A Cryogenic Sample Environment for the TARUMÃ Station at the CARNAÚBA Beamline at Sirius/LNLS

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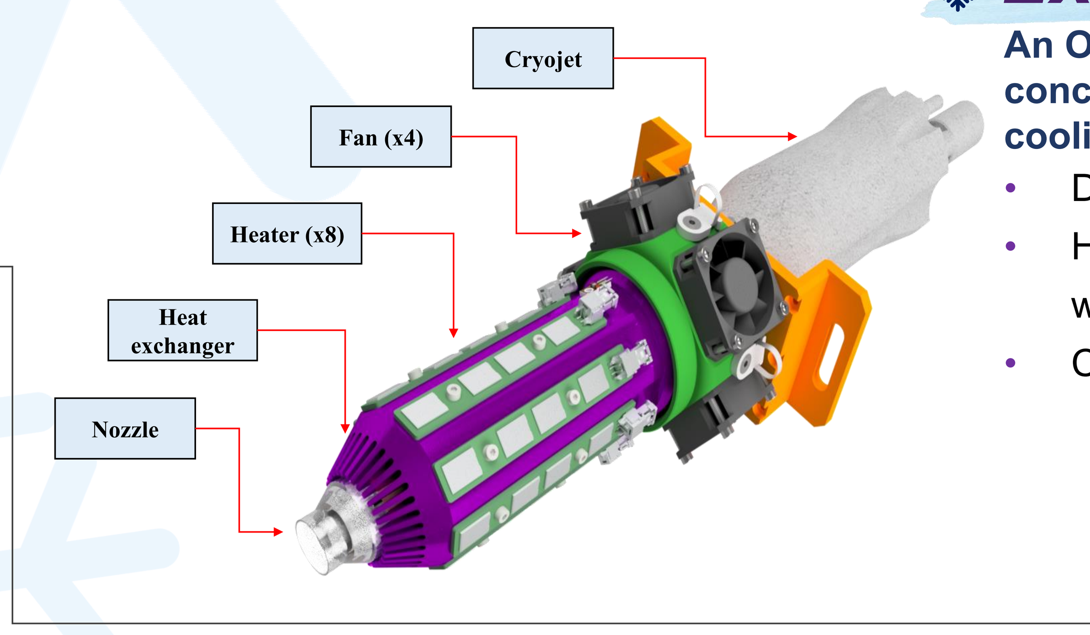
Abstract

TARUMÃ [1,2] is the sub-microprobe station of CARNAÚBA (Coherent X-Ray Nanoprobe Beamline) at Sirius at the Brazilian Synchrotron Light Laboratory (LNLS). Covering the tender-to-hard energy range from 2.05 to 15KeV with achromatic fixed-shape optics, the fully coherent submicron focused beam can be used for multiple simultaneous advanced micro and nanoscale X-ray techniques that include ptychography coherent diffraction imaging (ptycho-CDI), absorption spectroscopy (XAS), diffraction (XRD), fluorescence (XRF) and luminescence (XEOL). Among the broad range of materials of interest, studies of light elements present in soft tissues and other biological systems put TARUMÃ in a unique position in the Life and Environmental Sciences program at LNLS. Yet, to mitigate the detrimental effect of the high photon flux of the focused beam due to radiation damage, cryocooling may be required. Here we present the design and first results of a novel open-atmosphere cryogenic system for online sample conditioning down to 110 K. The high-stiffness and thermally-stable sample holder follows the predictive design approach based on precision engineering principles to preserve the nanometer-level positioning requirements, whereas a commercial nitrogen blower is used along with a cold gas flow exhaustion system that has been developed in order to avoid unwanted cooling of surrounding parts and water condensation or icing.

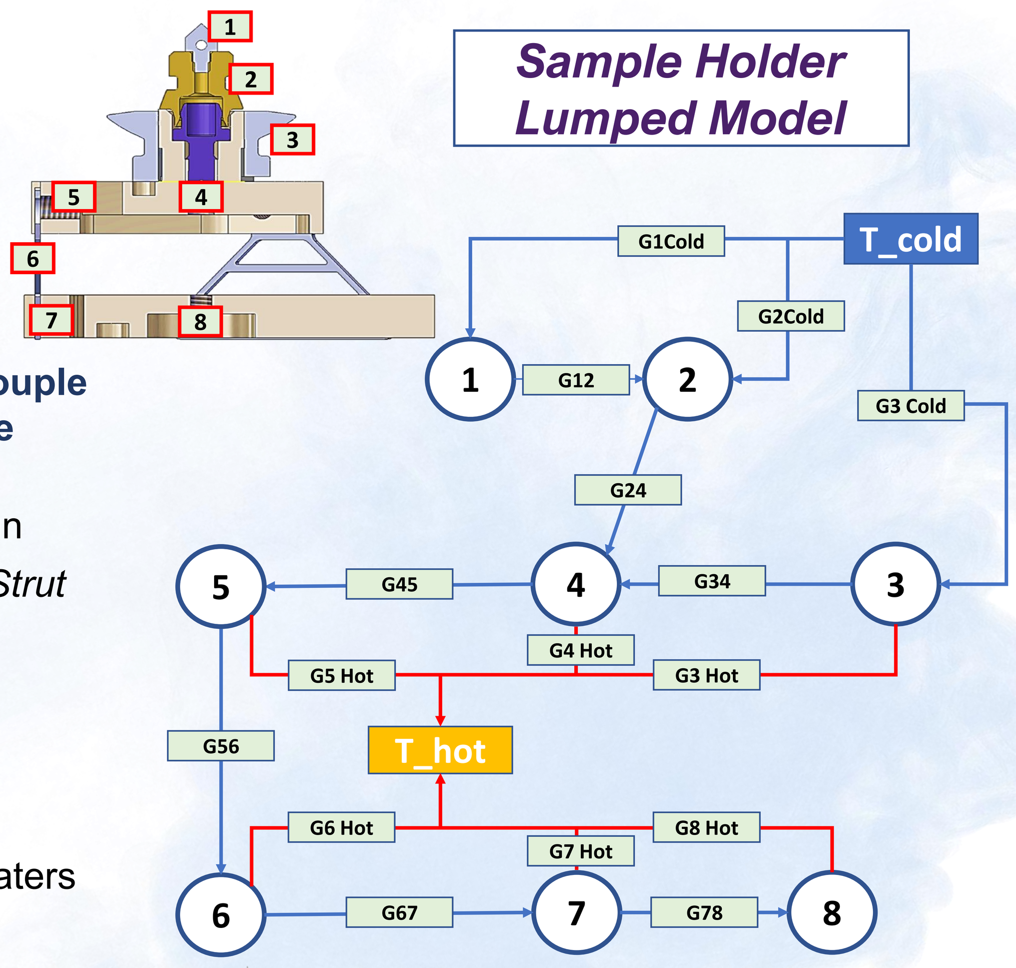
Exhaustion System



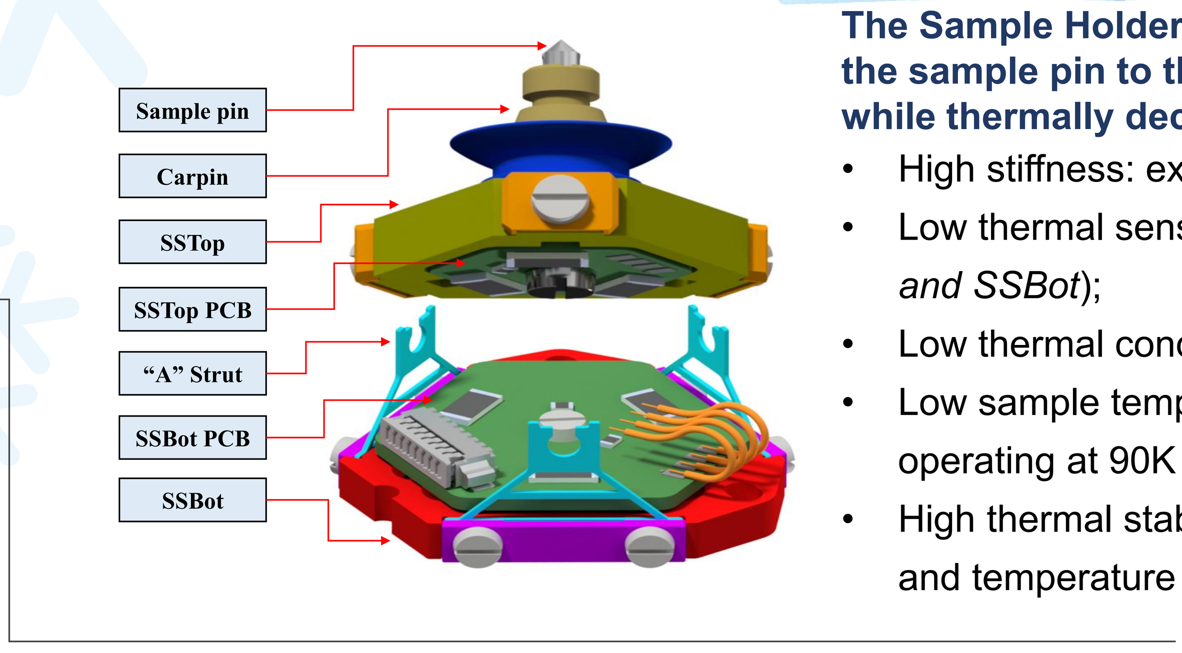
An Oxford Cryojet5 is used as the cooling instrument. Due to its open-atmosphere concept, an auxiliary exhaustion system for the cold gas was designed to prevent cooling and icing in nearby components.

- Drawing of the cold gas, using a set of 4x Sanyo cooler fans;
- Heating of the gas to room temperature (RT) via 8x metalcore circuit boards (PCBs) with power resistors (190W total) coupled to a custom aluminum heat exchanger;
- Cold flow stabilization using a 3D printed nozzle.

Sample Holder Lumped Model



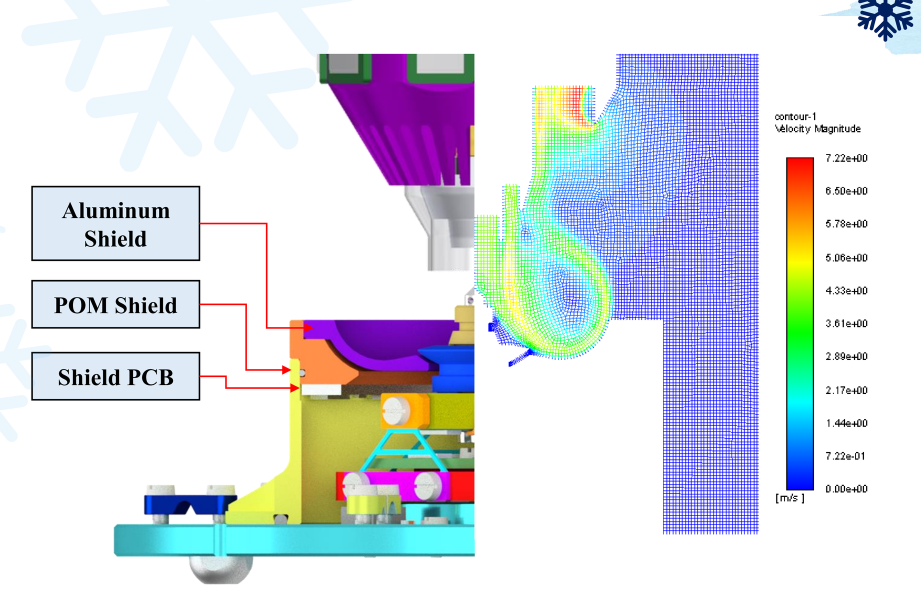
Sample Holder



The Sample Holder is intended to mechanically couple the sample pin to the PI P-563 XYZ scanning stage while thermally decoupling it.

- High stiffness: exactly-constrained, all metal design
- Low thermal sensitivity: Invar 36 alloy (*SSTop, A-Strut and SSBot*);
- Low thermal conductivity: thin, 0.3mm weak links;
- Low sample temperature: 110K with the cryojet operating at 90K (lumped elements estimate);
- High thermal stability: 2x metalcore PCBs with heaters and temperature sensors for active control.

Holder Shield



The holder shield has 2 main functions: guiding the cold gas flow back to the exhaustion systems; and providing a dry environment to avoid icing on the sample holder parts.

- Controlled atmosphere, with dry nitrogen gas purge;
- Shield PCB to heat the aluminum shield, avoiding condensation;
- POM structure to thermally decouple the shield;
- Pressure transducer to interlock the nitrogen purge;
- Humidity sensor interlocking the cryojet according to the shield dew point;

The TARUMÃ Station

Results

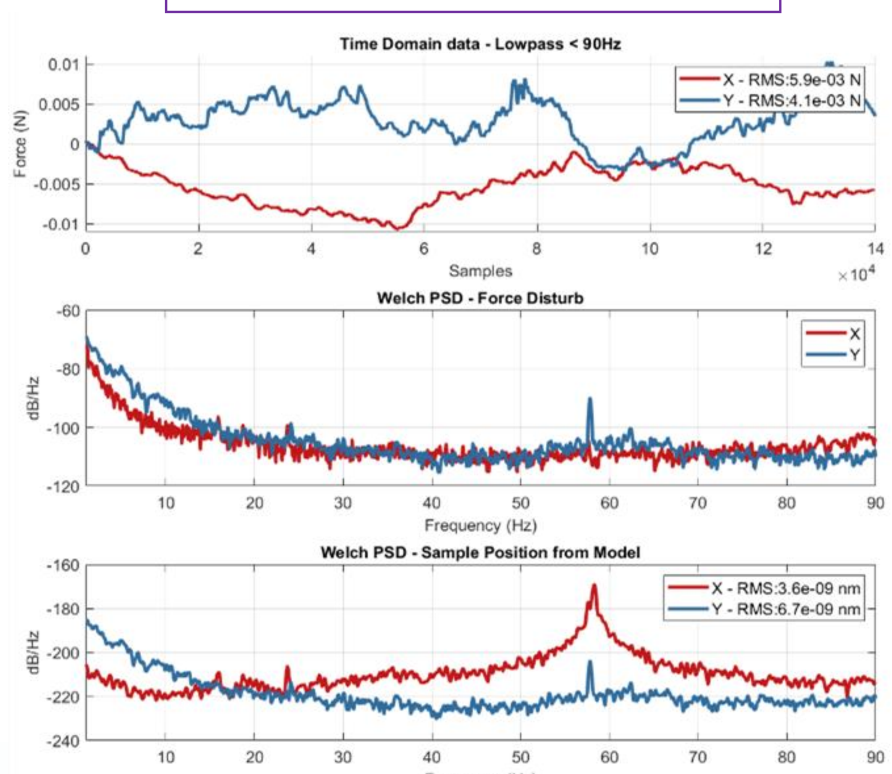
To validate the system shield flow and induced disturbances before the final commissioning at the beamline, a mockup of the system was 3D printed and a parallel flexure-based precision load cell with a Lion Instruments capacitive probe was made. Both the in plane and vertical force disturbances were measured for multiple sample configurations and flow conditions.

- Load cell resolution: < 0.6μN;
- Load cell first mode: 102 Hz;
- Cold flow: 7 and 21 l/min;
- RT flow 5.7 and 18.9 l/min.

The filtered force data (0 - 90 Hz) was fed to a TARUMÃ dynamic model with results within the design budgets: 3.6 and 6.7nm RMS for X and Y, respectively;

Mockup test

Test force and model data



Conclusion

We present the main design remarks regarding the integration of a commercial cryojet into a cryogenic sample setup for cooling sensitive samples at the TARUMÃ station, with focus on position stability and condensation/icing management. Although the final assembly has not yet been fully commissioned due to delays related to the COVID19 pandemic, the first force disturbance results with a mock-up system endorse the correct use of design-for-stability concepts. It can also be highlighted the extensive use of SMD components with metalcore PCBs as heating elements and temperature sensors, leading to the needed setup compactness for the strict space-constraints in the station.

Acknowledgement

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References

[1] H.C.N. Tolentino, et al., "Innovative instruments based on cryogenically cooled silicon crystals for the CARNAÚBA beamline at Sirius-LNLS," AIP Conference Proc. 2054(1), 060026, 2019.
[2] R. R. Geraldes, et al. "Design and Commissioning of the TARUMÃ Station at the CARNAÚBA Beamline at Sirius/LNLS," presented at MEDSI 2020, Chicago, USA, 2021, this conference.

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