Defect-free Ultra-Rapid Polishing/Thinning of Diamond Crystals Radiator Targets for Highly Linearly Polarized Photon Beams

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Outline

- Introduction- Sinmat
  - Sinmat-overview
  - Diamond Technology
- STTR Project
  - Objectives
  - Results
- Conclusions and Future Directions

Overview: Sinmat Inc.

- University of Florida Spin-off. Developing planarization technologies the semiconductor industry
- Winner of four R&D 100 Awards 2004 & 2005, 2008, 2009
- Employees and consultants: 30
- **Global leader in SiC polishing slurries** (> 50% of global market): electronics for inverters, hybrid cars and SSL
- Approx 50 % revenue from commercial products : Growth rate > 50%/year.
- Developing several CMP centric technologies – LEDs; Power/RF devices; Ultra large wafer polishing

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)

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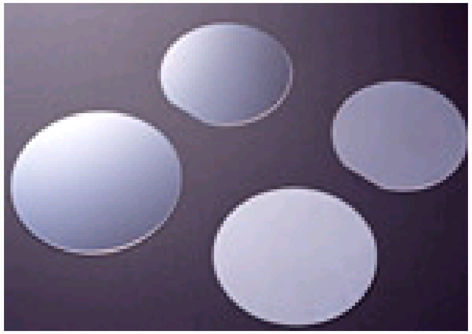
Ultra-hard substrates for electronic & optics

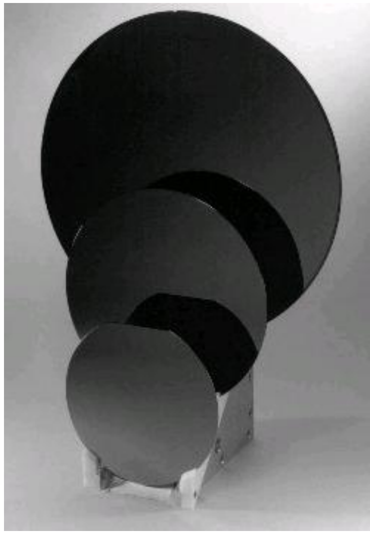
Silicon Carbide (SiC)

Gallium Nitride (GaN)

Sapphire (Al_2O_3)

Diamond Substrates





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

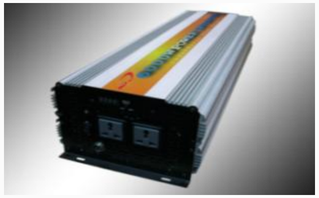
Wide Band Gap Materials (SiC, GaN, Sapphire & Diamond)

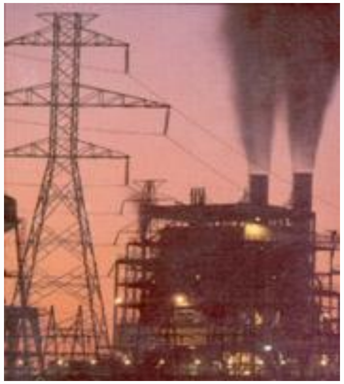
Power Devices

AC-DC Converter

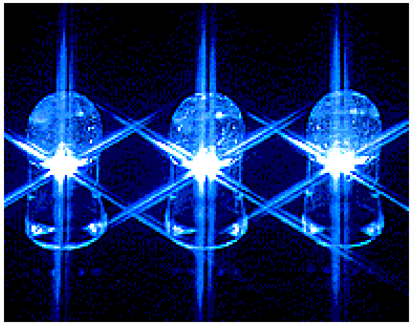
DC-DC Converter

Inverters





Light Emitting Devices (LEDs)



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

- Coherent bremsstralhung radiators for high energy polarized photon beams
  - Nuclear experiments at JLAB and elsewhere
- Beam tracking detectors
  - National Superconducting Cyclotron Lab, Michigan State (US), GSI Darmsdat Germany
- 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**

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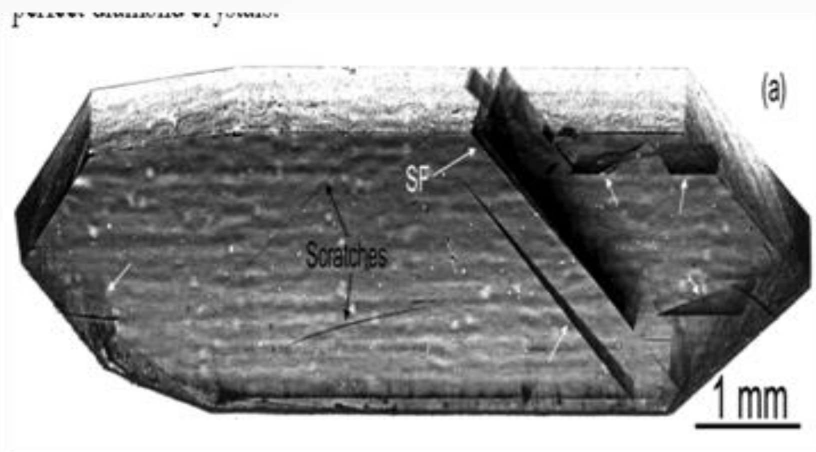
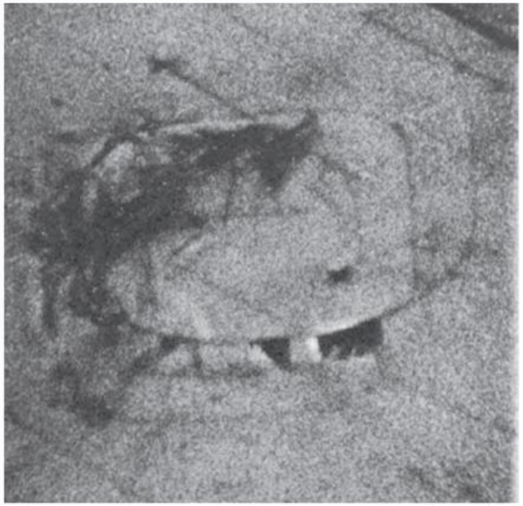
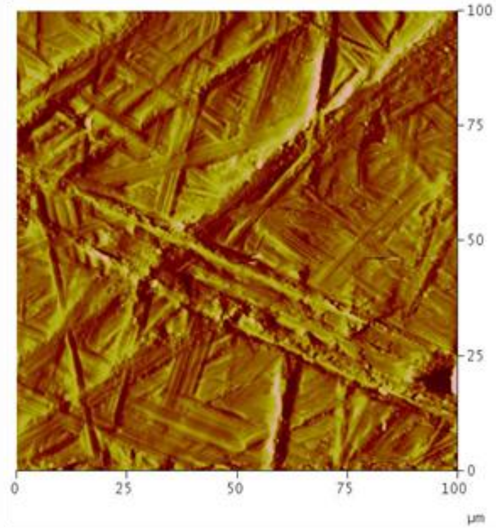
Surface Scratches and Dislocations



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

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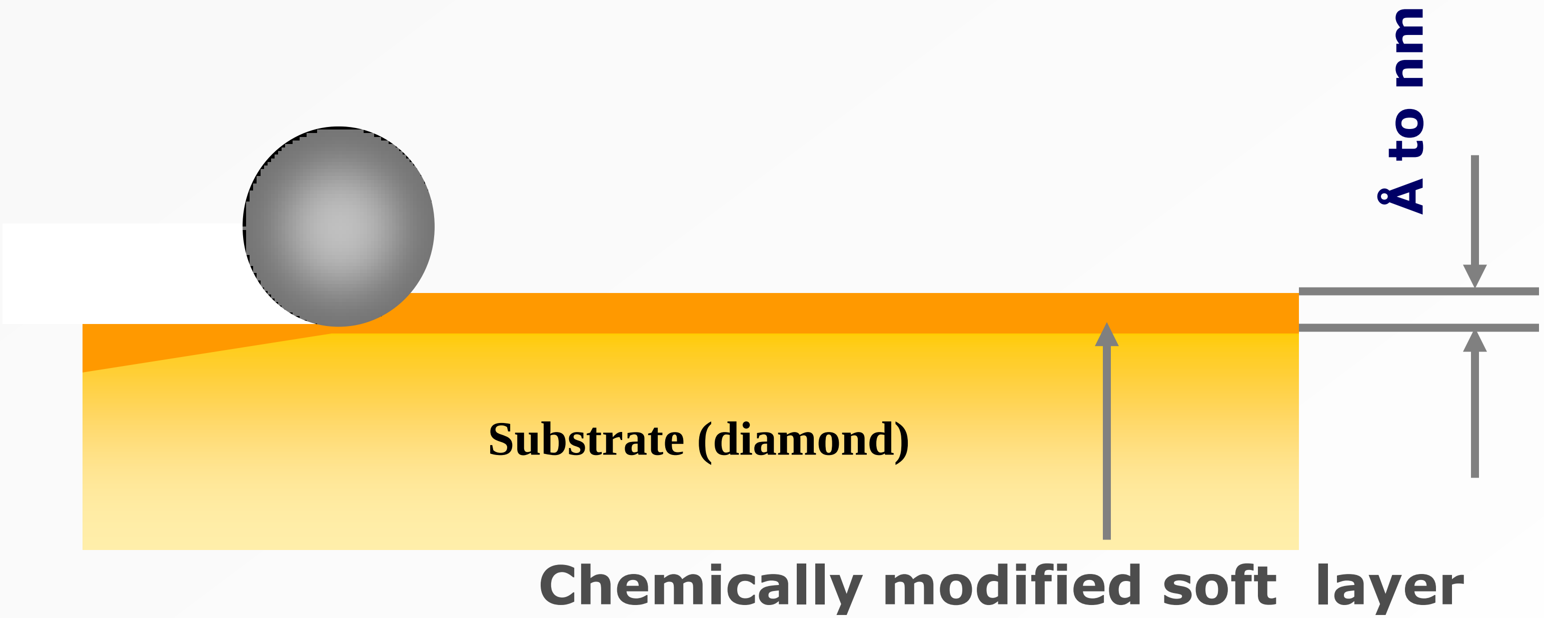
Reactive CMP (RCMP): Soft layer Polish



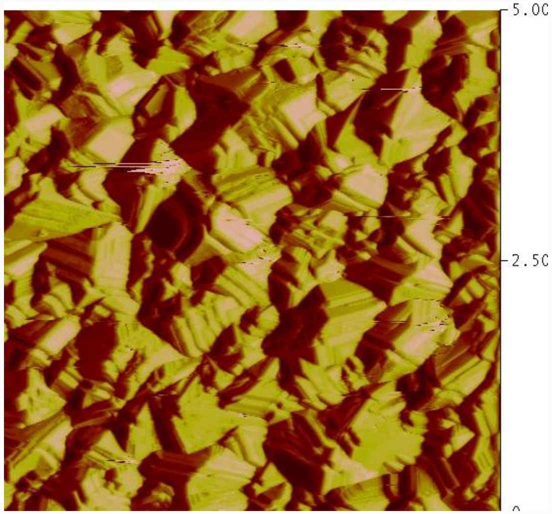
- Chemically convert hard Diamond into a soft-layer
- Use nanoparticles
- Remove Soft layer

  Achieve High Removal Rate

  No Scratches
- Single Component Slurry

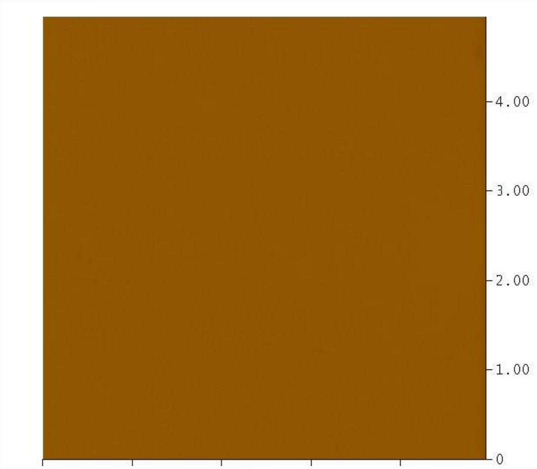
Roughness Reduction of Micro Crystalline samples with RCMP

Before Polishing



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

After Polishing

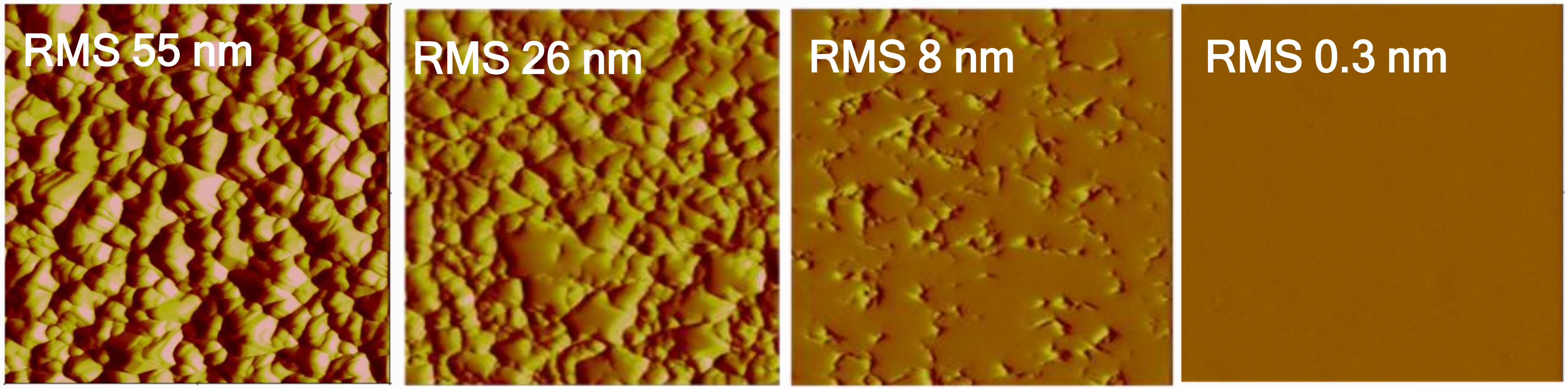


Img. Rms (Rq) 0.335 nm
Img. Ra 0.222 nm

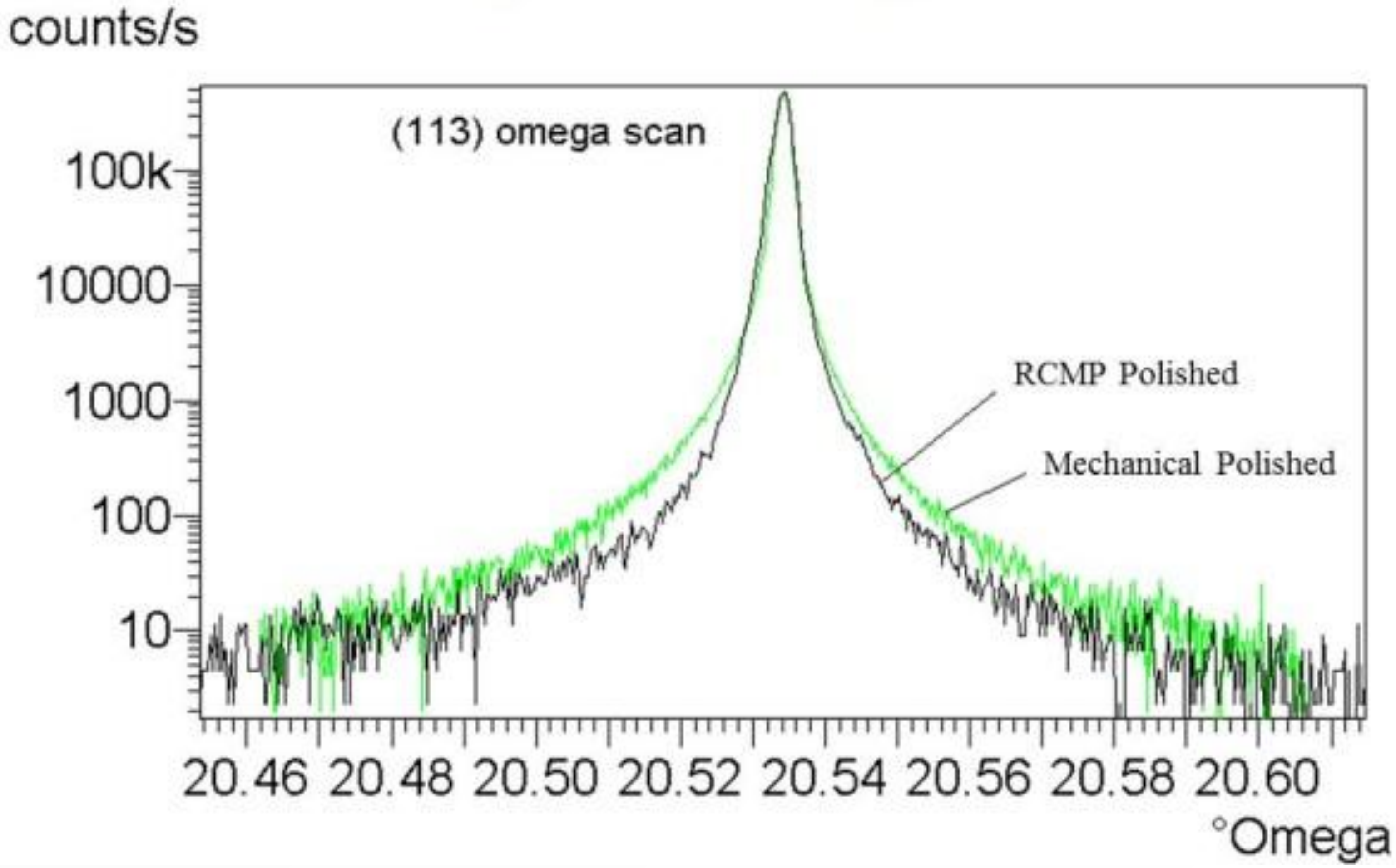
Silicon on Diamond Substrates

Diamond - Reactive chemical mechanical polishing process

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



X-ray Rocking Studies



Step 2 Process –reduced X-ray rocking curve width

Timeline of Diamond Growth & Polishing

1952
First Demonstration of diamond by Synthetic 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)

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

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

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

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

1950 1960 1970 1980 1990 2000 2010

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Sinmat's Diamond Strategy

- **Leverage its novel diamond polishing technology to fabricate high performance diamond based devices for Nuclear Phsysics Applications**
  - Ultra-Thin (< 50 microns) Diamond radiator crystals
  - Diamond Detectors
  - 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**

Outline

- STTR Project
  - Objectives
  - Results

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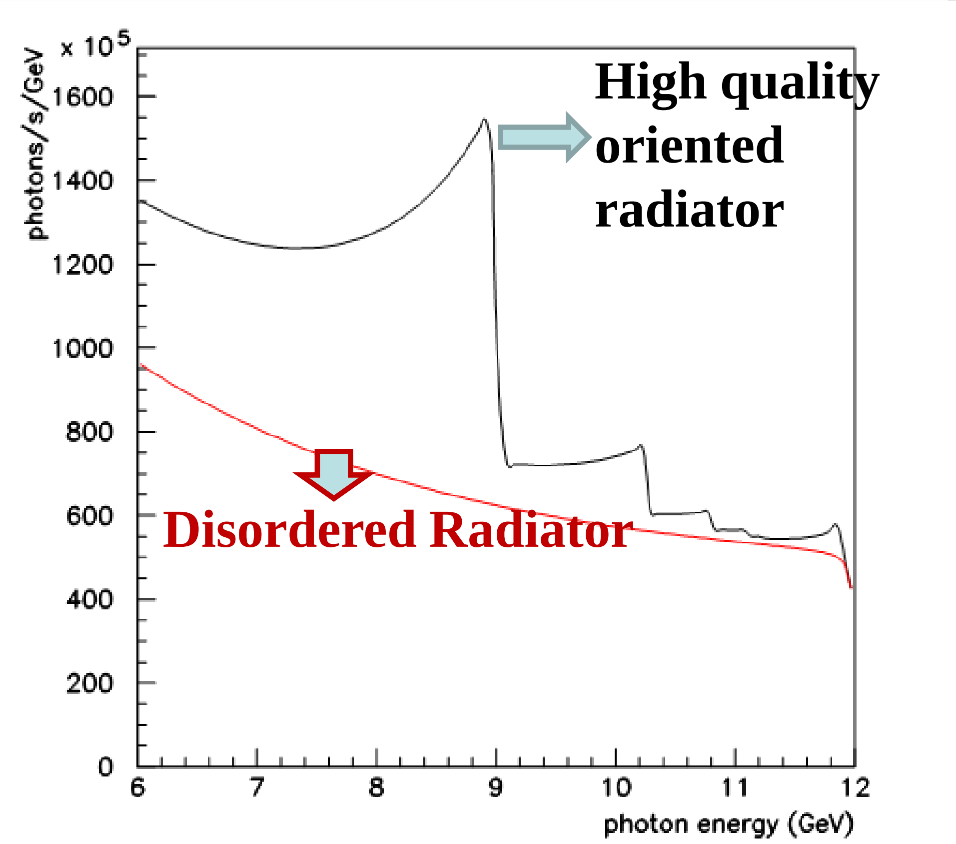
STTR Phase II Project Objective

- Fabrication of thin diamond (20 micron thick) coherent bremsstrahlung radiator targets for the GlueX experiment at JLAB-12GeV
- Requirements
  - Large area: 4x4 mm^2
  - Small thickness: <20μm
  - Ultra-flat crystal planes: <20 μr RMS

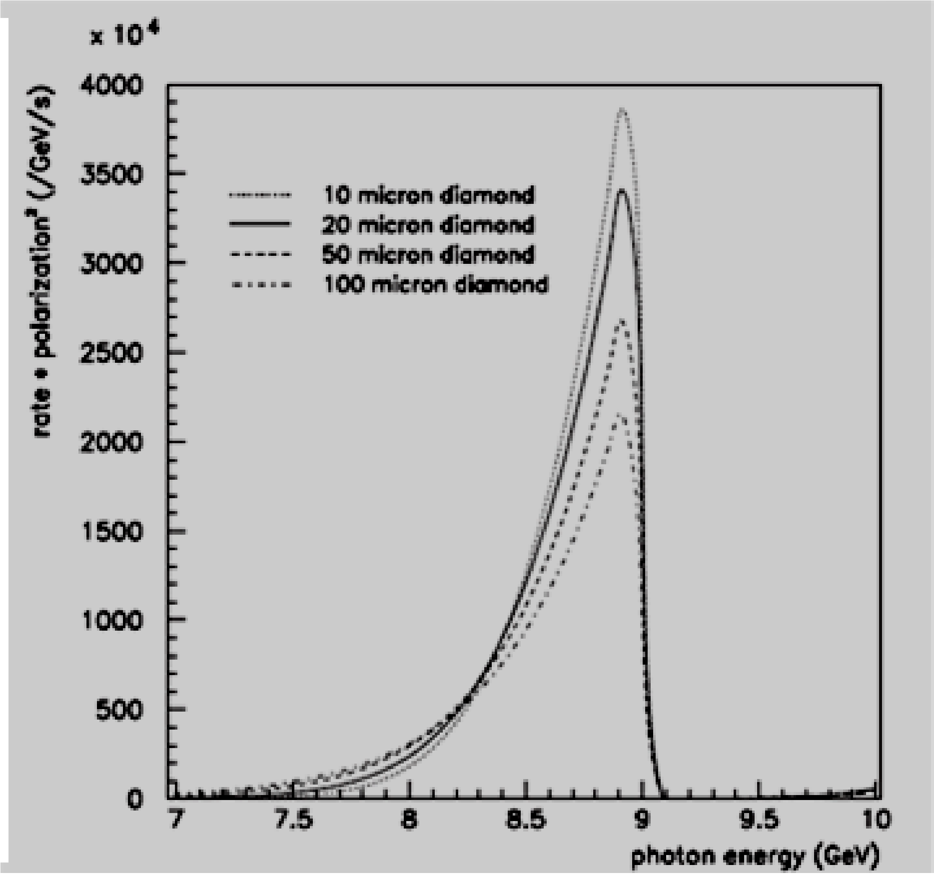
These two are in tension

- Current state of the art can provide either high flatness or low thickness but not both together

Quality/Thickness of Diamond Vs Radiator Performance



Bremsstrahlung spectrum with and with out oriented crystal radiator



Polarization Figure of merit as function of diamond radiator target thickness

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Technical Metric

DIAMOND PLANARIZATION/THINNING	
Property	**Proposed Polishing Metrics**
Dimension	> 4mm x 4mm
Surface finish (roughness)	<1.5A measure area 5x5 μm by AFM
Sub-surface damage	Non-existent when measured optical polarization and cathodoluminescence
Thickness	**<20μm**
TTV	±1μm
(220) RC peak width	**<20μr whole-sample RMS**
Polish rates	>3μ/hr
Vapor Phase Etch rates	>75μ/hr
Other features	Multiple sample polish capability

APPROACH 1

2-step process to achieve project goals

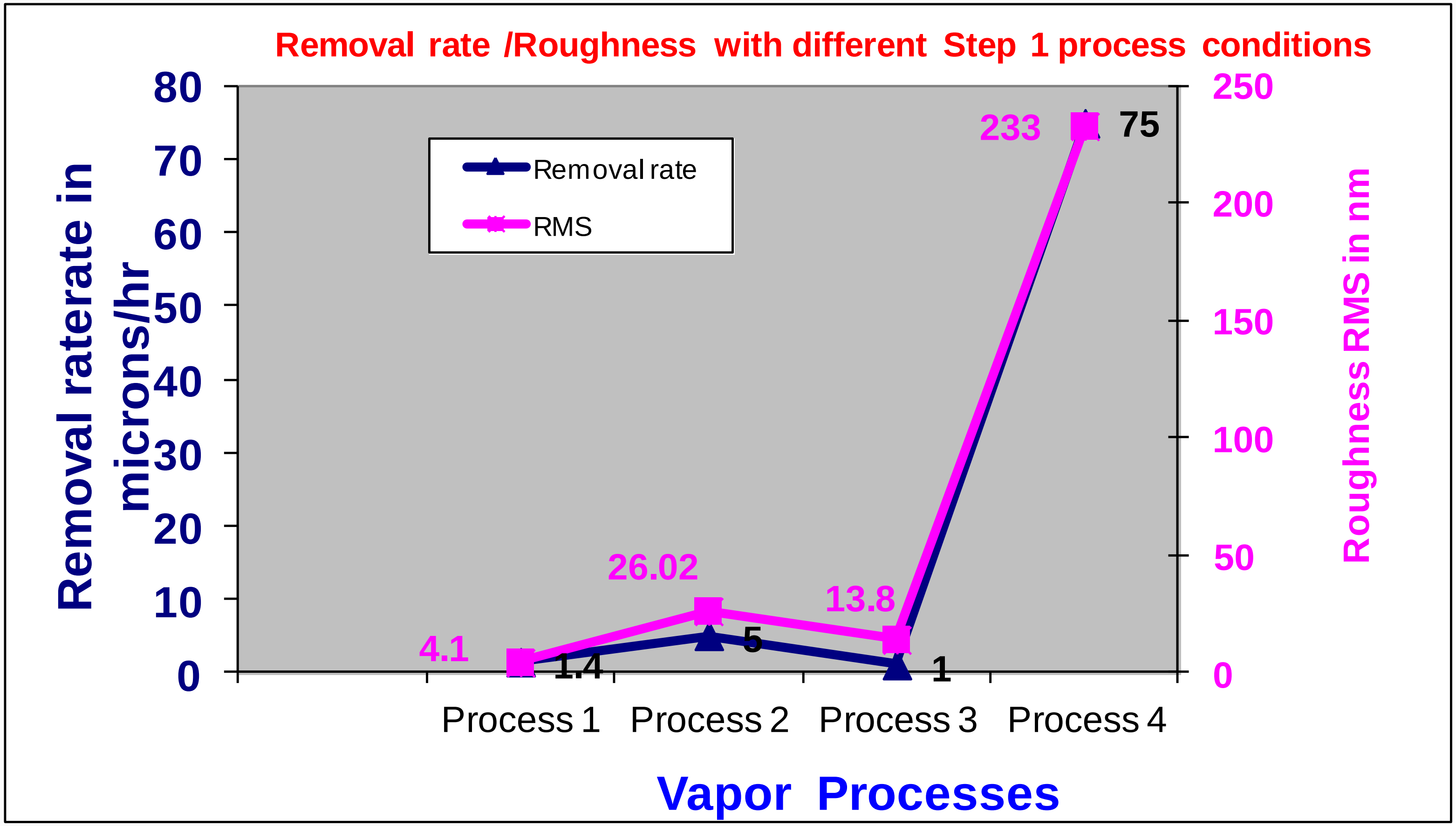
- Step 1: Ultra rapid thinning using Vapor phase technique
  - Removal rates ie., >50 microns/hr
  - Surface may have high roughness (20-100 nm rms)
- Step 2: achieve ultra-smooth, defect-free surface using RCMP process
  - Help removing the roughness created by step 1 process rapidly
  - Creates defect /damage free surfaces

100 micron thick sample → **30 micron thick sample** → **20 micron thick sample**

Step 1: Rapid Thinning using Vapor phase technique

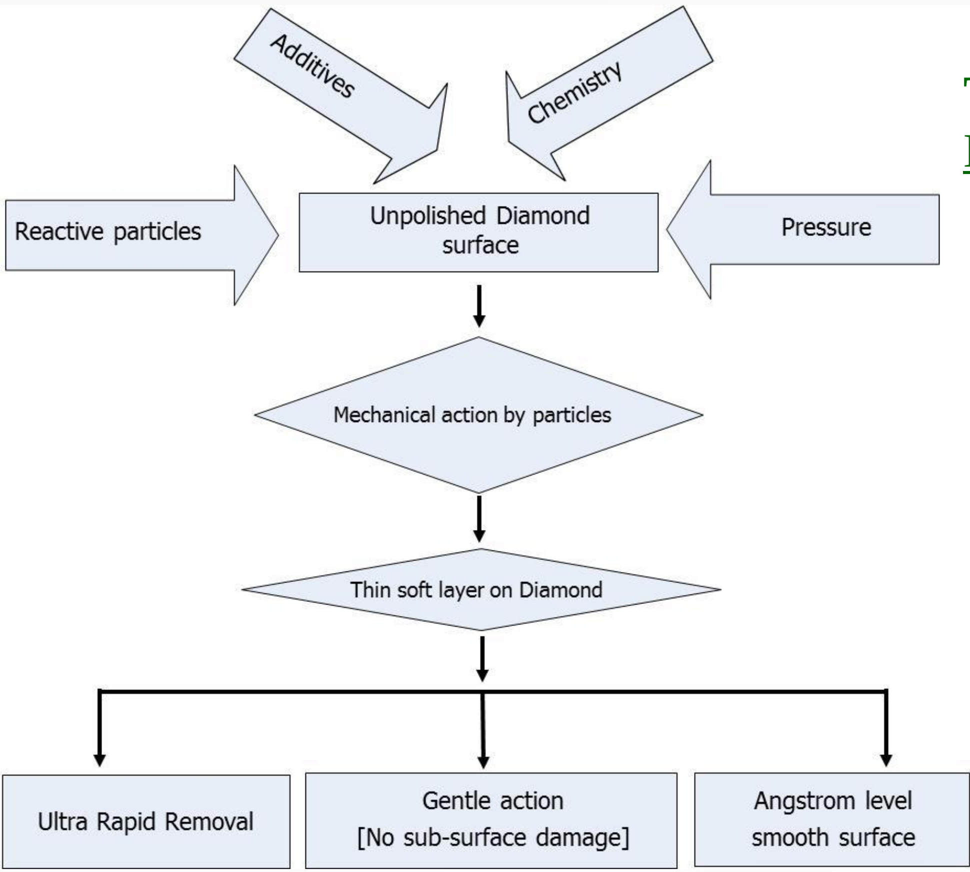
Step 2: Rapid Defect-Free, Ultra-smooth polishing using RCMP process

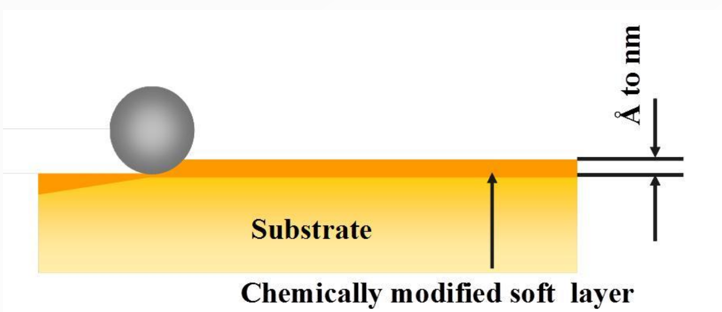
Step 1 Processes



- The higher the removal rate, the higher the roughness with vapor phase process
- The roughness caused by this step will be removed by Step 2 process

Sinmat's Reactive Chemical Mechanical Polishing (RCMP) Process





Technological Innovation: Reactive Chemical Mechanical Planarization (RCMP) process

Diamond + Particles + Chemistry $\xrightarrow{\text{Chemical Action}}$ **Soft Layer**

Soft Layer + Particles $\xrightarrow{\text{Mechanical Action}}$ **Polishing of Diamond**

Unique Aspects of the RCMP Process

- Surface finish 1 – 10 Å achieved
- Large area (2 inch – 8 inch substrates)
- Low friction
- Nanoparticles based process
- No sub-surface damage
- Applicable to all types of Diamond films
    - Single-crystal
    - Micro-crystal
    - Nanocrystal

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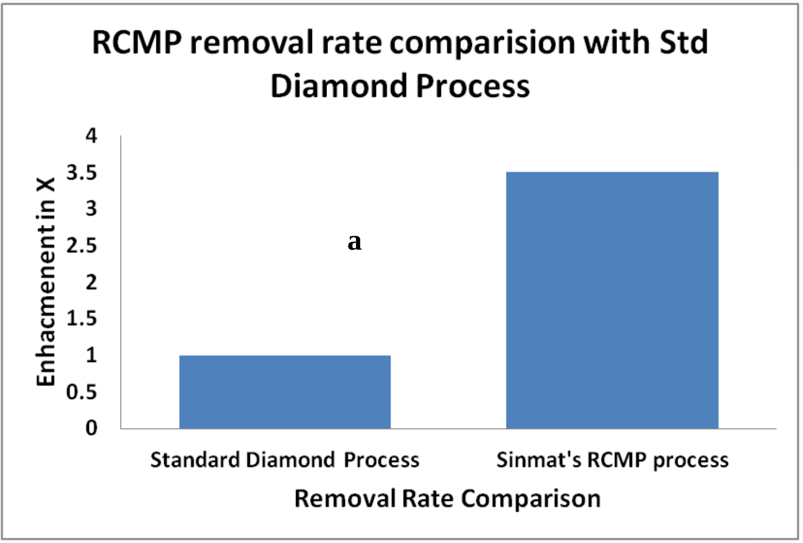
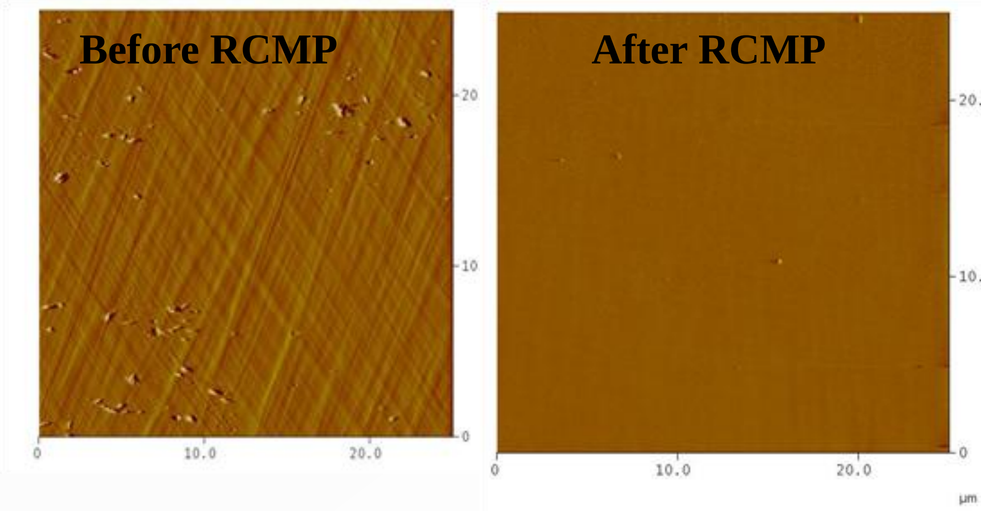
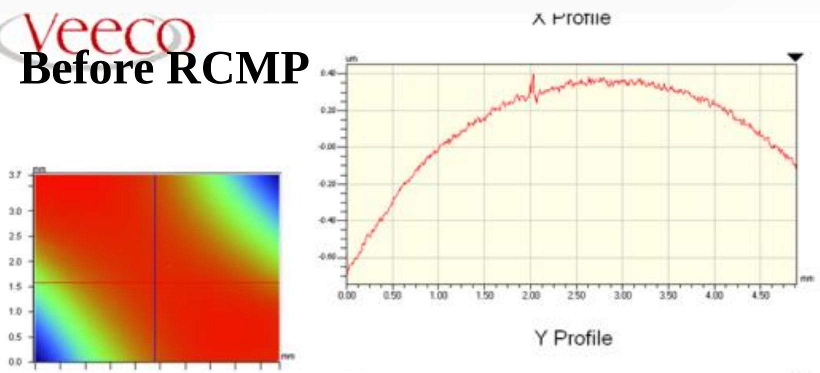
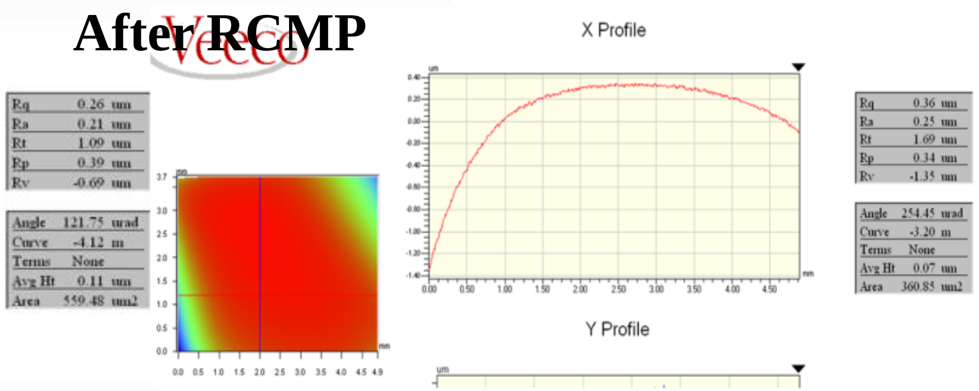
RCMP Polishing on Single Crystal Polishing





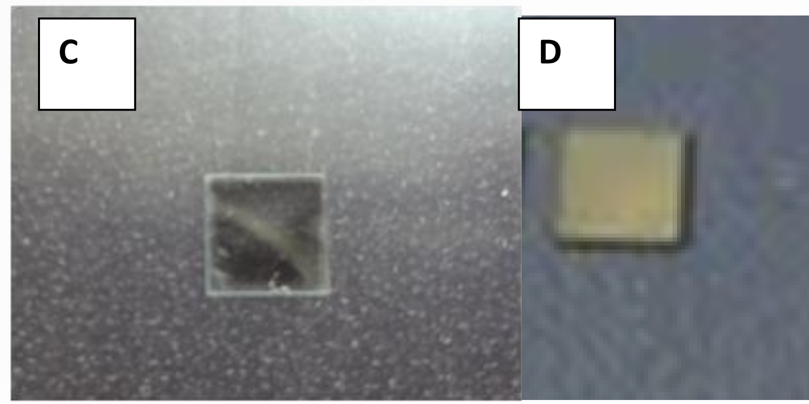




1) Enhanced removal,
2) elimination of scratch
3) Improved Flatness

First results: 4 samples thinned, assessed

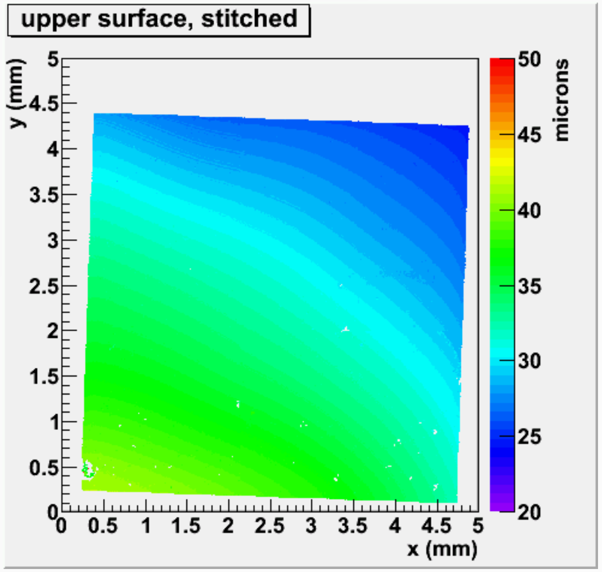
- Samples thinned and polished at Sinmat
- Surface, thickness profiles measured with Zygo interferometer
- Samples taken to CHESS for X-ray rocking curve topographs

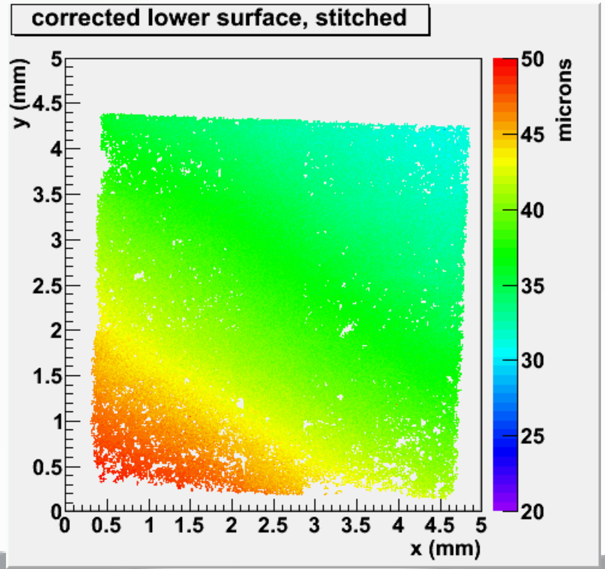


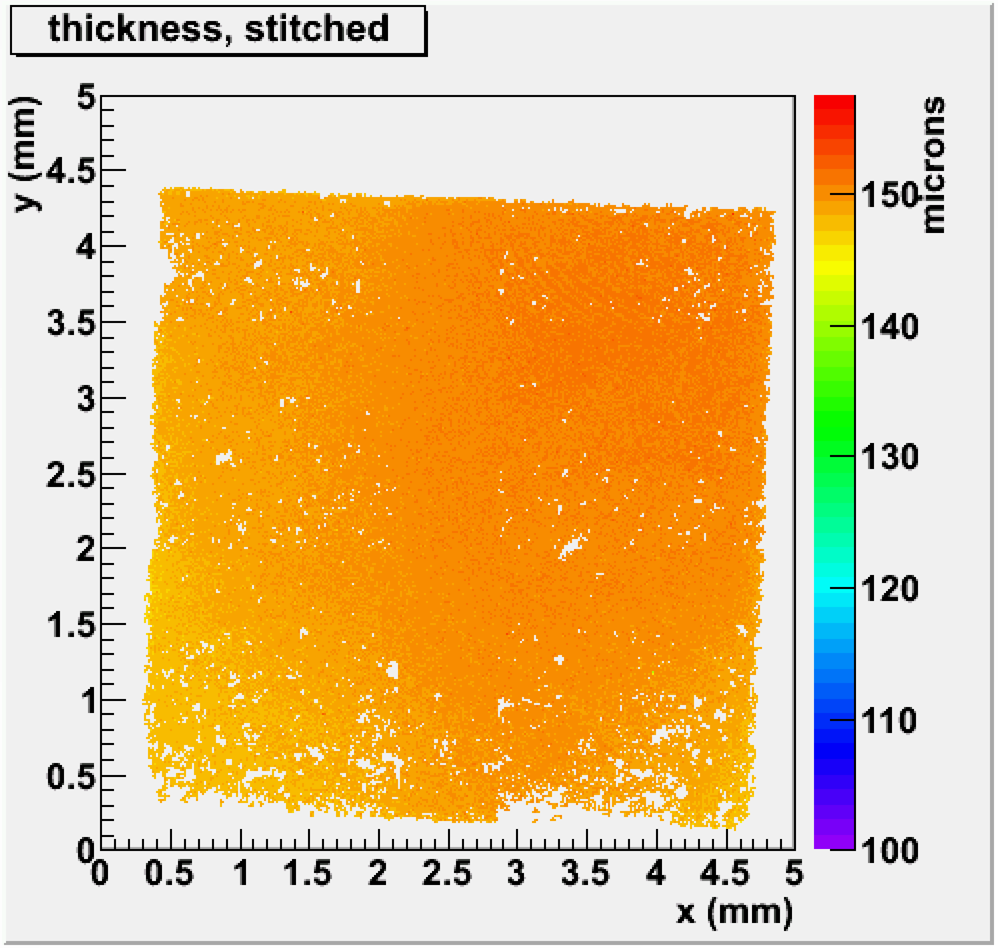
4 different crystals fabricated with using RCMP & VPE etching (A) 150 micron thick (B) 90 micron crystal (C) 30 micron thick crystal, (D) 10 micron thick crystal

Sample A: 150 microns thickness

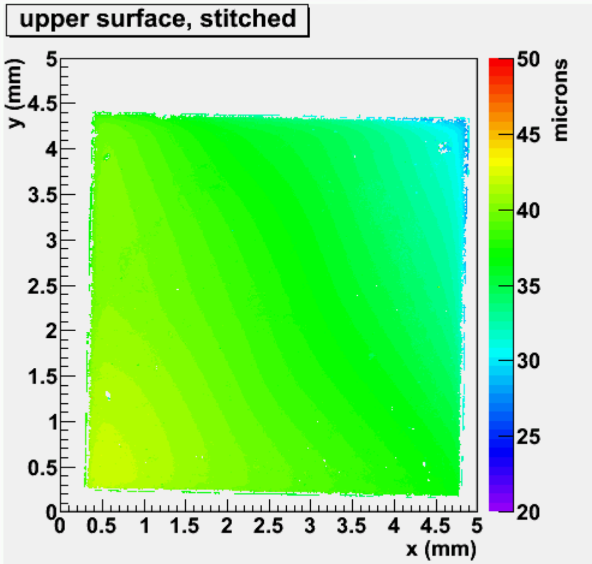
surface and thickness profiles (Zygo 3D)

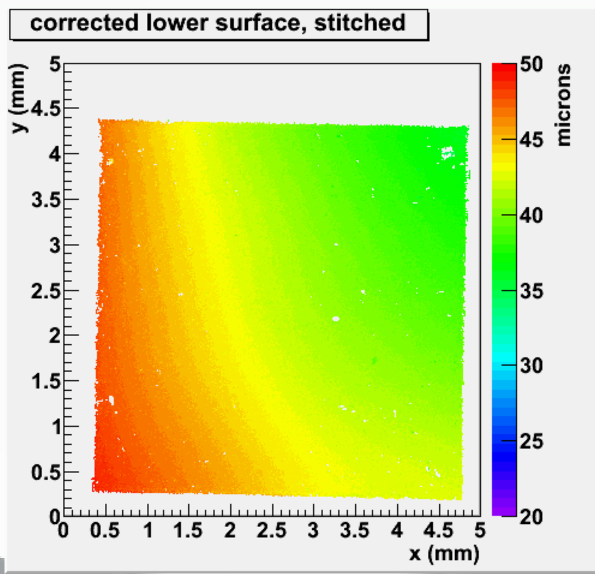




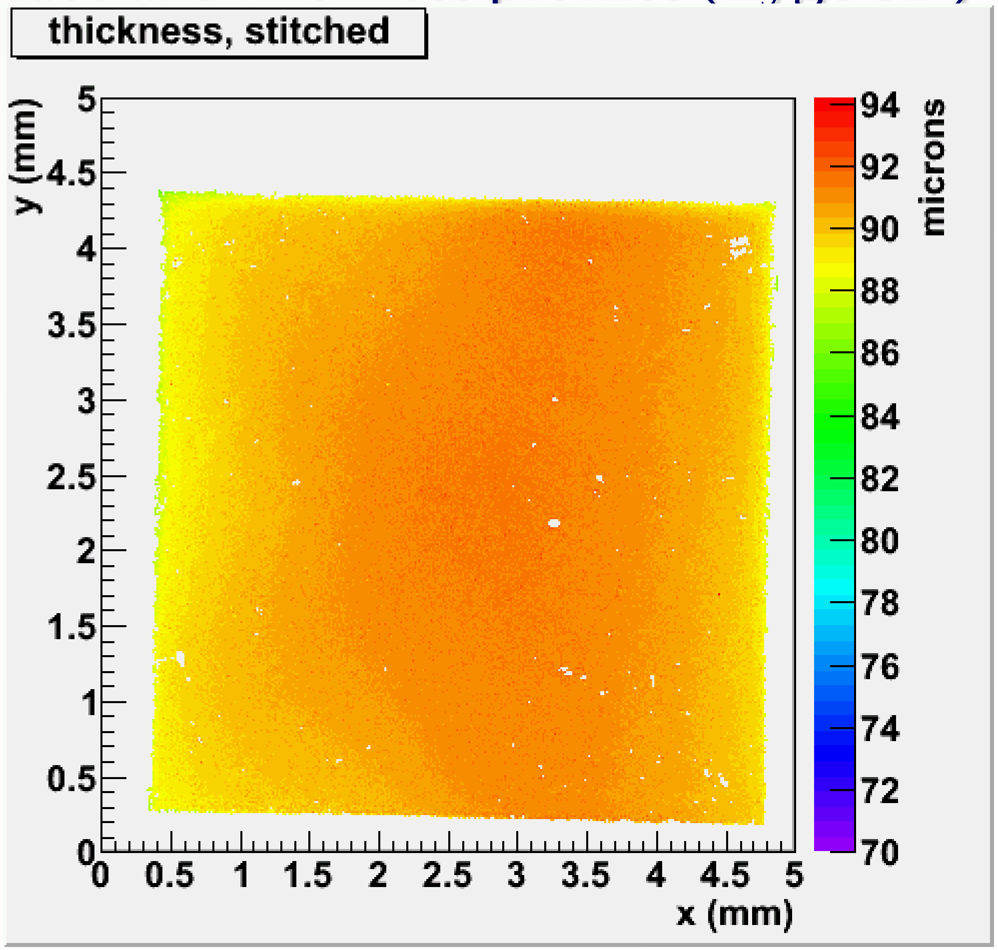


Sample B: 90 microns thickness

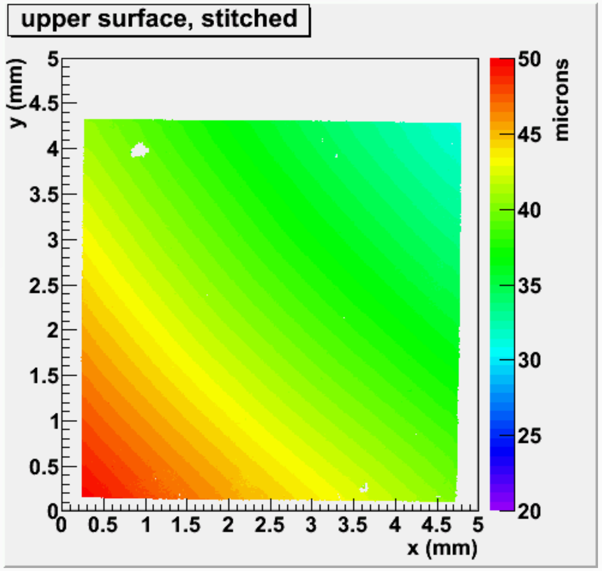


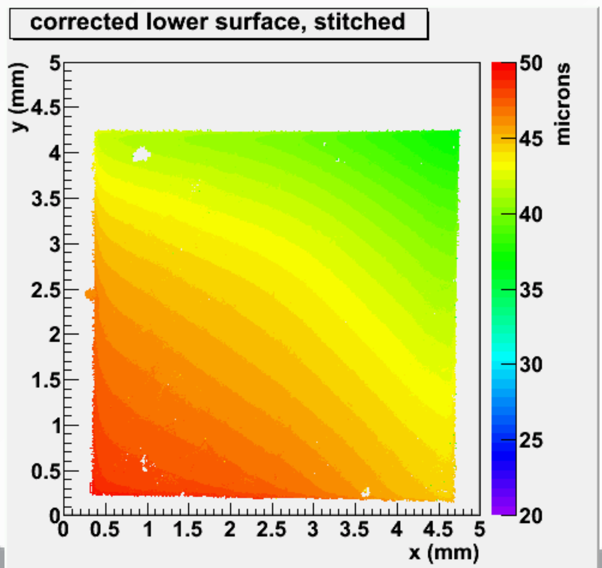


surface and thickness profiles (Zygo 3D)

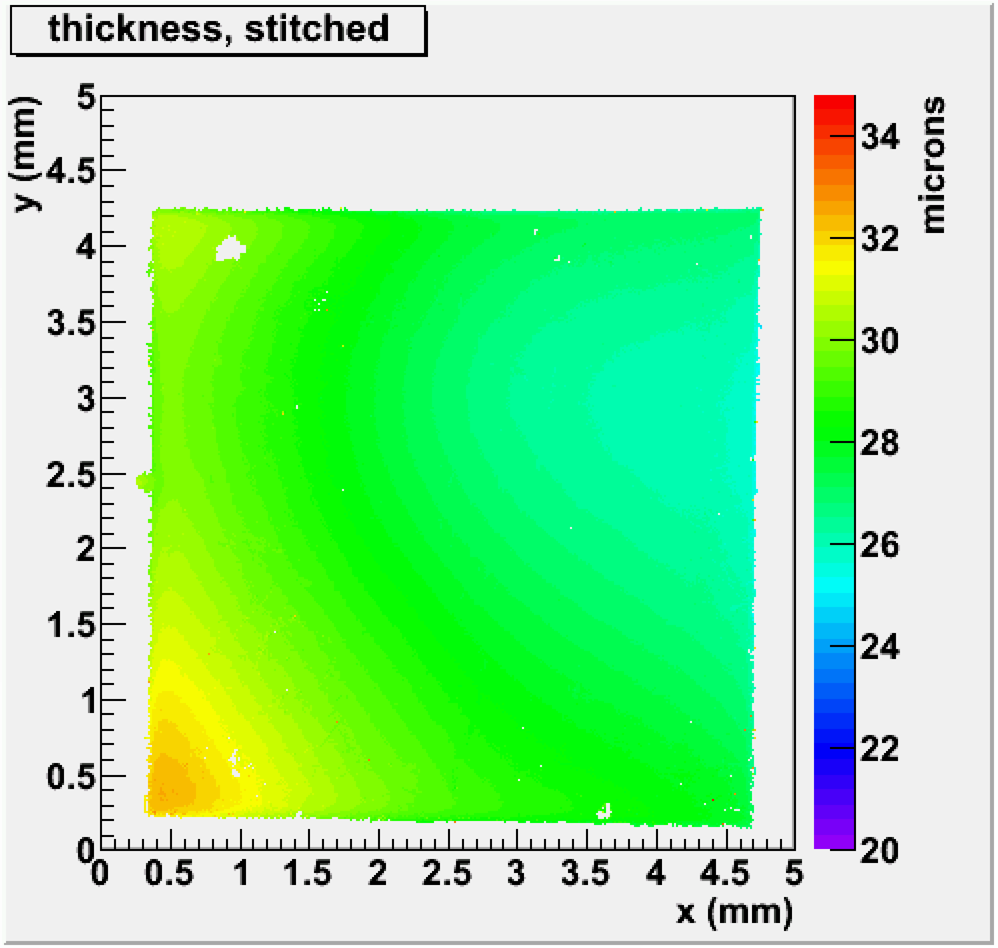


Sample C: 30 microns thickness





surface and thickness profiles (Zygo 3D)



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X-ray diffraction assessment June 2012

- measurements at Cornell High Energy Synchrotron (CHESS)diffraction end-station C
- special monochromator setup and diffractometer configured for these measurements
- thanks to CHESS Staff Scientist Ken Finkelstein

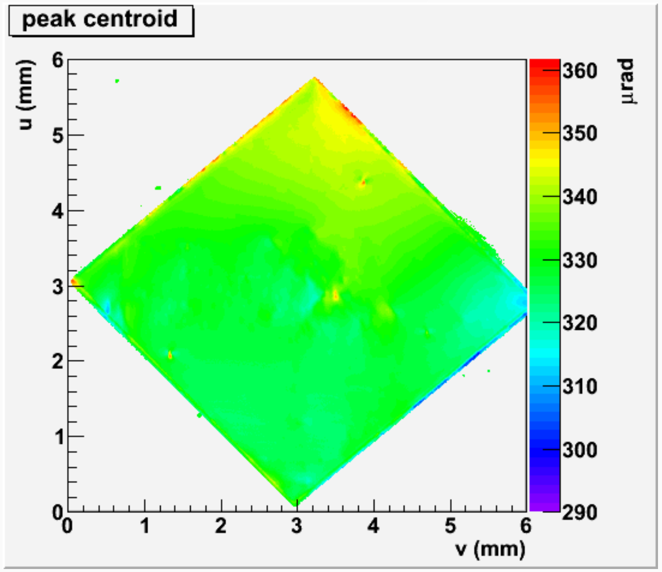
S150 – thick reference standard

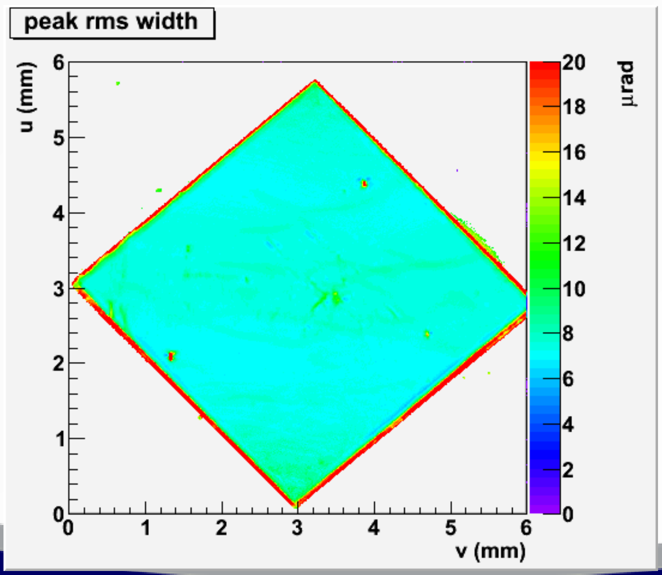
S90 – intermediate reference

S30 - near GlueX requirement

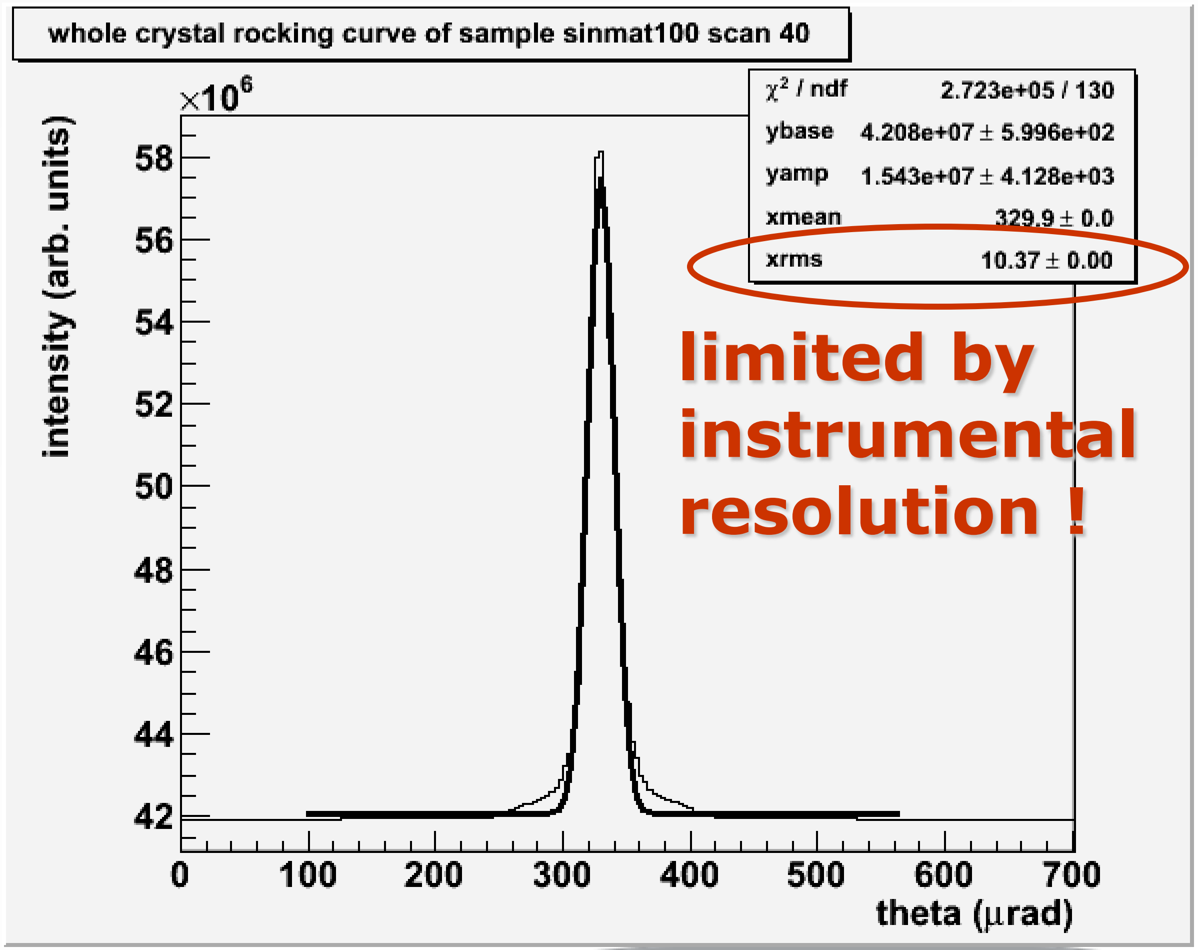
S10 - primary sample of interest

X-ray assessment: S150

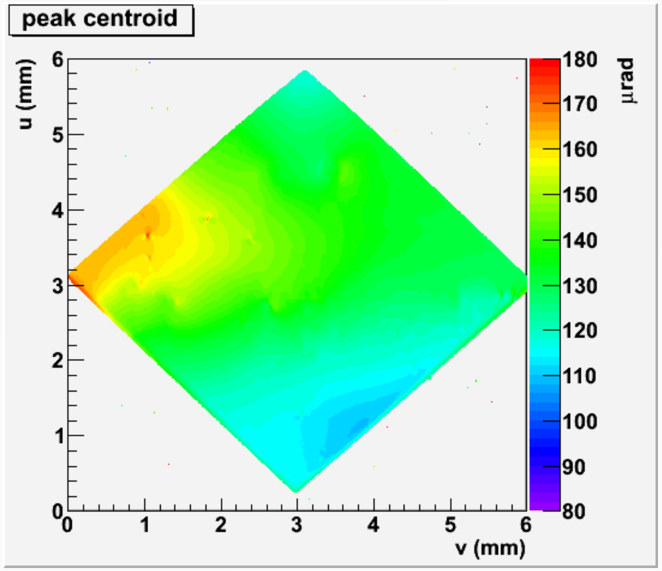


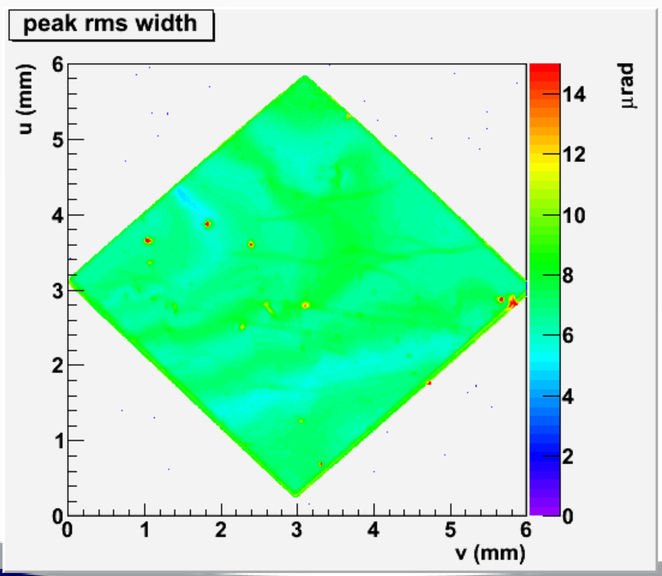


surface of S150 was polished with RCMP process - Sinmat

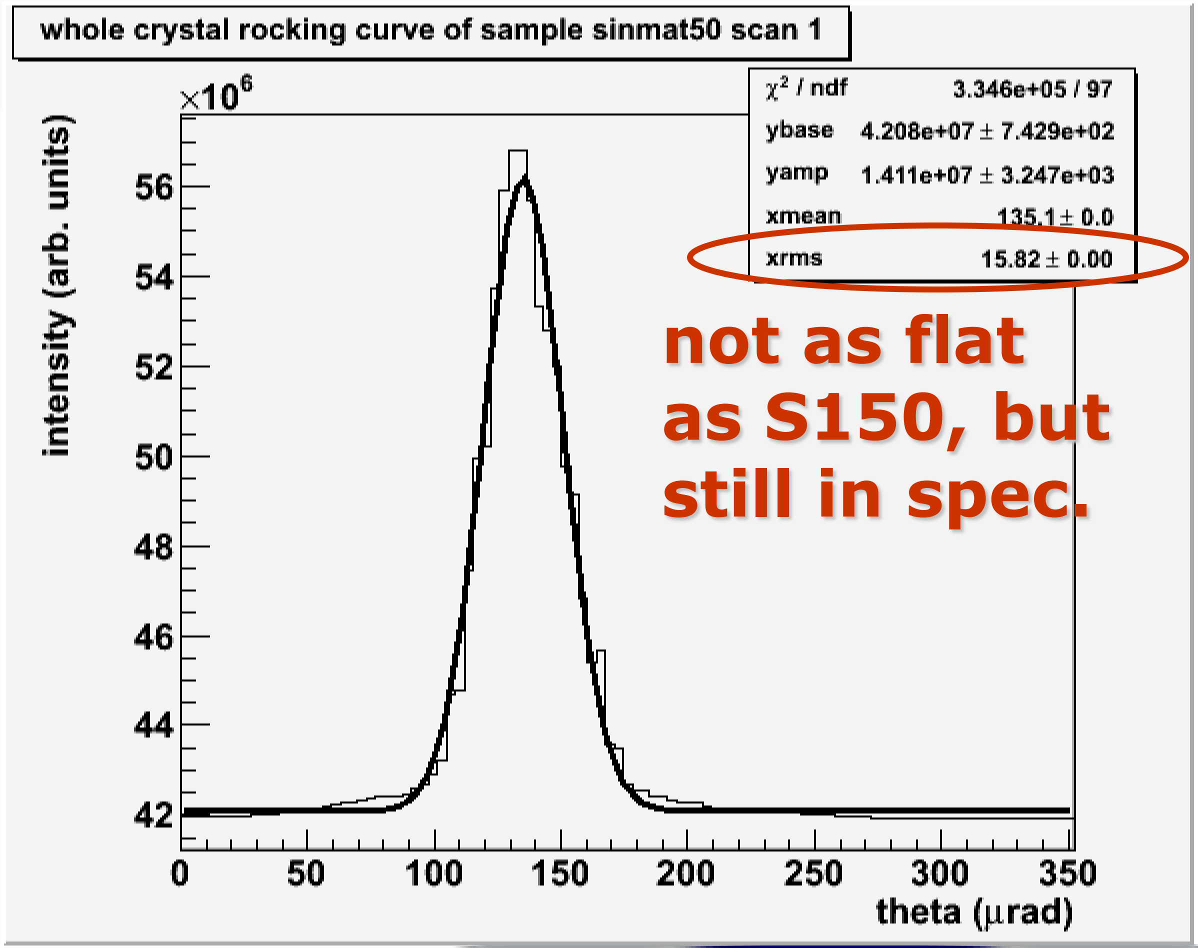


X-ray assessment: S90



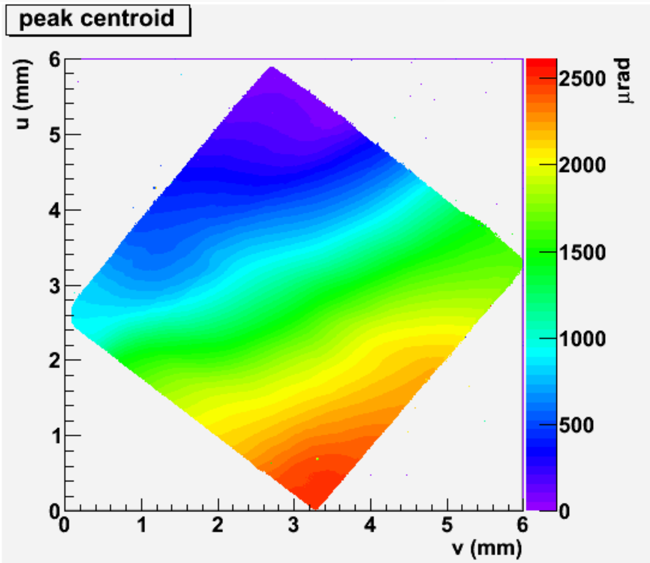


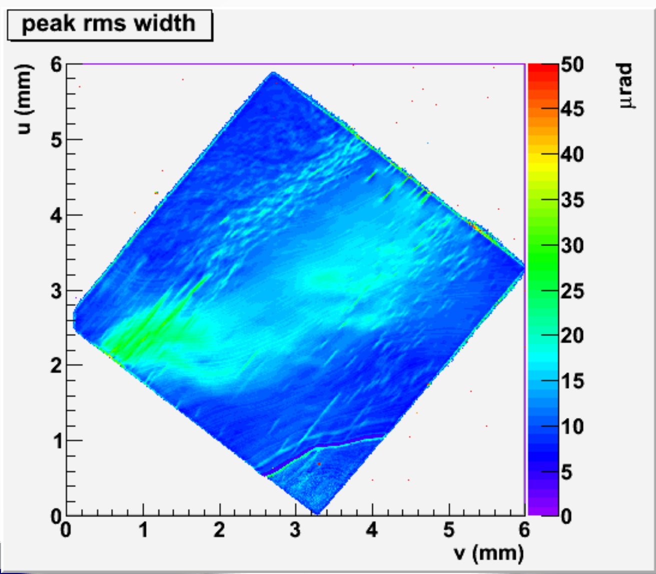
surface of S90 Sinmat



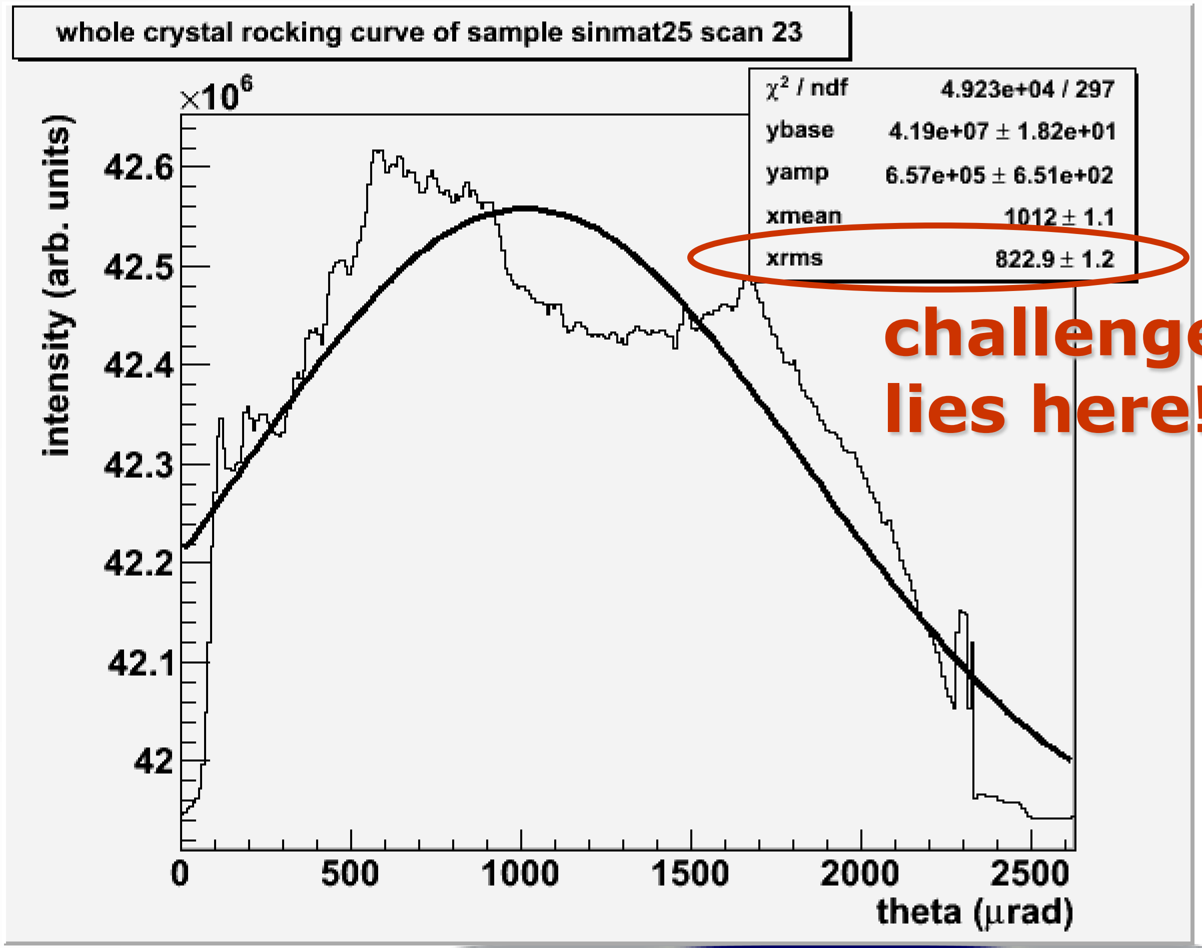
not as flat as S150, but still in spec.

X-ray assessment: S30 – close to GlueX specs



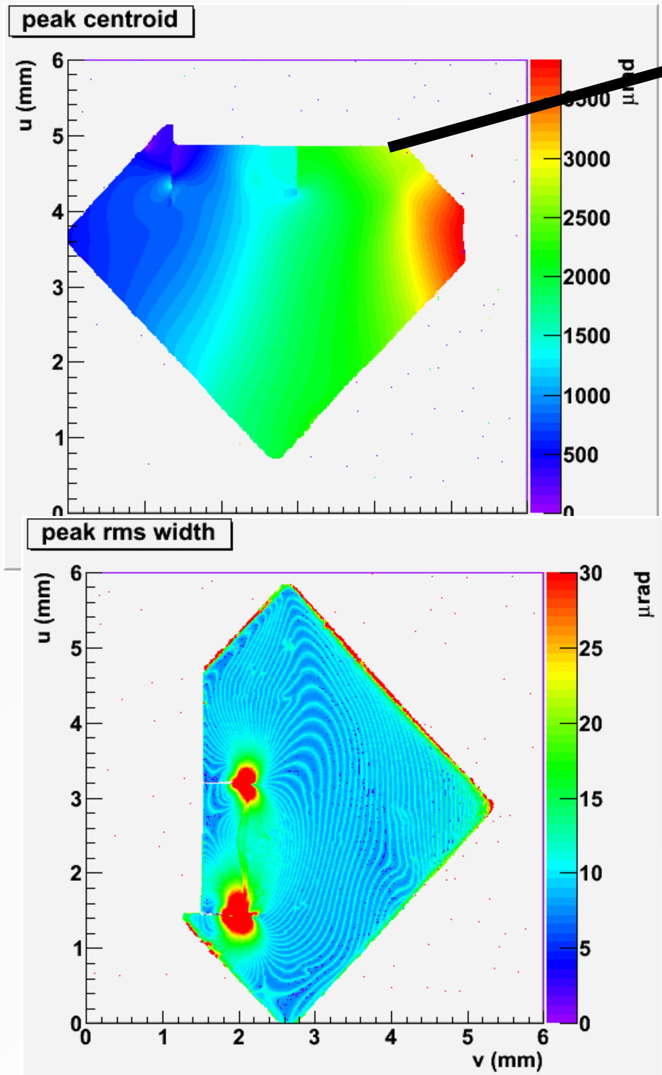


surface of S30 Sinmat

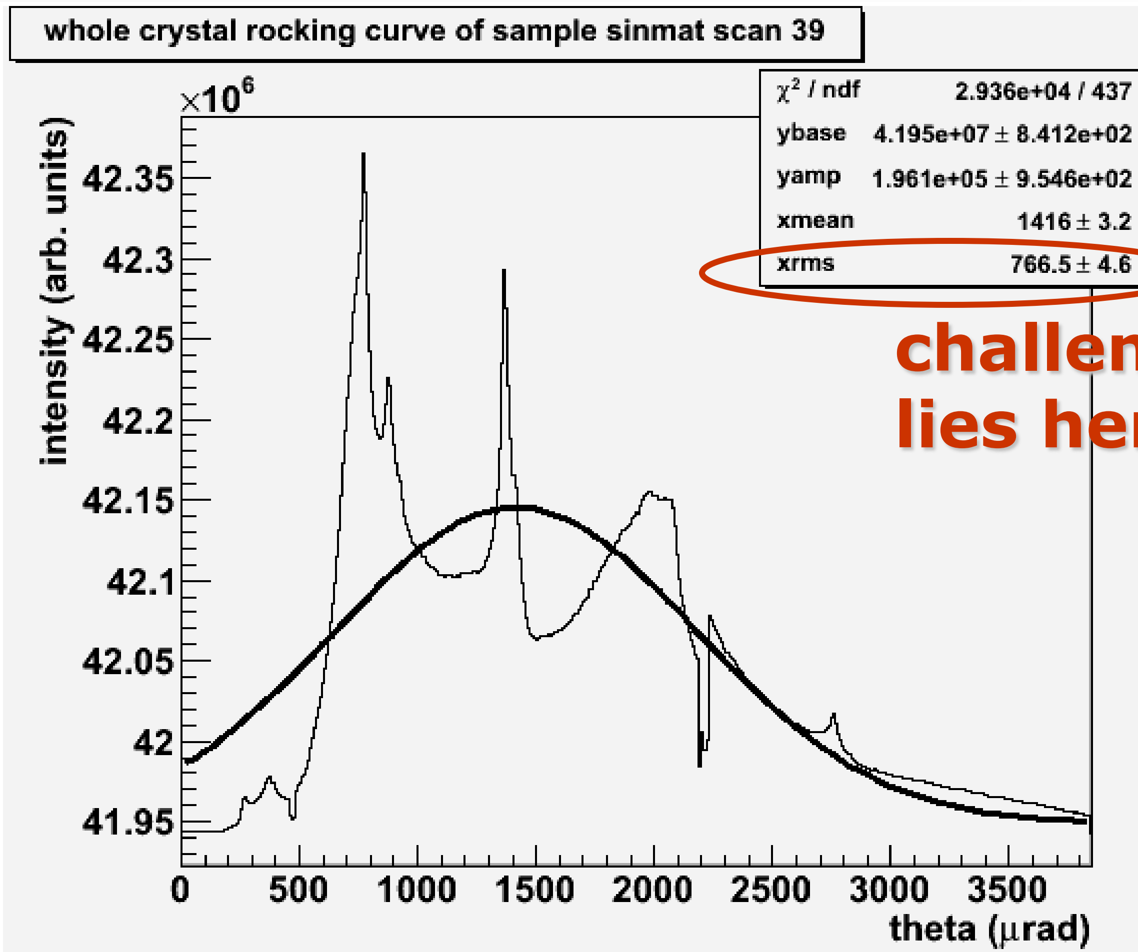


challenge lies here!

X-ray assessment: S10 – Limits of Fragility



Sample broke due to fragility



challenge lies here!

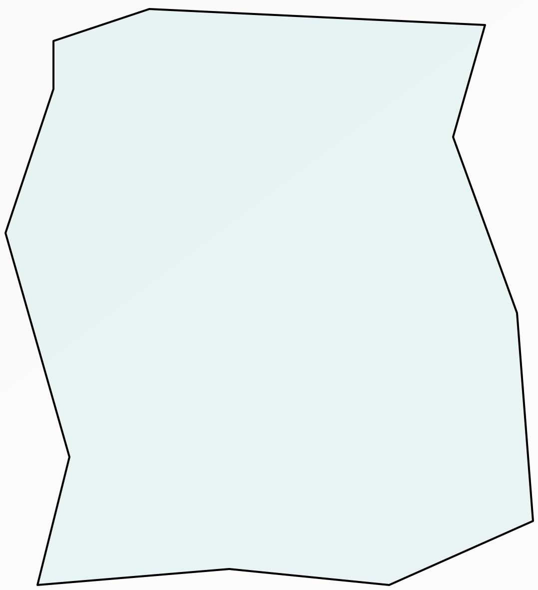
Approach 1 Summary

Crystal Thickness in µm	GlueX Thickness Requirement	GlueX Crystal Quality Requirement
150	X	✓
90	X	✓
30	✓	X
10	✓	X

- ❑ Crystals with 10 micron and 30 micron thickness are not qualified for the GlueX due to the high X-ray RC rms
- ❑ This because the crystal warps when the thickness is reached
- ❑ ***New Approach Needed!***

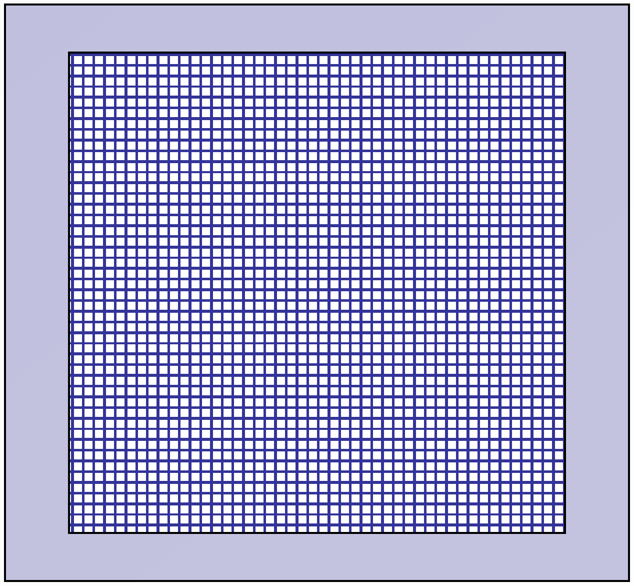
New approach tested in 2012: *add a frame*

diamonds appear to warp severely when thinned to 20 microns



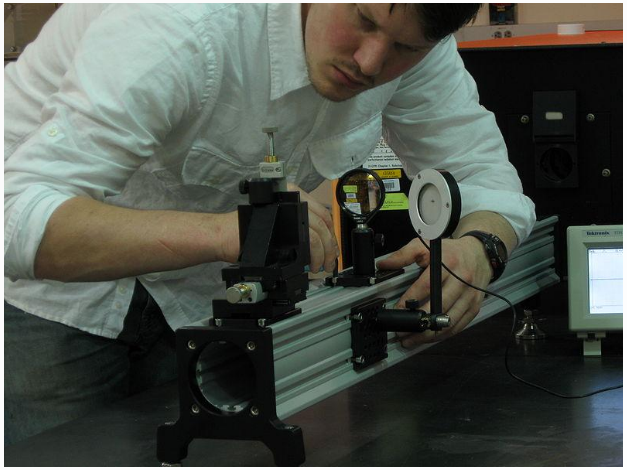
warping is from combination of mounting and internal stresses

try to stiffen the diamond by leaving a thick outer frame around the 20 micron region



frame around 20 micron is still part of the single crystal, maintains planarity

New Approach: 2 techniques explored



- **New approach 2a:**
  - laser ablation (UV-Excimer 193nm)
  - capability developed at U.Conn under STTR Phase I

- **New approach 2b:**
  - RCMP+ vapor phase etching
  - using capabilities at Sinmat & University of Florida

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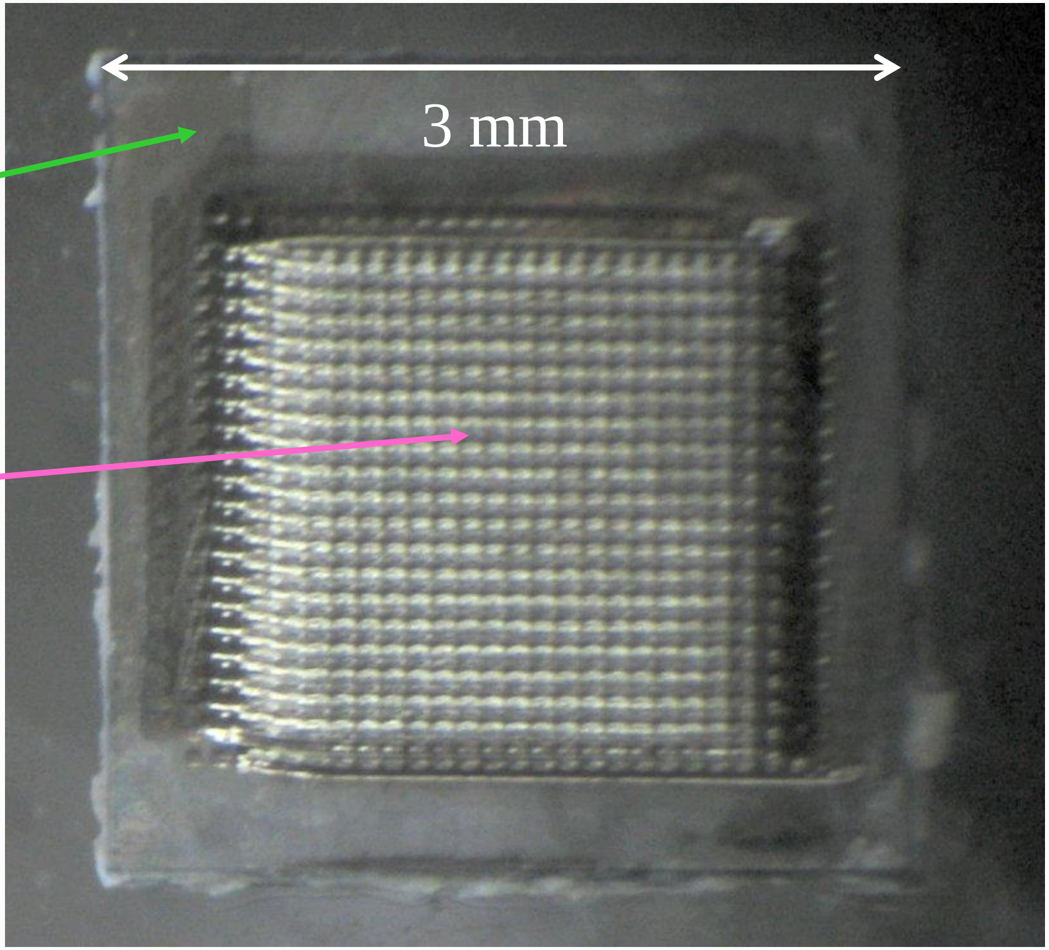
Approach 2a: First ablated sample: U40

300 micron frame around outside edge

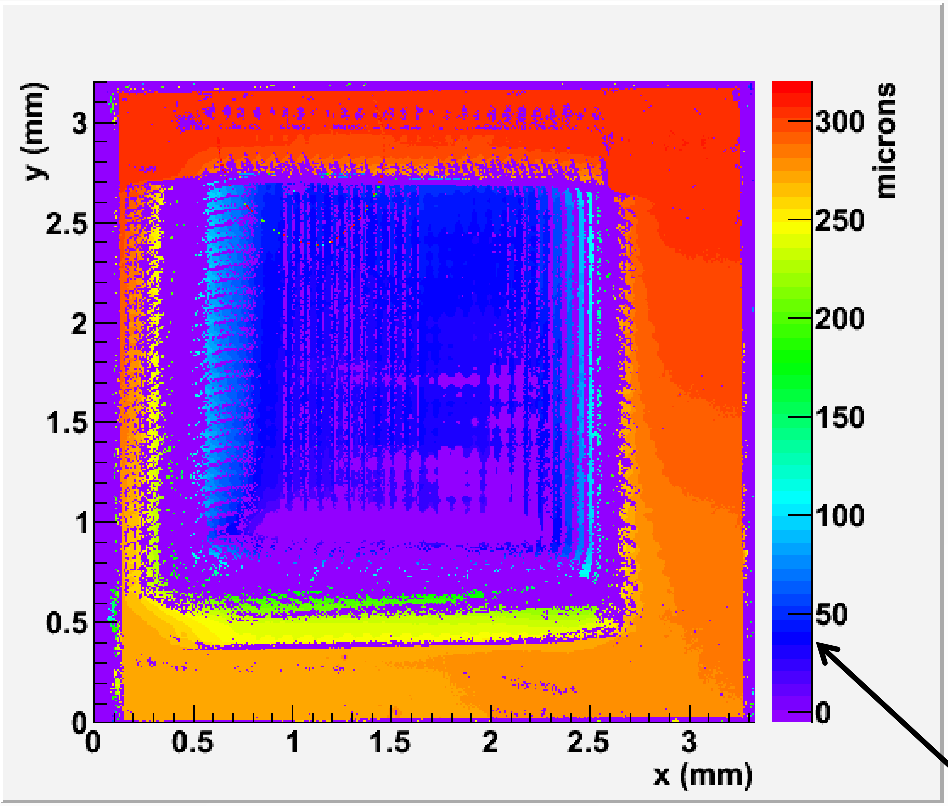
thinned inner rectangular window

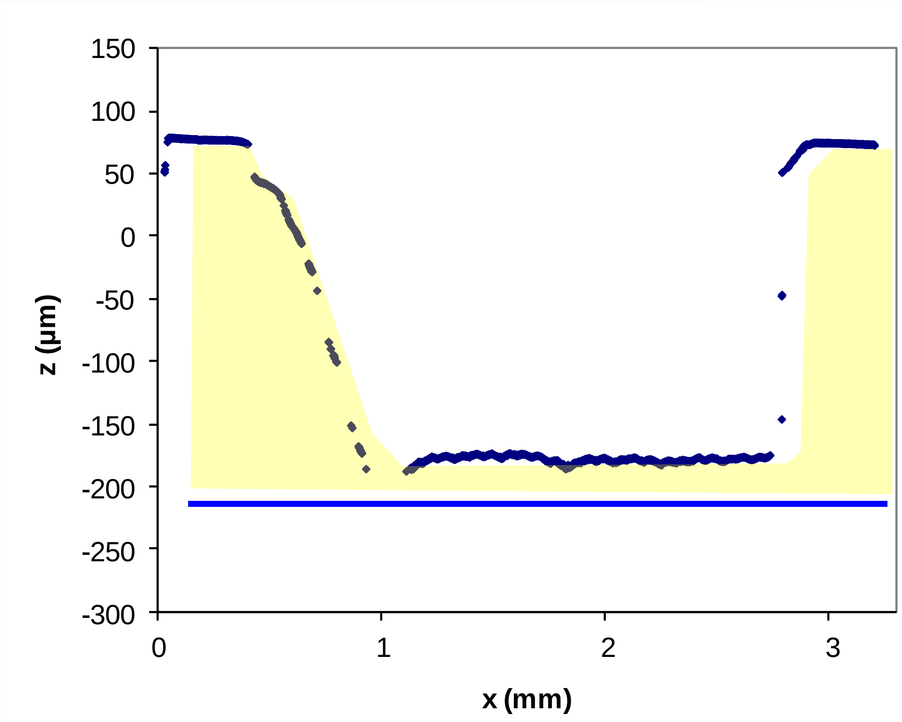
residual raster pattern is from a coarse laser step size



3D Zygo Images of U40

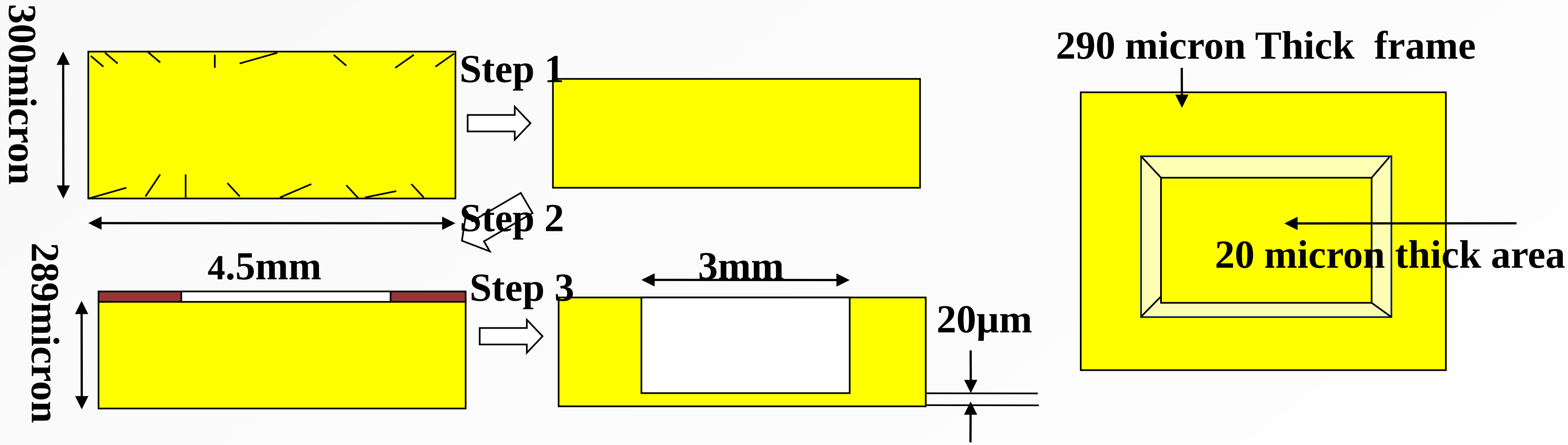
White-light interferometer gives surface and thickness profiles





40 microns

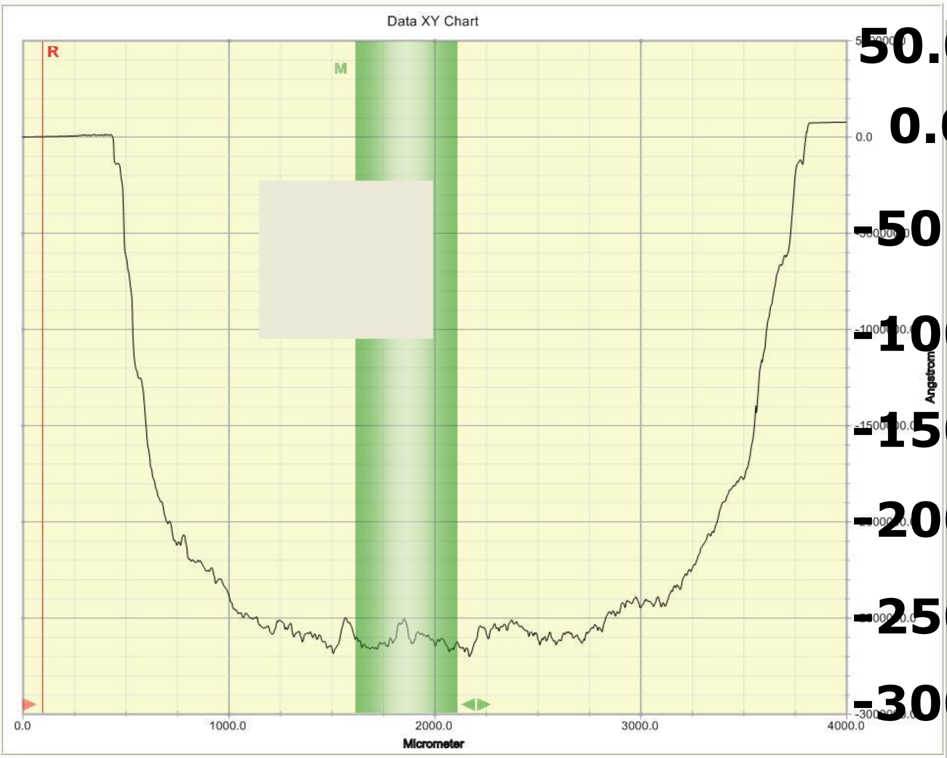
Approach 2b: RCMP + Vapor Phase etch

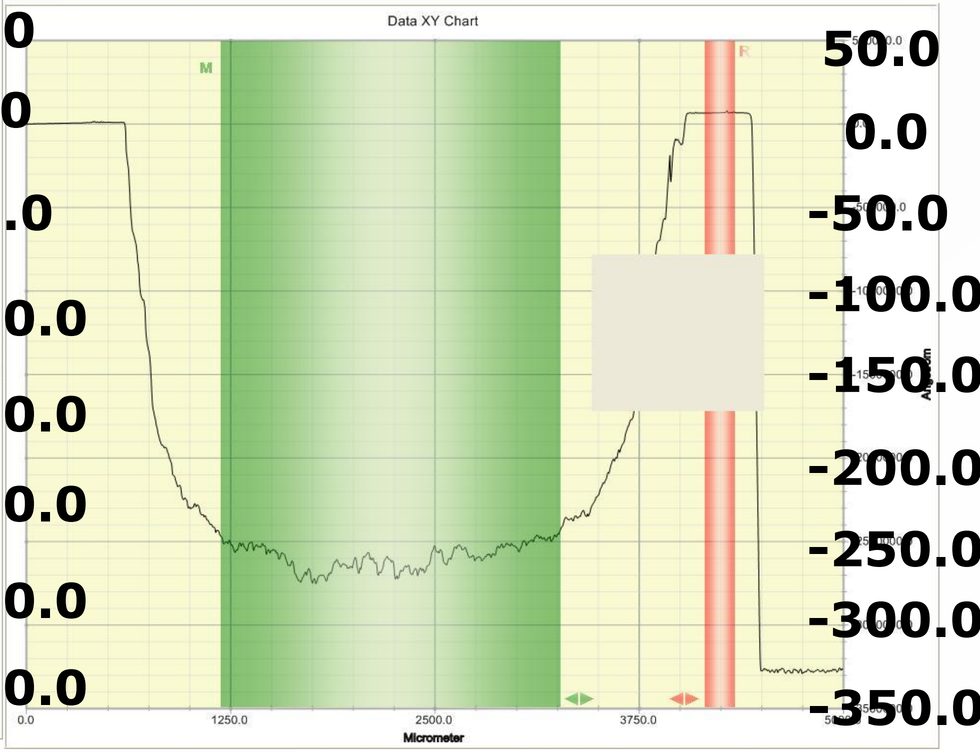


Self- framed diamond crystal radiator fabrication

- Step 1 **RCMP** to eliminate scratches/surface/surface damage
- Step 2 masking the area that will be used as a frame
- Step 3 etching the window area down to 20 microns

Profilometer Data of Sample by Approach 2b

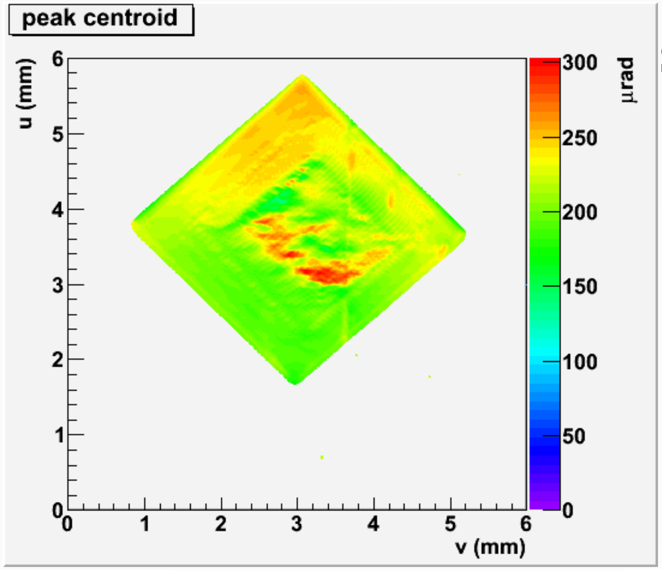


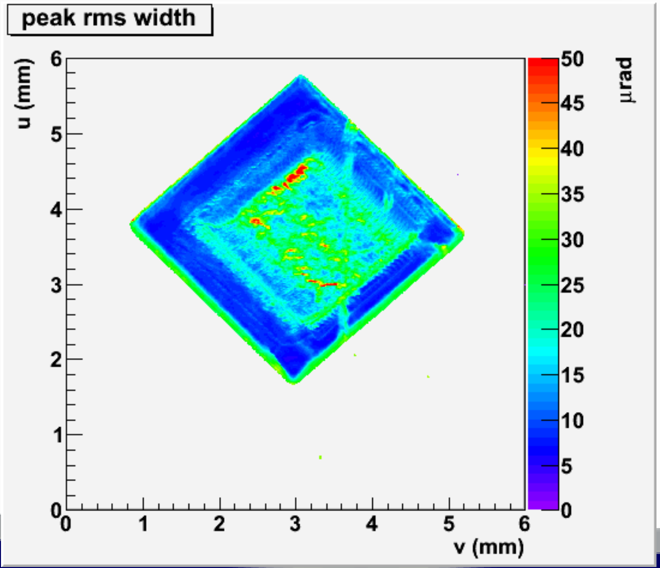


Bottom shows self framed 40 micron radiator fabricated by approach 2

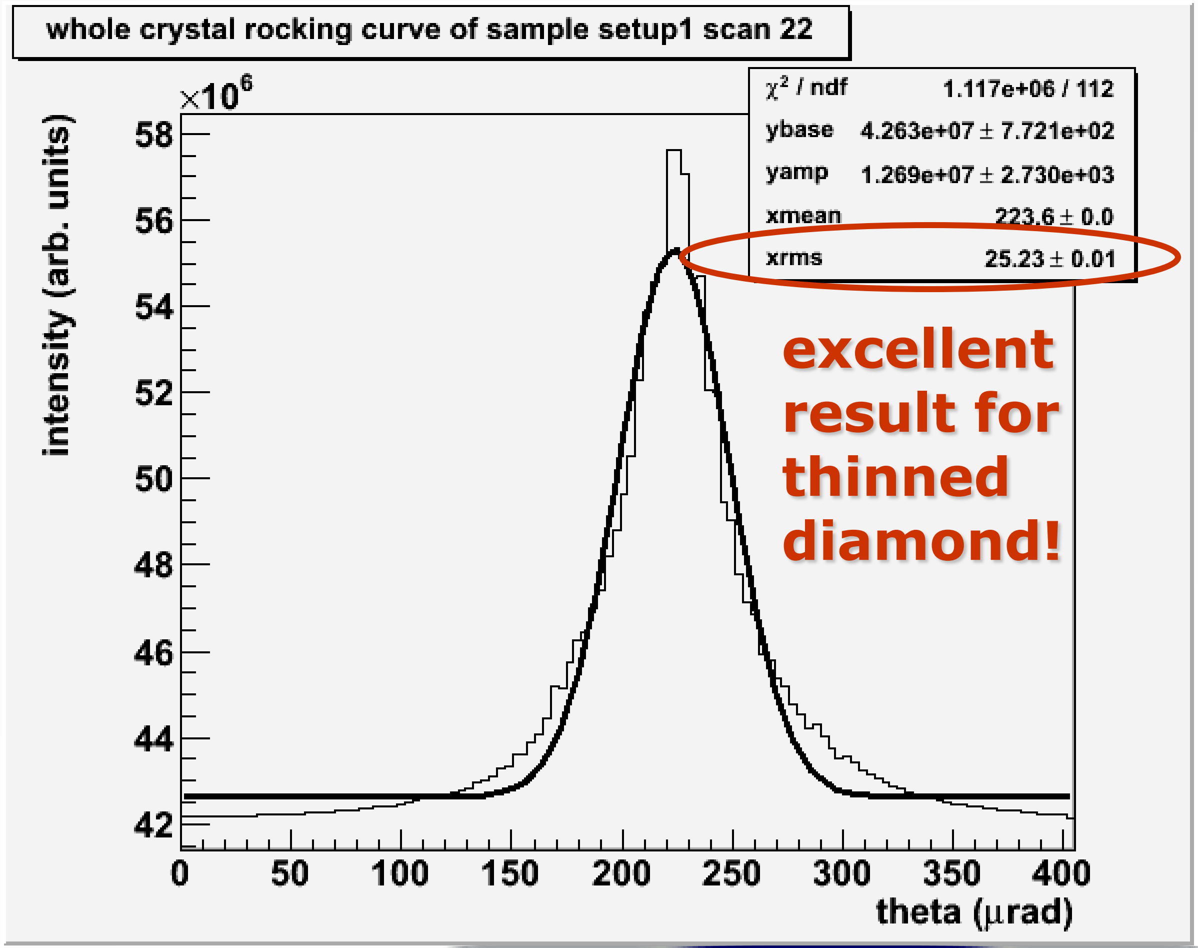
Problems : High roughness & slow etching rate due to mask evaporation and re-deposition on the active area

X-ray rocking curve for U40





surface of U40 was not treated after ablation



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CONCLUSION

Developed a two-step process thin diamond samples

Approach 1

- Step 1 Vapor Phase etching process (75 micron/hr)
- Step 2 RCMP Process
    - No surface topography/features, with AFM rms < 5 A
    - Low FWHM in x-ray rocking curve studies
- Fabricated 10,30, 90,150 micron thin diamond crystals by approach 1
- Thinner the crystal higher the RC RMS due to warpage

Approach 2

Self Framed Crystal Radiator: Feasibility tested

Self framed fabricated Crystal using Laser Ablation at U.Conn met the GlueX target requirements

Future Work

- Achieve flat crystals with thickness 20 microns
- Optimizing the etching/polishing parameters
- Combining Sinmat/U.Conn process
- Multiple crystal fabrication

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