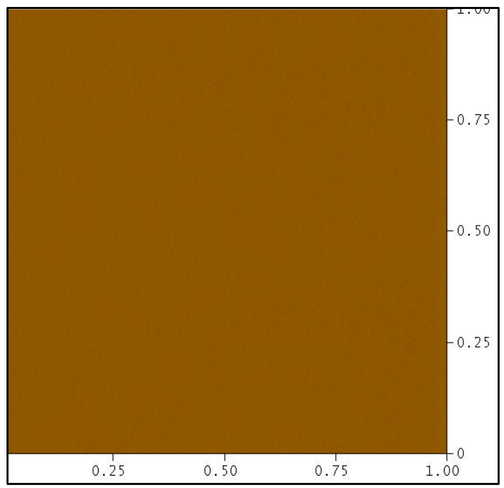
# Single Crystalline Diamond Polishing

| As-received Lapped Sample | AFM Image | Wyko Image |
|---|---|---|



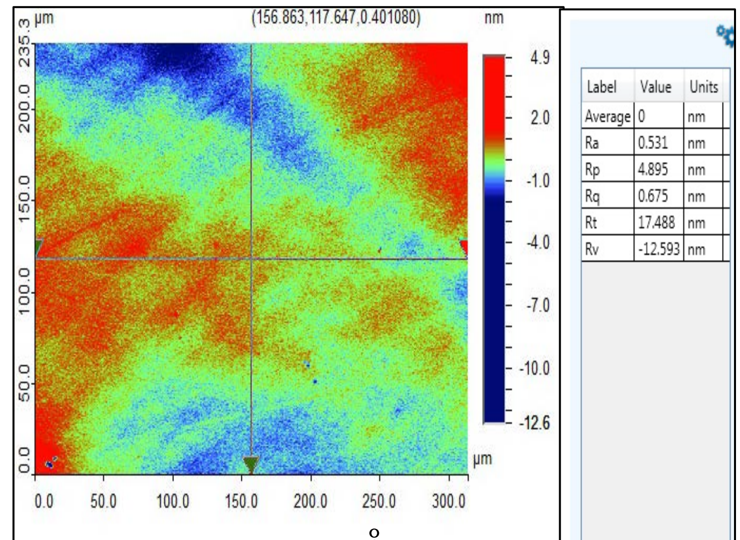

RMS 119.6 nm

**(5x5 sq.µm)**




RMS ~ 1.3 Å

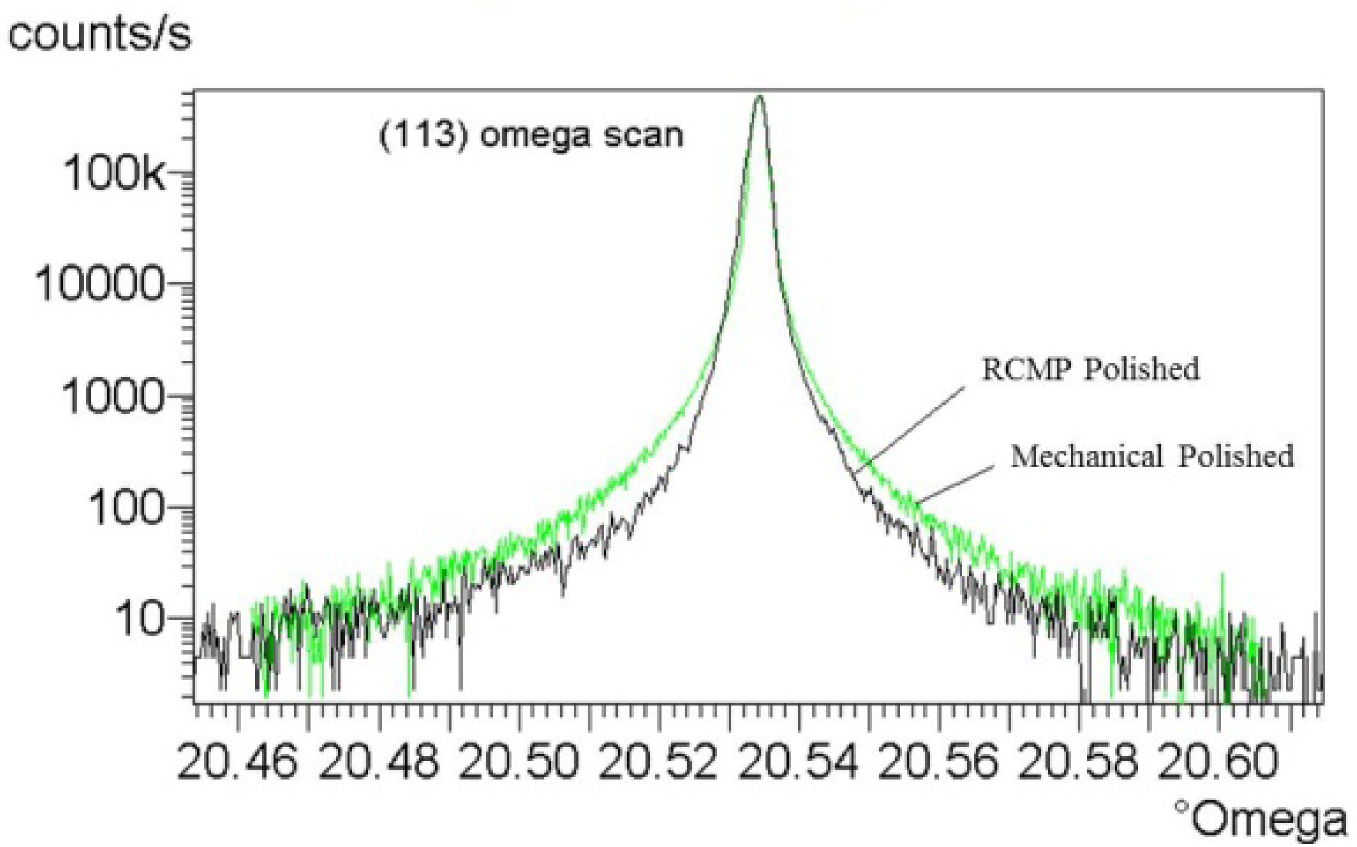
**(1x1 sq.µm)**




Ra ~ 5.3 Å

**(300 µm x 235 µm)**

# X-ray Rocking Curve Studies

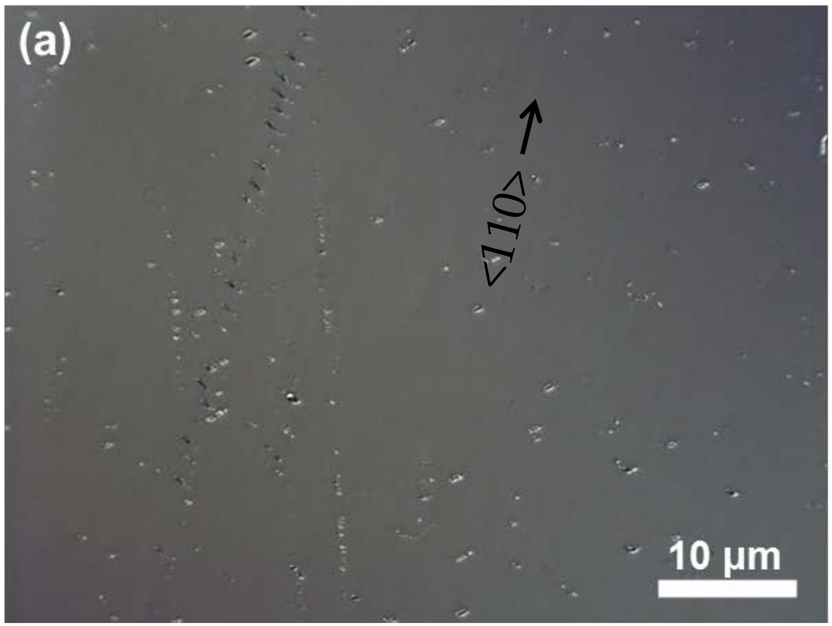



**RCMP Process –reduced X-ray rocking curve width**

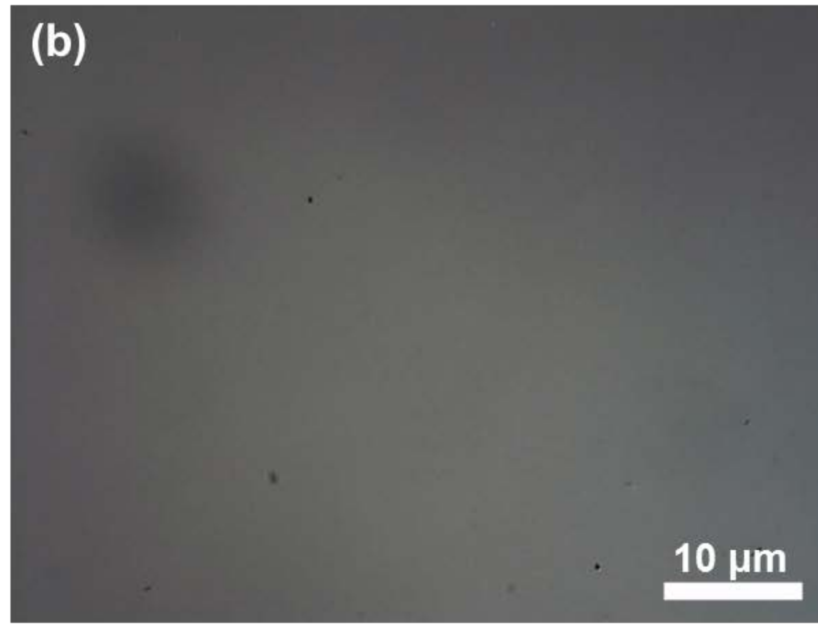
# Optical Microscope Images

- Surface morphology of HPHT diamond before and after RCMP

Mechanical polishing




Reactive CMP




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

- Scratch lines absent
- Traces of fracture points
- Completely devoid of striation marks

# SEM & Panchromatic CL (HPHT)

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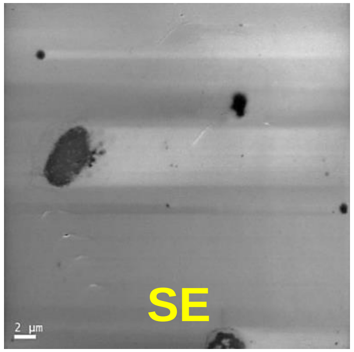
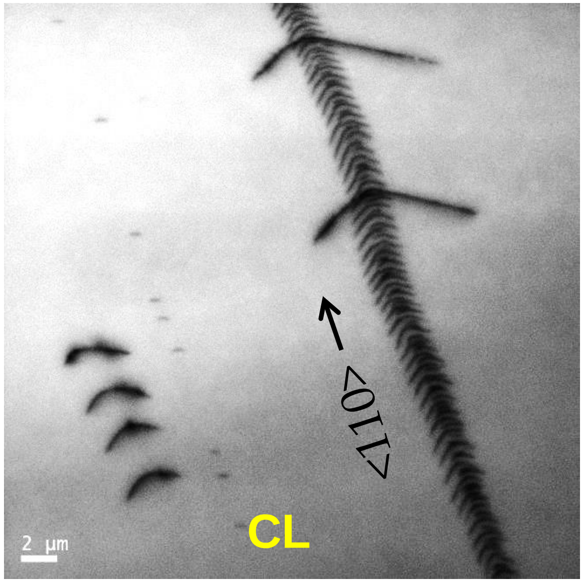
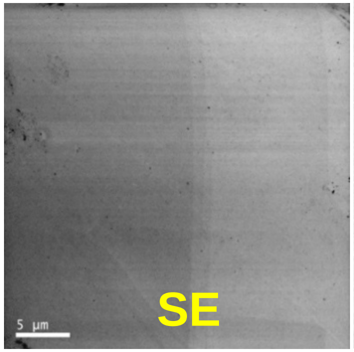
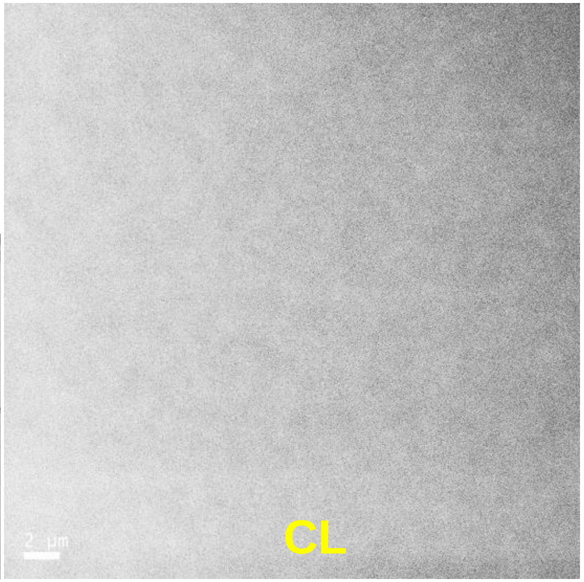
**Mechanical polishing**




**Reactive CMP**




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

- No dark spots
- Completely free of fracture defects

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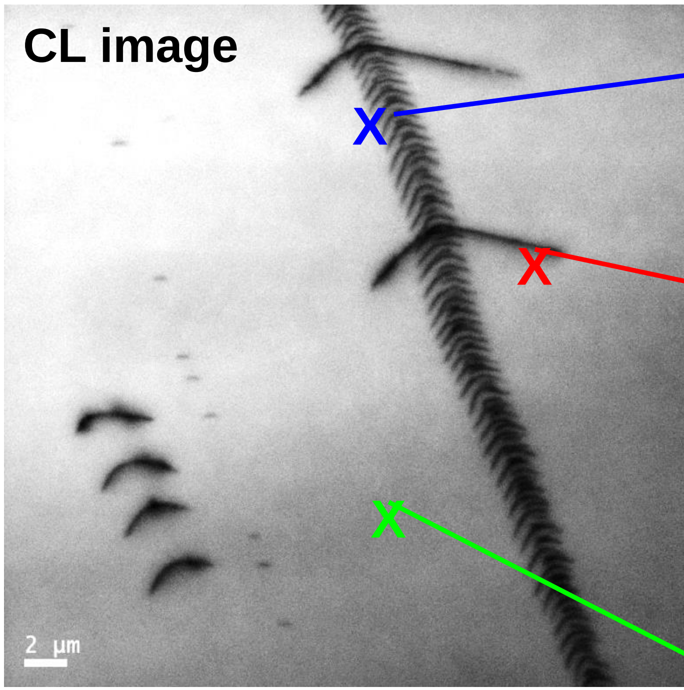
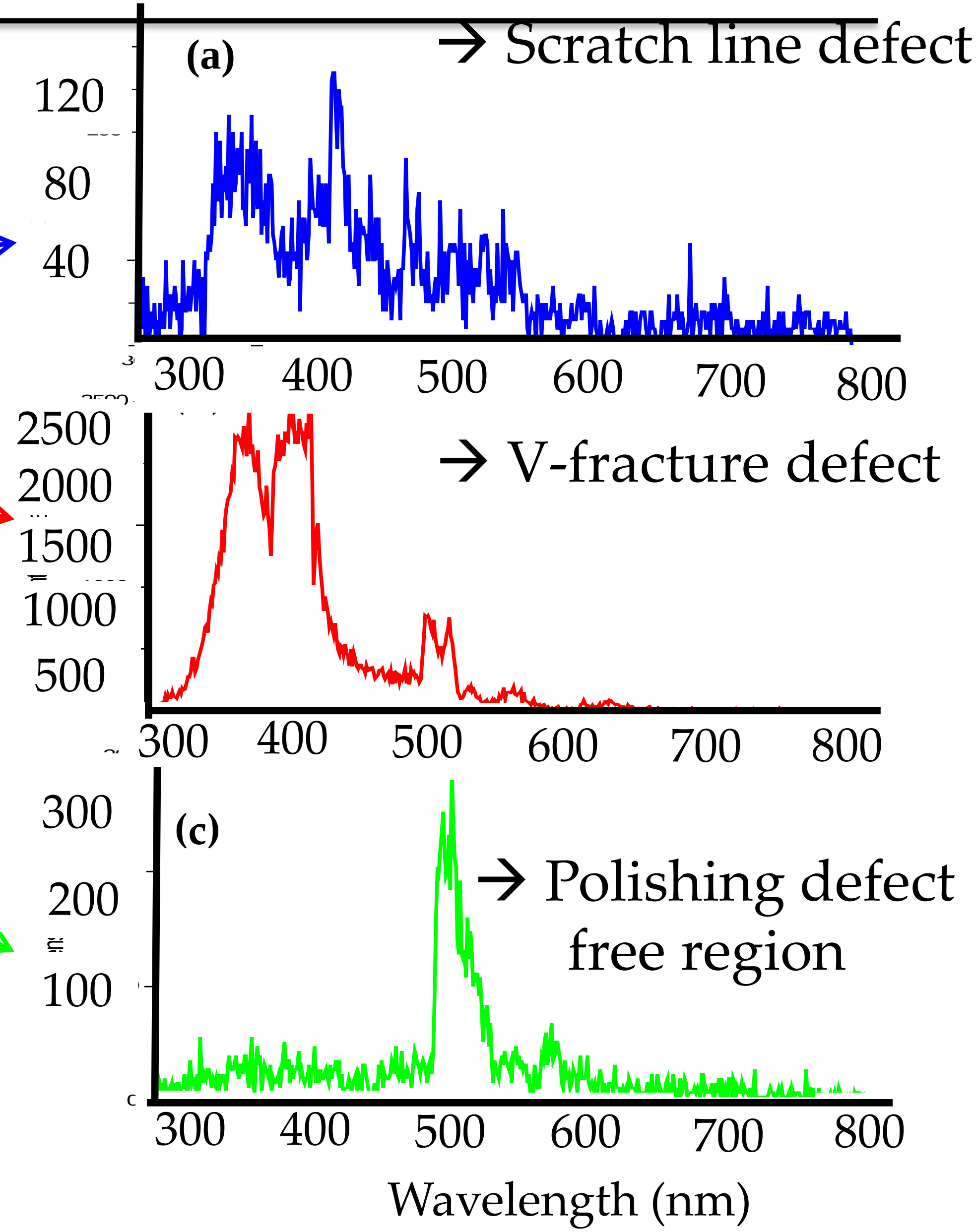
# CL Spectra (Spot Area Mode)




**Mechanical Polished**
**(5kV, 4000X)**





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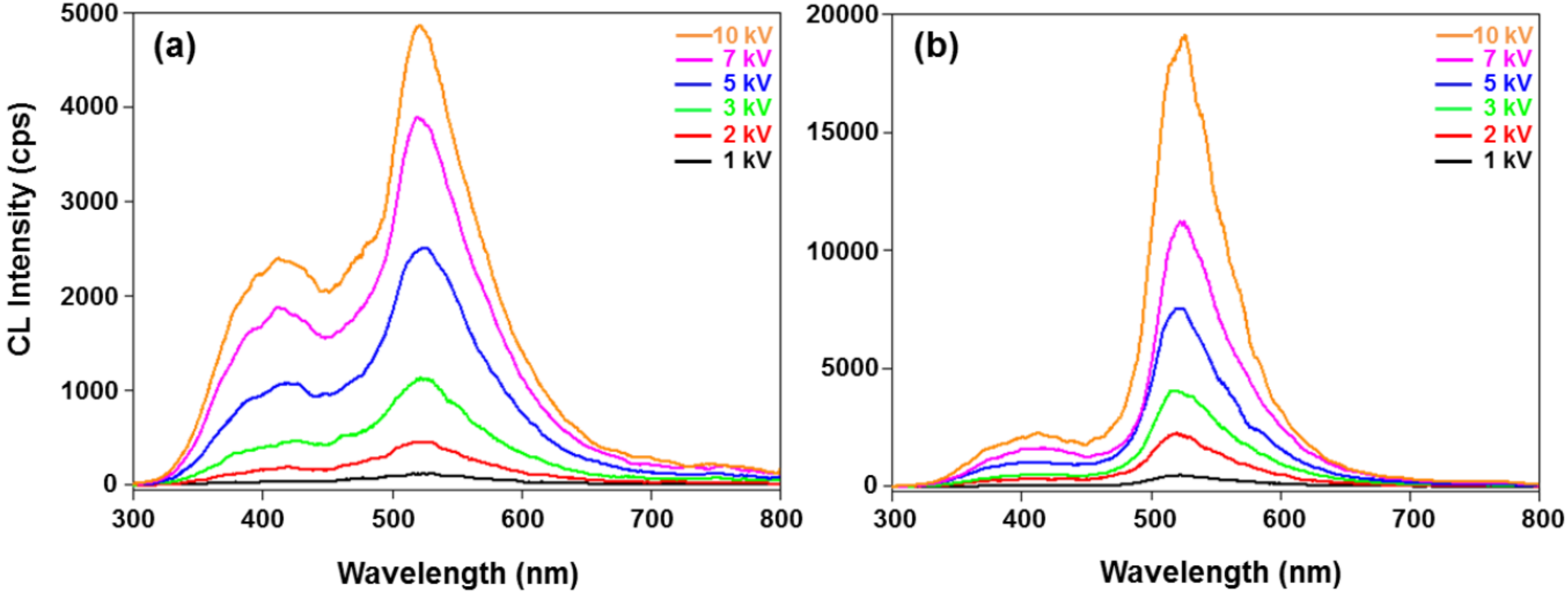
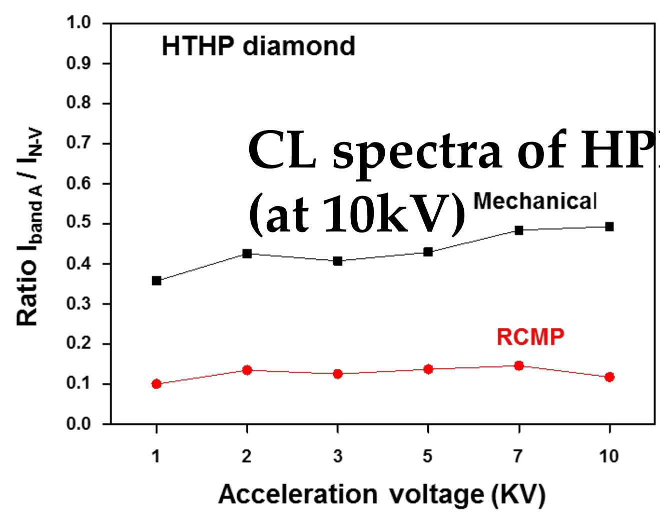
# CL spectra (Mechanical vs. RCMP)




**CL spectra of HPHT diamond (at 5kV)**

**CL spectra of HPHT diamond (at 10kV)**

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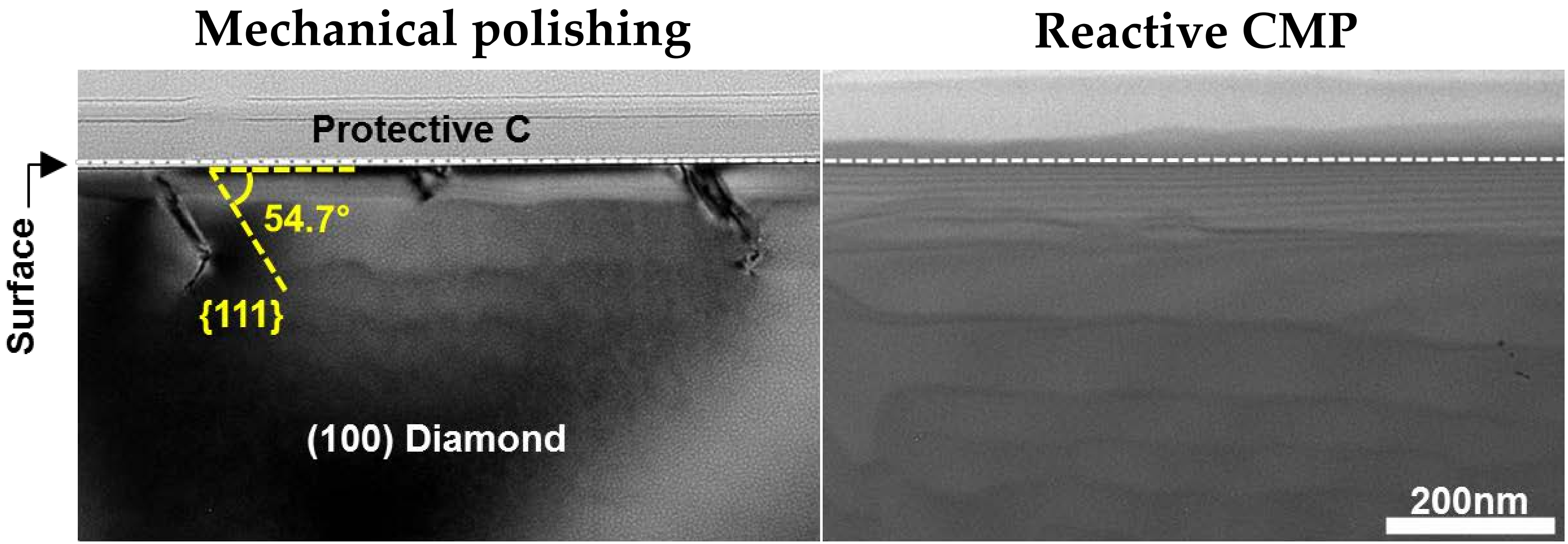
# Cross-sectional TEM




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

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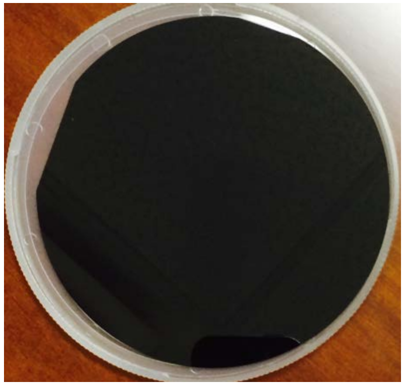
# RCMP Scale-up

## 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**

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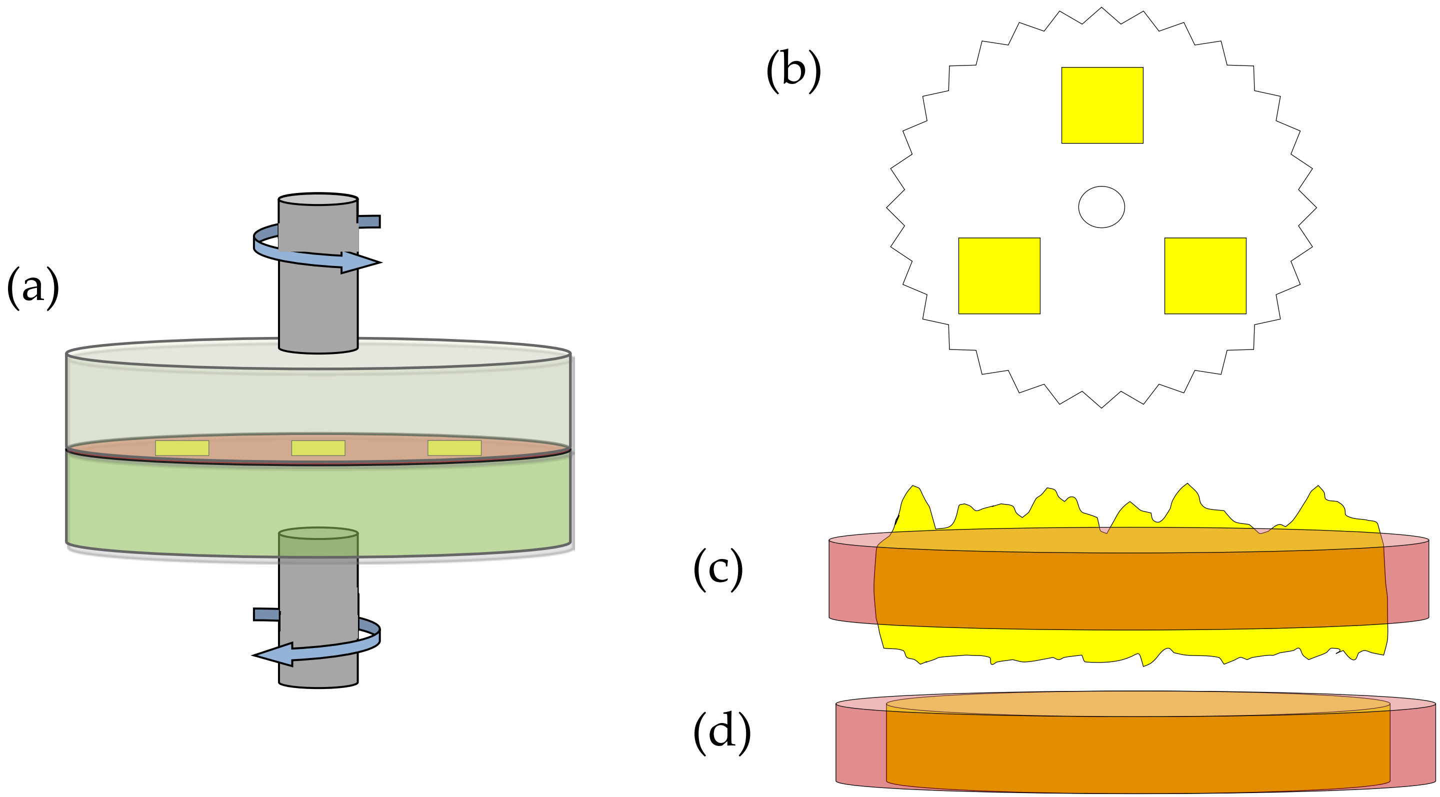
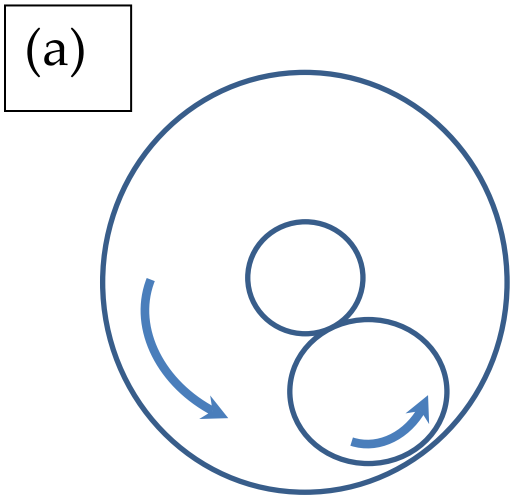
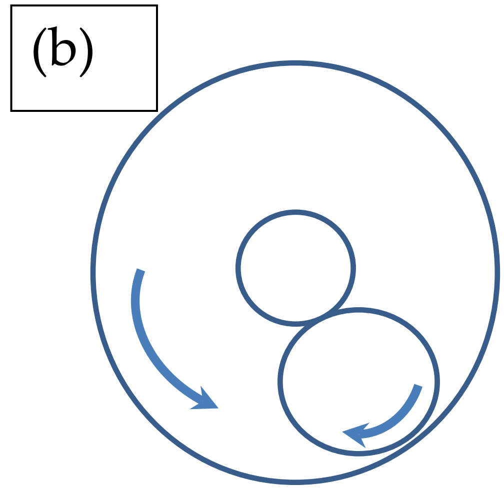
# Solution 2: Double-sided polishing



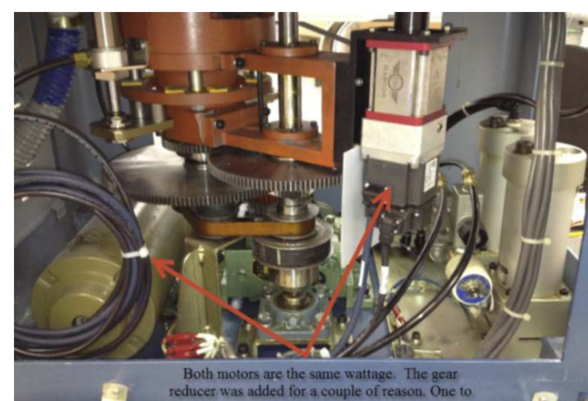

Schematic of (a) double sided polishing (b) Gear sample fixture (c) unpolished samples (d) double sided polished sample with ultra low TTV

# Custom Double-sided polishing



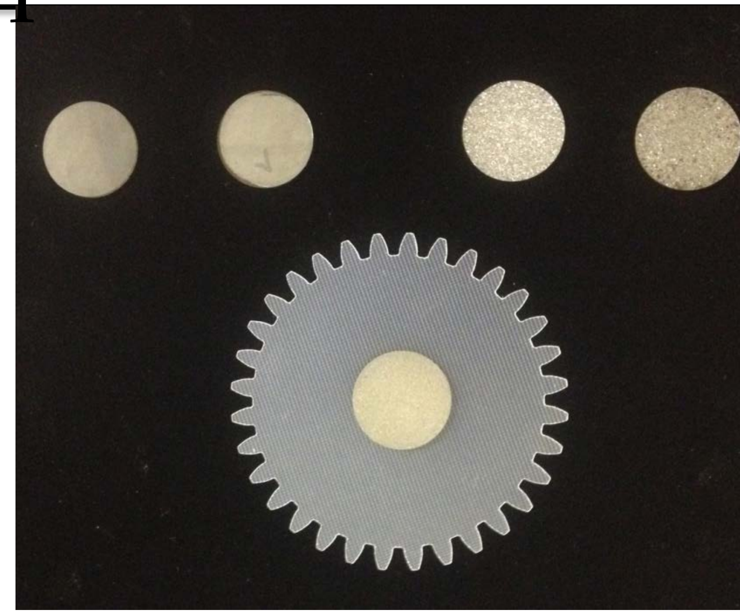



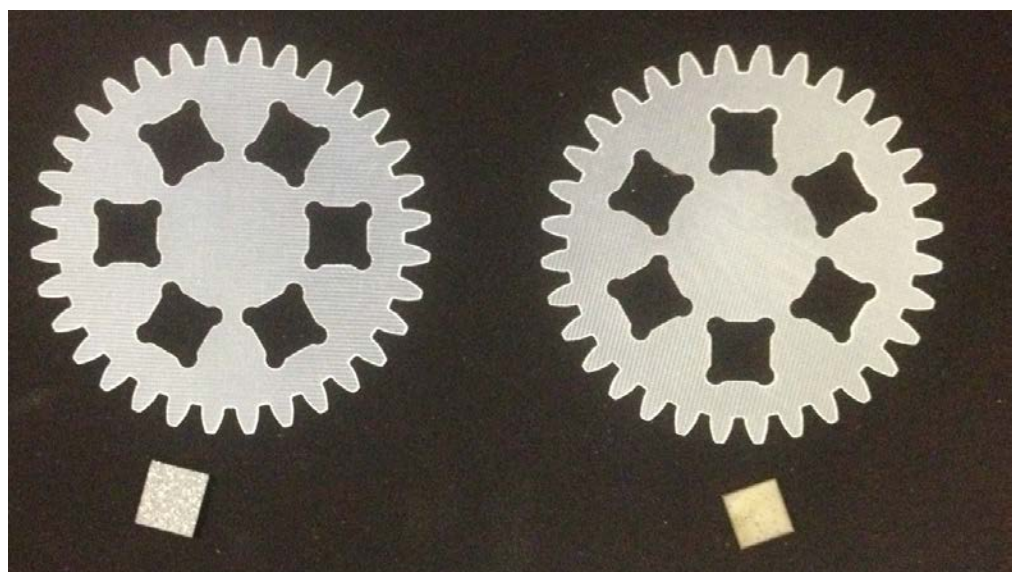

(a) Rotation of carrier and plate in the same direction & Plate will wear concave (b) Carrier and plate rotation in opposite direction & Plate will wear convex

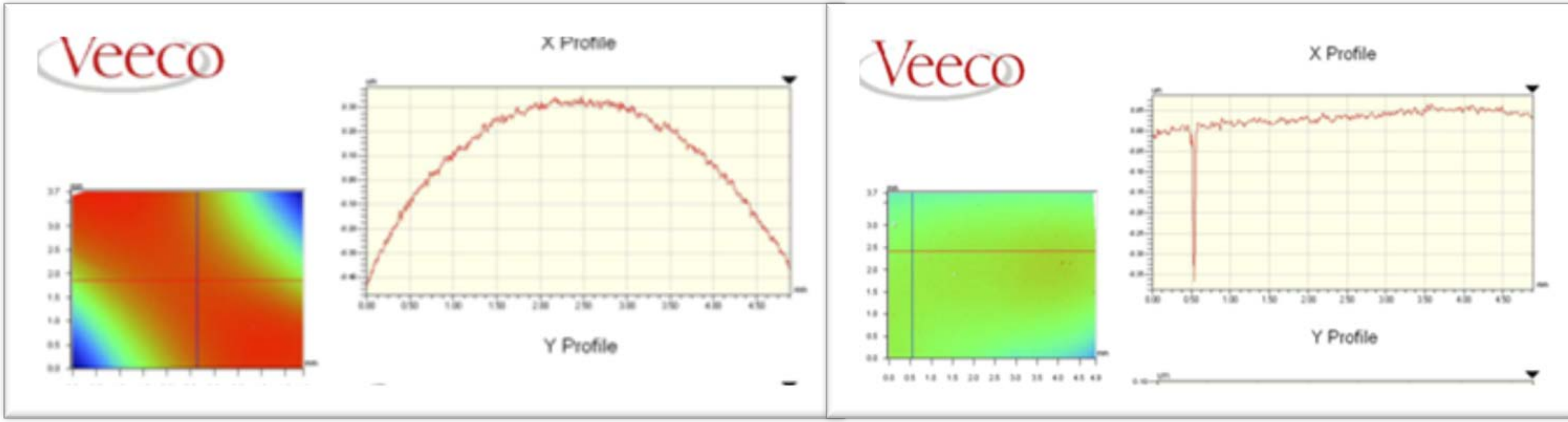
# Custom Double-sided polisher


Samples
Polished





# Ultra Flat Polishing

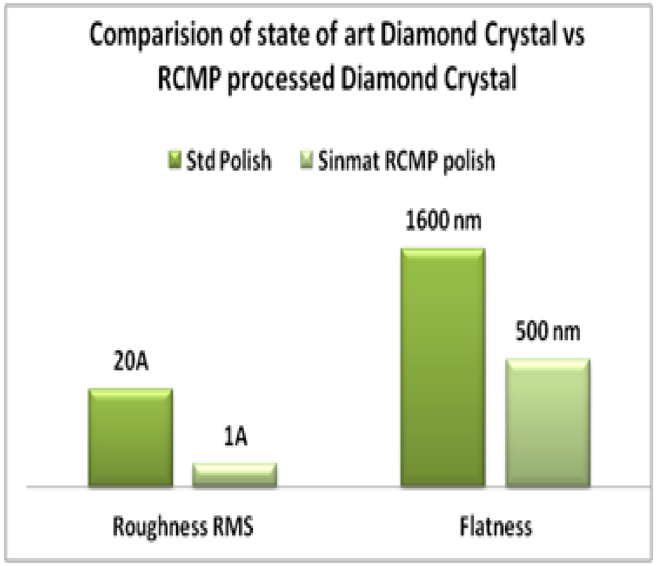


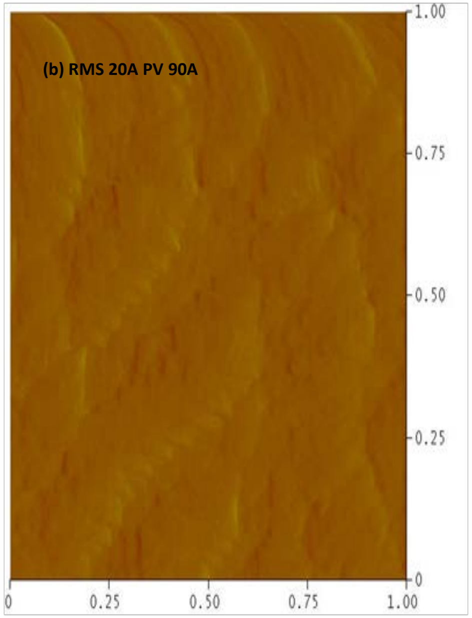

Peak to Valley Roughness
2.9 micron

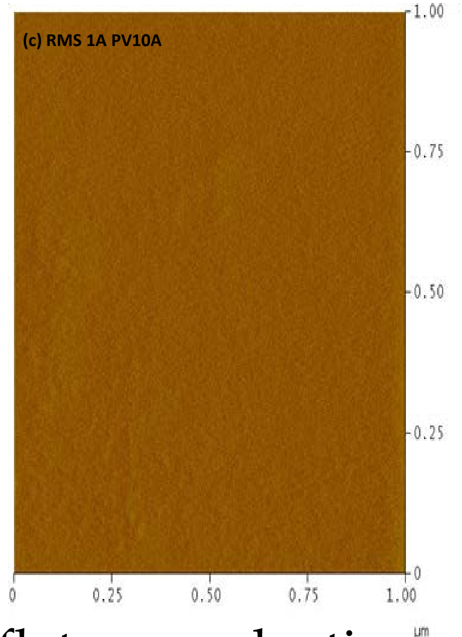
Peak to Valley Roughness
0.55 micron

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



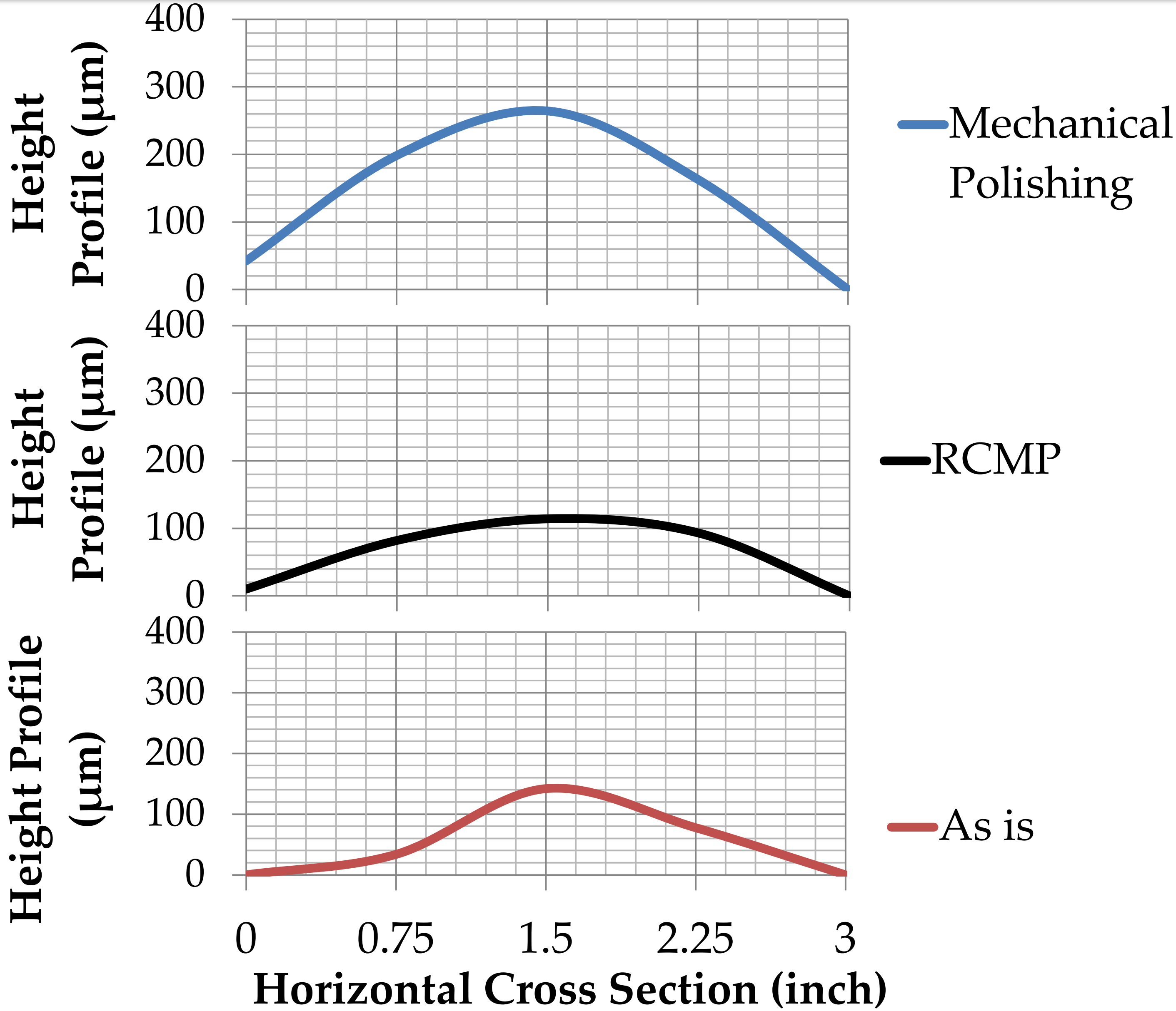




RMS roughness reduction by 1 order and optical flatness reduction from 1.6 micron to 0.5 micron with Sinmat's Reactive CMP (RCMP) polishing, (b) AFM picture of standard polish showing fractured surface (RMS 20A, (Peak to Valley) PV 90A) and (c) AFM picture of RCMP surface shows atomic smooth surface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

# Stress Free Polishing- Reduced Bow

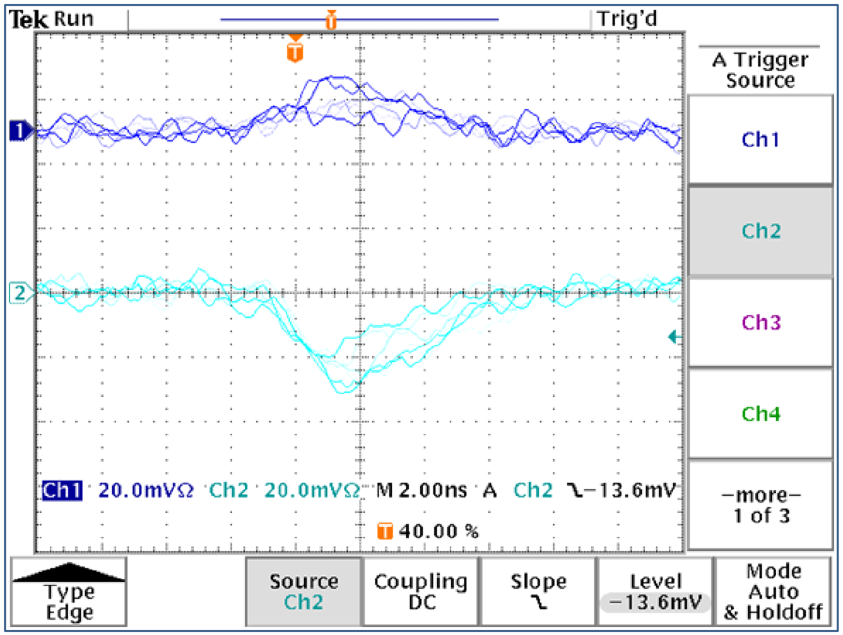
Sinmat
*Innovative CMP solutions*



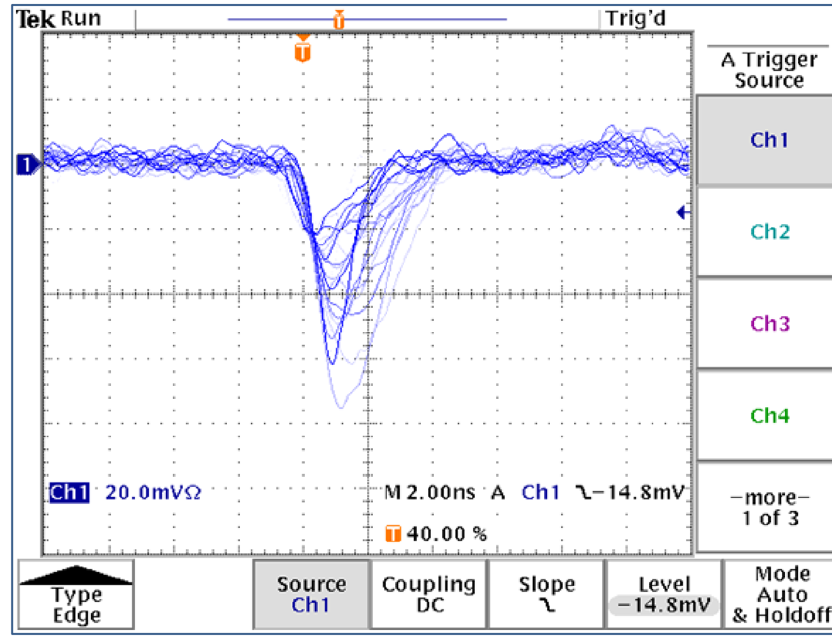
Sinmat
*Innovative CMP solutions*

# Detector Testing-NSCL

**Standard diamond**




**Sinmat finish diamond**

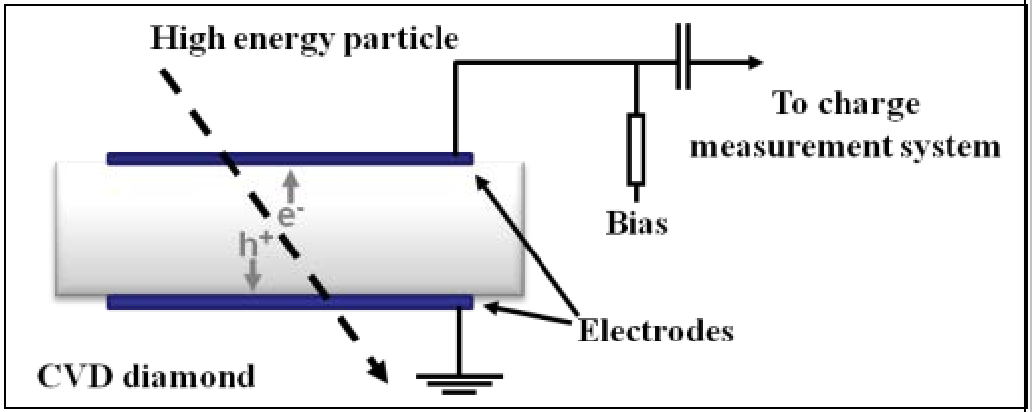



Detector response for U232 Alpha source at 100V bias (a) vendor polished sample – showing pulse height of 20-30 mV (b) RCMP polished diamond sample showing pulse height of 80mV. Both the plates were approximately 100μm thick **(Courtesy: Dr.Stolz , at NSCL)**

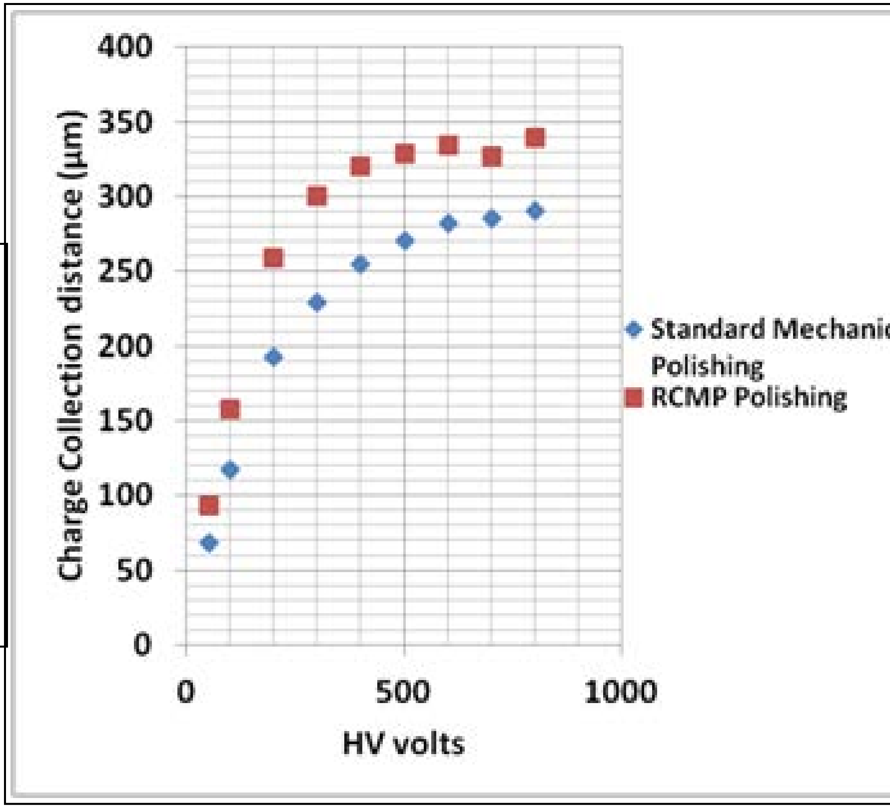
Polishing did not degrade the detector performance

# Detector Testing-OSU

## $^{90}$Sr source

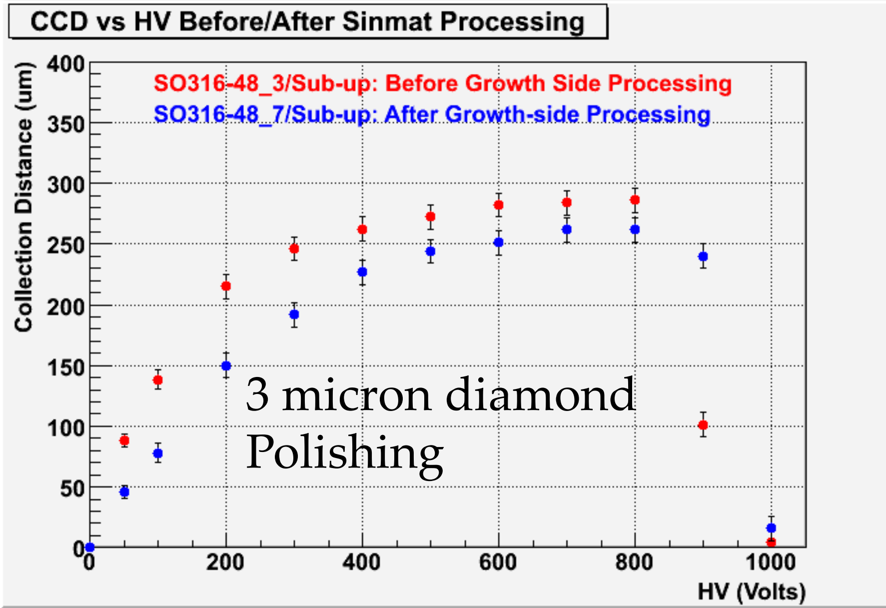


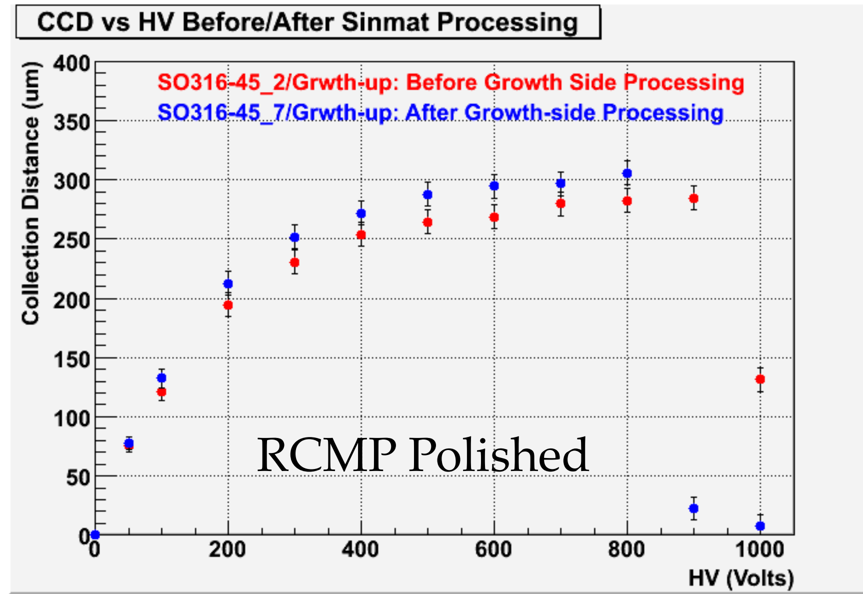

Set-up to Measure Charge Collection Distance

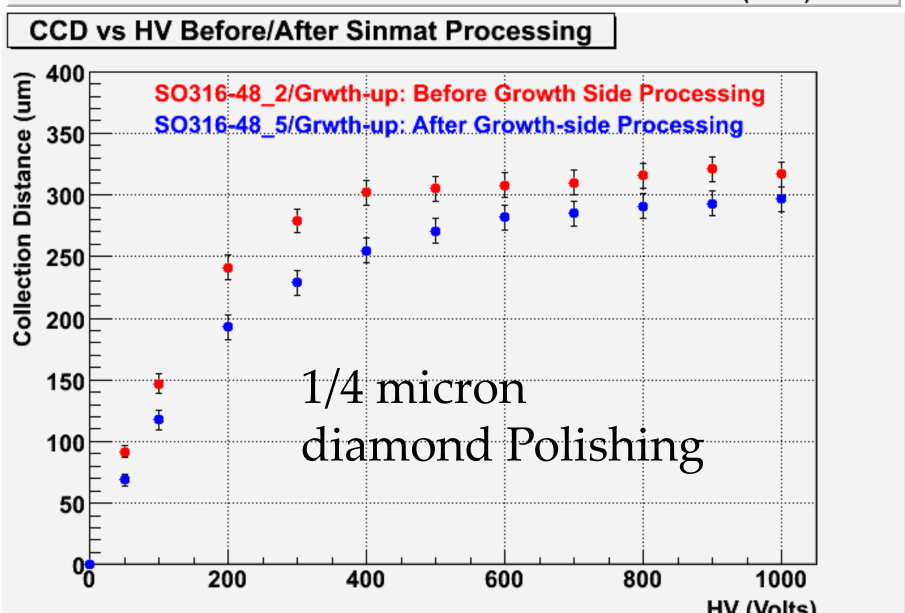



RCMP Polished Sample showing Higher Charge Collection Distance: Courtesy: Dr.Harris Kagan OSU

# Detector Testing-OSU



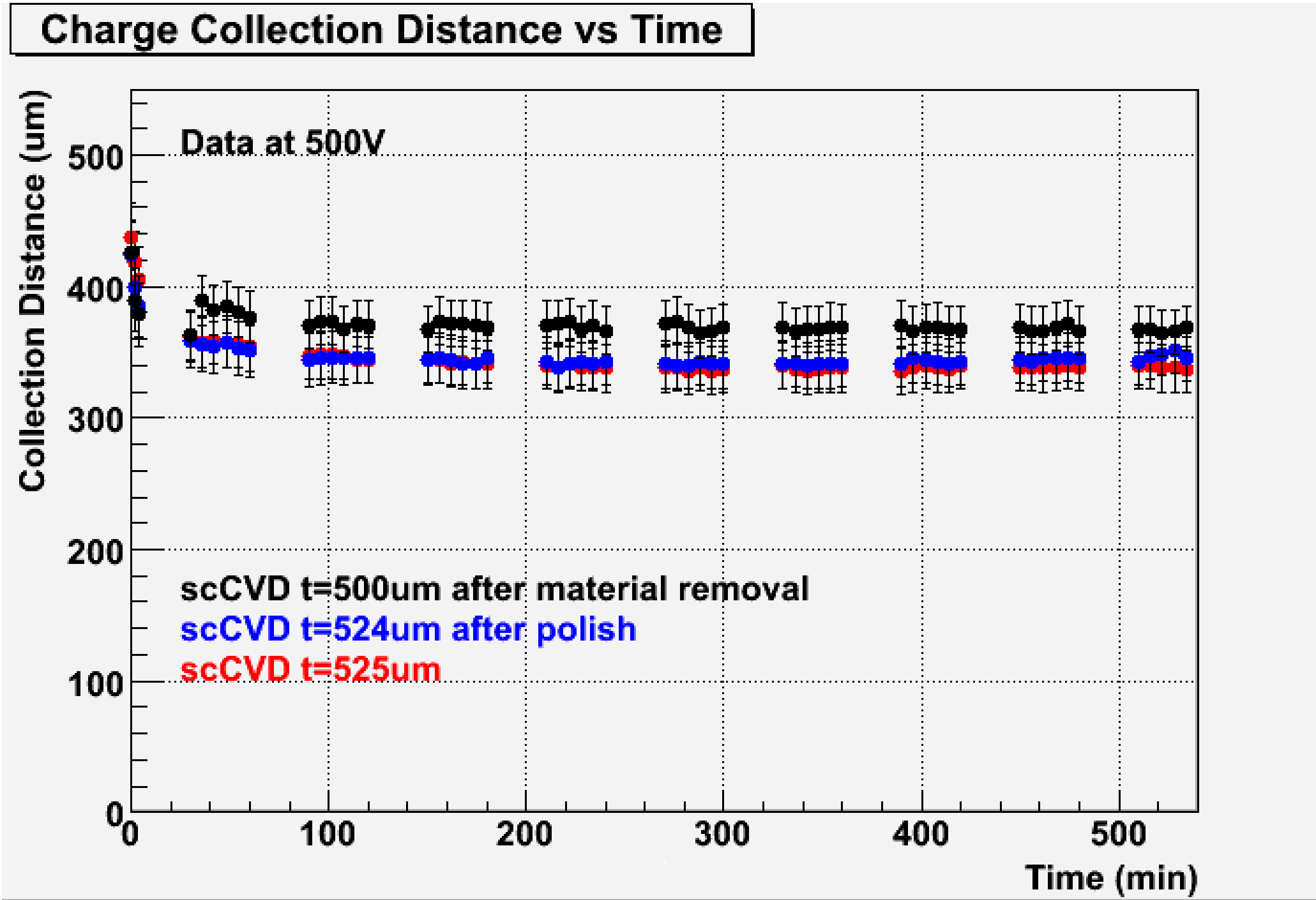




- RCMP Improves Detector Performance Significantly
- Mechanical Polishing Degrades Detector Performance

Courtesy: Dr. Harris Kagan OSU

# Detector Testing-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