

DESIGN OF VACUUM CHAMBER WITH CRYOGENIC COOLING OF SAMPLES



for Bragg-Plane Slope Error Measurements

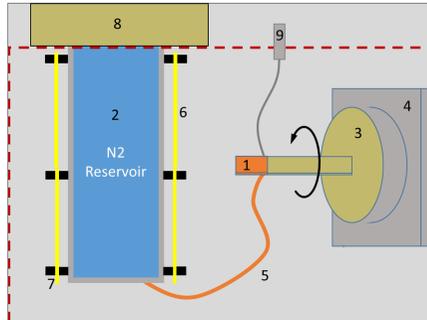
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ABSTRACT

Wavefront preservation is essential for numerous X-ray science applications. Research is currently underway at the Advanced Photon Source to characterize and minimize Bragg-plane slope errors in diamond crystal optics [1]. Understanding the effect of cooling the optics to cryogenic temperatures on Bragg-plane slope errors is of interest to this research. Through the use of a finite element model a custom, compact vacuum chamber with liquid nitrogen cooling of samples was designed and is being manufactured. The design process and initial results are discussed with this poster.

DESIGN REQUIREMENTS

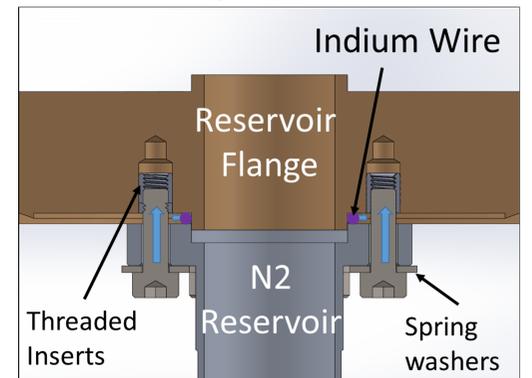
- Rotate crystal surface(1) $\pm 45^\circ$
- Keep sample temp. $< -150^\circ\text{C}$ long enough to conduct X-ray experiment (approx. 30 min.)
- Operate in high-vacuum environment



- | | |
|-----------------------------|----------------------------|
| 1. Crystal Sample (Diamond) | (OFHC) |
| 2. N2 Reservoir | 6. Rad. Shielding (Mylar) |
| 3. Crystal Mount (PEEK) | 7. Spacer/Clamps (PEEK) |
| 4. Rotation Stage | 8. Reservoir Flange (PEEK) |
| 5. Thermal Conductor | 9. Temp. Sensor (RT100) |

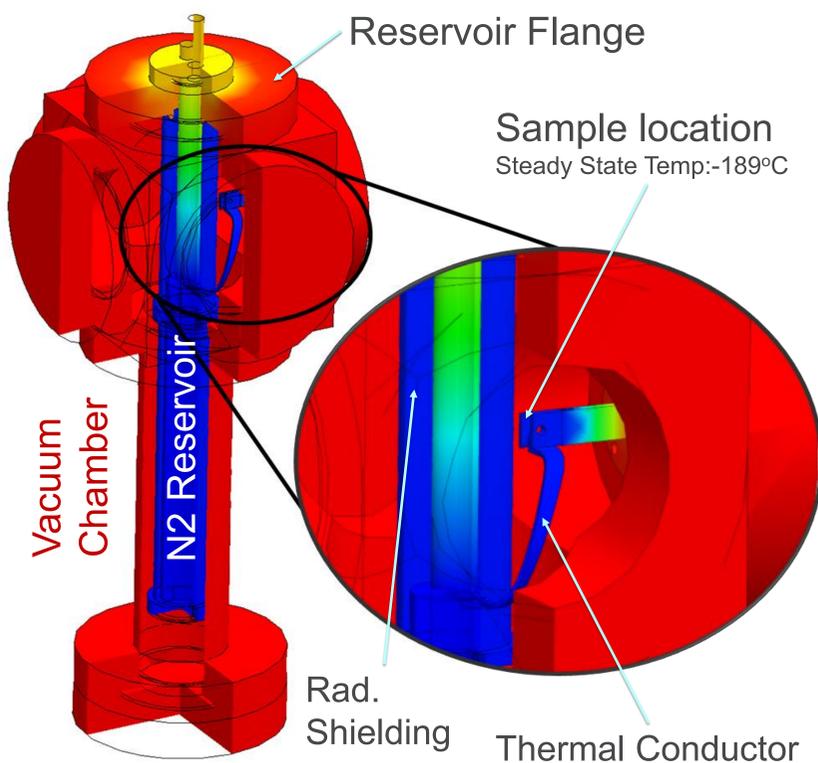
CHALLENGES

- Size: Compact and light as possible so it can be installed on current beamline stages
- Vacuum seal at cryogenic temperatures: Relatively large thermal contraction will happen between the N2 Reservoir (AL) and the Reservoir Flange (PEEK). A vacuum seal using an indium wire allows the seal to hold even at very low temperatures.



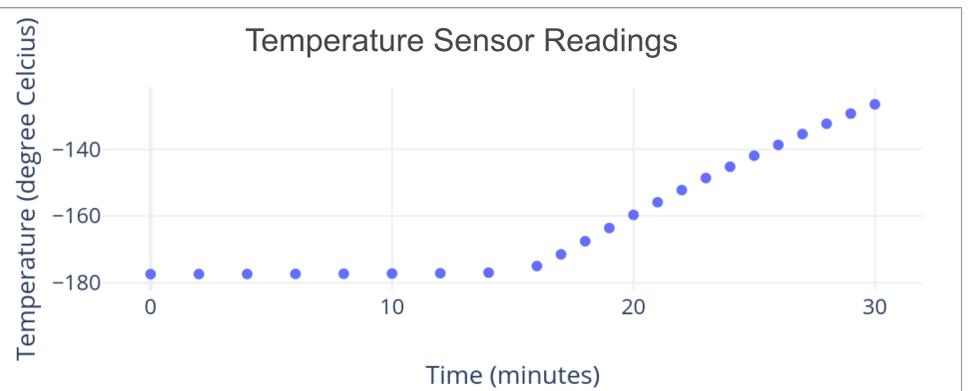
THERMAL FINITE ELEMENT ANALYSIS

- A Finite Element Model was created in SolidWorks® Simulation software and a Steady State Thermal analysis was run.
- Conduction and Radiation heat transfer were of primary concern for this analysis.
- Shell Elements were used for the N2 Reservoir, the Mylar Radiation Shielding, and a section of the vacuum chamber to reduce the overall number of elements in the model and thus the run time.
- At the sample location a steady state temperature of -189.1°C is predicted. It was estimated from the predicted power into the FE model and the latent heat of Liquid Nitrogen that this temperature (-189°C) would be constant for approx. 16 minutes.



PROTOTYPE TESTING

- From the Finite Element Analysis results a prototype chamber was designed and built to test the cooling mechanism. An RT100 thermal sensor was fastened to the sample location to measure temperature.
- The Prototype Chamber test results were remarkably similar to the FEA. The sample location was cooled to about -180°C for about 15 minutes before starting to warm (the $\sim 10^\circ\text{C}$ discrepancy between FEA and Prototype results is due to the thermal conductor length needing to be extended for the prototype)



CONCLUSIONS

- Using finite element analysis a compact, [approx. 120 mm x 170 mm x 300 mm] cryo-cooling vacuum chamber was designed built and tested.
- The sample location was cooled to below -150°C for more than 20 minutes (This is shorter than design requirement it should still be acceptable)
- Utilizing an indium wire seal at low temperature joints high-vacuum was achieved

NEXT STEPS

- Complete Design Requirements: A final design for the chamber has been completed. The design includes a rotation stage and beryllium windows to allow the X-ray beam through to the sample. The final design is currently being manufactured.
- Complete Bragg-Plane slope error measurements at the beamline with X-rays.

REFERENCES

- [1] T. Sun, *et al.*, Nat. Photonics **6**, 586 (2012).
- [2] D. Shu, *et al.*, this conference.

Final Design

