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

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Aug 6, 2014
Gaithersburg, MD

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)

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

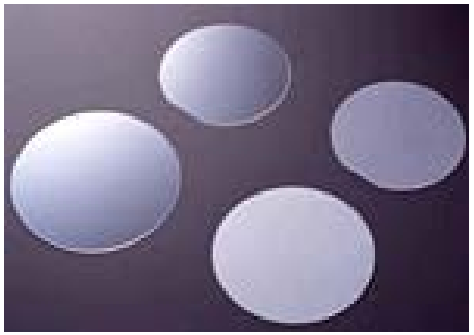
Ultra-hard substrates for electronic & optics

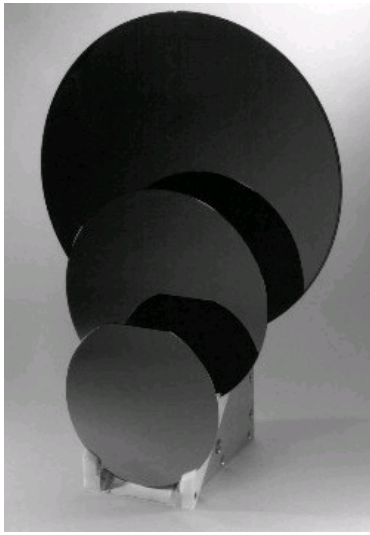
Silicon Carbide (SiC)

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

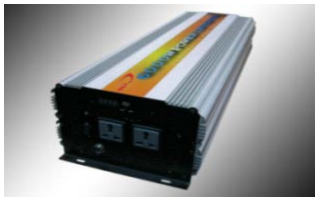
Applications of (SiC, GaN, Sapphire & Diamond)

Power Devices

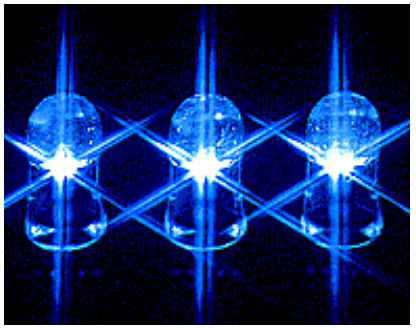
AC-DC Converter

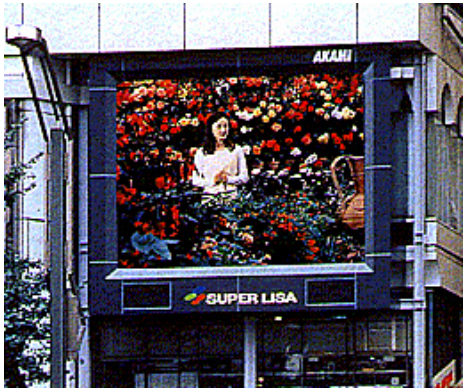
DC to DC Converter

Inverters

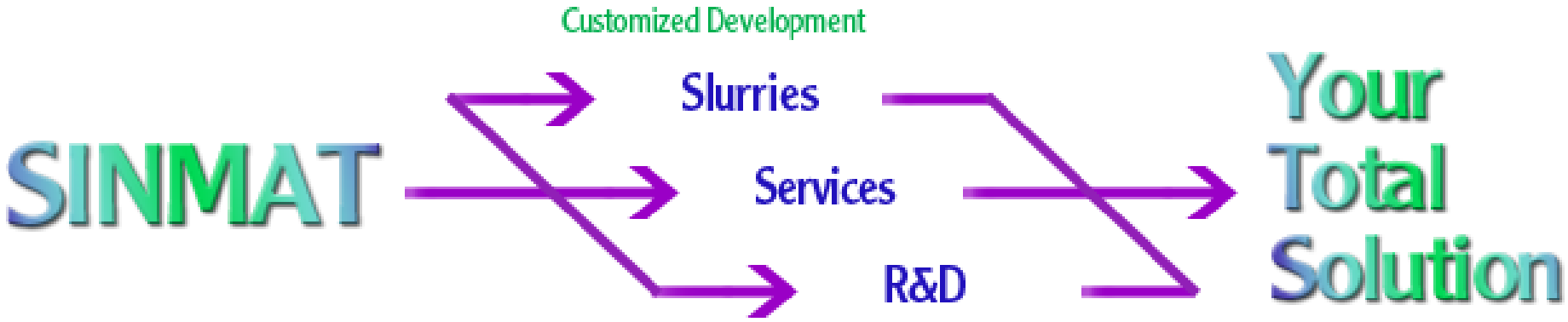


Light Emitting Devices (LEDs)





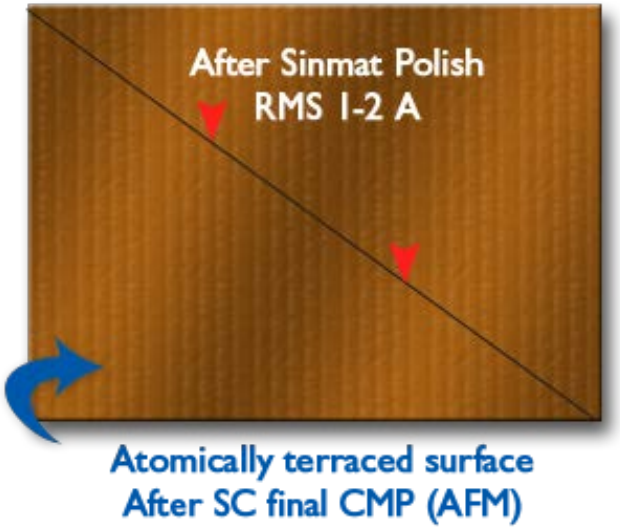
Sinmat Inc



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

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

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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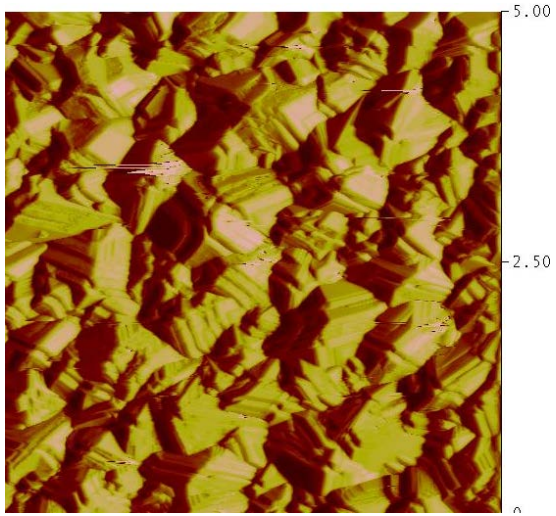
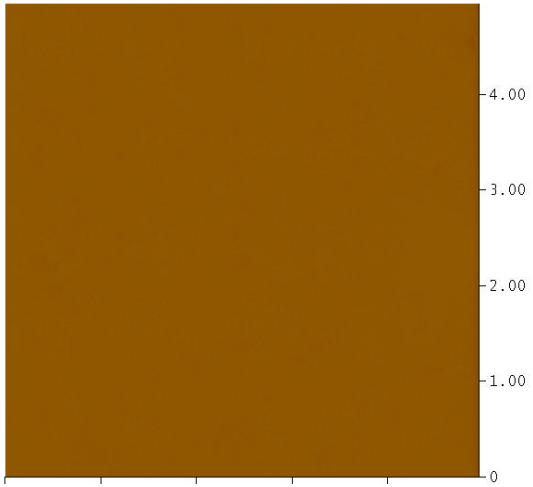
Innovative CMP solutions

- Before Polishing



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

- After Polishing



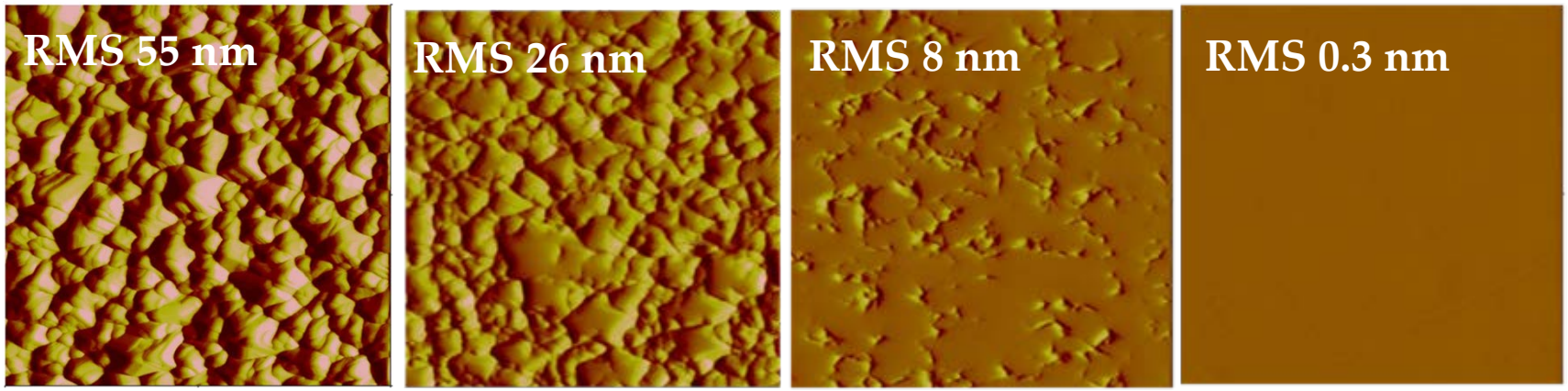
Img. Rms (Rq)	0.335 nm
Img. Ra	0.222 nm

Prior to Project

Silicon on Diamond Substrates

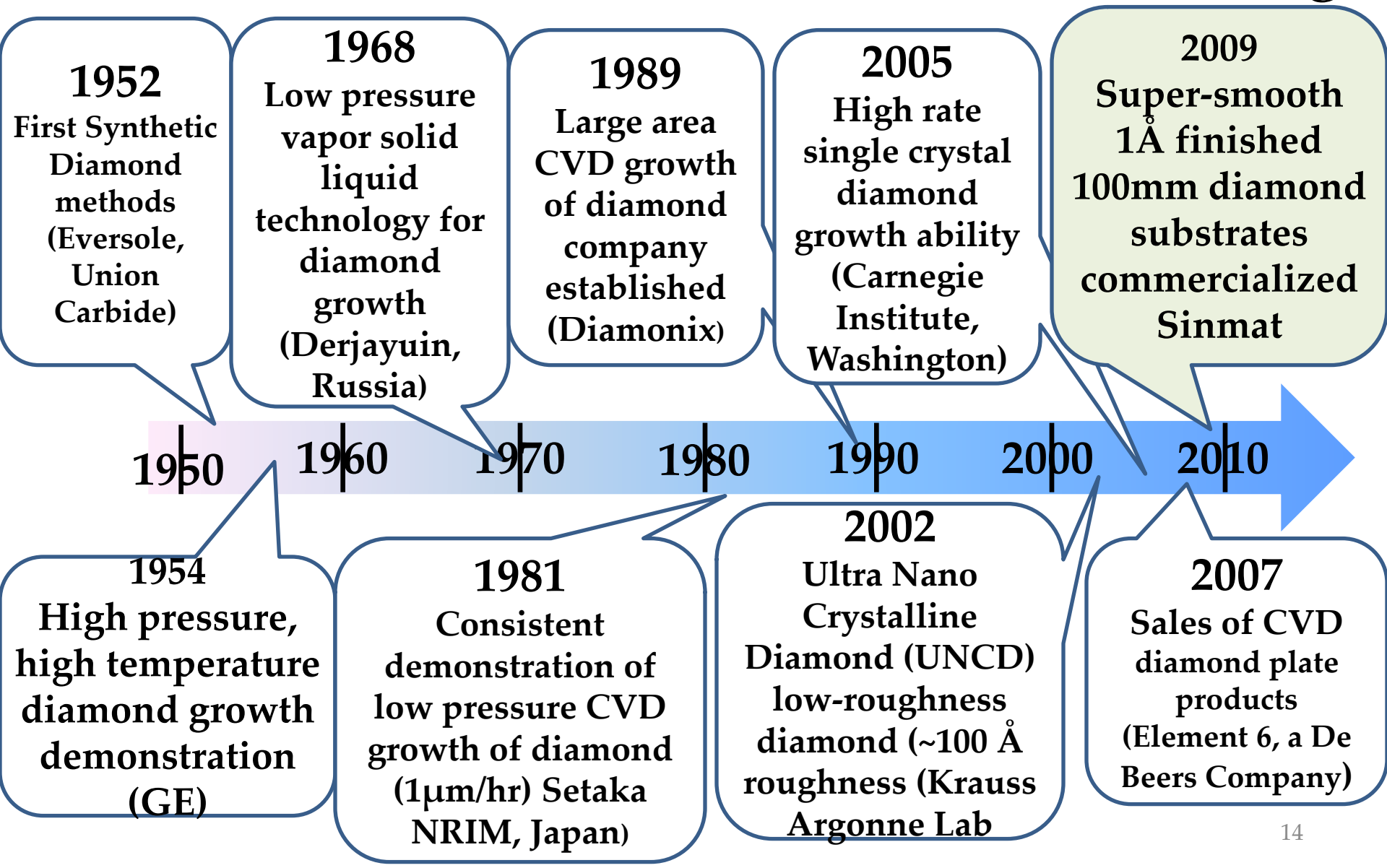
Diamond - Reactive chemical mechanical polishing process

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



Prior to Project

Timeline of Diamond Growth & Polishing



Sinmat's Diamond Strategy

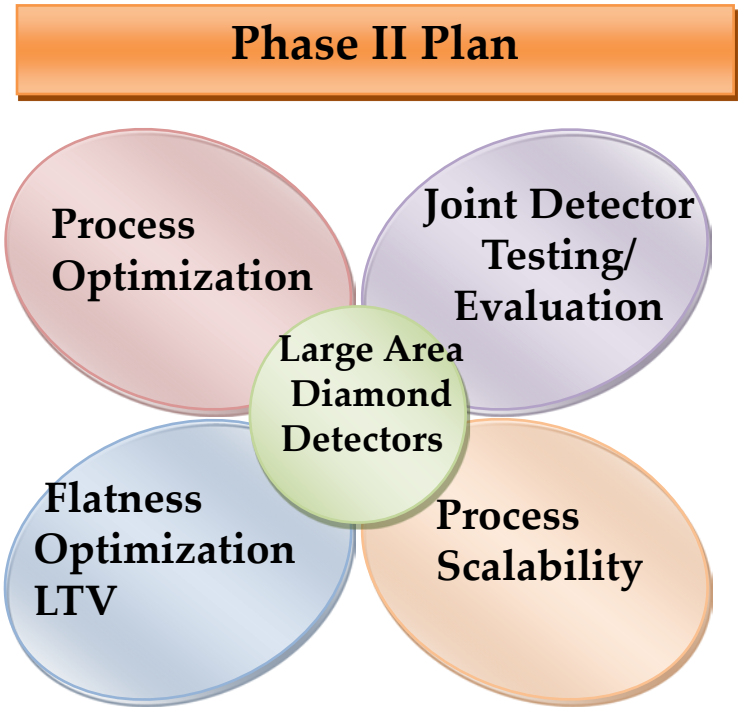
- Leverage its novel diamond polishing technology to fabricate high performance diamond based devices for Nuclear Physics Applications
  - Diamond Detectors
  - Ultra-Thin (< 50 microns) Diamond radiator crystals
  - Diamond X-ray Optics
  - High thermal conductivity substrates

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

SBIR Phase II Project Objective

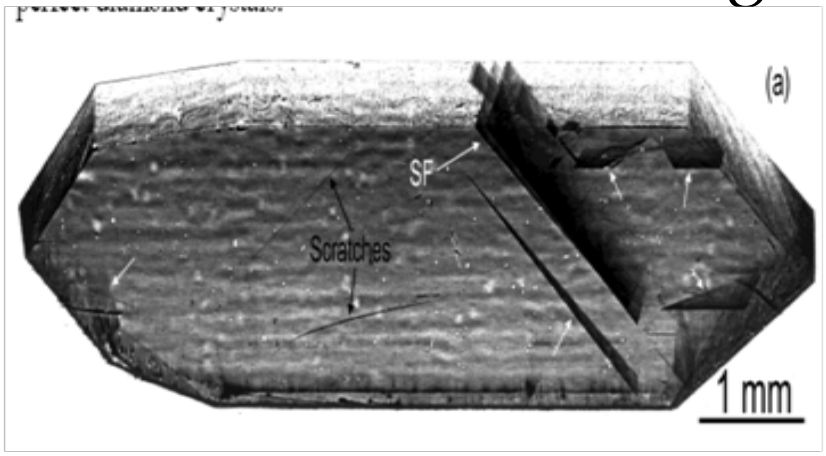
Use Sinmat's RCMP process to fabricate and evaluate diamond based detectors for high energy beam tracking application

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

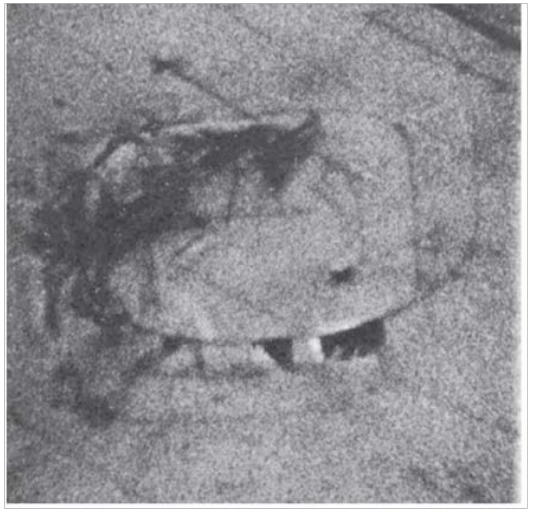


Problem

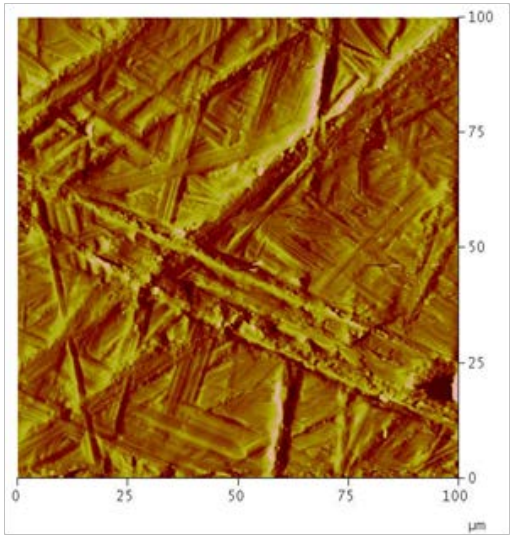
Surface Polishing



X-ray topograph of single crystal diamond showing scratches



Cathodoluminescence image of subsurface damage caused due to diamond based polishing

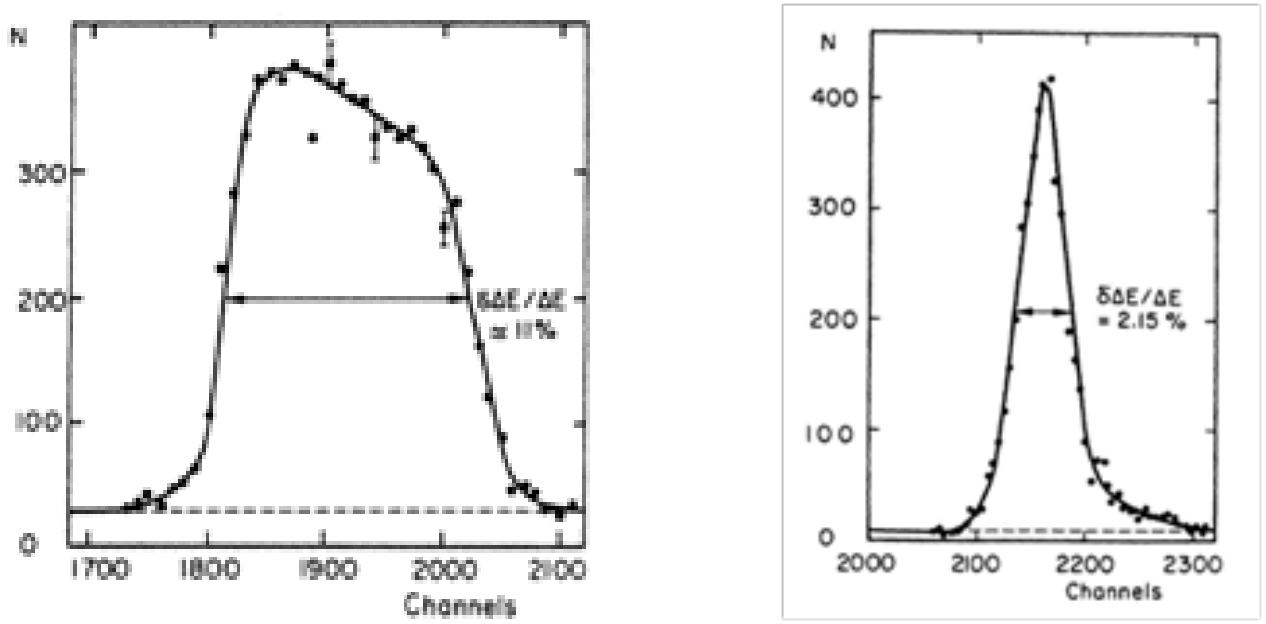


AFM Picture shows surfaces scratch on diamond

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

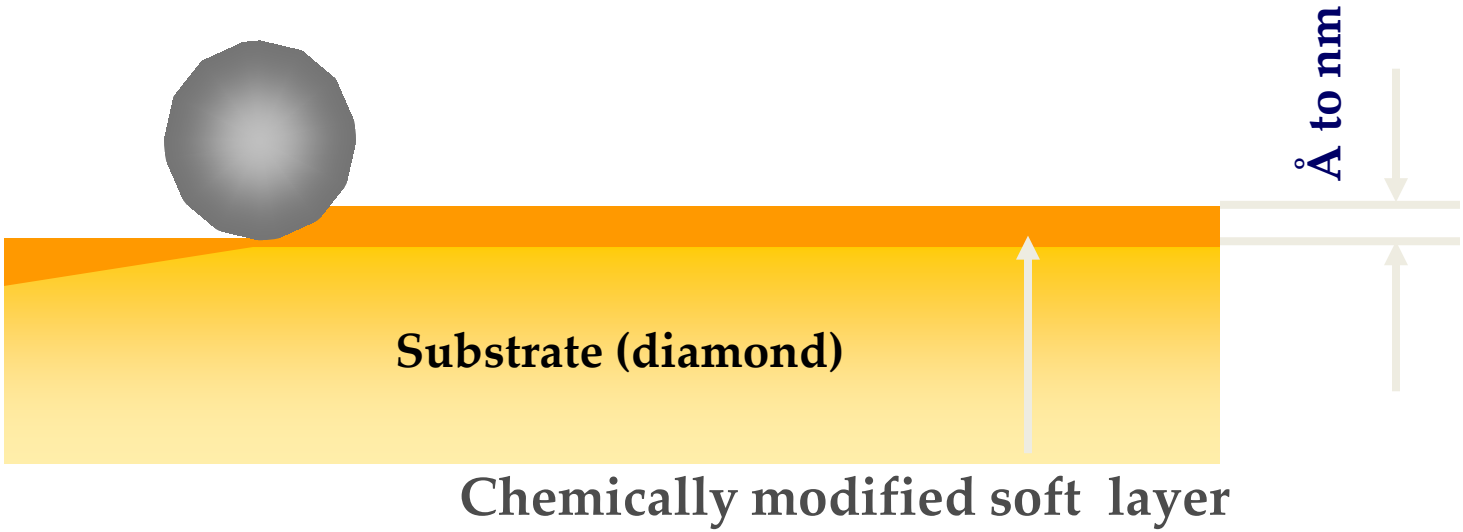
Problem

Flatness and Thickness Tolerance Variation



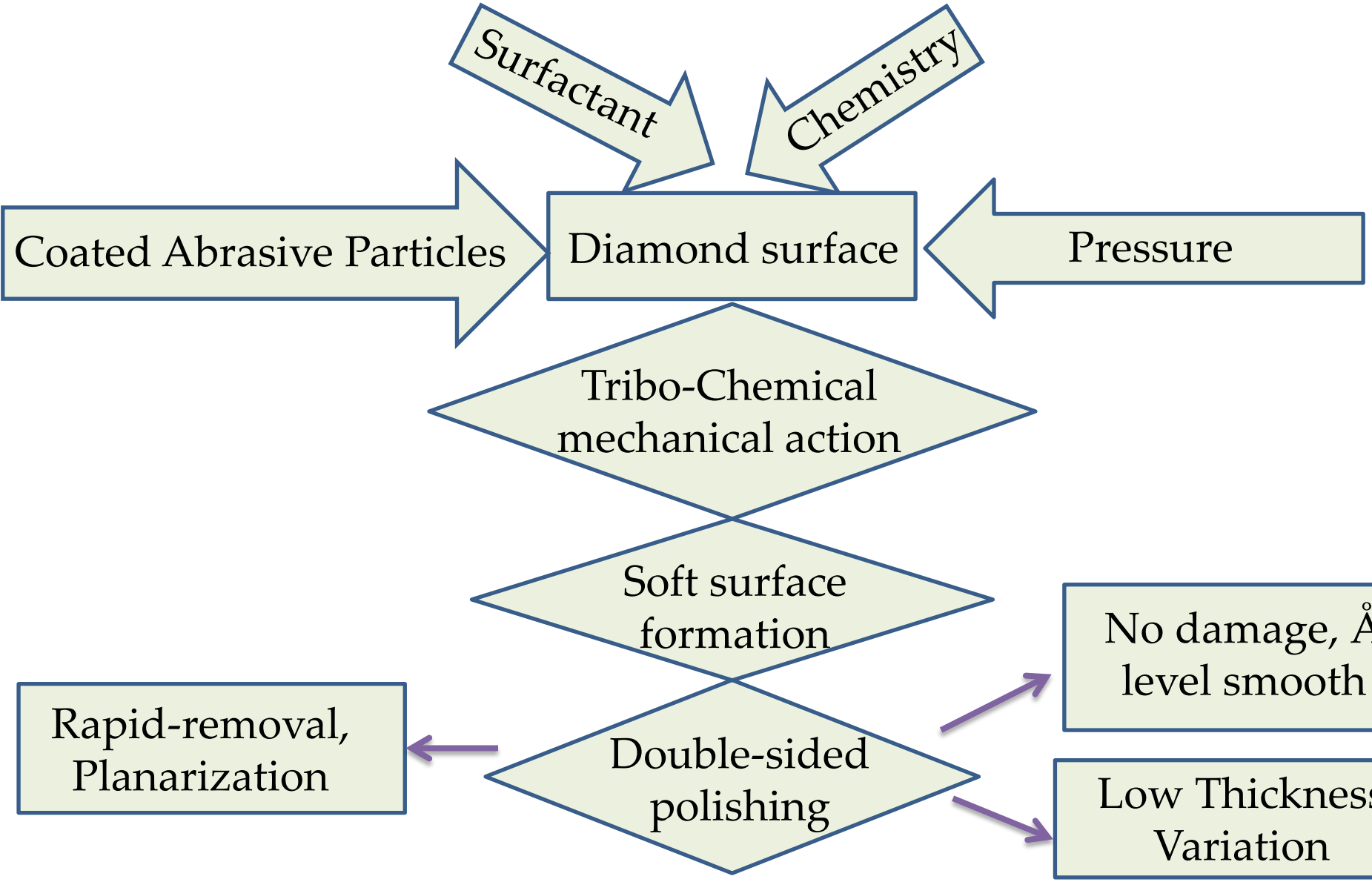
Example of energy straggling in detectors (a) showing poor energy resolution due to energy straggling in the detector and (b) Showing better energy resolution with lesser energy straggling [Muller]

Reactive CMP (RCMP): Soft layer Polish

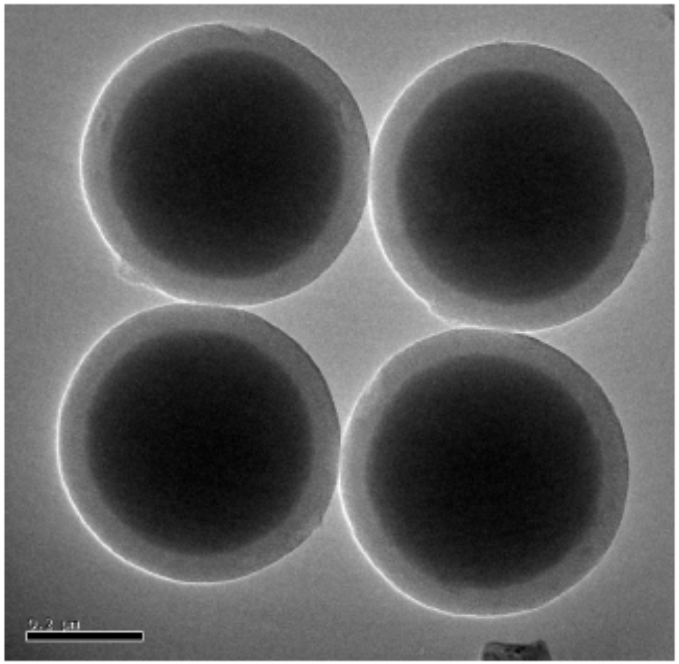


- Chemically convert hard Diamond into a soft-layer
- Use nanoparticles
- Remove Soft layer
  - Achieve High Removal Rate
  - No Scratches
- Single Component Slurry

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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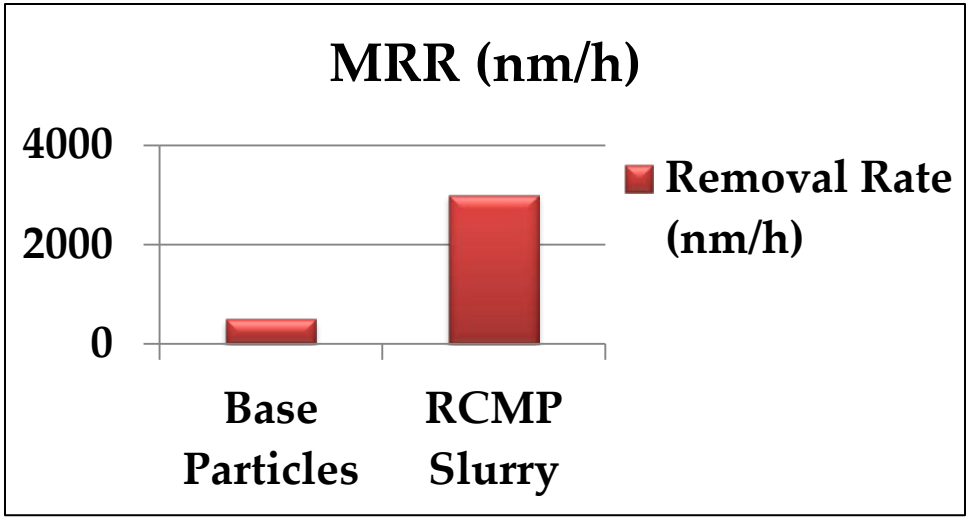
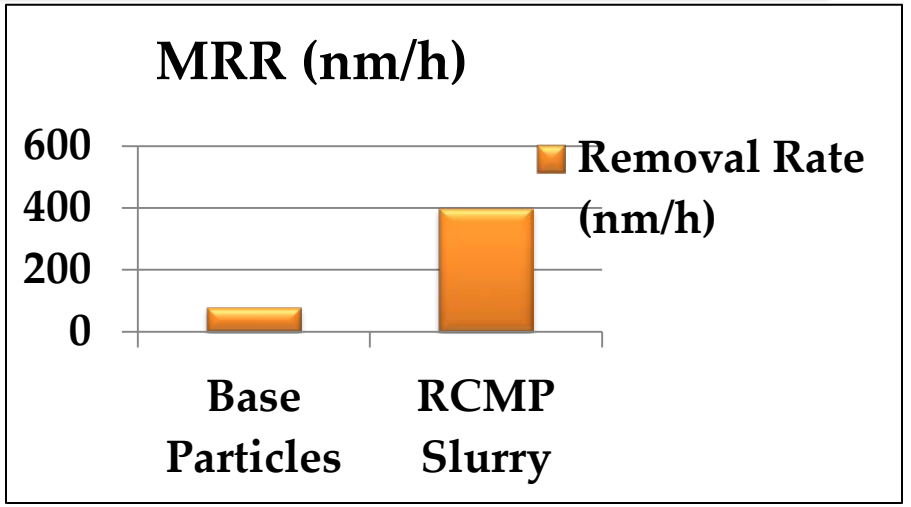
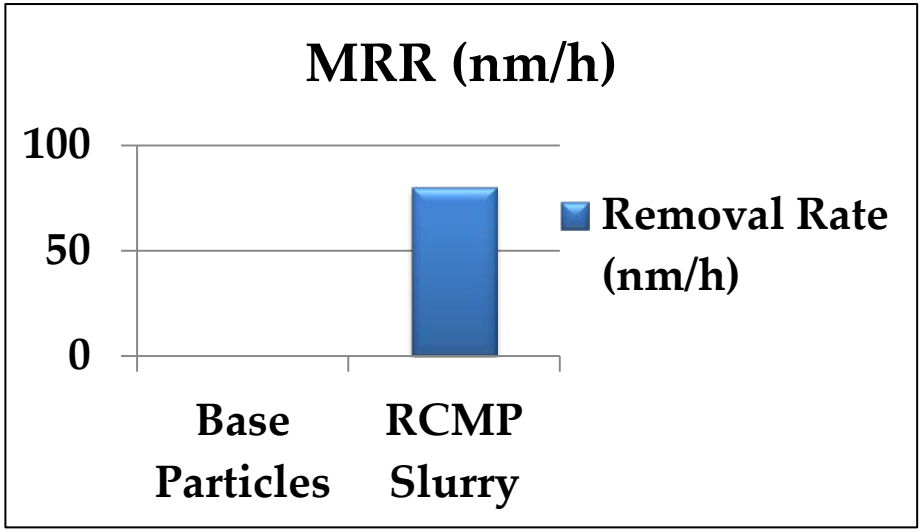
Innovative CMP solutions



High Removal Rate RCMP Slurry



Medium Removal Rate RCMP Slurry



High Finish RCMP Slurry

p-Crystalline Diamond Grain Flattening using RCMP

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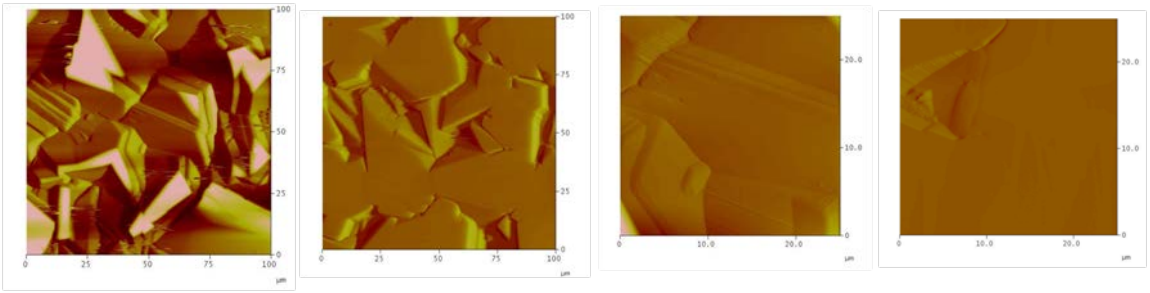


RMS 1.2 micron

RMS 0.6 micron

Thickness variation on PV 5 micron

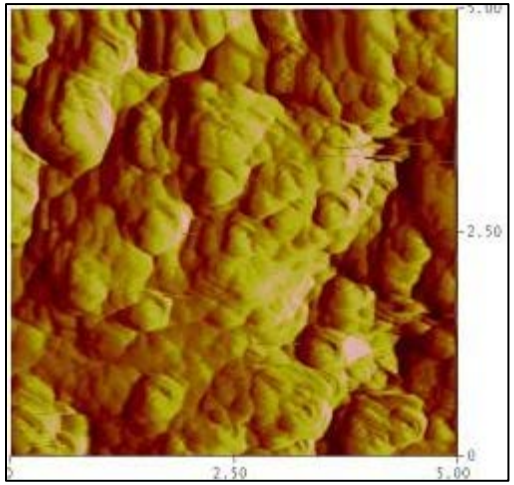
Thickness variation on PV 1.5 micron

(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).

p-Crystalline Diamond Polishing

<25 micron grains

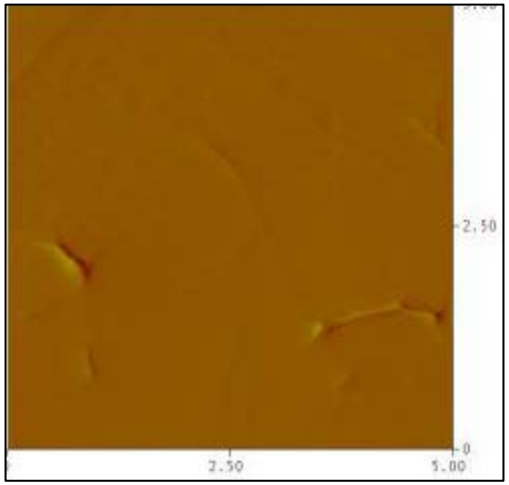
As-received
Lapped Sample



RMS ~ 59.4 nm

(5x5 sq.μm)

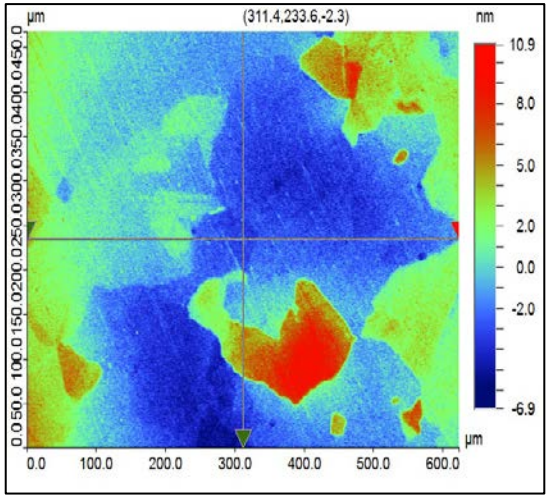
AFM Image



RMS ~ 4.6 nm

(5x5 sq.μm)

Wyko Image



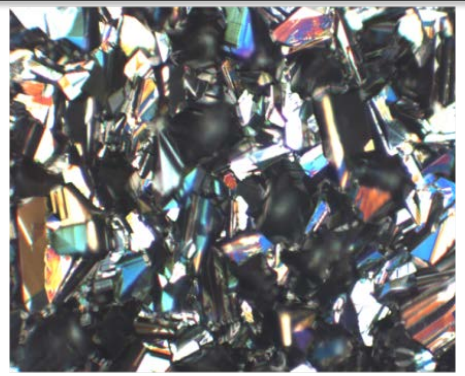
Label	Value	Units
Average	0	nm
Ra	2.263	nm
Rp	10.889	nm
Rq	2.837	nm
Rt	17.745	nm
Rv	-6.856	nm

Ra ~ 2.2 nm

(600 μm x 450 μm)

Large Grain Flattened by RCMP

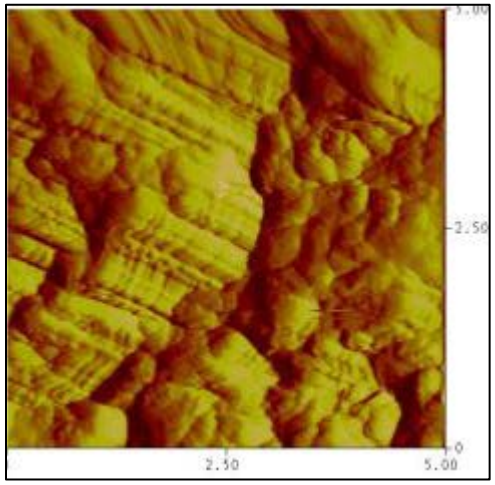
> 100 micron grains



The initial surface and progressive changes over time during polishing

Single Crystalline Diamond Polishing

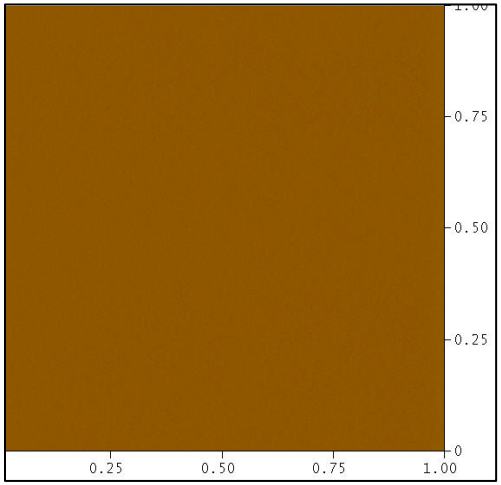
As-received
Lapped Sample



RMS 119.6 nm

(5x5 sq.µm)

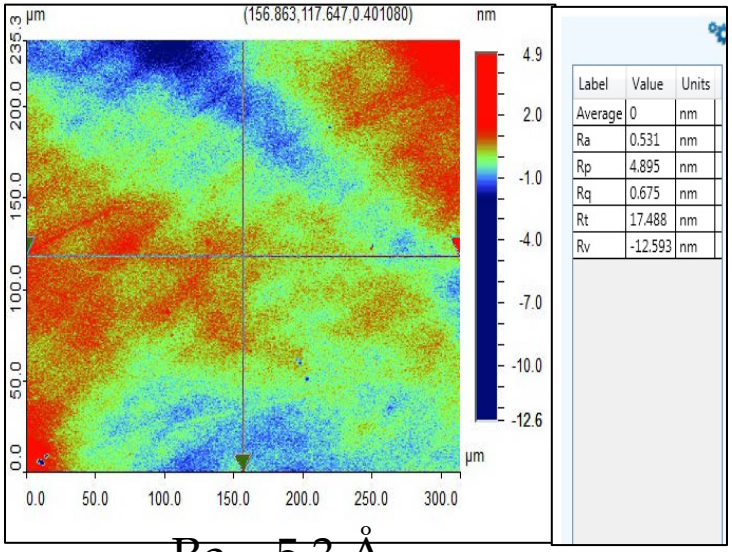
AFM Image



RMS ~ 1.3 Å

(1x1 sq.µm)

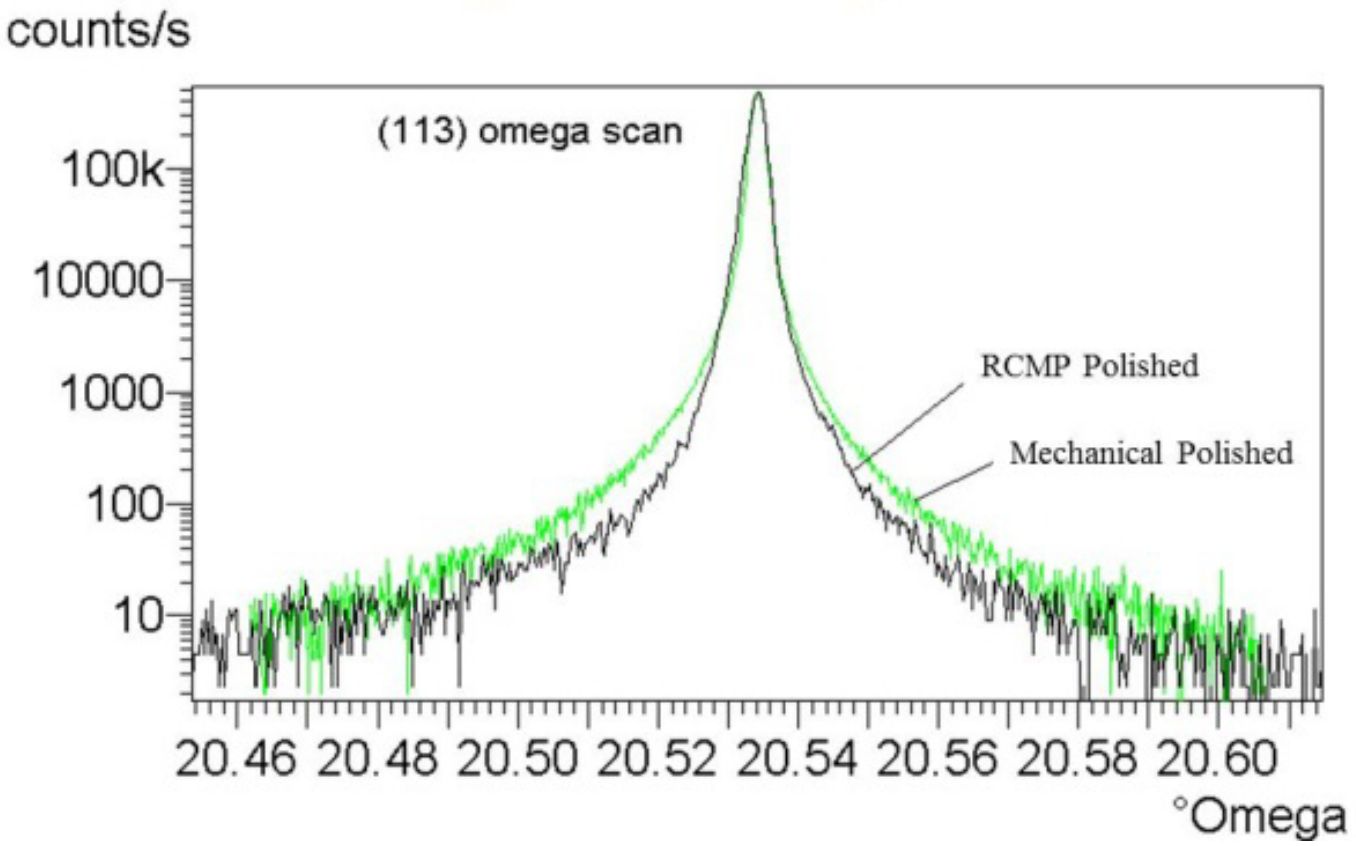
Wyko Image



Ra ~ 5.3 Å

(300 µm x 235 µm)

X-ray Rocking Curve Studies

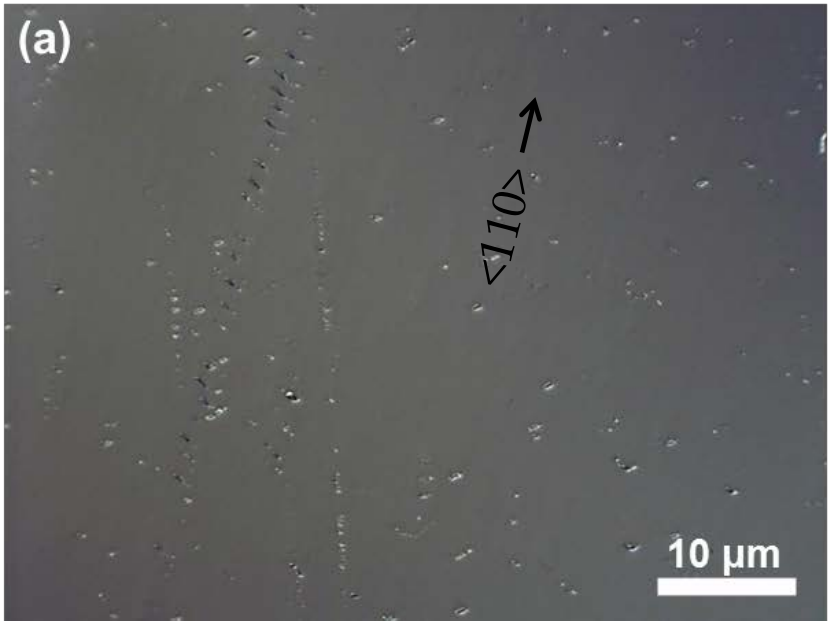


RCMP Process –reduced X-ray rocking curve width

Optical Microscope Images

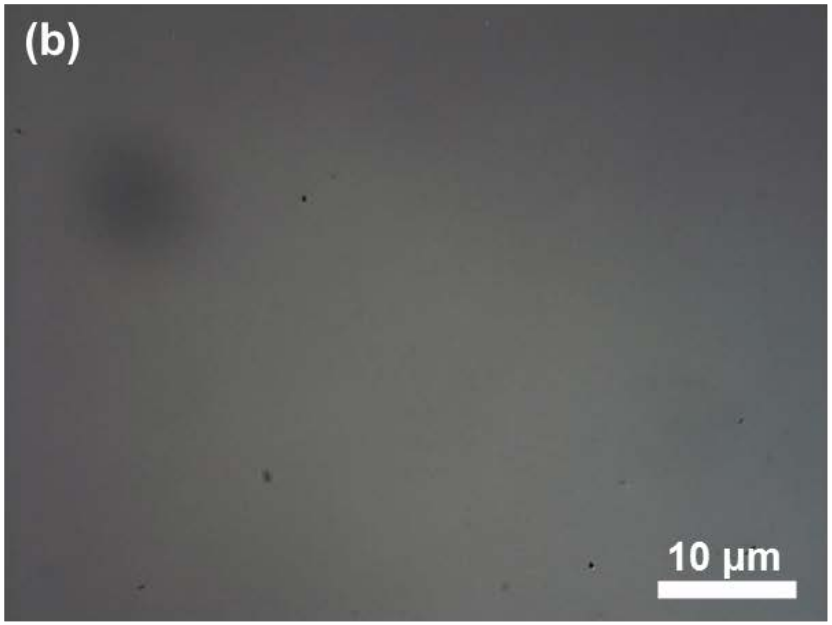
- Surface morphology of HPHT diamond before and after RCMP

Mechanical polishing



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

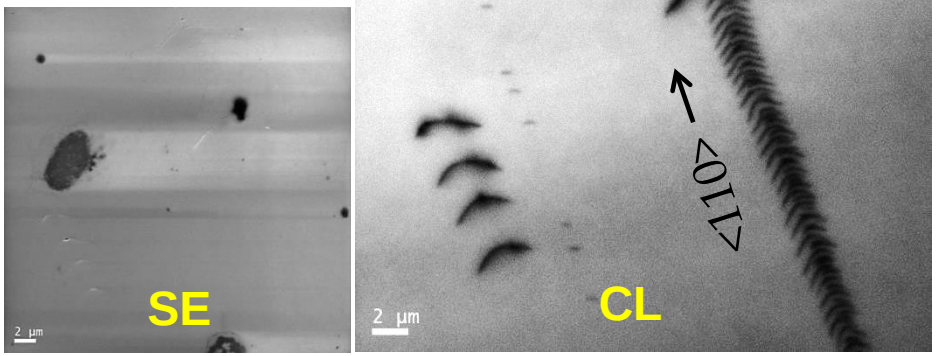
Reactive CMP



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

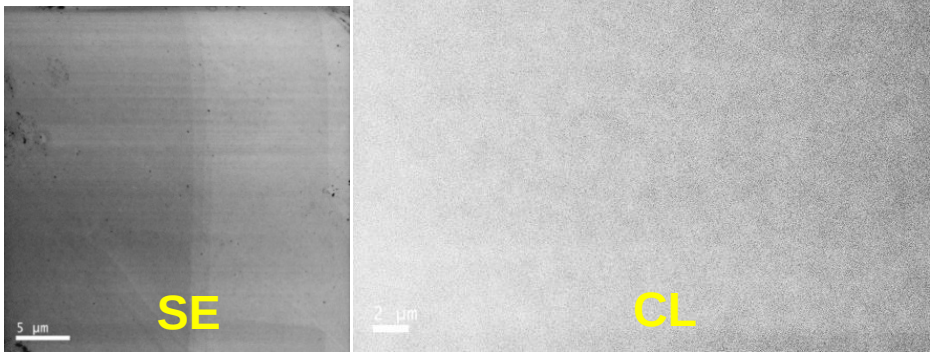
SEM & Panchromatic CL (HPHT)

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

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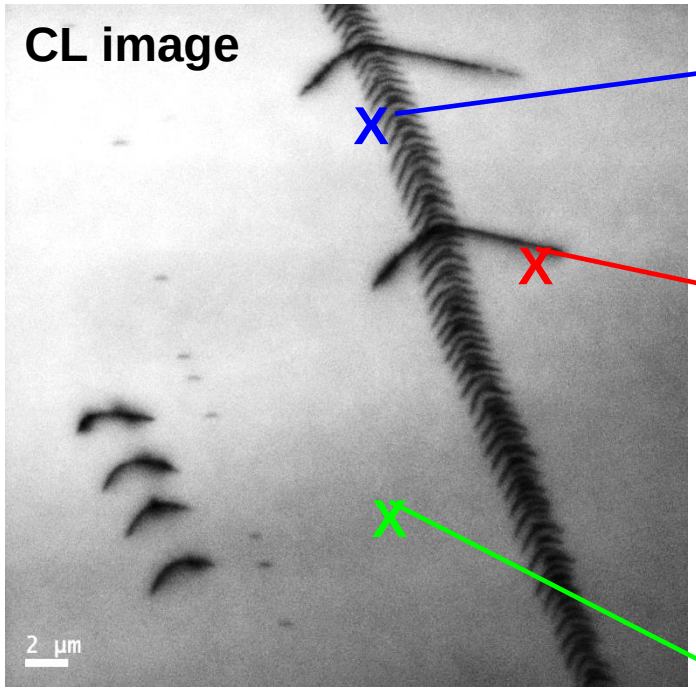
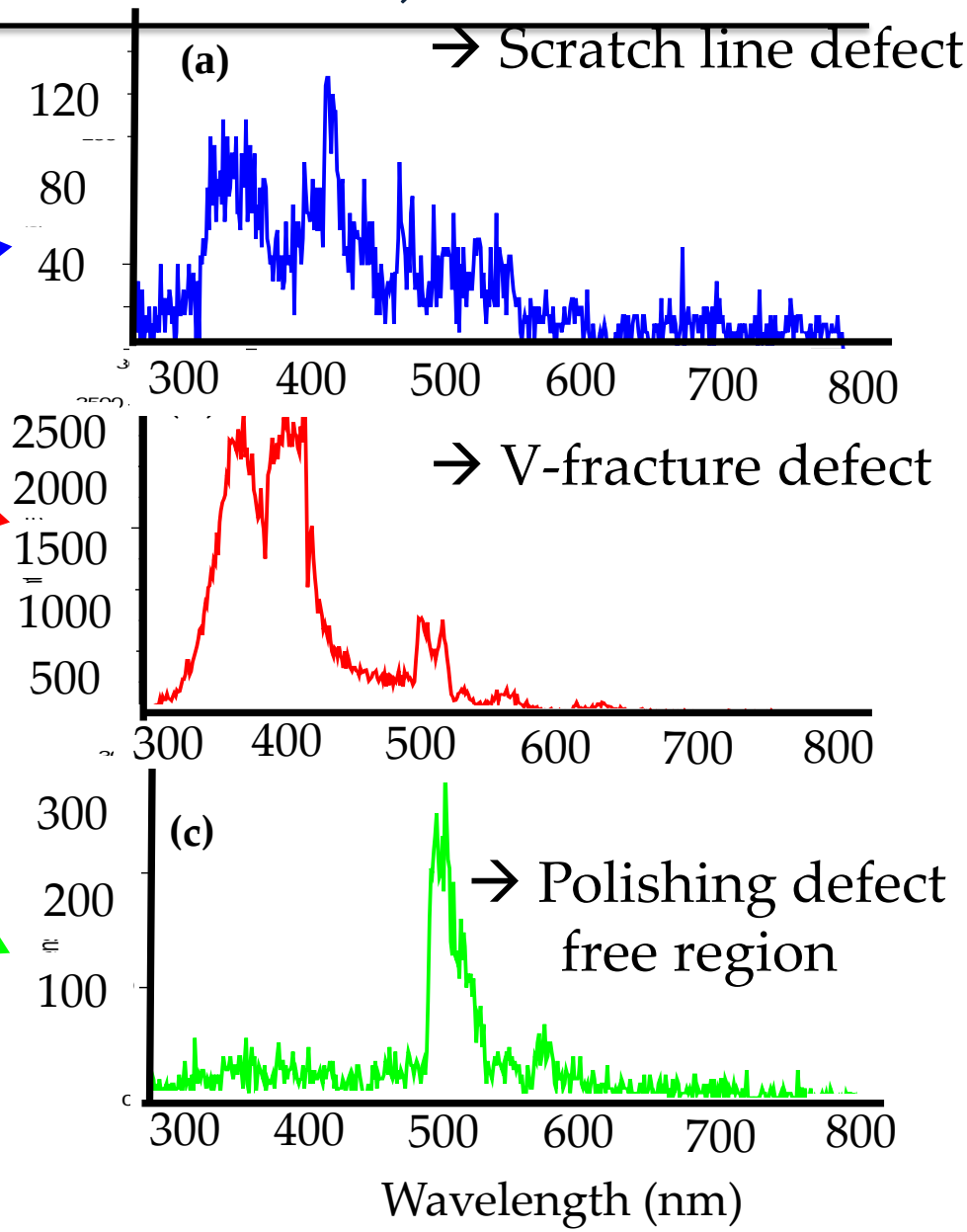
CL Spectra (Spot Area Mode)



Mechanical Polished (5kV, 4000X)



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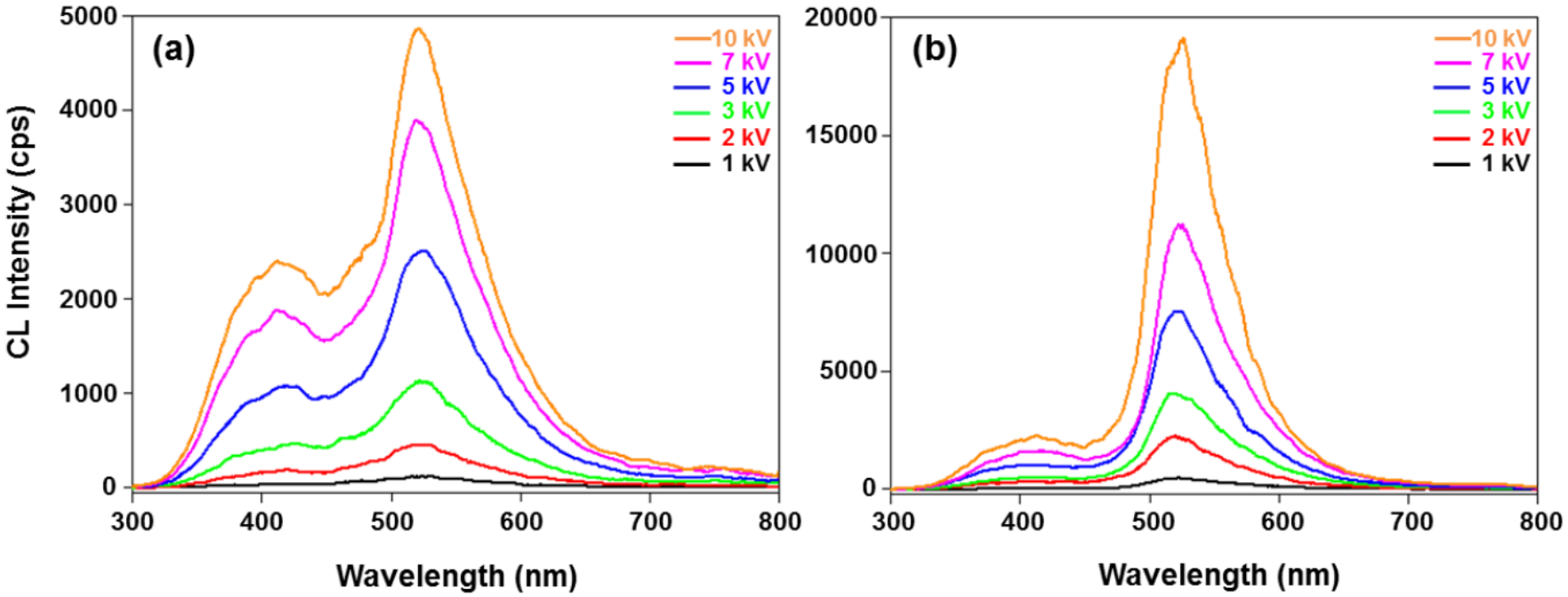
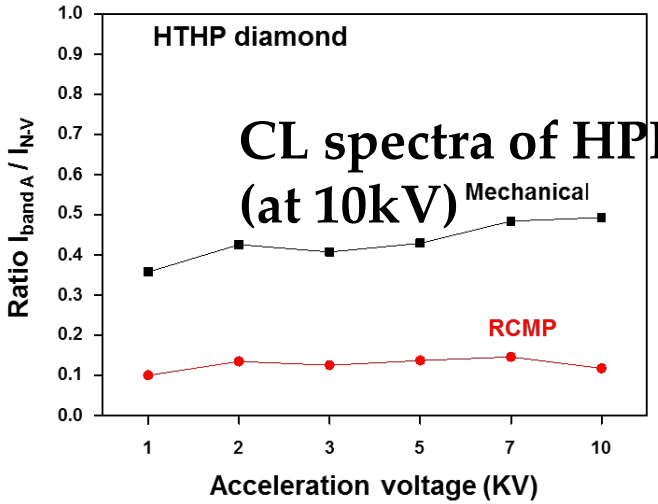
CL spectra (Mechanical vs. RCMP)



CL spectra of HPHT diamond (at 5kV)

CL spectra of HPHT diamond (at 10kV)



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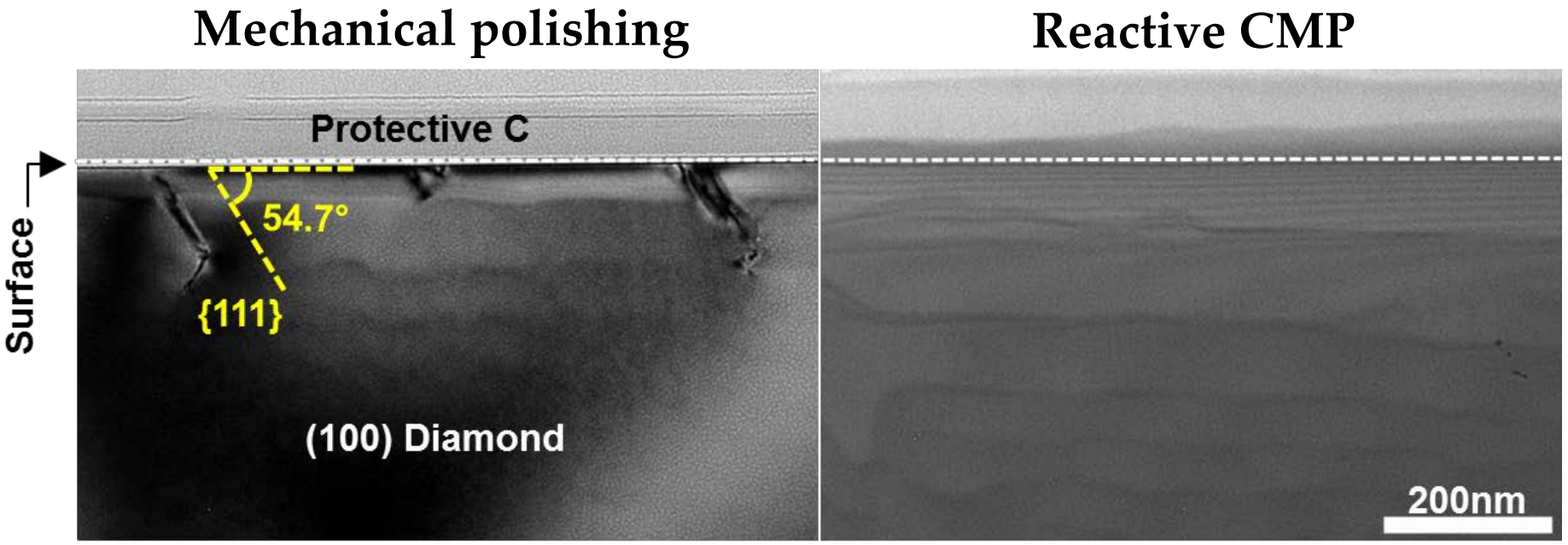
Cross-sectional TEM



- 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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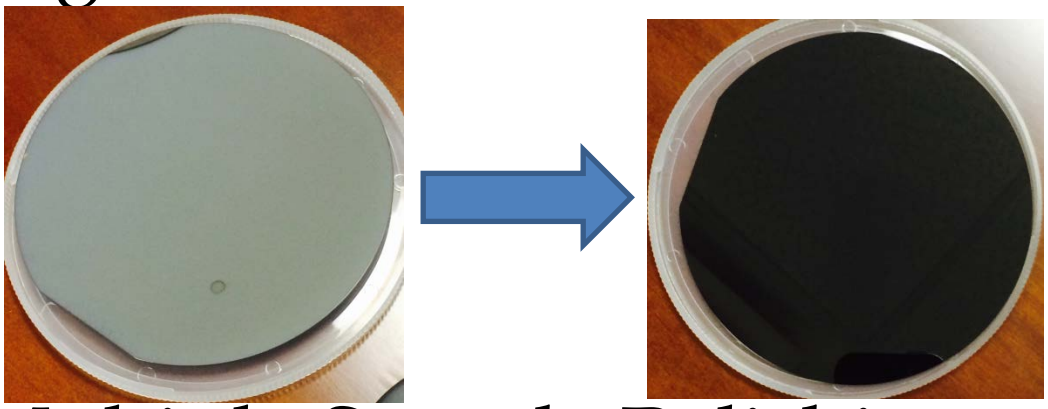
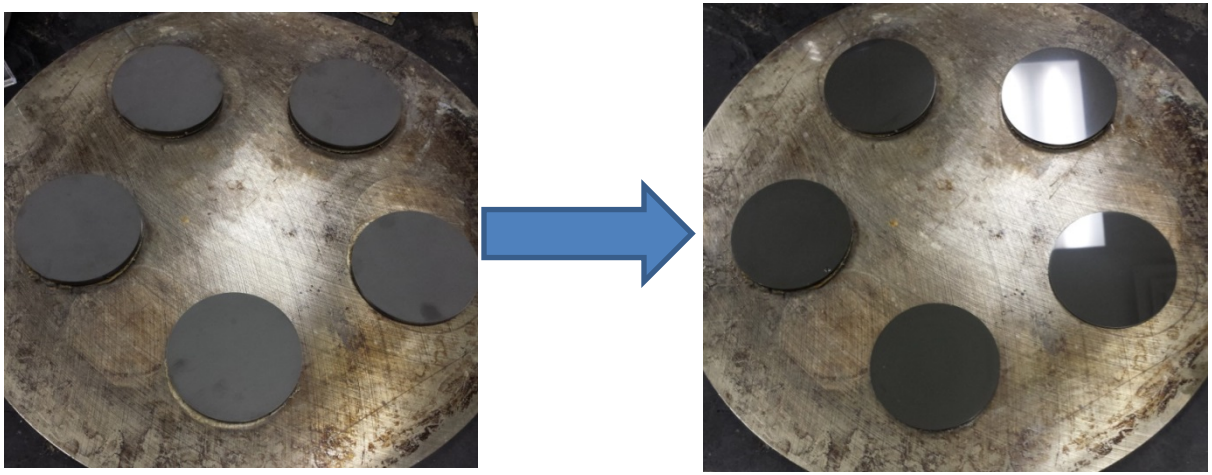
RCMP Scale-up

Large Area Substrate Polishing



4 inch sample polishing

Multiple Sample Polishing

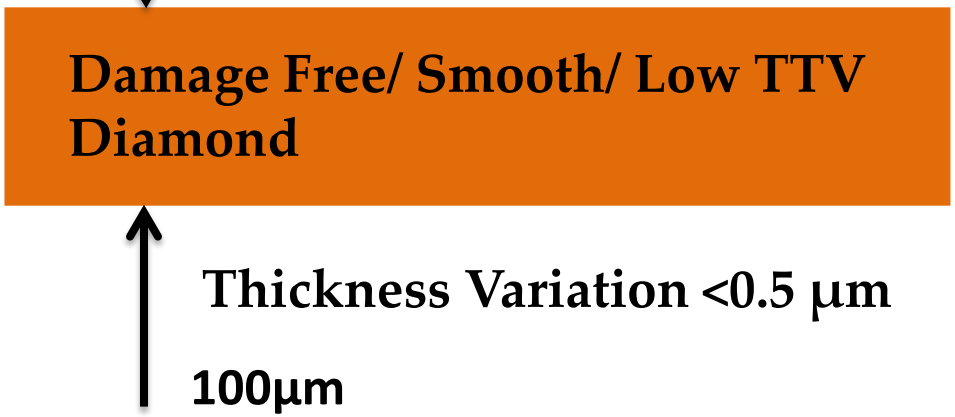


Five 3″ inch samples polishing

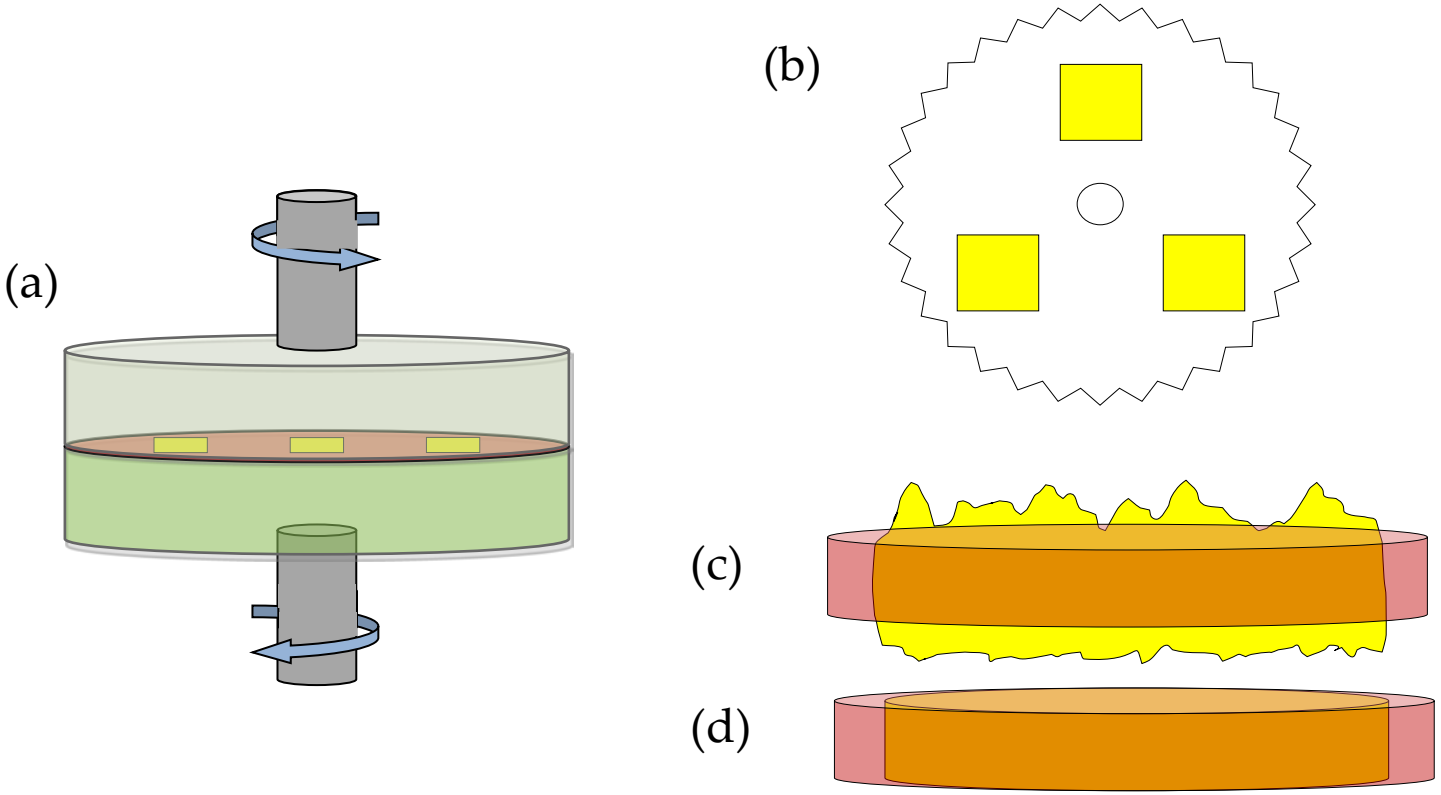
Solution 1: Ultra Flat Holder

Proposed RCMP Processes using specialized holder

Low TTV holder

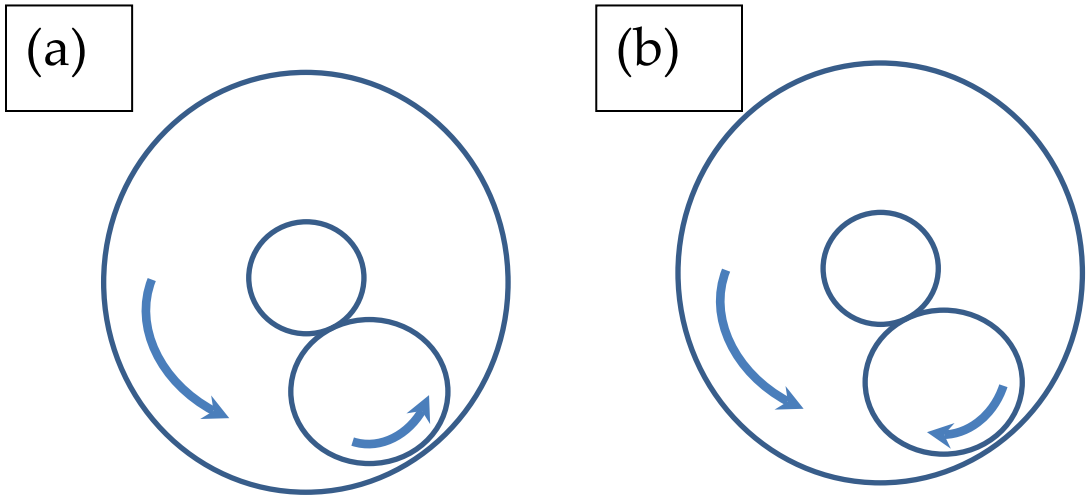


Solution 2: Double-sided polishing

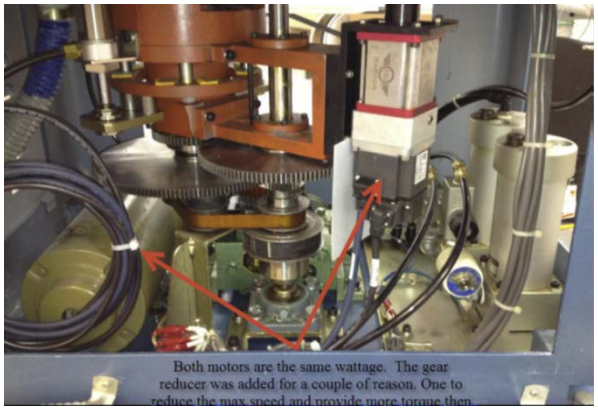


Schematic of (a) double sided polishing (b) Gear sample fixture (c) unpolished samples (d) double sided polished sample with ultra low TTV

Custom Double-sided polishing

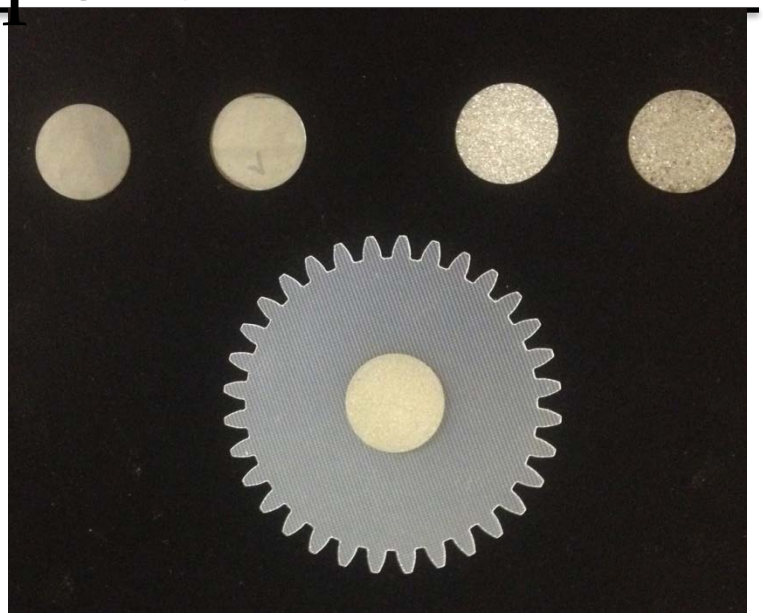


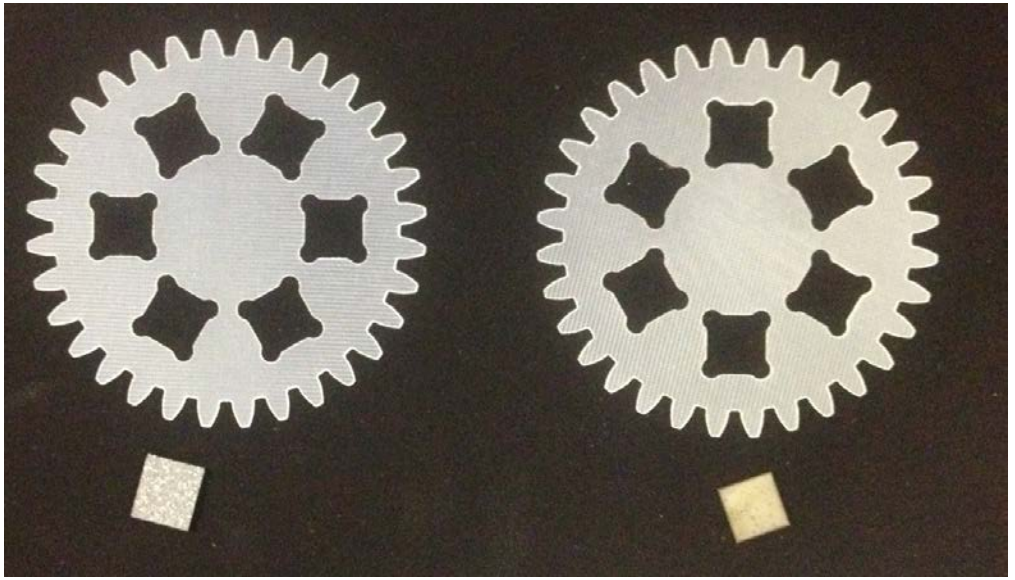
(a) Rotation of carrier and plate in the same direction & Plate will wear concave (b) Carrier and plate rotation in opposite direction & Plate will wear convex



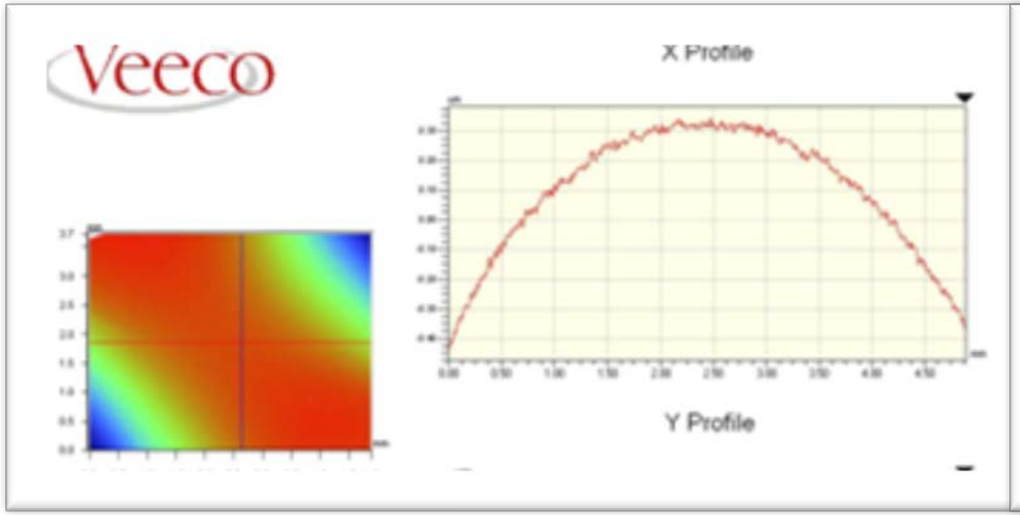
Custom Double-sided polisher

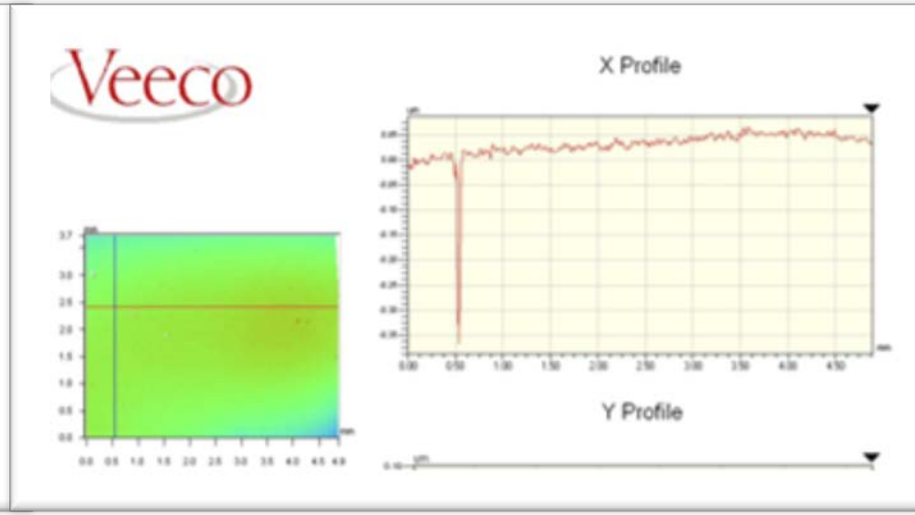
Samples
Polished





Ultra Flat Polishing



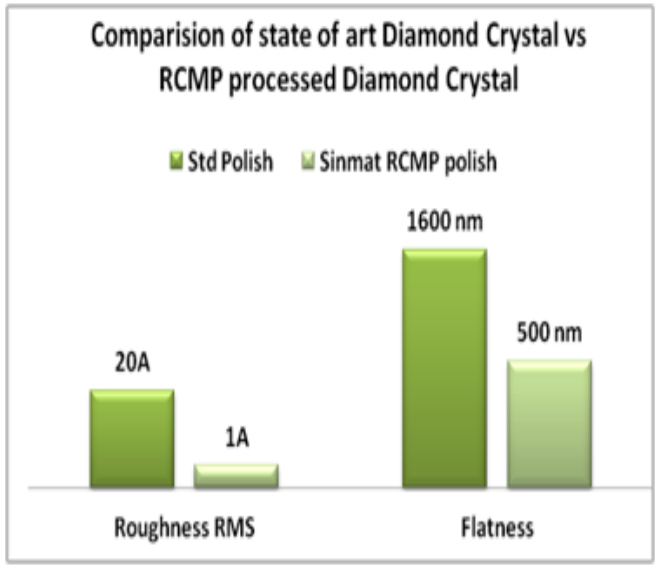


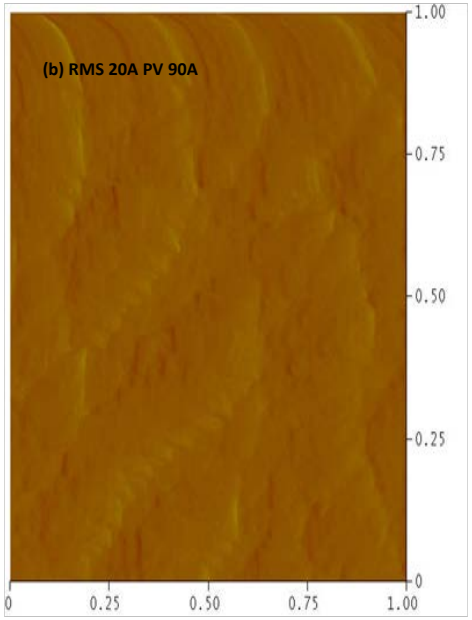
Peak to Valley Roughness
2.9 micron

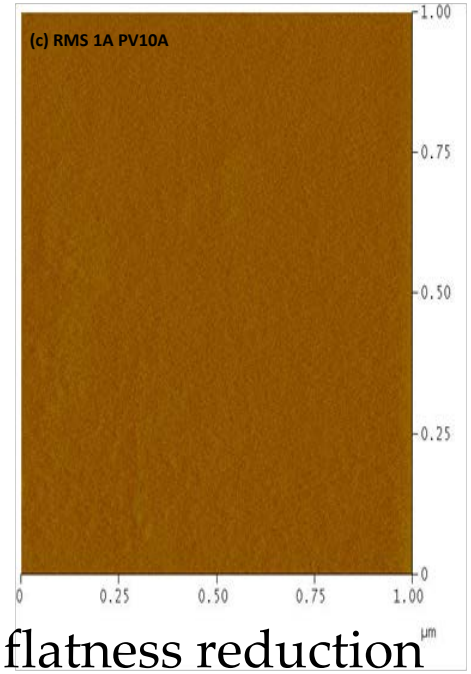
Peak to Valley Roughness
0.55 micron

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



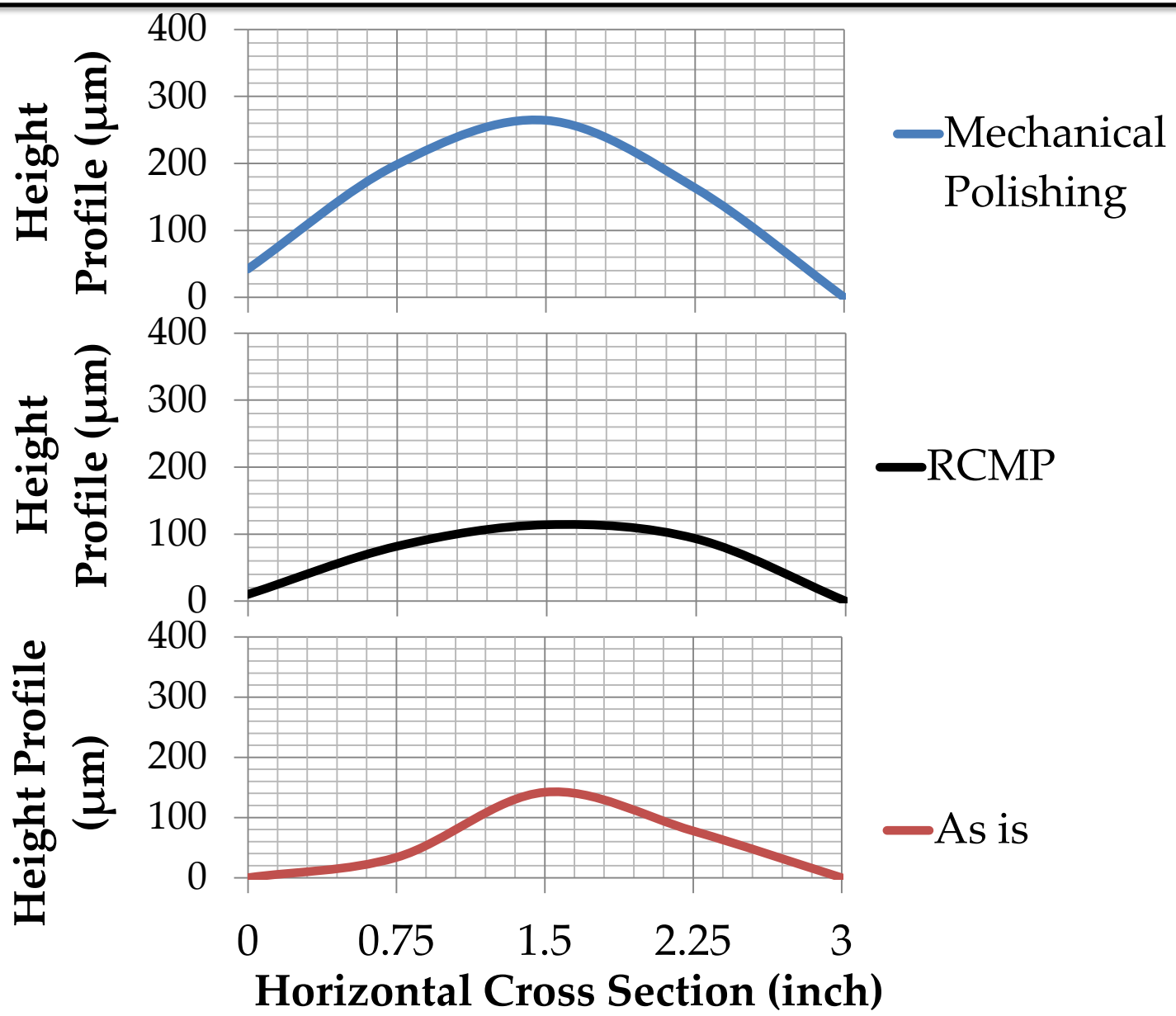




RMS roughness reduction by 1 order and optical flatness reduction from 1.6 micron to 0.5 micron with Sinmat's Reactive CMP (RCMP) polishing, (b) AFM picture of standard polish showing fractured surface (RMS 20A, (Peak to Valley) PV 90A) and (c) AFM picture of RCMP surface shows atomic smooth surface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

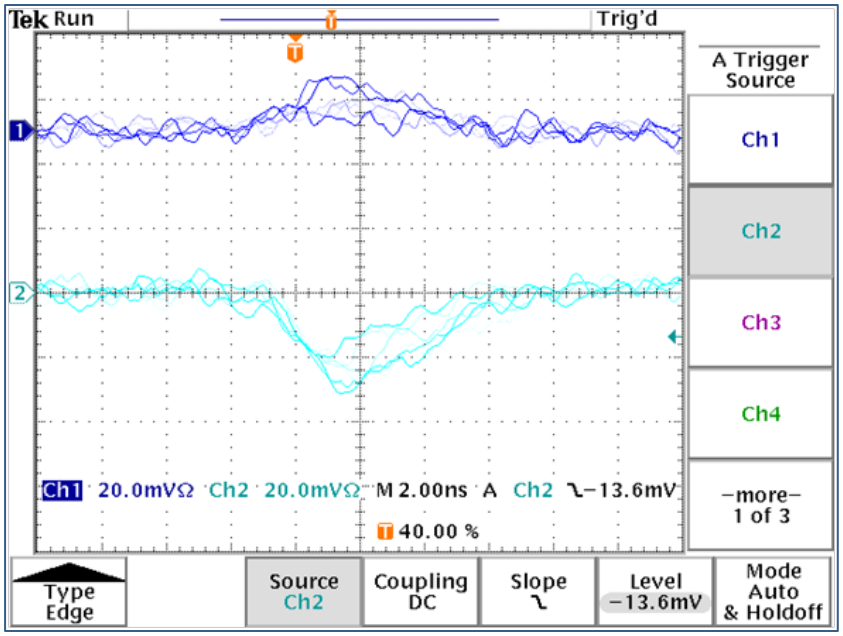
Stress Free Polishing- Reduced Bow

Sinmat
Innovative CMP solutions

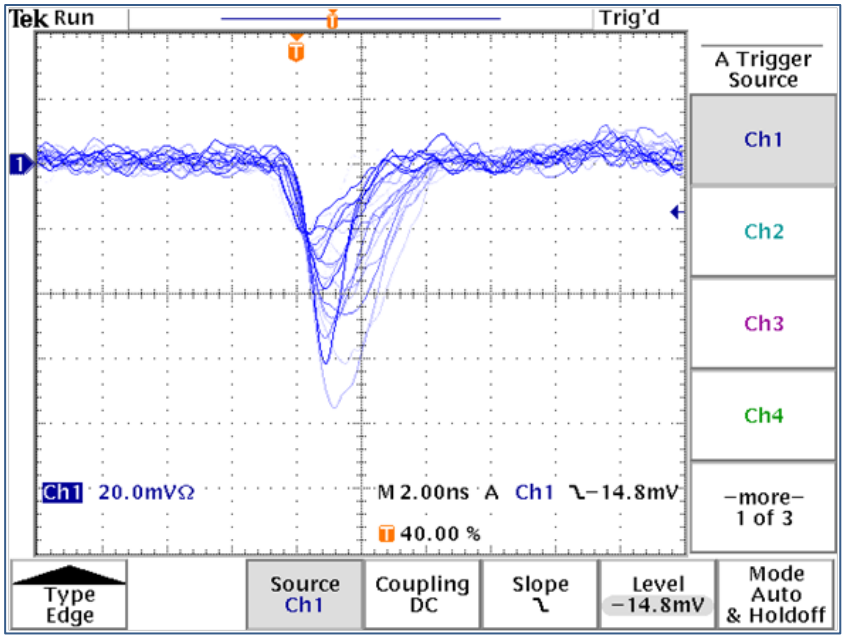


Detector Testing-NSCL

Standard diamond



Sinmat finish diamond

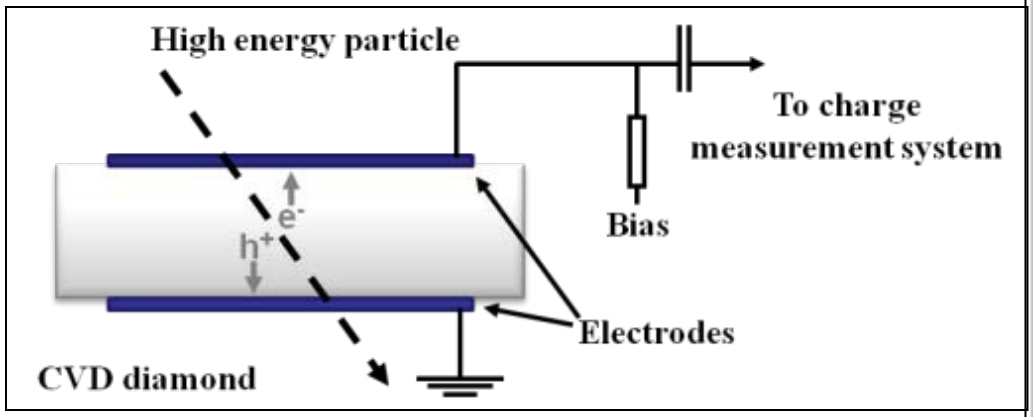


Detector response for U232 Alpha source at 100V bias (a) vendor polished sample – showing pulse height of 20-30 mV (b) RCMP polished diamond sample showing pulse height of 80mV. Both the plates were approximately 100µm thick **(Courtesy: Dr.Stolz , at NSCL)**

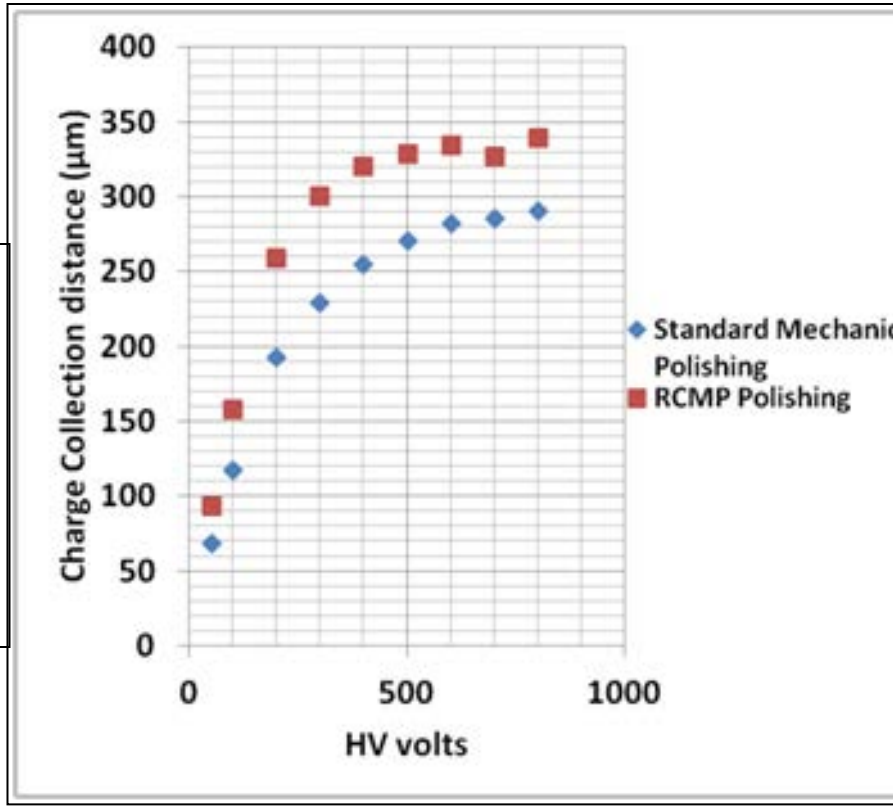
Polishing did not degrade the detector performance

Detector Testing-OSU

^{90}Sr source

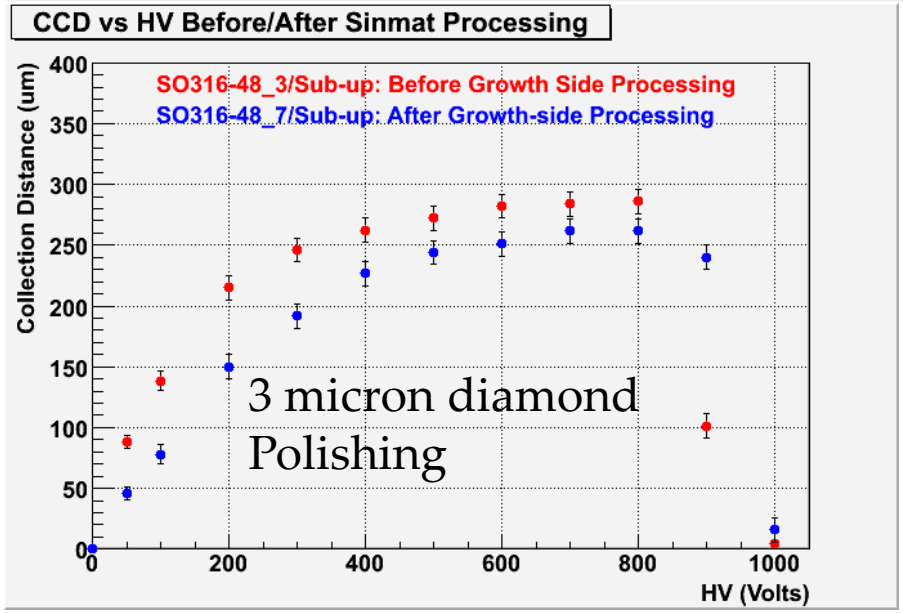


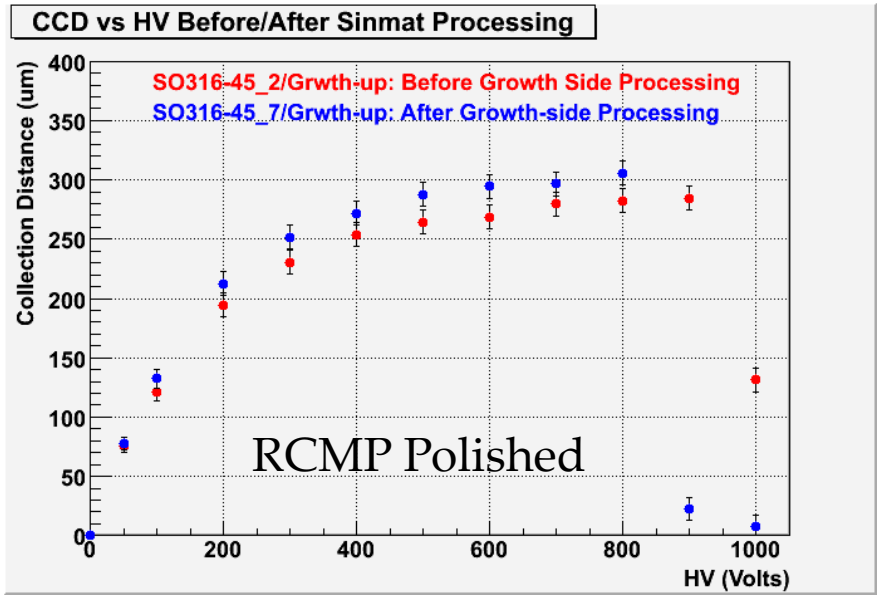
Set-up to Measure Charge Collection Distance

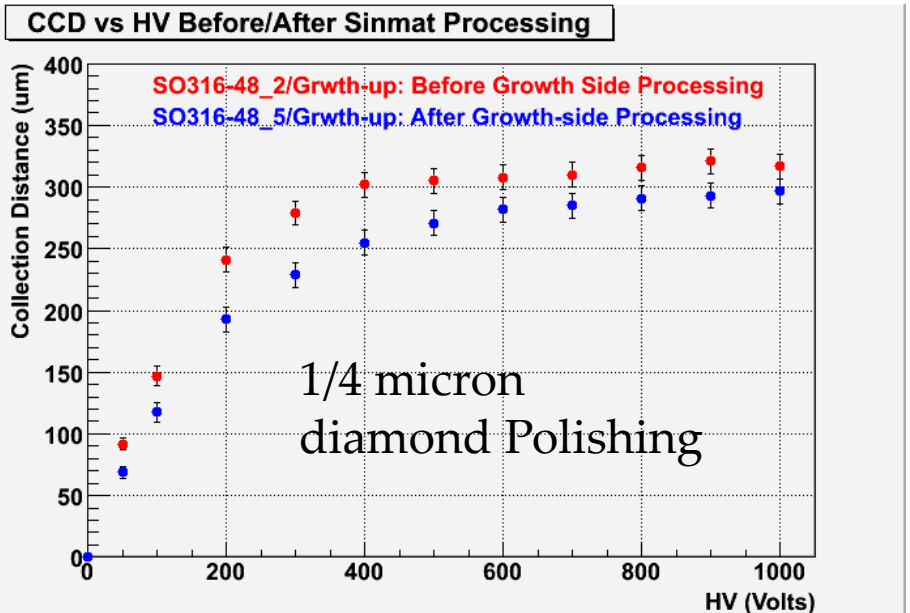


RCMP Polished Sample showing Higher Charge Collection Distance: Courtesy: Dr.Harris Kagan OSU

Detector Testing-OSU



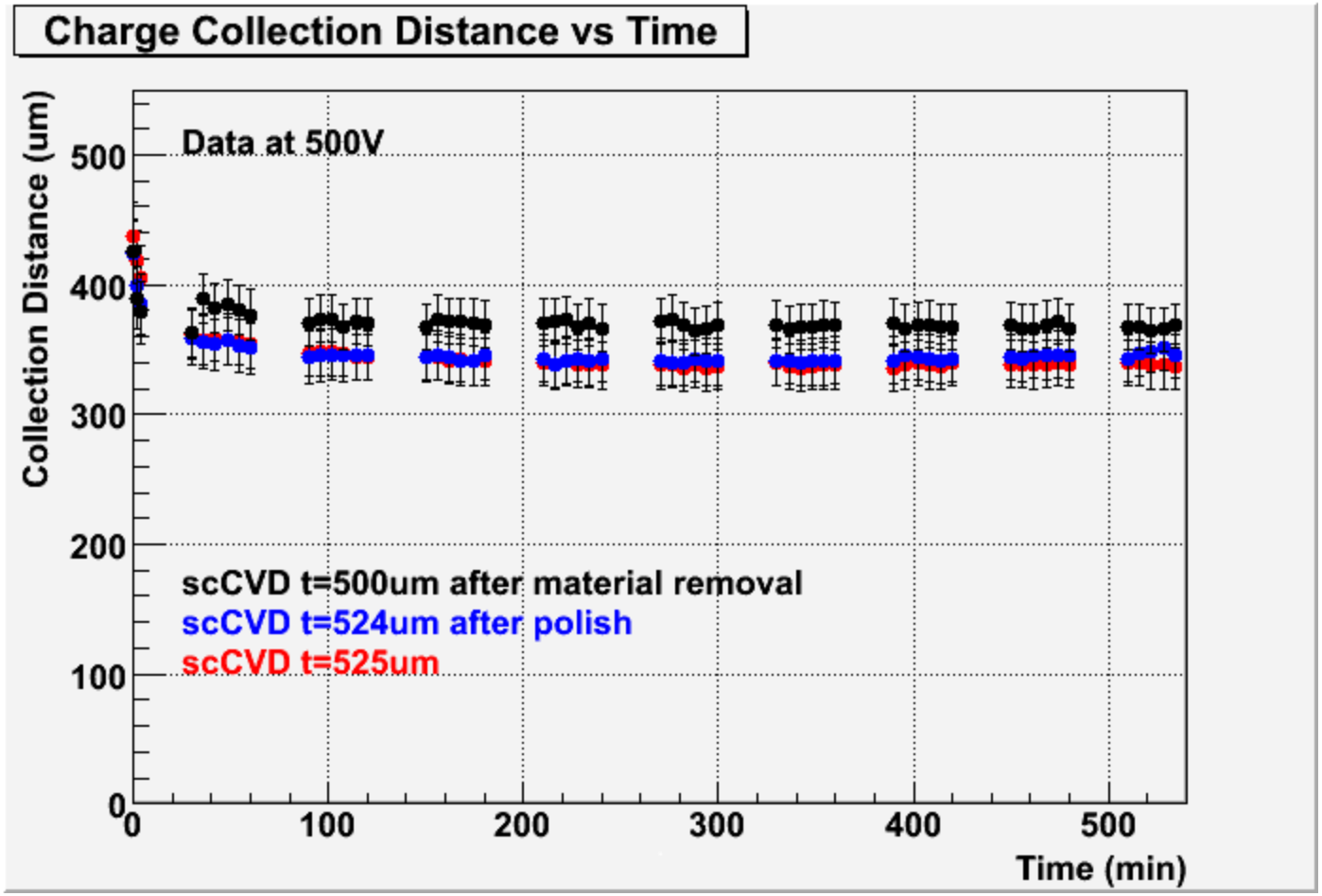




- RCMP Improves Detector Performance Significantly
- Mechanical Polishing Degrades Detector Performance

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