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A CRYOGENIC SAMPLE ENVIRONMENT FOR THE TARUMÃ STATION AT THE CARNAÚBA BEAMLINE AT SIRIUS/LNLS

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Abstract

TARUMÃ 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 15 keV 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 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.

INTRODUCTION

With the advancements of low-emittance 4th-generation synchrotron light sources, small X-ray probes with higher photon flux are made possible [1]. Here we bring the case of the TARUMÃ station [2, 3] at the CARNAÚBA (Coherent X-ray Nanoprobe Beamline) [3, 4] beamline at Sirius at the Brazilian Synchrotron Light Laboratory (LNLS), where the ultra-high vacuum (UHV) KB optics is capable of delivering the submicrometric focus of 550 to 120 nm while yielding a high photon flux of up to 1e11 ph/s/100mA [4]. In addition, the large working distance of 440 mm after the KB set allows a broad range of sample environments outside vacuum (see [2]).

Working in the tender X-ray region, the station can be used for soft tissue and light elements multi-instrumentation probing. One of the most appealing techniques is the nano ptycho-CDI, in which the referred probe, combined with the beamline optics and the characteristics of the PiMEGA or MobiPix Medipix-based area detectors [5], can result in spatial resolution in the nanometer range [2]. Yet, this resolution limit is directly impacted not only by the relative position stability between the sample and the probe, bringing the TARUMÃ stability requirements down to the same order, but also by the characteristics of the sample itself over time. This results in two main challenges: ensuring the sample-to-probe spatial stability, that is mostly addressed by the high-stiffness and exactly-constrained optics [6]; best effort over the commercial stages composing the sample manipulator (with a complementary metrology frame) [2] and sample setups based on precision engineering principles; and mitigating the dose-induced sample degradation due to the high photon density and absorption in the tender energy range, especially in biological samples. For the latter, cryocooling the sample has been proved very effective, greatly reducing the dose damage, and improving temporal preservation during long scanning times [7]. In the following sections we present the in-house development and pre-commissioning tests of the TARUMÃ cryogenic sample environment.

SYSTEM OVERVIEW

Aiming at implementing the needed sample cryocooling functionality for the tender X-ray TARUMÃ station and unlocking the possibility of studying soft organic and other dose-sensitive materials, a commercial liquid nitrogen (LN_2) based Cryojet-5 from Oxford Cryogenics system was chosen as the cooling instrument. This choice was made because of the open-atmosphere condition of the sample stage and space constraints limiting other conductive options. Yet, despite the simplicity of the system and easily achievable gas temperatures as low as 80K, the high density of instruments around the sample (see [3]) made its integration at the station a challenging task.

Firstly, the open-atmosphere concept of the commercial cryojet might lead to thermal drift, condensation and/or icing issues in the sample or in sensitive nearby instruments, if the cold gas outflow would not be properly managed. At TARUMÃ, this is prevented with the implementation of an exhaustion system. Then, the open flow nature of this solution might be conflicting with the nanometric sample positioning requirements. This is addressed by a thermo-mechanical design that decouples the high-stiffness sample holder assembly from an auxiliary gas shield with optimized aerodynamics to minimize flow-induced disturbances. In addition to the cryojet and the sample itself, the TARUMÃ cryogenic setup (Fig. 1) is composed of three main subsystems: the *sample holder*, the *holder shield* and the *exhaustion system*.

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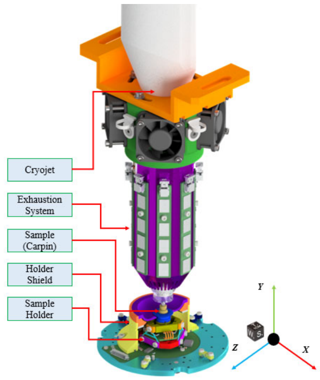

Figure 1: TARUMÃ cryogenic setup main subsystems.

As TARUMÃ is a scanning station that may take experiments reaching several hours, the thermal drift must be Minimized. Moreover, to archive a high throughput of the beamline with fast sample exchange, the cooldown and heat up times must be limited to few minutes. These aspects are addressed via embedded thermal management.

SAMPLE HOLDER

The sample holder system (Fig. 2) comprises the CARNAÚBA standard sample pin (Carpin), a magnetic coupling system for an aerodynamic bridge to the shield, two frames (SSTop and SSBot), a set of thermal decoupling flexures (A-struts), and temperature sensors and heaters on printed circuit boards (PCBs). To speed up setup time by making it as modular and easily exchangeable as possible, the standard mounting plate for the holder was made compatible with the TARUMÃ standard interface plate that lies on the PI P-563 XYZ piezo scanning stage.

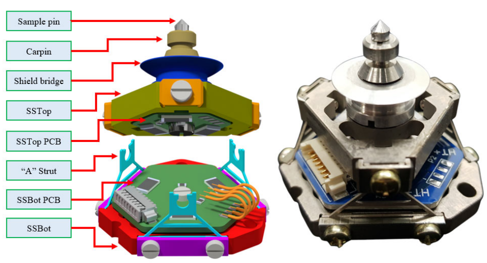

Figure 2: Left: overview of the sample holder with its main subcomponents. Right: picture of the assembly.

Alternatively, the holder was also designed to be compatible with the ptycho-Bragg-CDI setup, in which it is instead fixed to a tip-tilt Attocube piezo assembly (ANGt101 and ANGp101) on a different mounting plate. This sets an additional requirement that the sample holder accepts rotations of about $\pm 4°$ in the Rx and Rz axes with respect to the holder shield around the sample nominal position.

In addition to low position drift in the sample, low thermal conductivity is requried between the sample and the room-temperature (RT) parts, so that sufficiently low temperatures can be achieved with a given heat exchange with the cold air. Thus, the main material in the holder (SSTop, SSBot and "A" Struts) is Invar 36. In spite of not being the most refractory metal available, analytical models and finite element (FE) simulations resulted in minimum sample temperature arround 110 K, whereas the maximum vertical position sensitivity should be in the range of 41 nm/K for constant variation either in the temperature of the cold gas or the RT base (SSBot). Other options, such as Ti6Al4V might reach even lower temperatures, but showed higher sensitiviy. All the Invar parts were nickel plated to mitigate corrosion and will be gold-plated to reduce radiation heat exchange.

Both SSTop and SSBot are equiped with custom aluminum metalcore PCBs with Riedon 1kOhm RTD (Resistance Temperature Detector) temperature sensors and Susumu 2512 SMD (Surface Mounted Device) power resistors acting as heaters. Each PCB can then deliver up to 4 W to the Invar parts. This is needed to control the cold SSTop temperature few degrees above the minimum achievable temperature to reduce thermalization times (220 s for 150 K, estimated) and drifts, and to maintain the SSBot at RT. Keeping both referred parts under control also allow the Invar A-struts internal gradient to be constant, as convection heat exange is minimum, greatly reducing position drifts. The SSTop PCB can also be used to heat all the cold parts for faster sample exchange.

HOLDER SHIELD

The sample holder shield (Fig. 3) has two main functions: guiding the cryojet cold gas back to the exhaustion system in a controlled way, thus, minimizing flow-induced disturbances in the holder and preventing nearby components from being cooled; and providing a dry environment to avoid icing on the sample holder cold parts.

The first is achieved by the appropriate design of a concave aluminum gas guide that forces the incoming cryojet flow back upwards, to where the low differential pressure of the exhaustion system can draw it, according to CFD simulations. Yet, as the shield is cooled by the cold gas in forced convection, an embedded heating system was designed to avoid unwanted icing by controlling it slightly above RT. 10x Susumu 2512 SMD power resistors connected in series in a circular aluminum metalcore PCB can deliver up to 40 W to the shield. For temperature feedback for a PID control loop, two 1 kΩ Riedon platinum RTD sensors are again used.

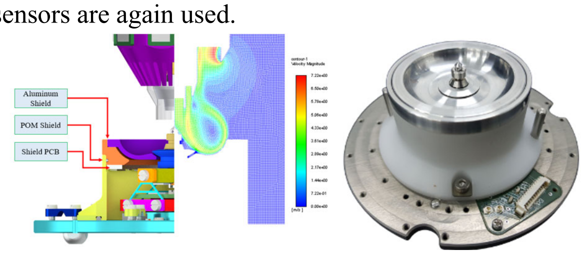

Figure 3: Overview of the Holder Shield including a CFD velocity plot example and the final assembly.

Next, the dry environment for the Sample Holder by is achieved by purging dry nitrogen gas into the Holder Shield. Sealing at the base is made via o'rings, whereas there is a small the gap of a few tenths of a millimeter between the holder and the shield, with unimportant leakage. This assures the mechanical decoupling between both parts and complies with the tip-tilt requirements for the holder.

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An analog pressure transducer KP216H1416XTMA1 by Infineon Technologies is used to ensure a minimum of 20 Pa of relative pressure is always kept, while an analog HIH-4031-001 air humidity sensor by Honeywell is used to constantly monitor if the internal atmosphere water content is below dew point. Both sensors are assembled in a FR4 PCB and used in an interlock system for the cryojet.

EXHAUSTION SYSTEM

The exhaustion system (Fig. 4) was design to create a negative pressure zone as close to the cryojet nozzle as possible, collecting the cold gas around the nozzle itself after cooling the sample to preserve the surroundings. The system also acts as a gas heater by exchanging heat through internal fins. A total heating power of 190 W can be used to rise the cold gas temperature back to RT. This is especially important to avoid further condensation inside the exhaustion channels and to mitigate any undesired cooling of secondary nearby instrumentation.

The module main frame is an aluminum heatsink machined via EDM (Electrical Discharge Machining) for the internal fins, which is equipped with 8 custom aluminum metalcore PCBs with four 100 Ω Ohmite D-Pack power resistors acting as heaters. The temperature control feedback is provided by two 1 kΩ Riedon RTD sensors assembled in aluminum PCBs. The negative differential pressure is achieved by a set of 4 Sanyo commercial cooling fans controlled by the same driver that is used for the heaters. According to CFD simulations, the fans can reach the needed static pressure of 36 Pa and flow of 113 l/min.

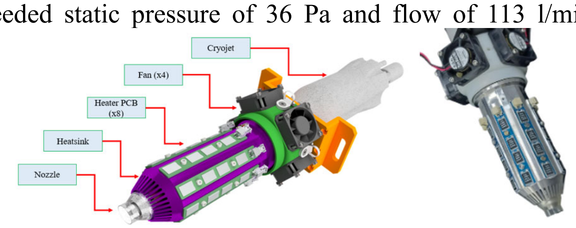

Figure 4: Exhaustion system render and final assembly.

FIRST 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 (< 0.6 μN resolution with first mode at 102 Hz) with a Lion Instruments capacitive probe was made (Fig. 5). Both the in-plane and vertical force disturbances were measured for multiple sample configurations and flow conditions: for the cold flow rates of 7 and 21 l/min, and the RT flow rates of 5.7 and 18.9 l/min.

The results suggest that the low-frequency contribution (< 10 Hz) dominates the disturbances for all flow rates and sample conditions. With the nominal sample holder position, the average 0-90 Hz RMS in-plane force level with the maximum cryojet flow was 2.2 ± 0.7 mN, whereas the vertical was 4.1 ± 2.1 mN. The force data from two X and Y average test runs (Fig. 6) was used to feed a dynamic model of the TARUMÃ sample stages to calculate the expected position disturbances at the sample position in with respect to the experimental bench (see [3]).

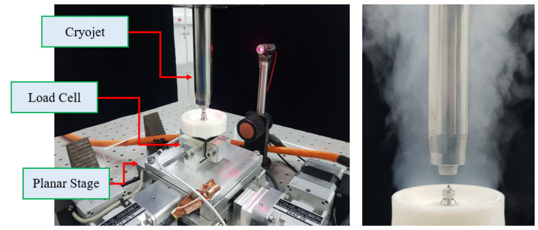

Figure 5: Test setup and mockup shield flow.

The resulting X position RMS value from 0 to 90 Hz was 3.6 nm, whereas the Y was 6.7 nm, which is acceptable within the design budget, as a fraction of the beam size and reasonable for ptychography.

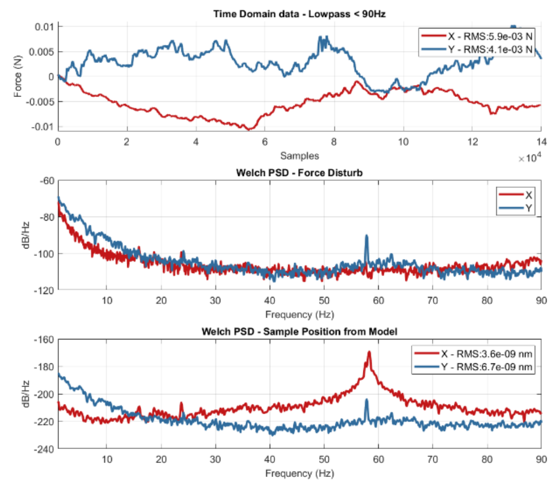

Figure 6: X and Y force time plot and PSD with the referred sample position PSD from the dynamic model.

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 and thermal managements. 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 resulting in acceptable predicted disturbances within the design budgets. It's also important to highlight the extensive use of SMD components with metalcore PCBs as heating elements and temperature sensors as well as ordinary (FR4) PCB with SMD pressure and humidity sensors for control and interlocking purposes, leading to the needed setup compactness for the strict space-constraints in the station.

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REFERENCES

[1] D. Einfeld, "Performance and Perspective of Modern Synchrotron Light Sources", in *Proc. 58th ICFA Advanced Beam Dynamics Workshop on High Luminosity Circular e+e- Colliders (eeFACT'16)*, Daresbury, UK, Oct. 2016, pp. 17-24. doi:10.18429/JACoW-eeFACT2016-MOOTH4

[2] H. Tolentino, R. R. Geraldes, G. Moreno, C. S. B. Dias, C. Perez, and M. M. Soares, "TARUMÃ station for the CARNAUBA beamline at SIRIUS/LNLS," *X-Ray Nanoimaging: Instruments and Methods IV*, vol. 11112, Sep. 2019, p. 5. doi: 10.1117/12.2531110

[3] R. R. Geraldes *et al.*, "Design and Commissioning of the TARUMÃ Station at the CARNAÚBA Beamline at Sirius/LNLS," presented at MEDSI2020, Chicago, IL, USA, Jul. 2021, paper WEPB13, this conference.

[4] H. C. N. Tolentino *et al.*, "CARNAUBA: The Coherent X-Ray Nanoprobe Beamline for the Brazilian Synchrotron SIRIUS/LNLS," in *Journal of Physics: Conference Series*, vol. 849, no. 1, p. 012057, 2017. doi:10.1088/1742-6596/849/1/012057

[5] L. Sanfelici *et al.*, "Solutions for the SIRIUS' beamlines in a nutshell," *AIP Conference Proceedings*, vol. 2054, p. 030033, 2019. doi:10.1063/1.5084596

[6] G. B. Z. L. Moreno *et al.*, "Exactly-constrained KB Mirrors for Sirius/LNLS Beamlines: Design and Commissioning of the TARUMÃ Station Nanofocusing Optics at CARNAÚBA Beamline," presented at MEDSI2020, Chicago, IL, USA, Jul. 2021, paper TUOB01, this conference.

[7] M. Odstrcil *et al.*, "Ab initio nonrigid X-ray nanotomography," *Nat. Commun.*, vol. 10, no. 1, p. 2600, 2019. doi:10.1038/s41467-019-10670-7