

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

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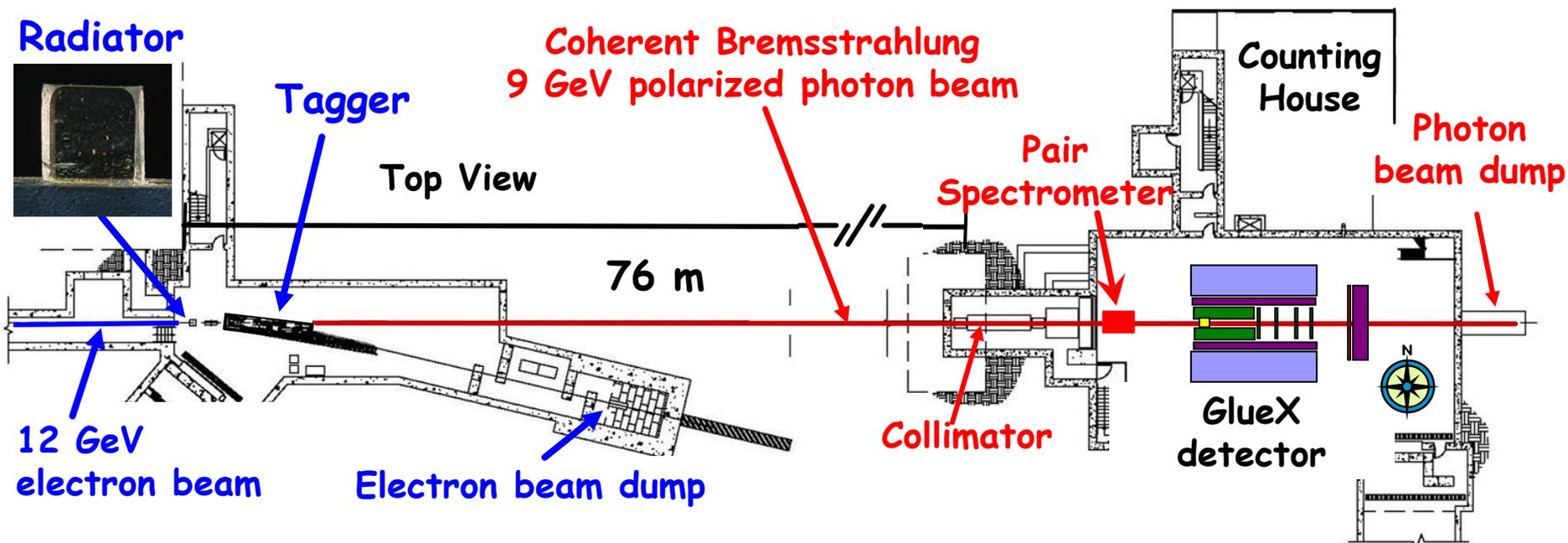
# Outline

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- **Application: high-energy polarized photon source**
  - ❑ uniqueness of diamond as a radiator
  - ❑ competing specifications: thickness vs. flatness
  - ❑ proposed solution: a thick frame around the radiator
- **Two approaches investigated**
  - ❑ vapor phase ion etching with mask (Sinmat)
  - ❑ milling by UV laser ablation (UConn)
- **Conclusions and Future Directions**



# Diamond - a high-energy polarized photon source



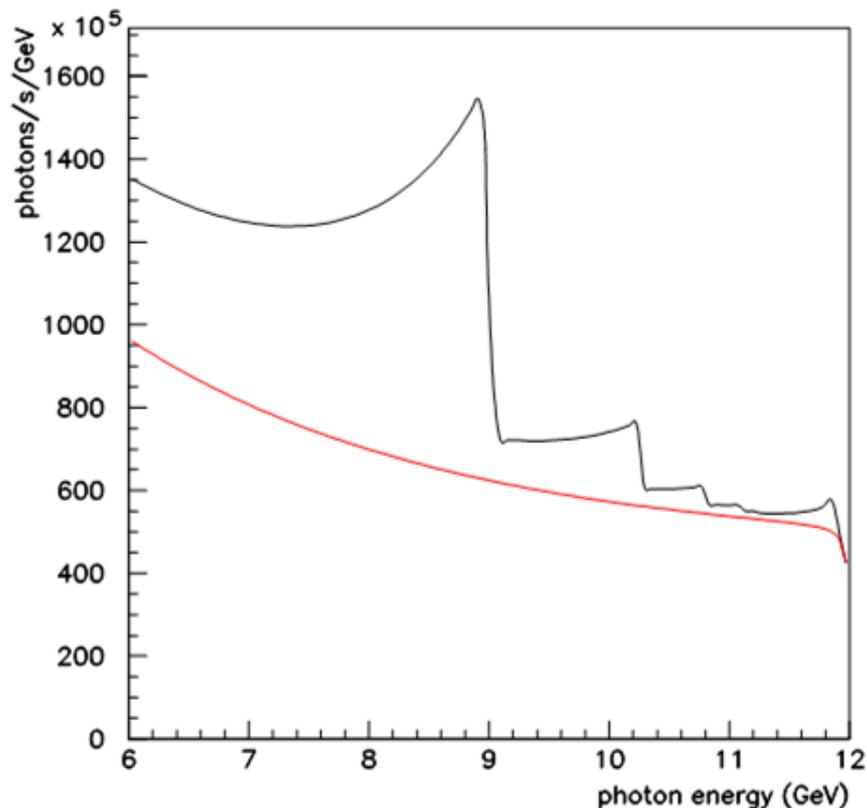
Crystalline radiator => electrons “bremsstrahlung” from entire planes of atoms at a time

1. discrete peaks in energy spectrum
2. photons are polarized within the peaks

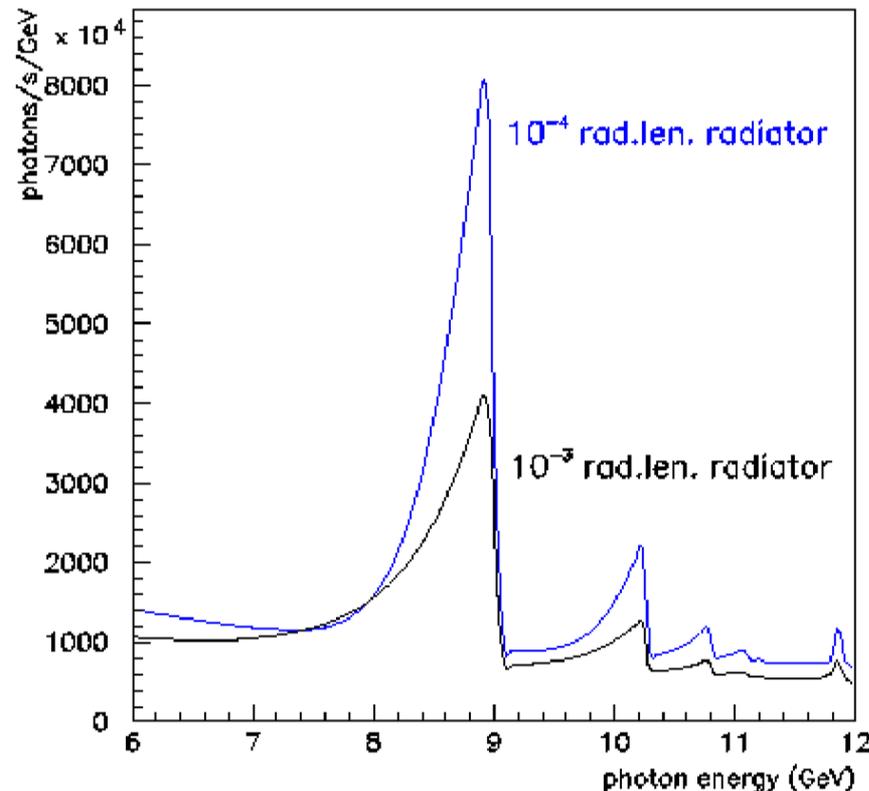
**Diamond is the unique choice for crystal radiator**

1. low atomic number
2. dense atomic packing
3. high thermal conductivity
4. radiation hard, mechanically robust, large-area monocrystals, ...

# Coherent bremsstrahlung beam properties



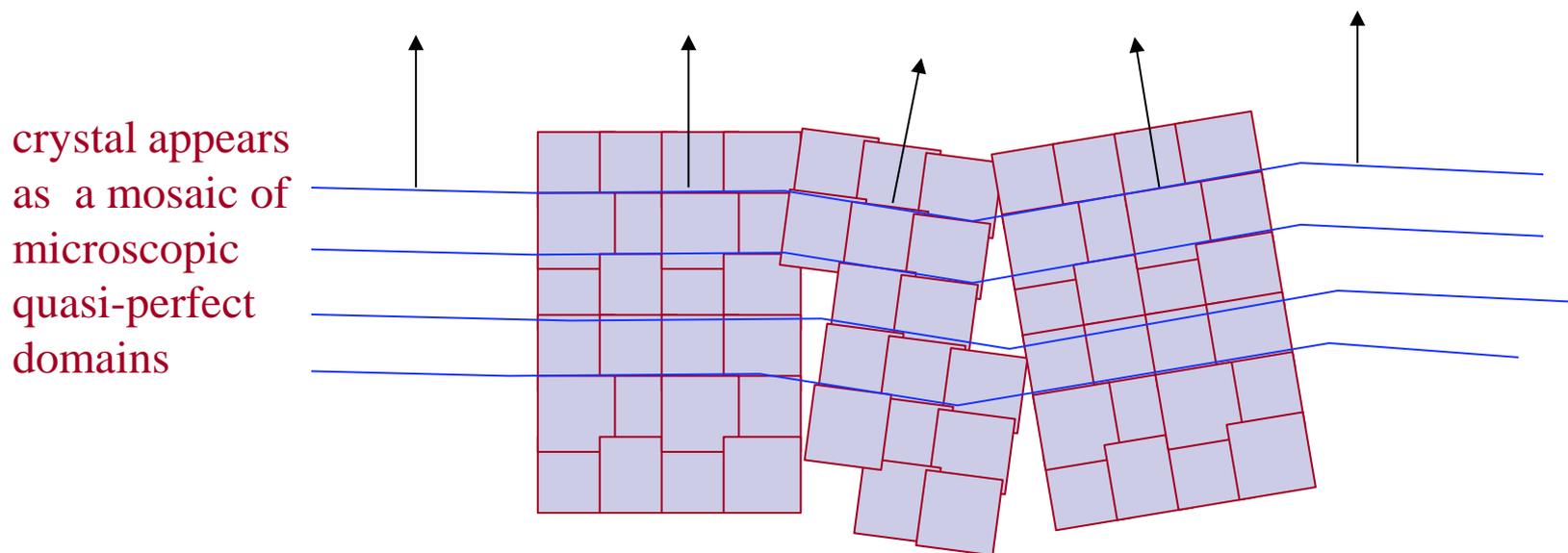
**Bremsstrahlung spectrum with (black) and without (red) an oriented diamond crystal radiator**



**Same spectrum, after cleanup using small-angle collimation**

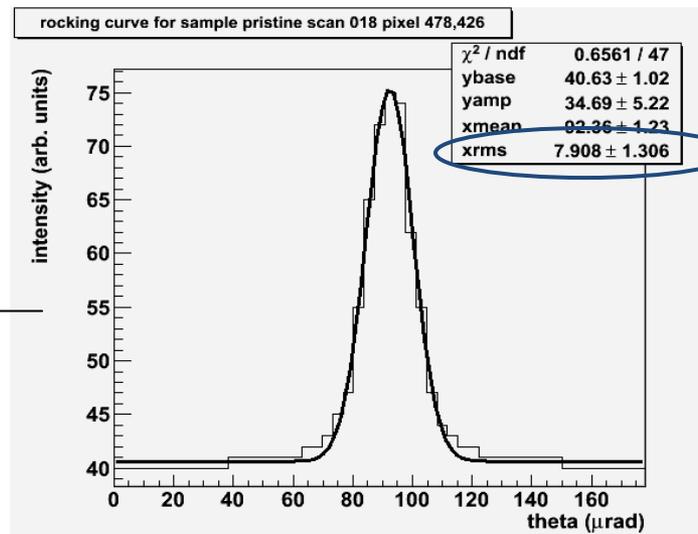
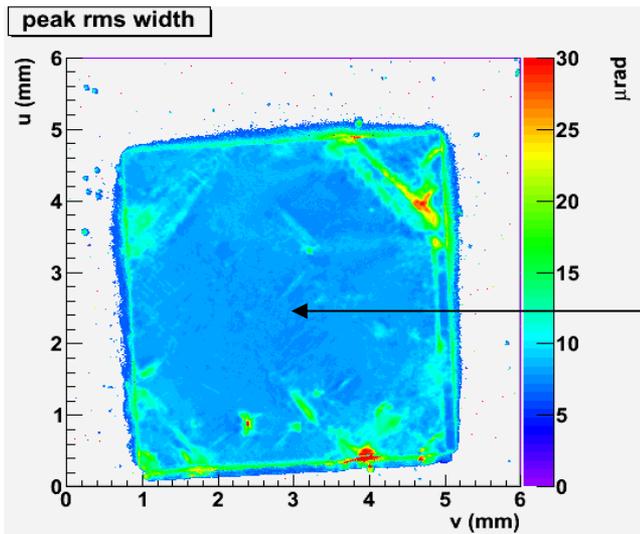
# Diamond radiator requirements

1. thickness  $10^{-4}$  radiation lengths  $\sim 20$  microns
2. “mosaic spread” of the crystal planes  $\sim 20 \mu\text{rad RMS}$



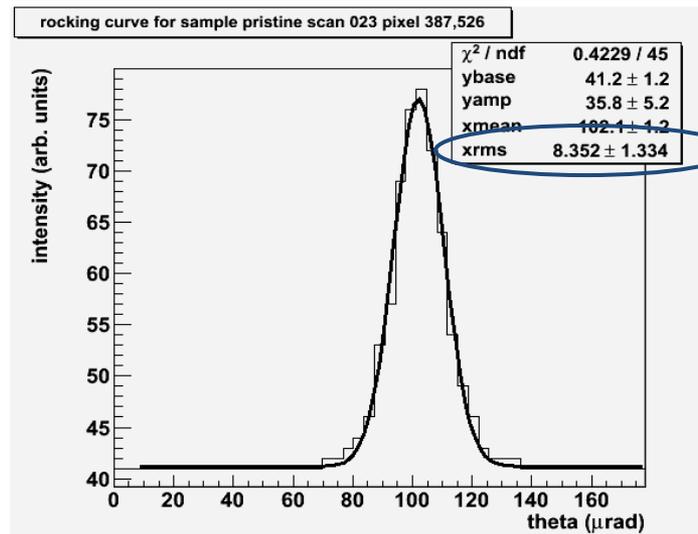
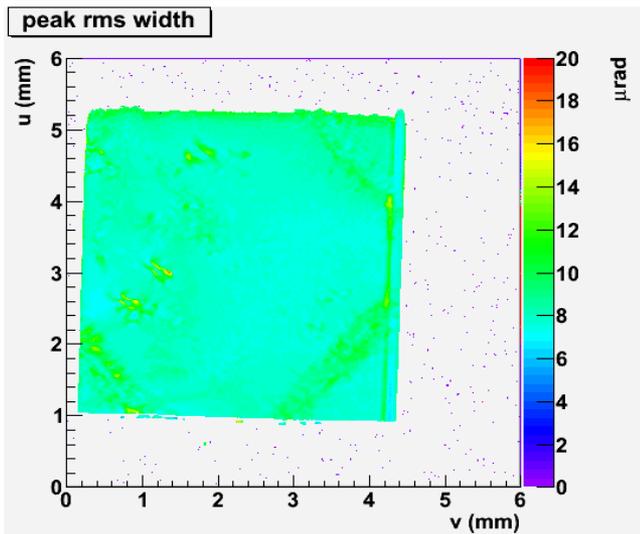
- ❑ Actually includes other kinds of effects
  - distributed strain
  - plastic deformation
- ❑ Measured directly by X-ray diffraction: “rocking curves”

# E6 single-crystal CVD(!) diamond



Very close to theoretical rocking curve RMS width for diamond !

but...



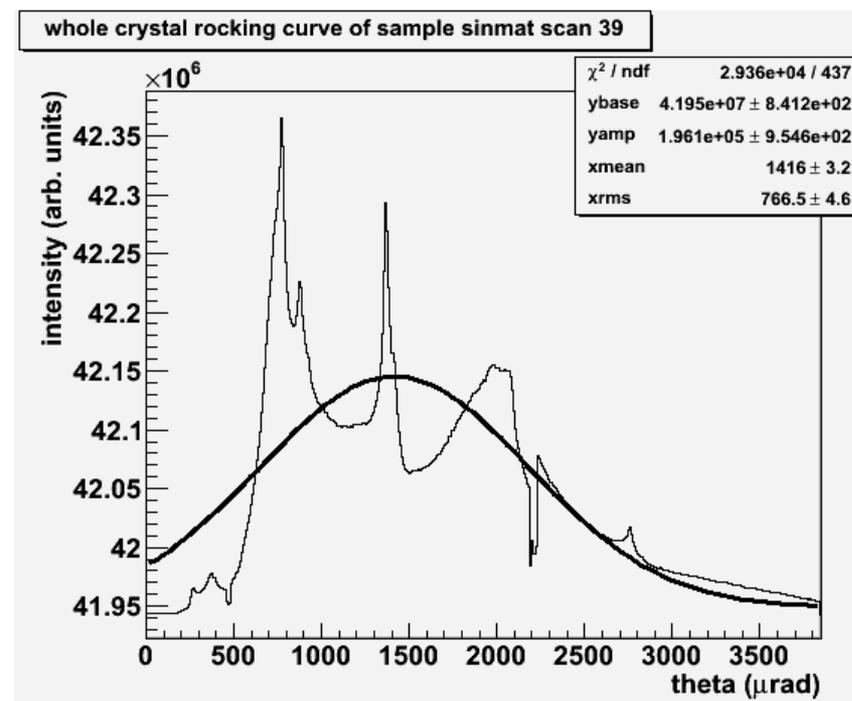
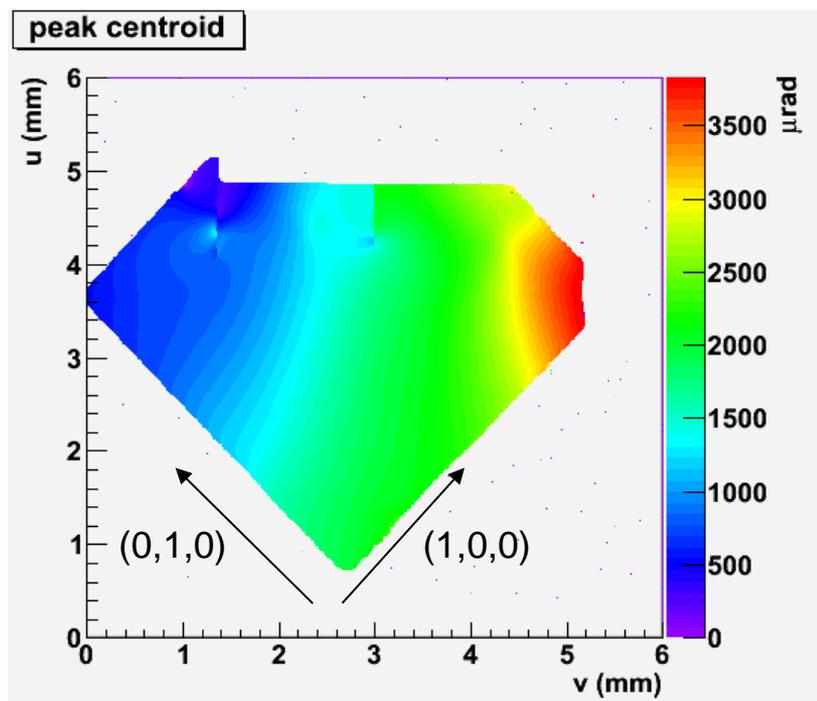
*These crystals are 300 microns thick.*

X-ray measurements performed at Cornell High Energy Synchrotron Source (CHESS)

# E6 CVD diamond thinned to 15 microns

Sample was thinned using proprietary Sinmat RCMP process (presented in early talk)

- very fragile -- notice the corner broken off
- rocking curve shows very large bending deformation



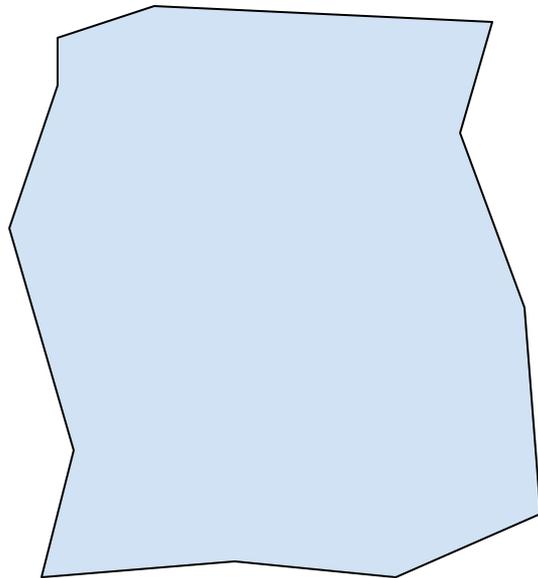
X-ray measurements performed at Cornell High Energy Synchrotron Source (CHESS) - *STTR phase 1*

# Primary R&D challenge in Phase 2

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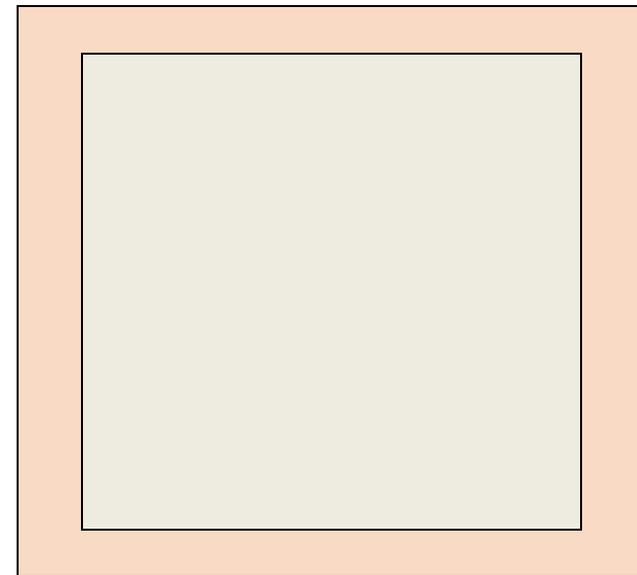
Understand and overcome the thin diamond warping problem.

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 window is still part of the single crystal, acts like a drum head.**

# Two-prong method of attack

## 1. Chemical etching using a mask - Sinmat

- Step 1: Deposit a metallic mask covering the outer frame region.
- Step 2: Etch masked sample using oxygen VPIE.
  - Monitor removal rates, expect >50 microns/hr
  - Watch when mask sputters away, when gone return to step 1.
- Step 3: Measure central thickness, remove residual mask when done.

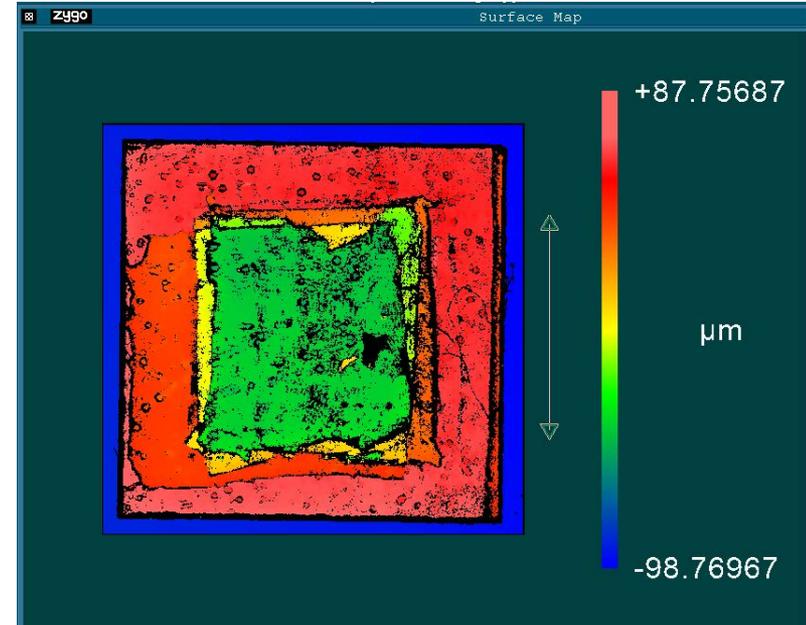
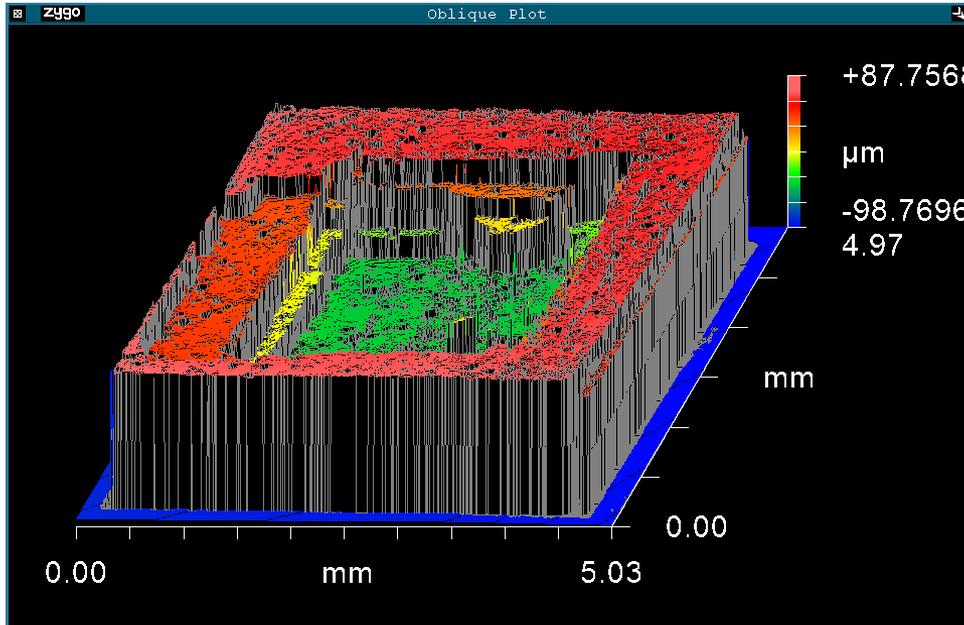


## 2. Precision milling with a UV laser - UConn

- New ablation facility built at UConn for this project
  - 5W pulsed excimer laser generates 5W at 193 nm
  - UV optics to expand beam, focus to 0.1mm spot
  - evacuated ablation chamber with tilted sample holder
  - 3D motion controls to raster the diamond across the beam
- Software developed to generate smooth flat ablated surface



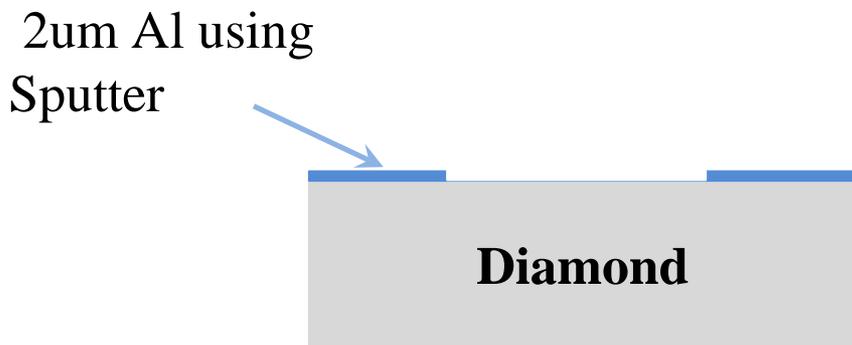
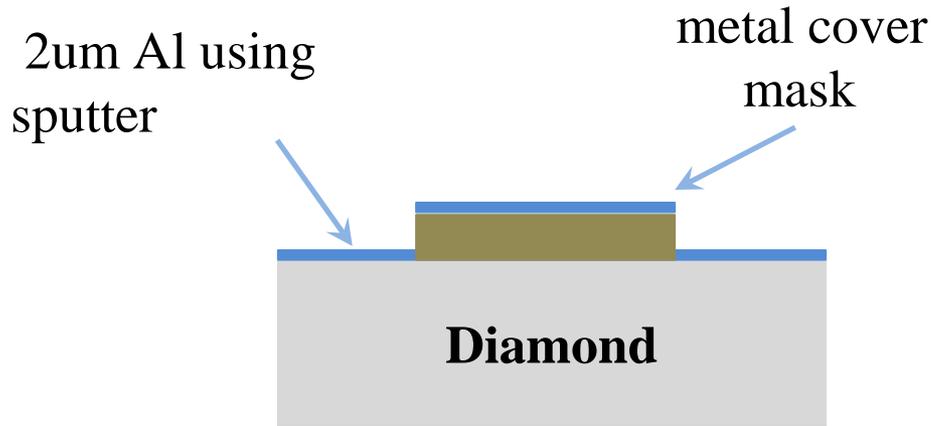
# APPROACH 1: Chemical etching



- Slight misalignment shows 4 masks were needed.
- Frame thickness is 185 microns
- Central region is 55 microns thick
- Etched surface shows significant roughness, pits 10 microns deep

*Careful monitoring needed to prevent burn-through below 50  $\mu\text{m}$*

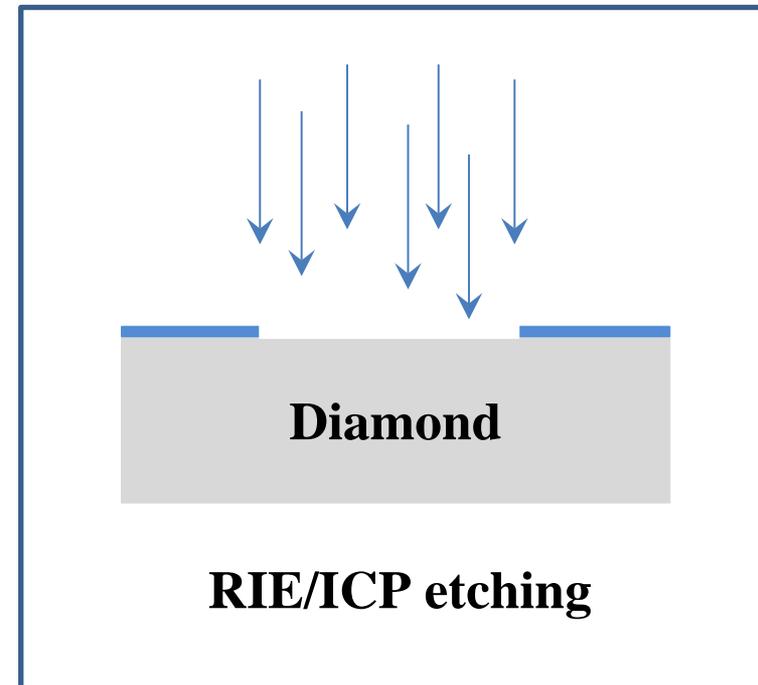
# Vapor Phase Etch Process



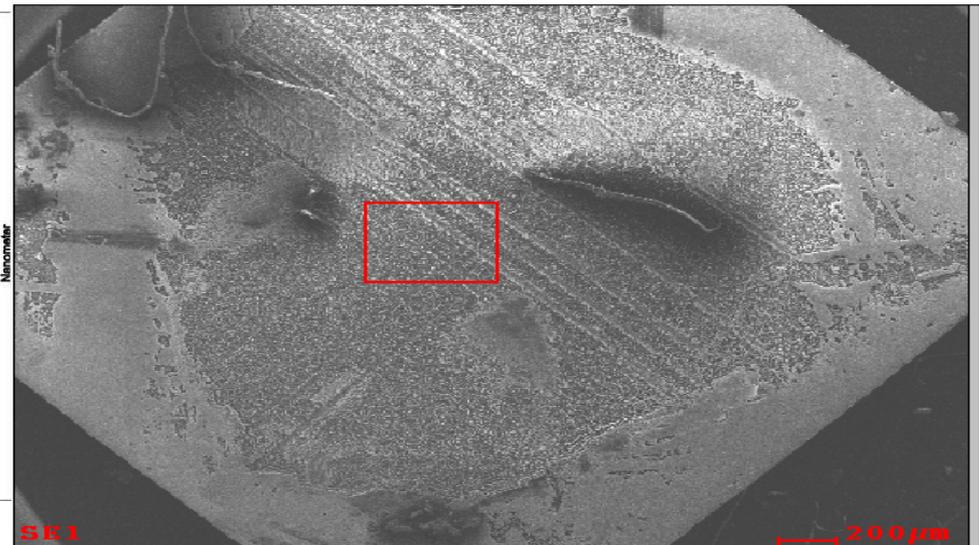
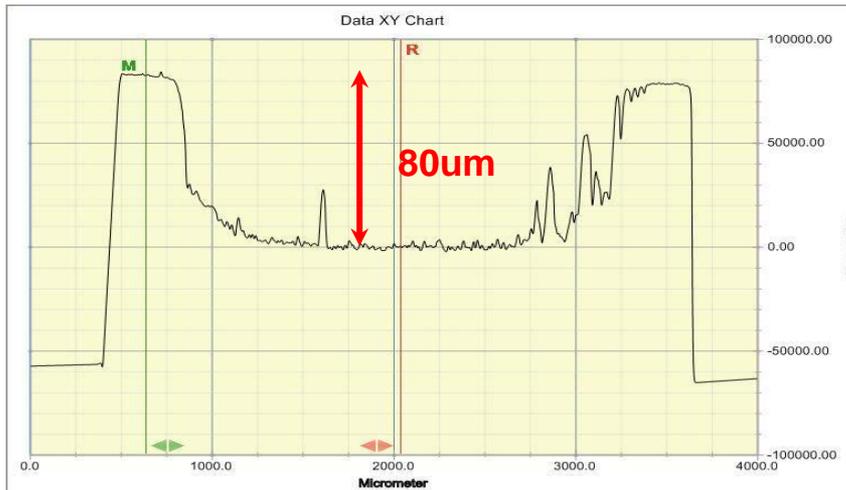
## ➤ Diamond etching recipe

Gases O<sub>2</sub> & Ar gas

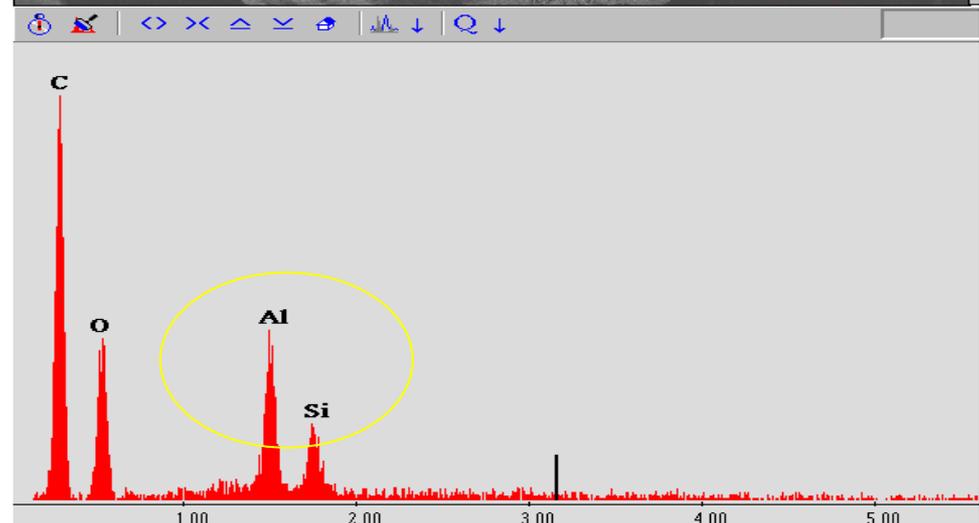
RIE/ICP= 500W / 1500W



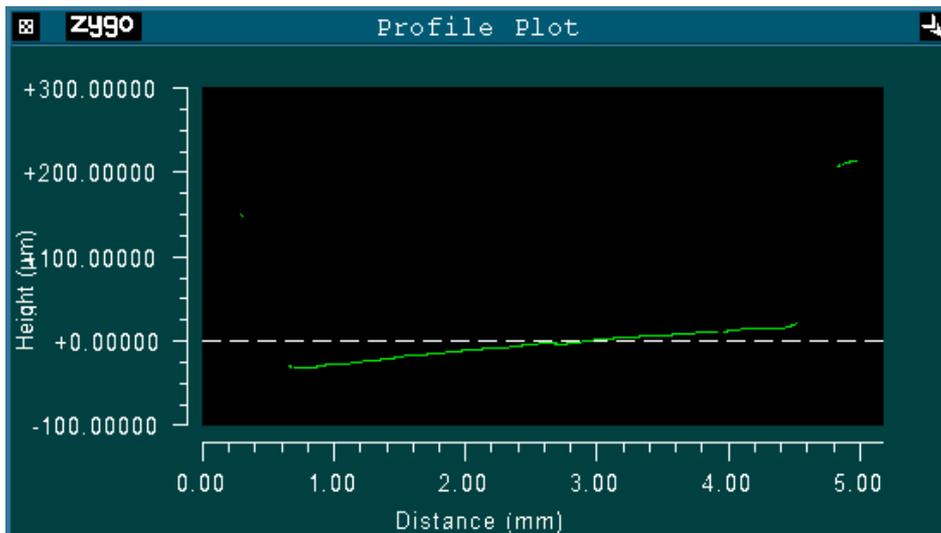
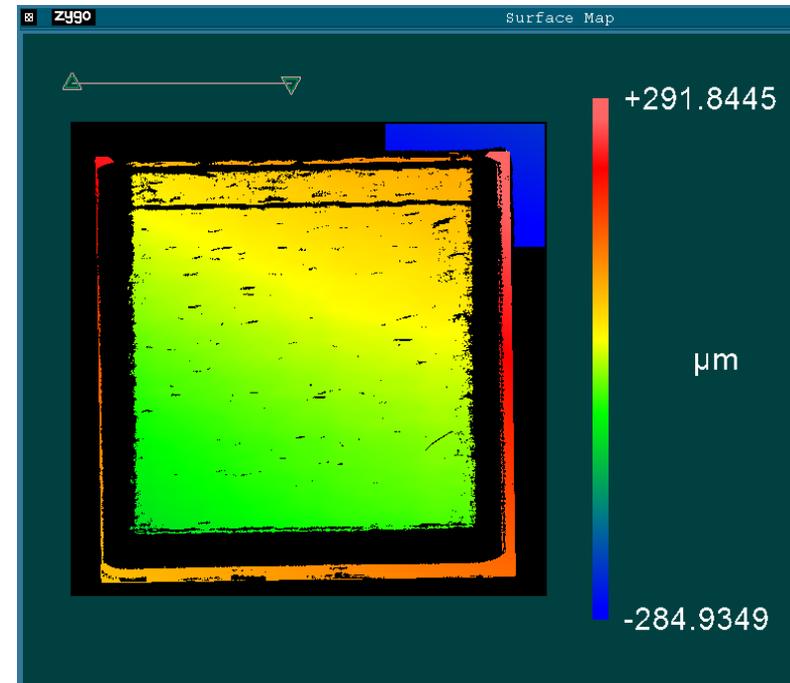
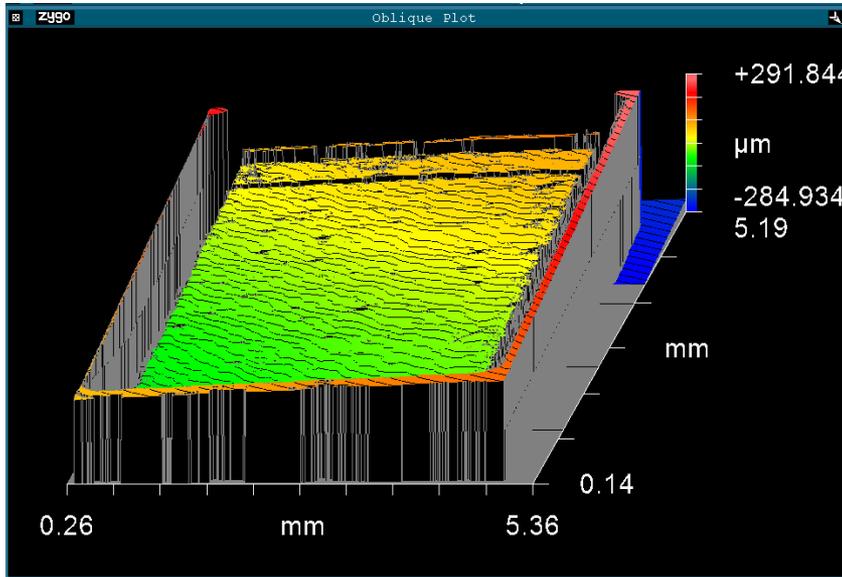
# Vapor Phase Etch Process



- ✓ Etch rate continuously reduced with progressive etching
- ✓ Al mask re-sputtered on the sample



# APPROACH 2: Laser ablation

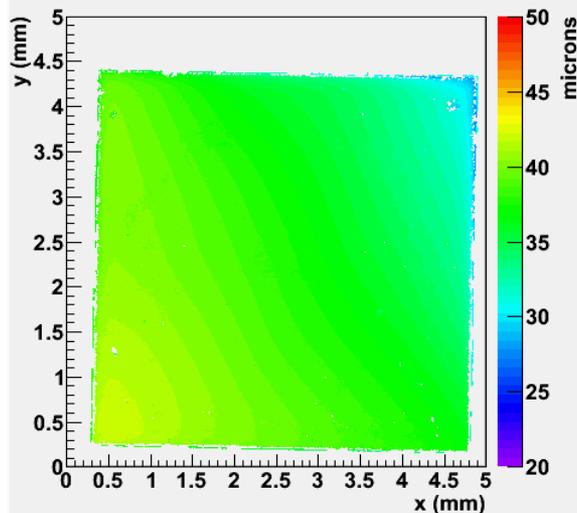


- 200 microns removed in 8hr
- surface roughness < 1 µm
- ***risk of burn-through below 50 µm thickness***

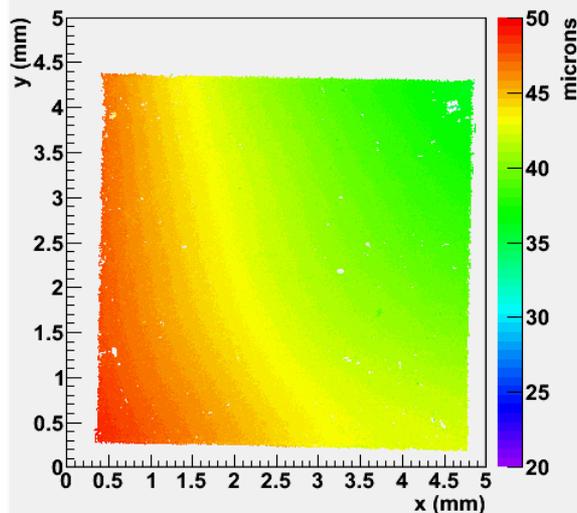
# Uniform sample S90

surface and thickness profiles (Zygo 3D)

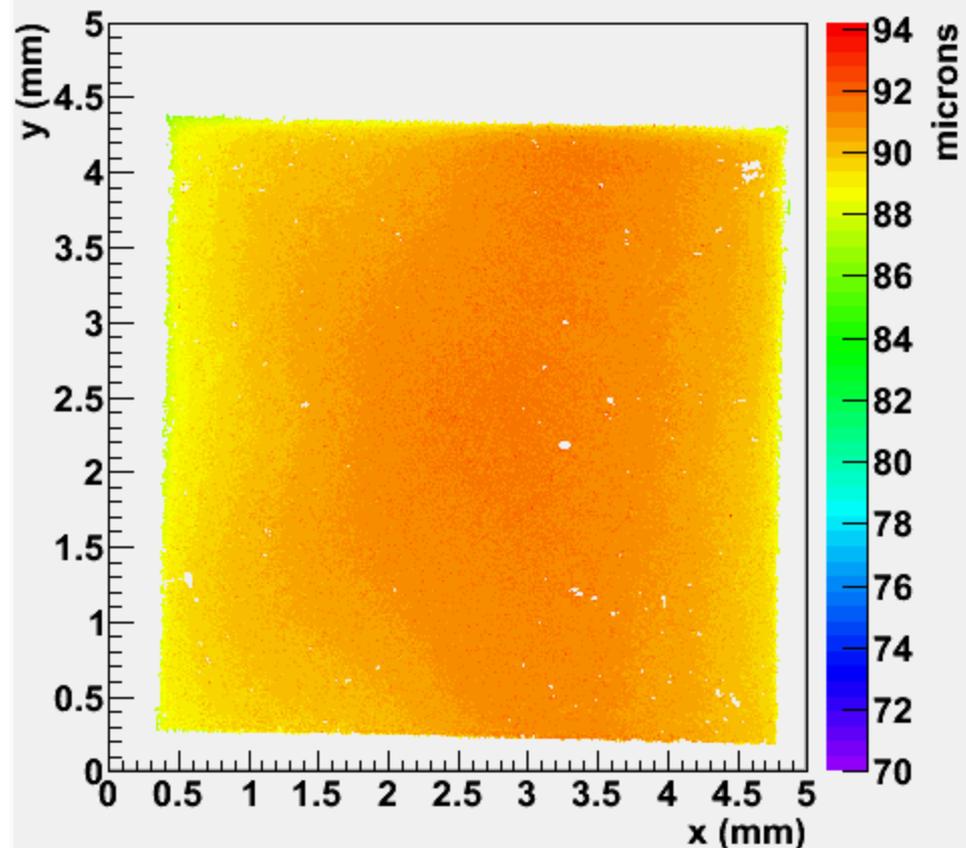
upper surface, stitched



corrected lower surface, stitched

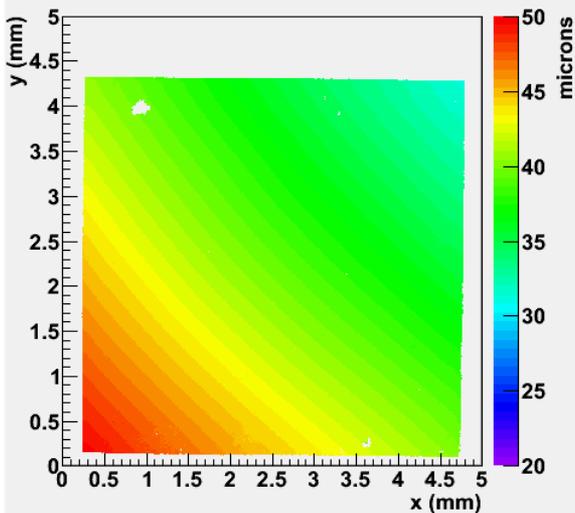


thickness, stitched

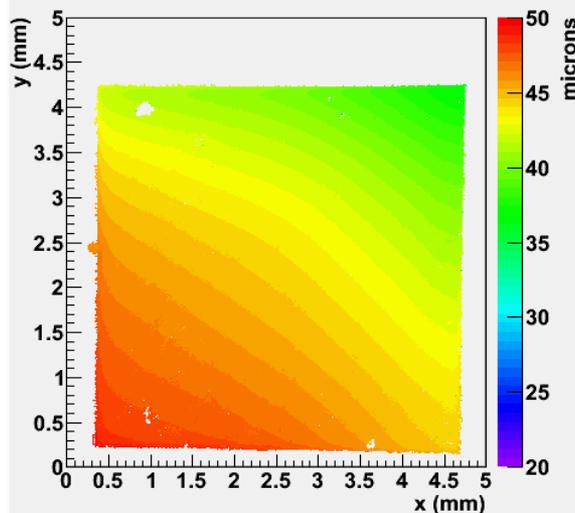


# Uniform sample S30

upper surface, stitched

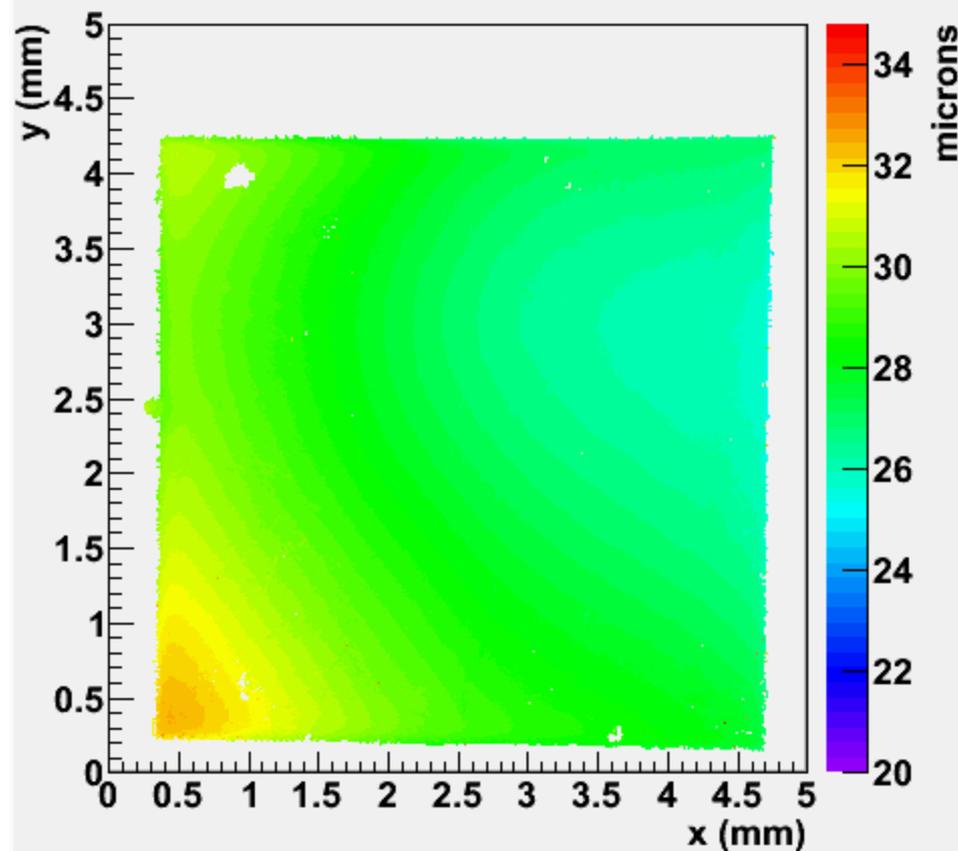


corrected lower surface, stitched

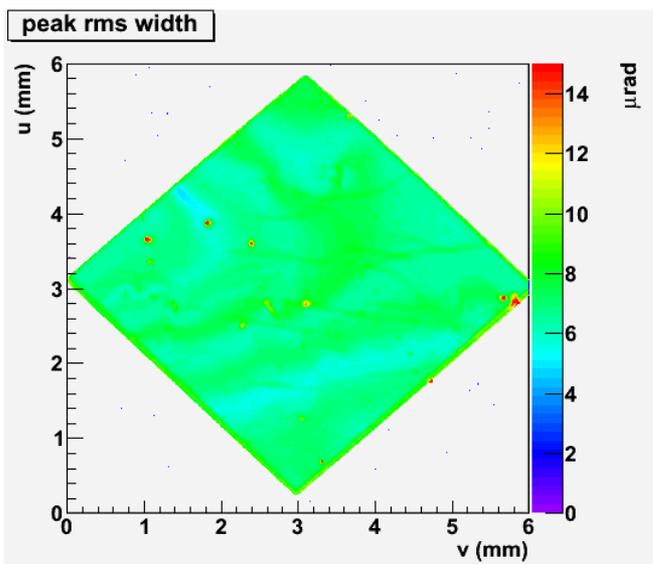
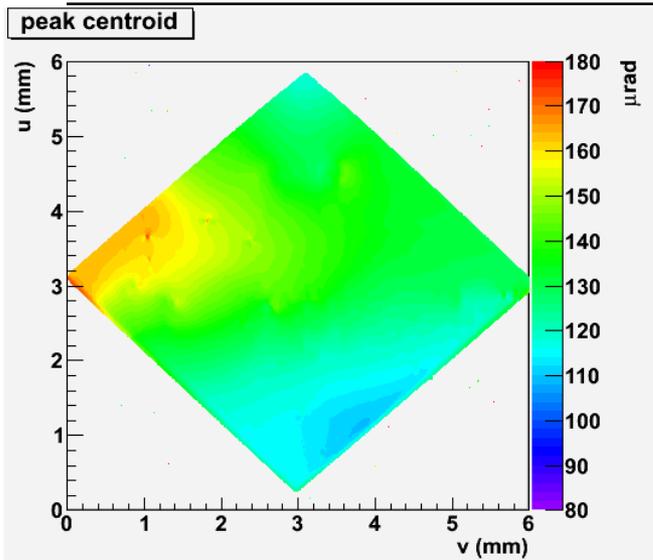


## Final polish with RCMP technique

thickness, stitched

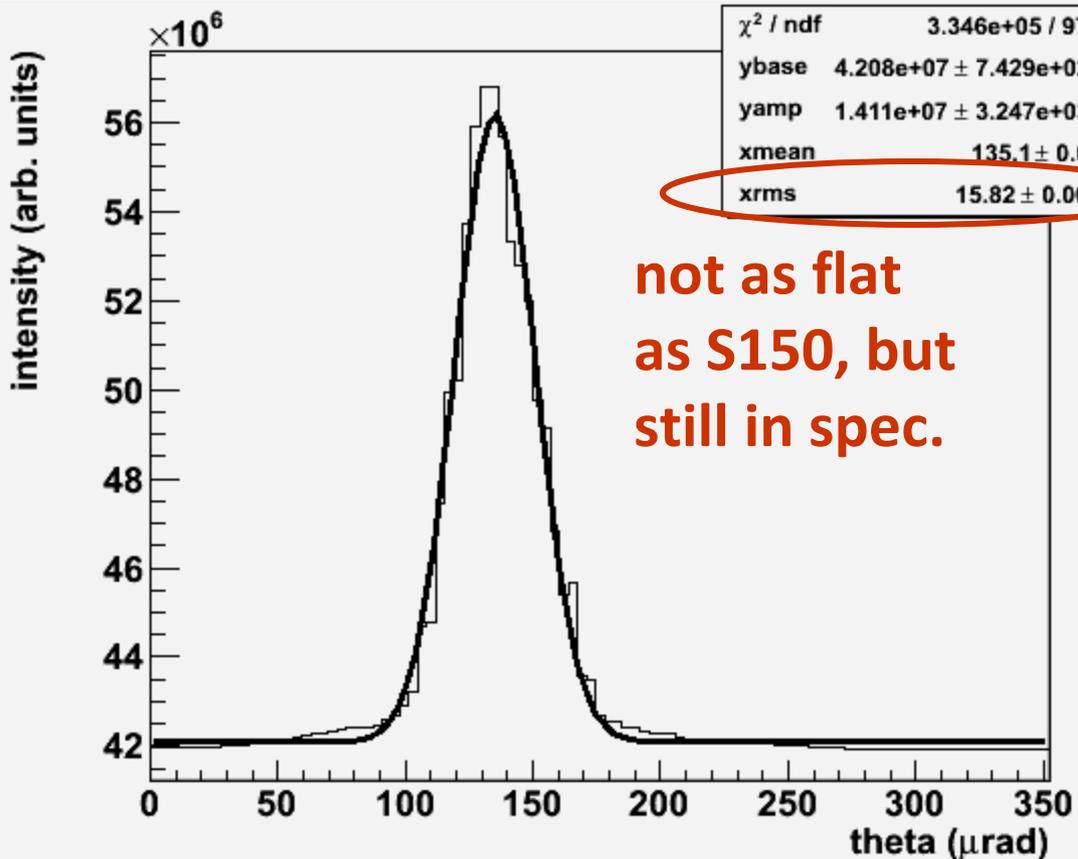


# X-ray assessment: S90



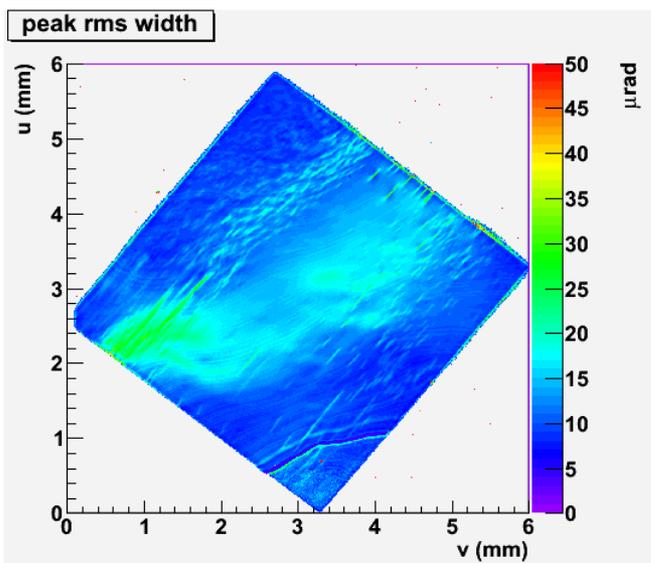
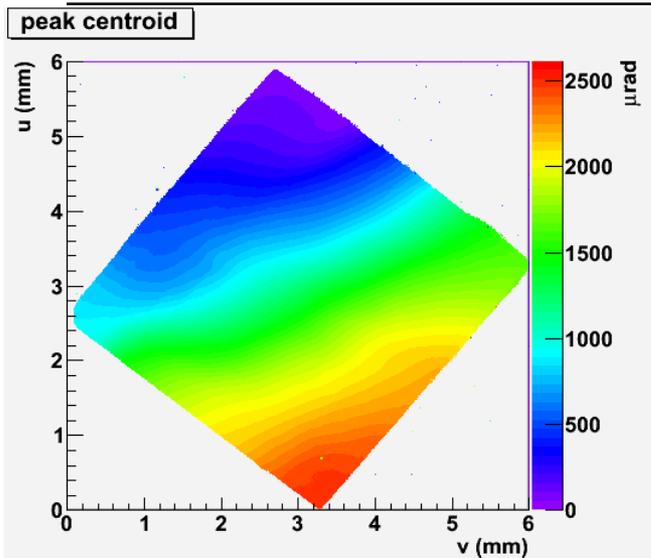
## whole-crystal rocking curve (220)

whole crystal rocking curve of sample sinmat50 scan 1

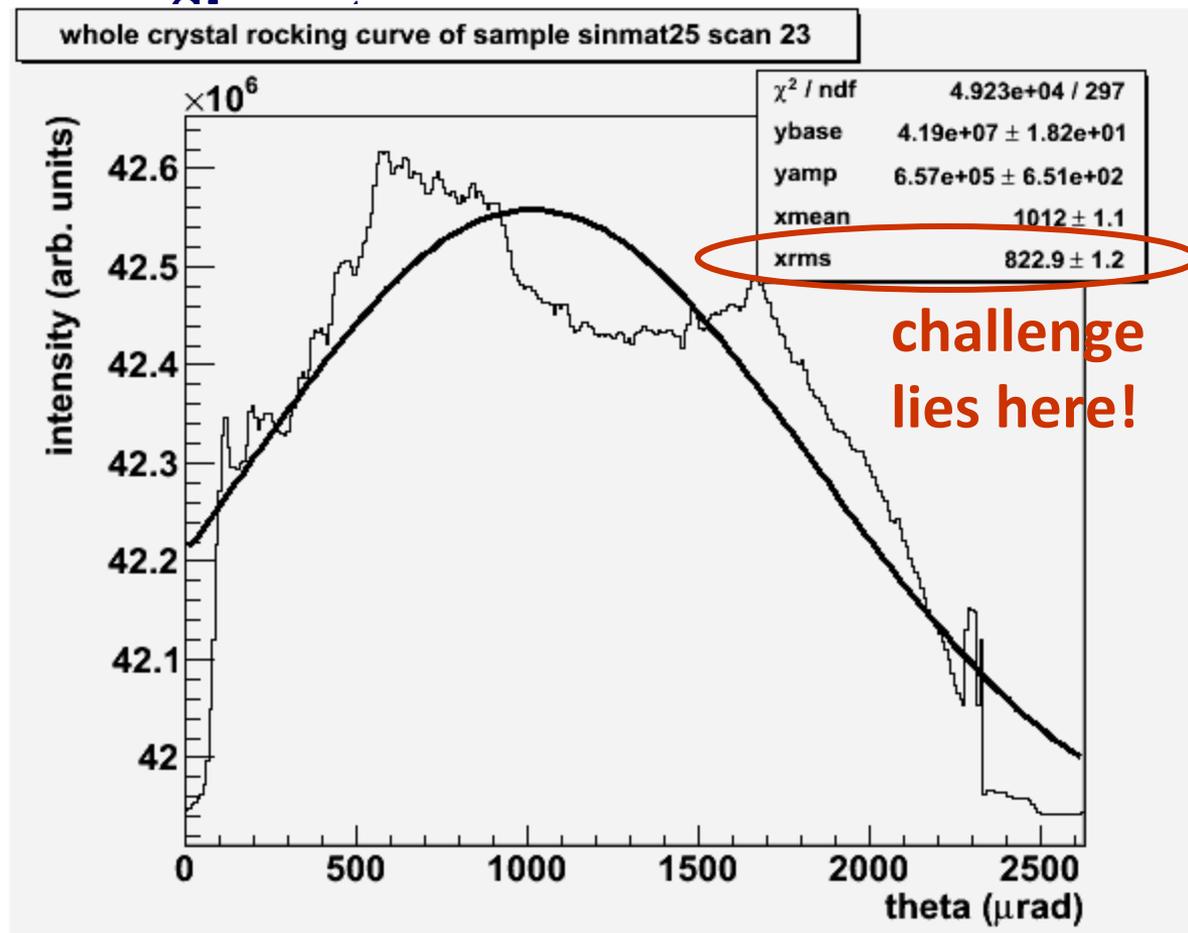


not as flat  
as S150, but  
still in spec.

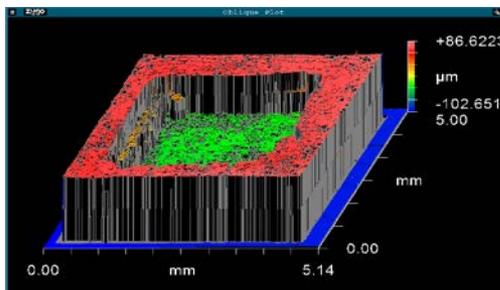
## X-ray assessment: S30



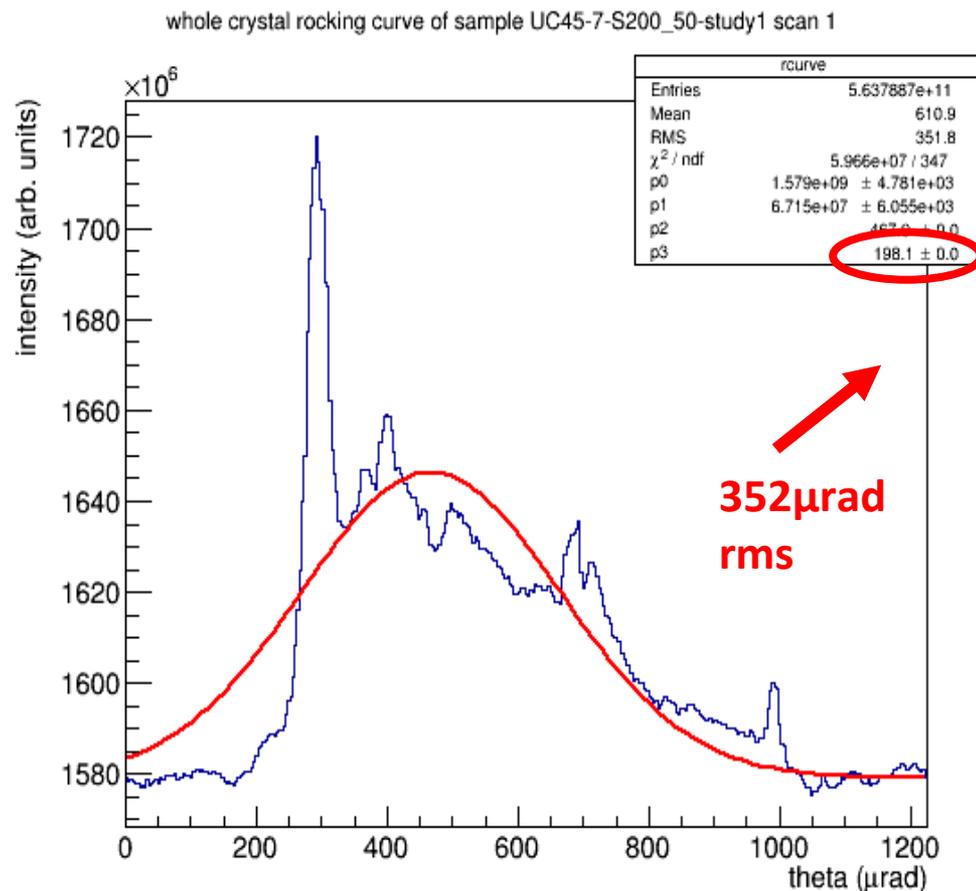
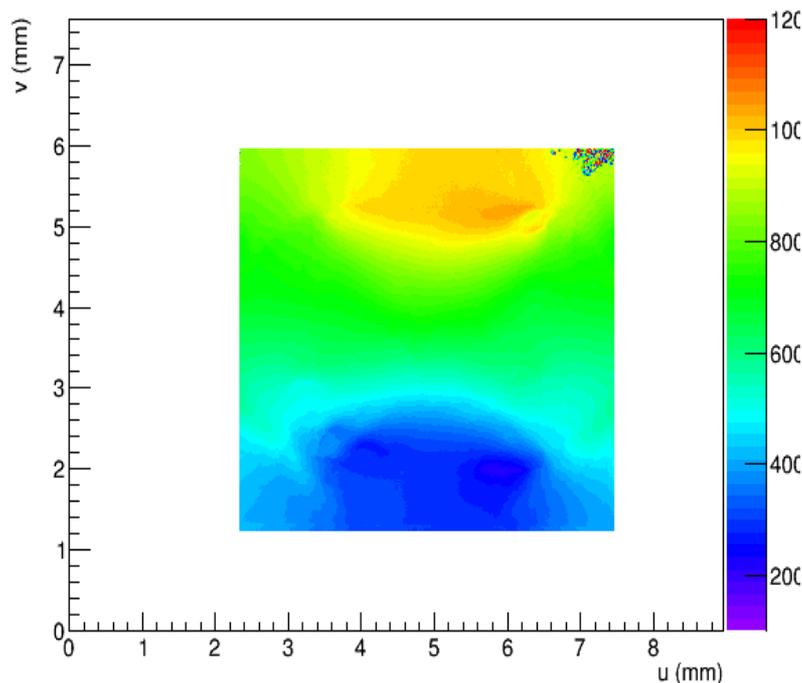
## rocking curve of S30



# X-ray diffraction: S200\_50

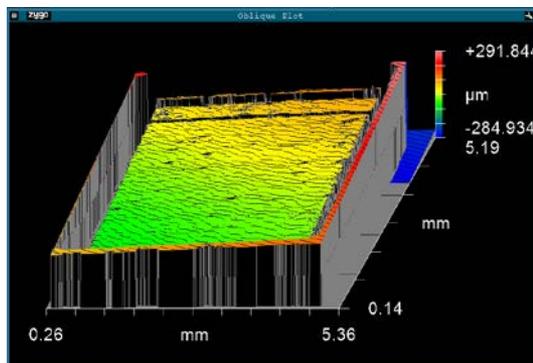


fit peak centroid UC45-7-S200\_50-study1\_001



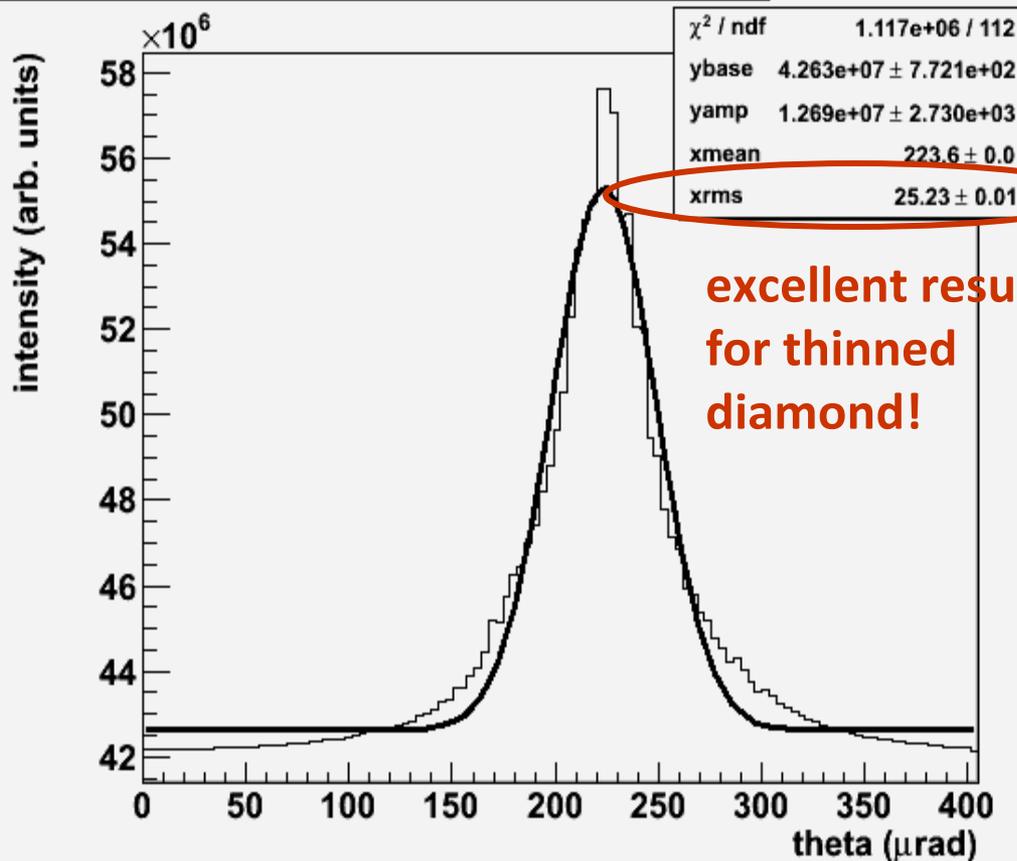
**result: large bending strain across crystal**

# X-ray diffraction: UC300\_40

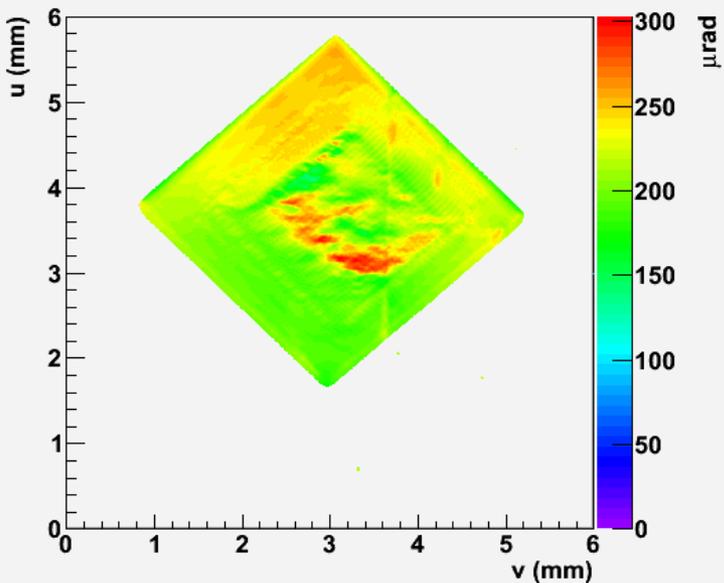


**surface was not treated after ablation**

whole crystal rocking curve of sample setup1 scan 22



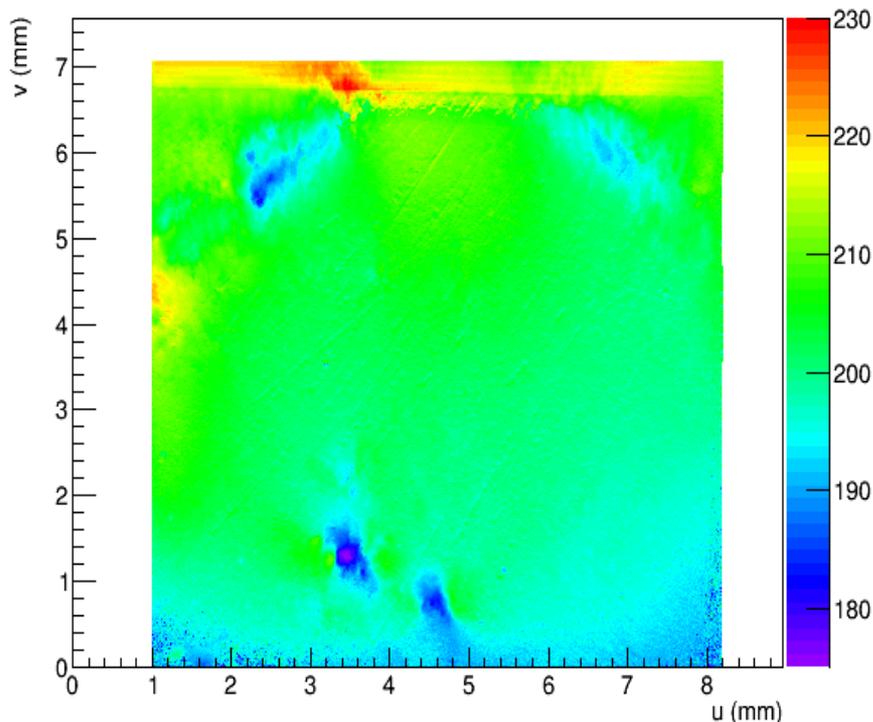
peak centroid



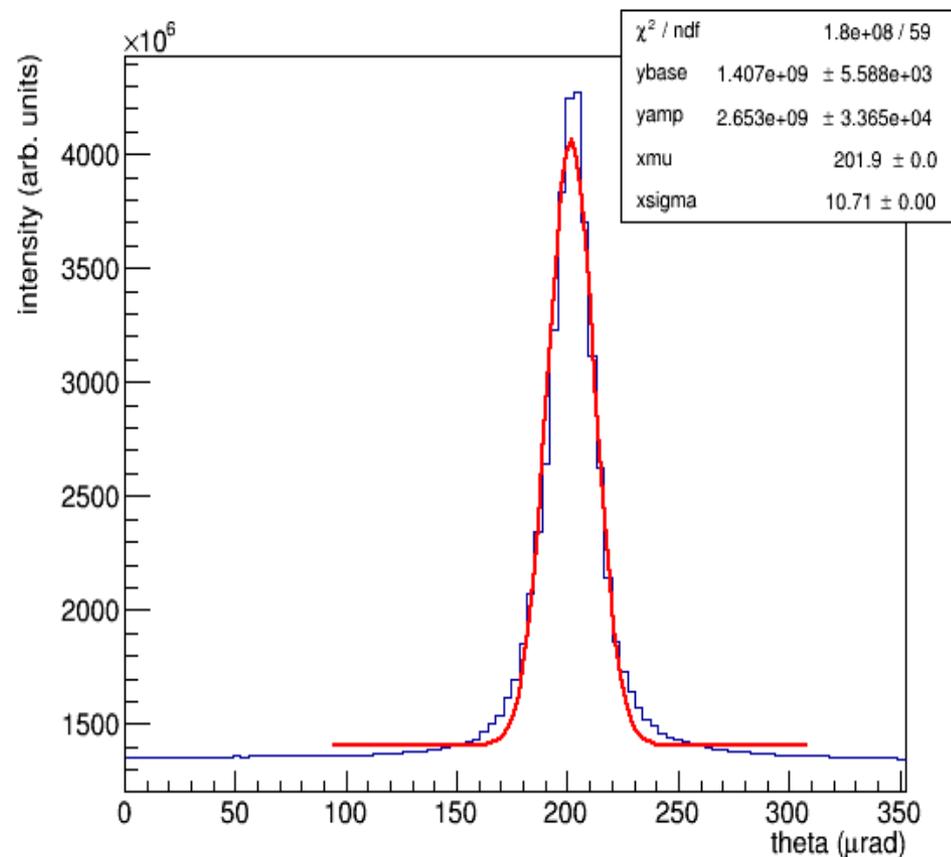
# Next steps: large-area 7x7 mm<sup>2</sup>

- excellent crystal quality
- very large thickness 1.2mm

fit peak centroid JD70-2-study1\_001



whole crystal rocking curve of sample JD70-2-study1 scan 1

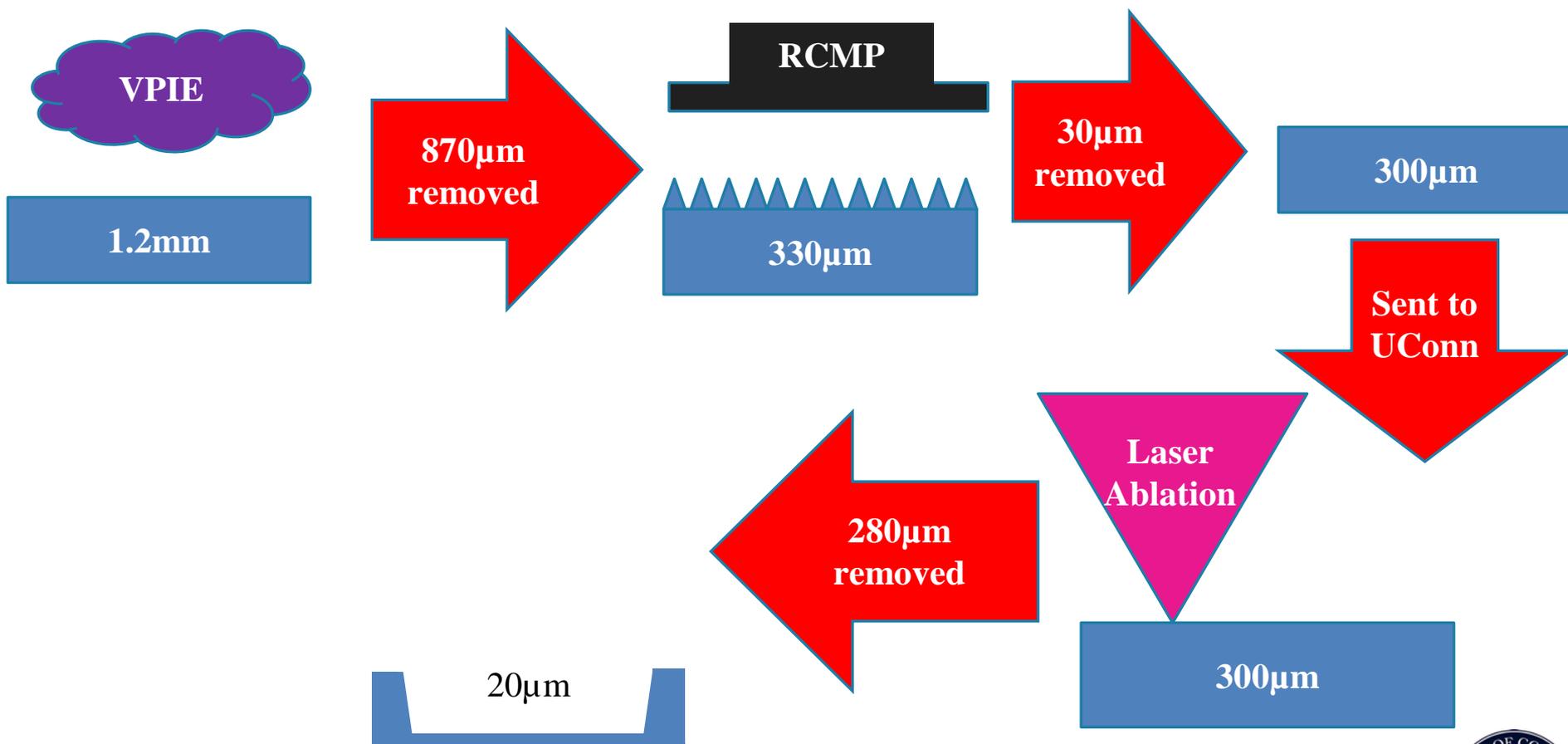


# Summary of Methods

parameters → method	20 μm thickness	cut rate	multiple sample	quality
↓ Reactive CMP	✓	✓	✓	X
Vapor Phase Etch process	✓	✓	✓	X
Laser ablation	✓	X	X	✓



# Plan: combine the 2 approaches



# Summary

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## Developed a three-step process to thin diamond samples

- Step 1: Vapor phase etching process (75 micron/hr)
- Step 2: Polish surface defects with RCMP process
- Step 3: Cut thin central window using laser ablation

## Validated results of all proposed steps using X-ray diffraction

- Designed a custom diamond diffraction setup at CHESS
- Optimized procedures to obtain 3 rocking curves per hour
- Developed analysis code to assess X-ray diffraction topograph
- Developed custom analysis code for 2-surface profilometry

## Expected by Phase 2 completion

- 3 production-quality crystals 7x7 mm<sup>2</sup> with 20 micron window
- draft publication for NP instrumentation journal



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- UConn students
  - **Brendan Pratt** (grad)
  - **Igor Senderovich** (grad)
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  - **Jong Cheol Kim** (grad)
  - **Minfei Xue** (grad)
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