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U.S. DEPARTMENT of ENERGY

Development of pTP Coatings for Wavelength Shifter with Industrial Scale in LAr Detectors

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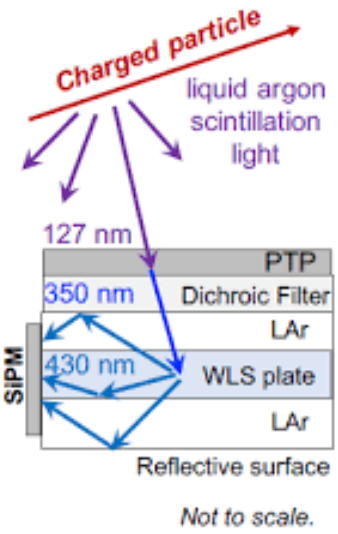
LaserFiberOptics LLC

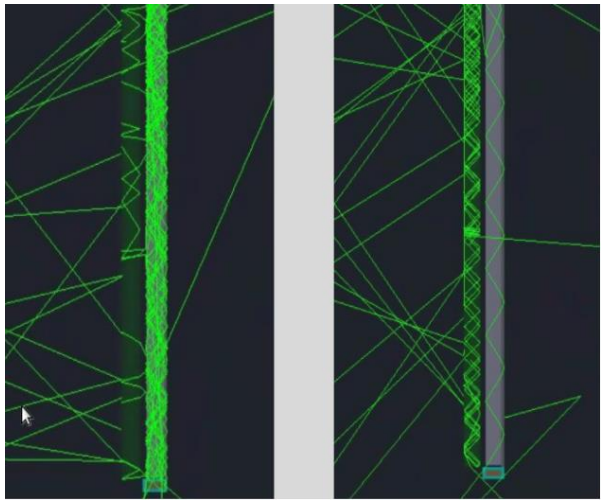
Outline

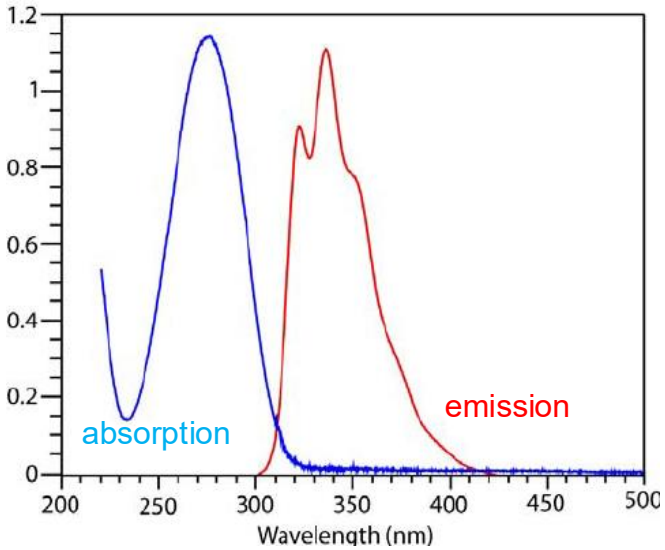
- Introduction on pTP coating
- Industrial scale production with PVD technique
- Filter performance test
  - Emission spectrum
  - Thickness
  - Thermal stress
  - Lesson learned
- Conclusions

Introduction

- The Deep Underground Neutrino Experiment (DUNE) the next-generation long-baseline neutrino oscillation experiment designed to address the key open questions, including the neutrino mass ordering and matter–antimatter asymmetry in the universe.
- A technical challenge → the efficient detection of scintillation light produced by neutrino interactions in liquid argon (LAr) with large area. The scintillation with 127nm light must be shifted to the visible spectrum range of photodetectors, for detection
- The DUNE FD3 APEX Photon Detection System requires a coverage of 2000m^2
- We worked with an industrial vendor LaserFiberOptics for mass production of the pTP coated filter with industrial facility with high quality, cost-effectivity and fast speed







PVD Optimization for PTP Coating

- **Process Enhancements**
  - Ultrasonic cleaning with solvents and DI water—>Standard cleaning procedure
  - Plasma treatment ($N_2/O_2/CF_4$ – 2:10:1, 15 min)—>Increase pTP bonding
  - Argon ion bombardment (10 min)—>remove trace organic residuals
- **Deposition Conditions**
  - Vacuum: 1.6E-4 torr
  - Boat Temp: 195°C
  - Rate: ~5 Å/s

An example for illustration purpose ONLY

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Heat Treatment & Scale-Up

- **Post-deposition Heat Treatment**
  - Covered heating >170°C preserves and densifies film
  - Uncovered film evaporates → enables recycling
- **Mass Production Setup**
  - 3-tier rotating dome (44 substrates/batch)
  - 2 coaters + 4-person crew = 5.4 m²/day (~220 pcs per day)
  - Meets DUNE FD3 project demand with 1 year of production time

three tier rotating dome

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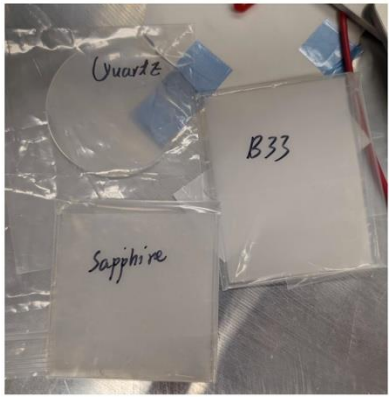
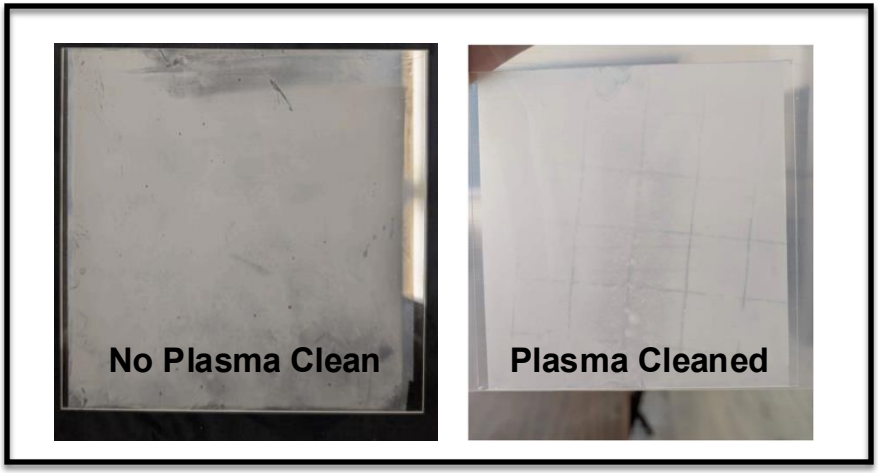
pTP coated samples

- **We received 3 batches of pTP coated filters from the vendor**
  - The coating has been processed with 3 types of substrate:
    - Quartz, Sapphire and B33 (substrate used in DUNE FD1)
  - 1st + 2nd batches coated with generic pTP (supplier claimed to be identical to TF product)
  - 3rd batch coated with Thermo-Fisher branded commercial pTP
  - The vendor keep optimizing the production apparatus and technique
  - Significant improvement in the coating quality over batches
  - High quality filters delivered from the vendor
  - Commercial mass production procedure of PTP coating on substrates is ready



1st Batch (3pcs)



2nd Batch (36 pcs)

3rd Batch (2 of 24 pcs)

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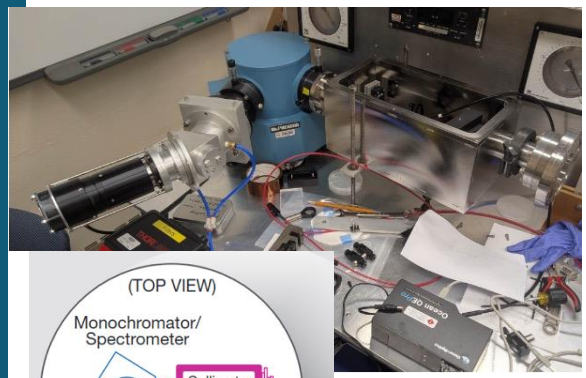
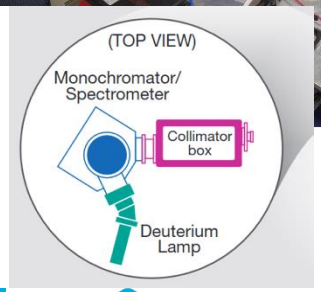
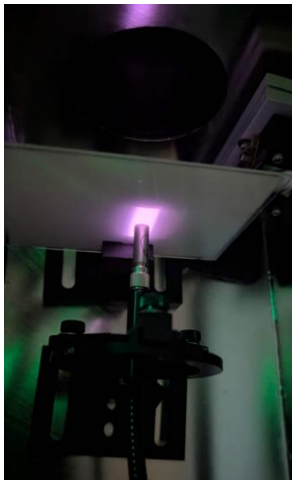
Emission Spectrum Measurement Setup

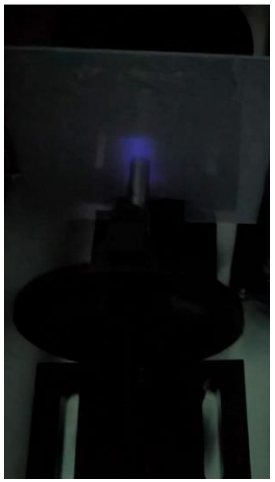
- **A UV monochromator system built by refurbishing a spare device with all new optical components**
  - Deuterium lamp providing a full spectrum of light (heat regulated using compressed argon gas)
  - A monochromator (with a motor controlled grating) to select single wavelength 50-570nm
- **Light emission measured in in two modes: transmission and reflection**
  - 266 nm wavelength selected as excitation
  - Emission light measured by a high sensitivity spectrometer with light collection with a UV collimator and quartz UV fiber input
  - Transmission mode results agrees with transmission—>substrate impact eliminated, Only transmission mode results reported here
  - Emission measured also tried with soft X-ray(see back up slides)



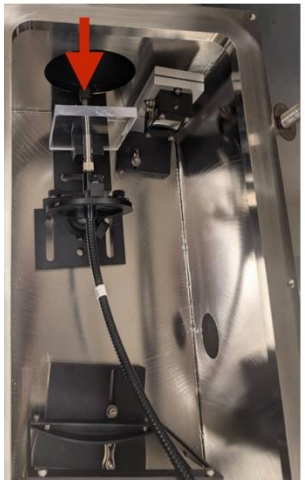




Full Deuterium Lamp spectrum



266 nm light (Taken with nightvision mode on Pixel 9)



Tranmission mode

Reflection mode

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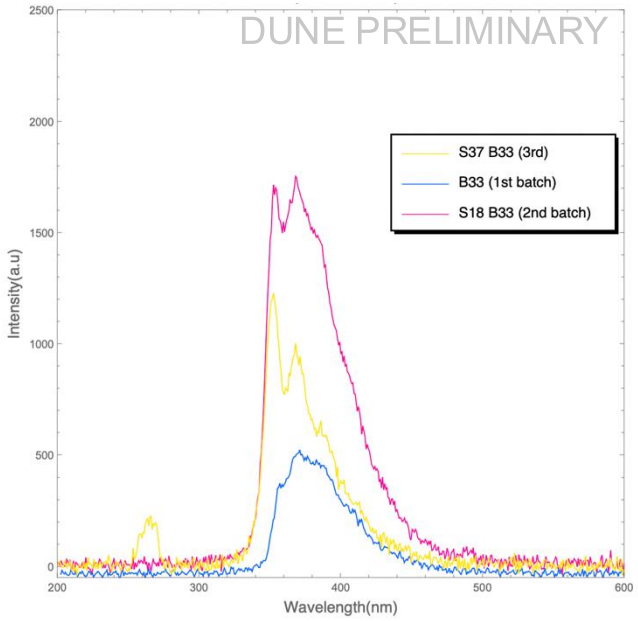
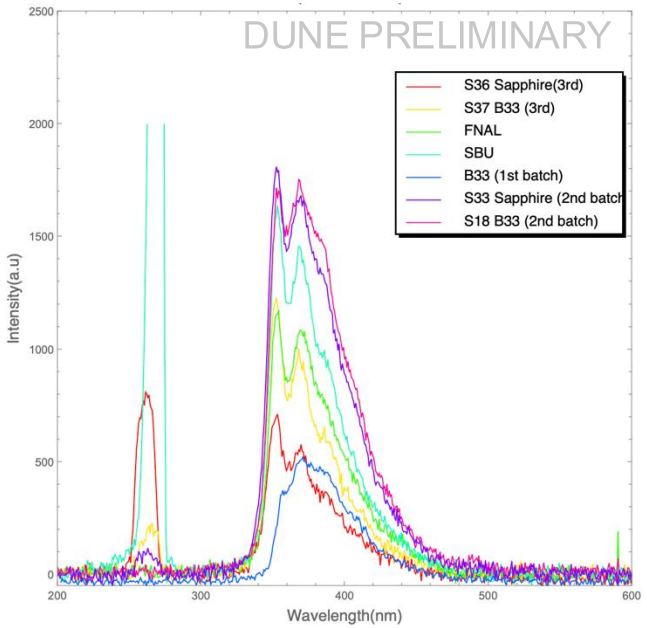
Emission Spectrum Results

- Thanks to our SBU colleagues, We got samples from other samples for comparison
  - 1x from FNAL(PVD coating on B33)
  - 2x from SBU (chemical coating on Acrylic) *See Rado's talk yesterday*
- The final 3rd batch show good agreement to the SBU and FNAL using the same pTP source from Thermal-Fisher
- pTP source is important





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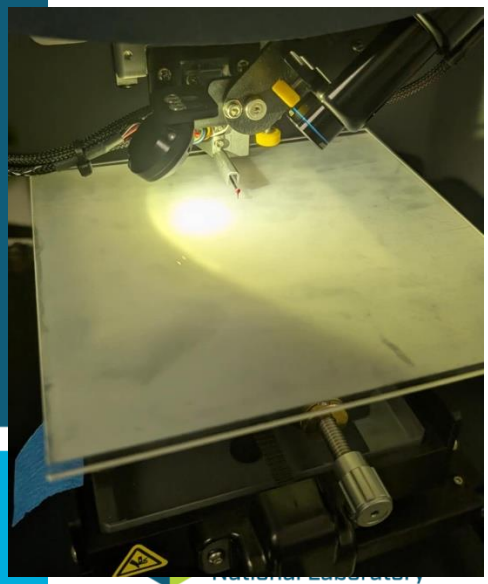
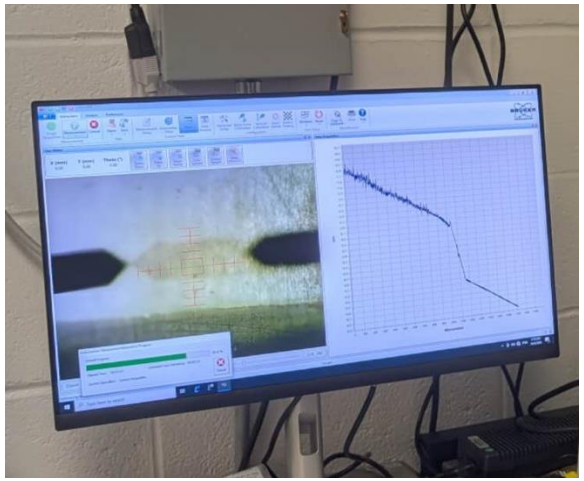
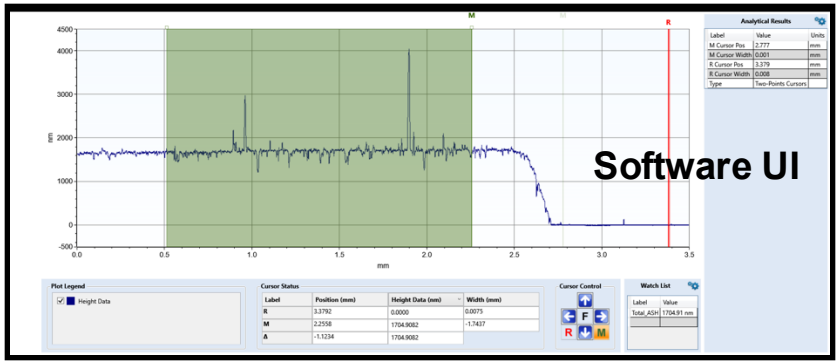
Thickness measurement with profilometer

- **Coating thickness measurement using profiler**
  - The profilometer uses a diamond probe with very little pressure on the surface ~ 3mg for the scanning
  - It is a differential measurement that requires a step
  - The the results requires a surface flatness correction(done by the software)
  - Can only measure the thickness at the edge

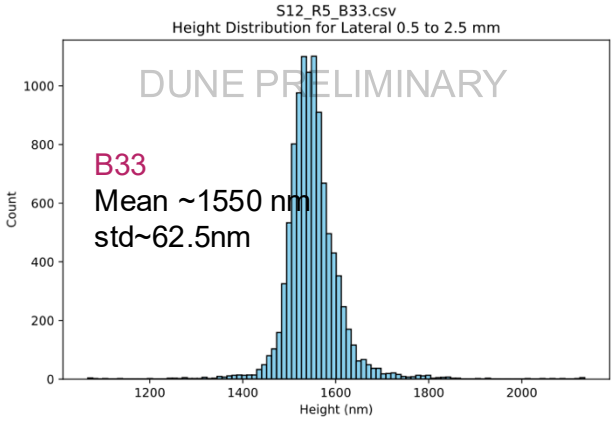


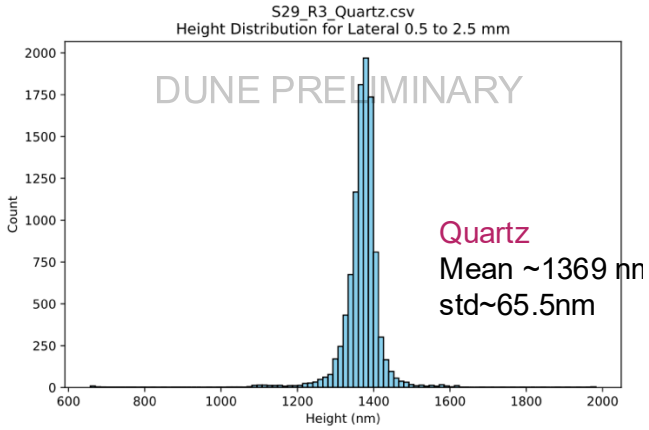


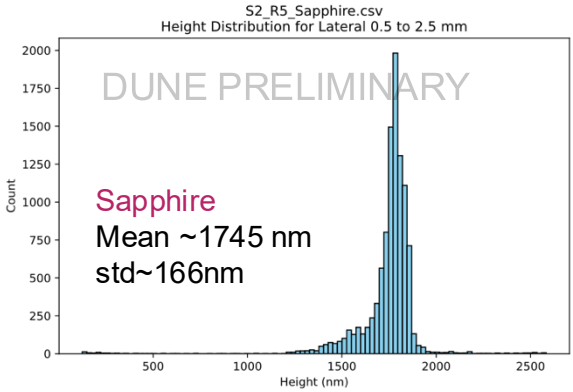


Thickness measurement results

- The target thickness for pTP coating is 1.5 um
- The thickness and uniformity improved significantly between 1st and 2nd batches
- Target thickness achieved
- Thickness measurement for 3rd batch in progress
- Will try larger area measurement with other approaches







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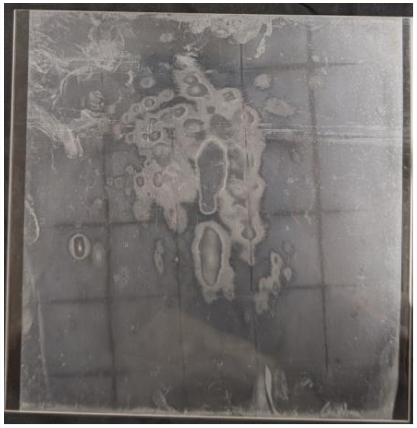
Thermal Stress Test

- Thermal stress test is important for the PDS
- Two configuration tried to simulate of the horizontal and vertically mount PD module immersion process
- Only tried with 2nd batch samples
- In an open mouth dewar filled with LN2
- The samples are pre-cooled on top of the LN2 to prevent thermal shot
- Immersion for about 24 hrs
- Air warmed and dried after pulling out of LN2

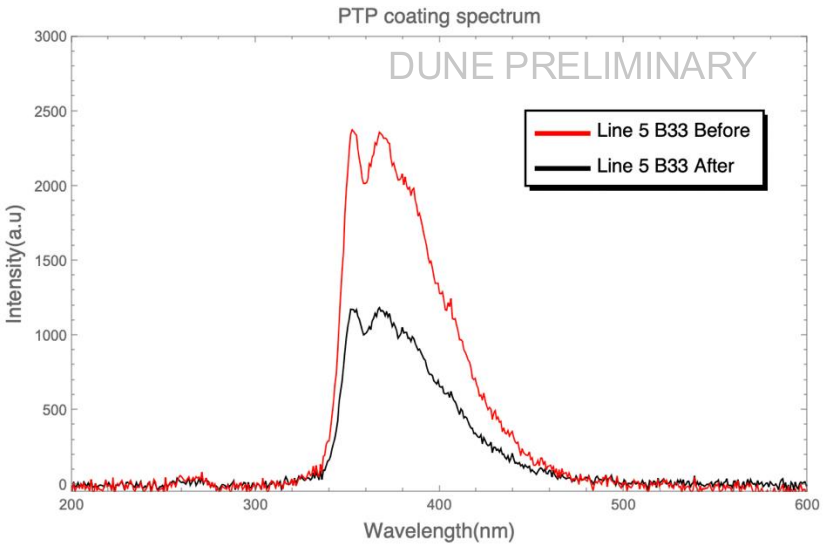
Thermal Stress Test Results

- All the substrates survived the thermal test with 2 cryogenic cycles
- Some degradation on the pTP coating observed
  - Need improved measurement approached for the future
- The light emission performance does not change significantly

Before



After



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Lesson learned

- pTP needs to be pure:
  - Although claimed to be identical by the pTP supplier, the generic and branded pTP are different
- The substrates need to be carefully cleaned
- The pTP coating needs to be conducted under stable temperature control process at low temperature for-high quality and high-volume production
- Cooling and cryogenic operation need to be done in dry atmosphere and slowly.

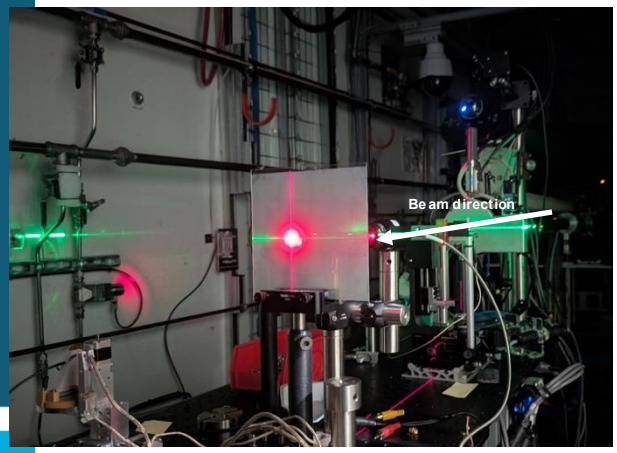
Conclusion

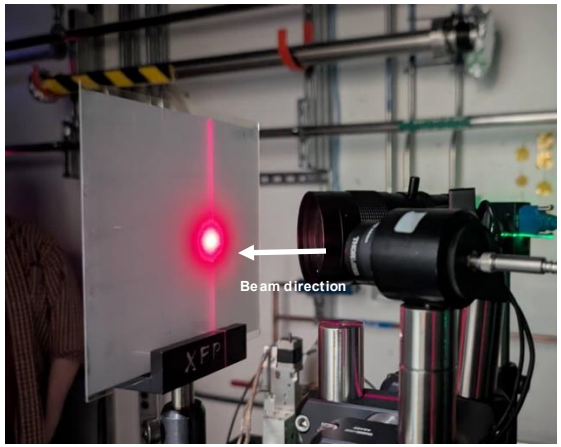
- A procedure established for mass production of PTP-coating with industrial scale with high quality and in a cost-effective manner
  - Significantly coating quality improvement achieved during the development
  - A report on commercial viability of PTP coating on substrates has been completed.
- Measurements on a large number of samples with coating variations have been completed for light emission and thickness.
- Cryogenic stability of the samples has been validated.
- Will continue with more characterization measurement
- Further collaboration on testing is welcome
- Journal publication in preparation

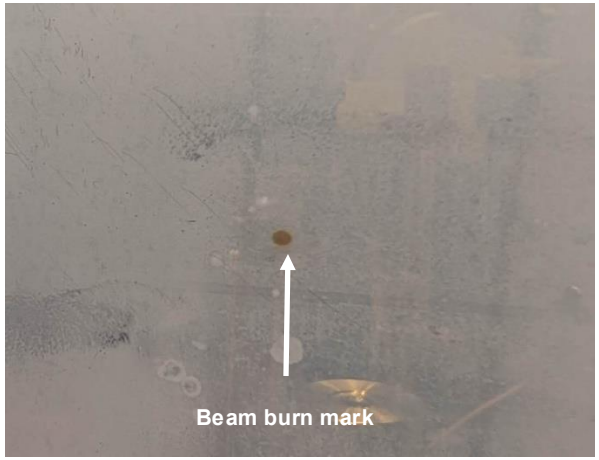
Back up

Sample Test at NSLS-II XFP beamline

- **LFO samples and new diamond samples test at NSLS-II**
  - s11,s27,s33 and all 3 diamond samples were measured
  - Also brought over our spectrometer but it doesn't work with their PC, the old UV spectrometer were used
  - Only measured in reflective mode
  - The beam burned a hole on the B33 sample
  - Strong emission with X-ray excitation, even the substrate got emission

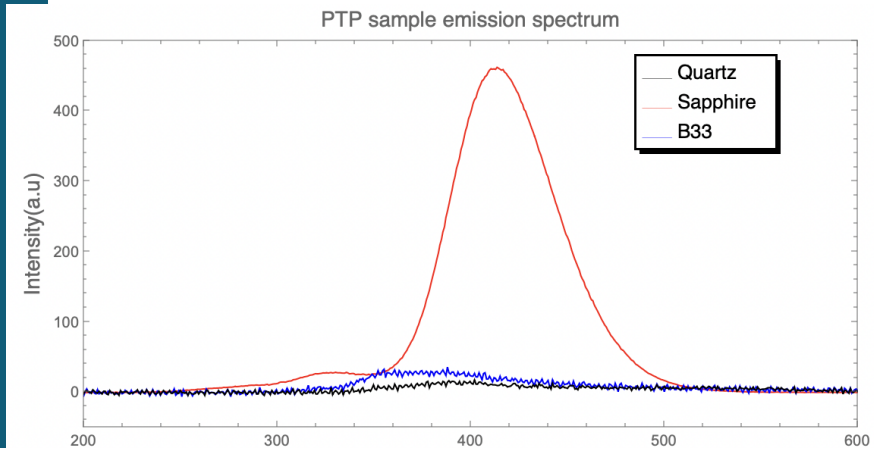


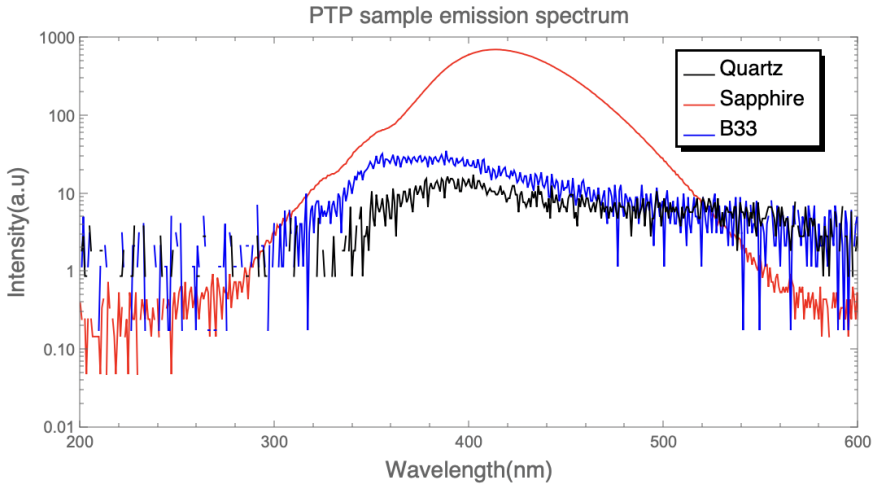


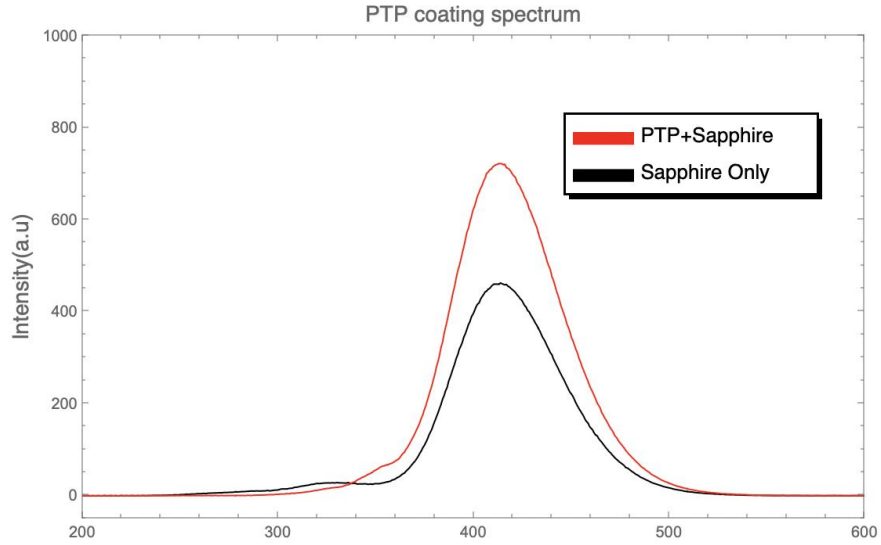


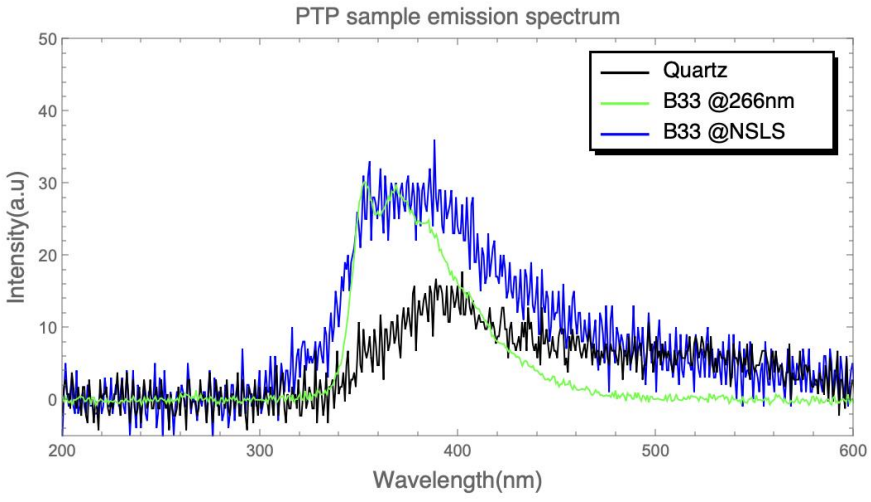
Sample Test at NSLS-II XFP beamline Results

- **Emission Spectrum**
  - Quartz and B33 shows similar shape with 266nm excitation
  - Sapphire shows huge emission due to direct excitation of the substrate

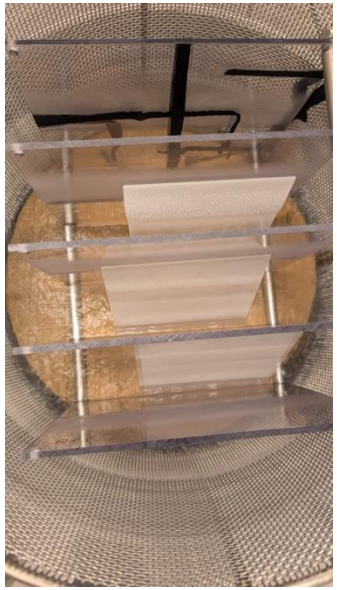








Thermal stress test (Vertical)



S28 Quartz

S4 Sapphire

Thermal results and observation

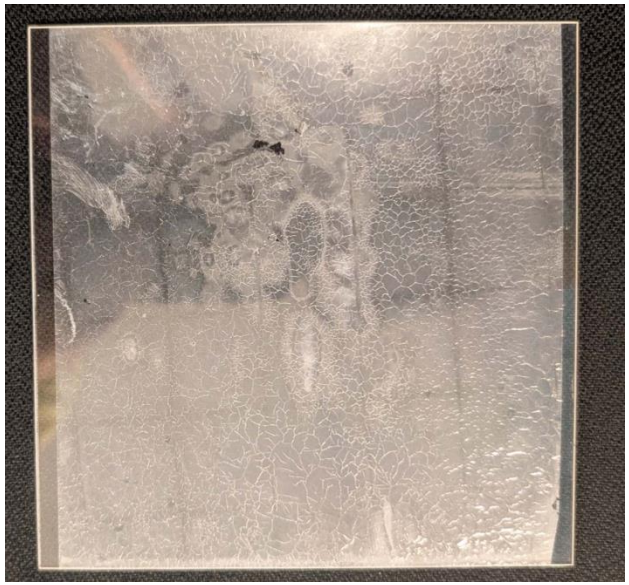
- All substrates survived the thermal stress test with the controlled slow cooling process, no visible damages
- Significant changes on the coating after the cryogenic cycle
  - Most significant on Sapphire, least impact on B33
- Water gets under the coating during warming up

S4 Sapphire

Before

1st cold

2nd cold

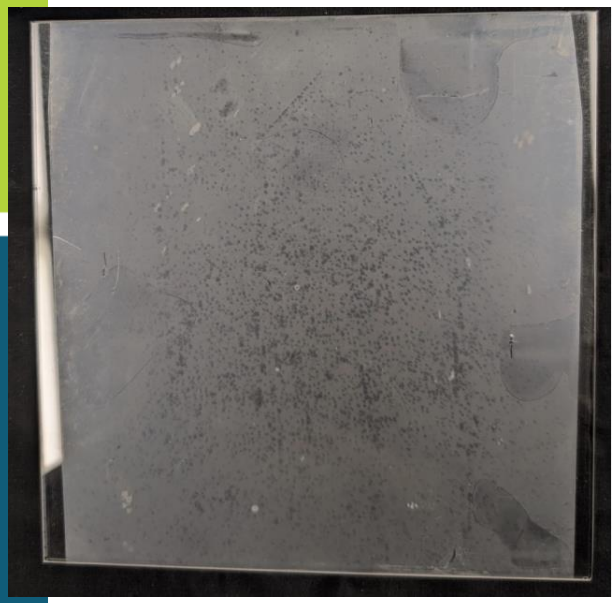


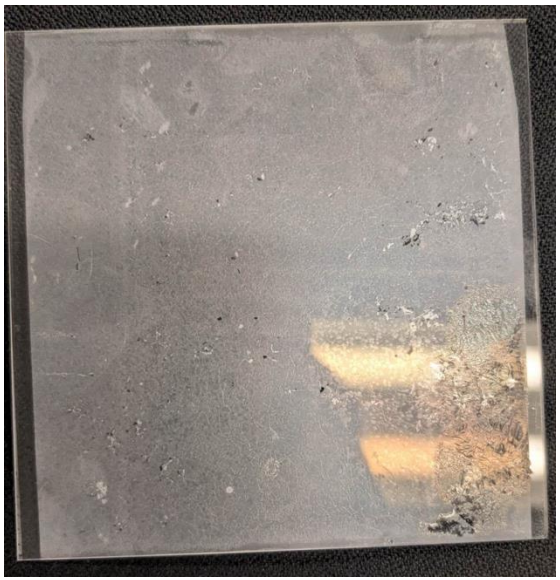
S28 Quartz

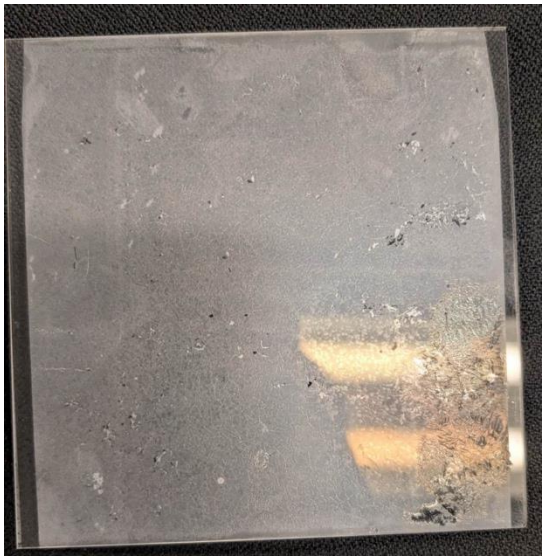
Before

1st cold

2nd cold

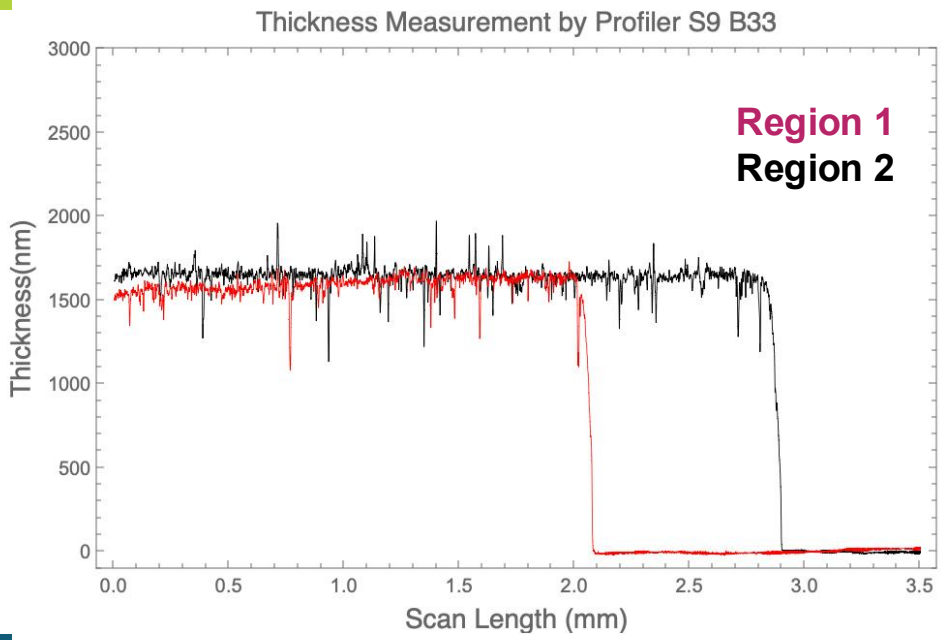


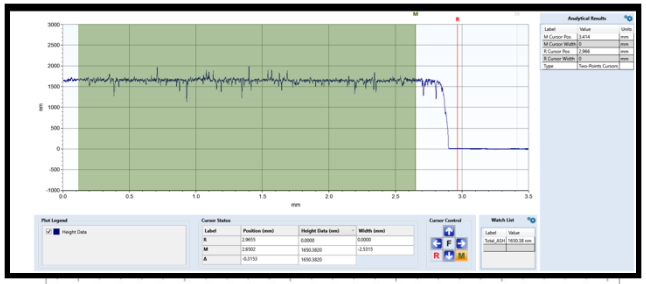


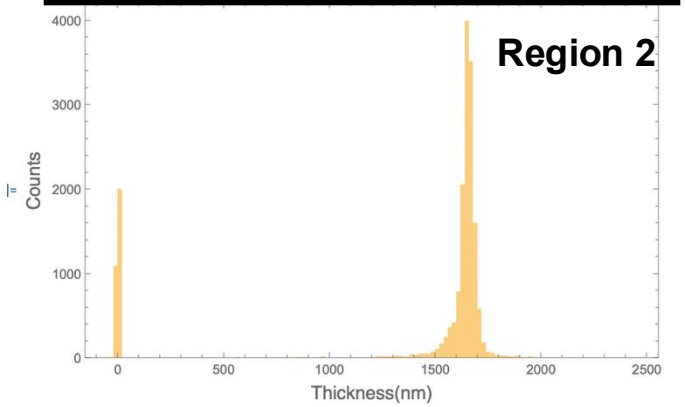


Line 5 B33

S9 B33







S21 Quartz