ISBN: 978-3-95450-229-5 ISSN: 2673-5520 doi:10.18429/JACoW-MEDSI2020-WEPB13

DESIGN AND COMMISSIONING OF THE TARUMÃ STATION AT THE CARNAÚBA BEAMLINE AT SIRIUS/LNLS

R. R. Geraldes†[1], C. S. N. C. Bueno, L. G. Capovilla, F. R. Lena, D. Galante, L. C. Guedes, L. M. Kofukuda, G. N. Kontogiorgos, S. A. L. Luiz, G. B. Z. L. Moreno, I. T. Neckel, C. A. Pérez, A. C. Piccino Neto, A. C. Pinto, C. Sato, J. L. Silva, A. P. S. Sotero, V. C. Teixeira, H. C. N. Tolentino, W. H. Wilendorf, Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM), 13083-970, Campinas, São Paulo, Brazil
[1]also at the Eindhoven University of Technology (TUe), 5612AZ Eindhoven, The Netherlands

Abstract

TARUMÃ, the sub-microprobe station at CARNAÚBA (Coherent X-Ray Nanoprobe Beamline) at Sirius at the Brazilian Synchrotron Light Laboratory (LNLS), has been designed to allow for simultaneous multi-analytical X-ray techniques both in 2D and 3D. A systemic approach, heavily based on precision engineering concepts and predictive design, has been adopted for first-time-right development, effectively achieving all-together: the alignment and stability requirements of the large KB mirrors with respect to the beam and to the sample; and the nanometer-level positioning, flyscan, tomographic and setup modularity requirements of the samples. This work presents the overall station architecture, the key aspects of its main components, and the first commissioning results.

INTRODUCTION

CARNAÚBA (Coherent X-Ray Nanoprobe Beamline) [1] belongs to the first set of beamlines of Sirius [2], the 4th-generation synchrotron light source at the Brazilian Synchrotron Light Laboratory (LNLS). It is meant for simultaneous multi-analytical X-ray techniques in 2D and 3D, with diffraction (XRD), spectroscopy (XAS), fluorescence (XRF), luminescence (XEOL) and ptychographic coherent diffractive imaging (ptycho-CDI). It is based on an all-achromatic optical design for the energy range from 2.05 to 15 keV, granting a flux up to 1e11 ph/s/100mA at the probe for high-throughput experiments with flyscans.

TARUMÃ is its sub-microprobe station, located at 136 m from the undulator source, in a satellite building, and reaching fully-coherent monochromatic beam sizes from 550 to 120 nm after the achromatic KB (Kirkpatrick-Baez) focusing optics. In addition to the multiple techniques available at TARUMÃ, a large working distance of 440 mm after the ultra-high vacuum (UHV) KB system allows for another key aspect of this station, namely, a broad range of decoupled and independent sample environments. Indeed, modular setups outside vacuum allow for *in situ*, *in operando*, cryogenic and/or *in vivo* experiments, covering research areas in biology, chemistry, physics, geophysics, agriculture, environment and energy.

SAPOTI will be the in-vacuum nanoprobe station at CARNAÚBA, located at 145 m from the undulator source. With an innovative manipulator developed in collaboration with MI-Partners, operation is expected by 2022.

† renan.geraldes@lnls.br

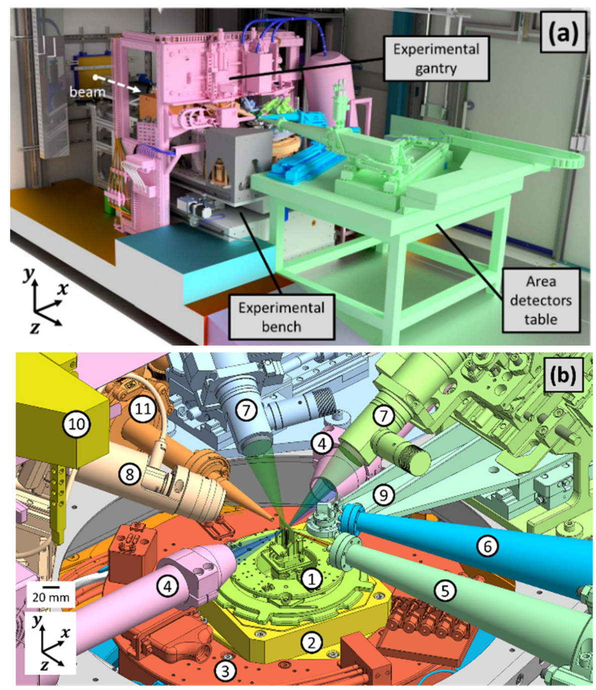

Figure 1: (a) TARUMÃ station at the experimental hutch. (b) Drawing of the region surrounding the sample, detailing: the sample setup (1), the sample stage (2 and 3), the fluorescence detectors (4), the flying paths for the transmission (5) and the diffraction (6) area detectors, the optical microscopes (7), the XEOL optics (8), a crystal analyzer spectrometer (9), the pick-and-place gripper (10), and the KB vessel exit port (11).

OVERVIEW

Figure 1 shows an overview of the TARUMÃ station. The experimental bench hosts: the high sensitivity elements, i.e., the KB vessel and the sample stage; and essential auxiliary elements, such as a complementary metrology frame and an auxiliary rotary stage for cable management (see Fig. 3), and an auxiliary table holding two optical microscopes, the XEOL lenses and a crystal analyzer. A separate table is used for two Medipix-based area detectors with pixel size of 55x55 μm^2: a PiMEGA, with 1536x1536 pixels at the working distance of 1.1 m, and a MobiPix, with 512x512 pixels at 0.44 m. These detectors can be alternated between transmission and diffraction positions, and the MobiPix can still be oriented towards the crystal analyzer at the distance of 0.42 m to work as a spectrometer

ISBN: 978-3-95450-229-5 ISSN: 2673-5520 doi:10.18429/JACoW-MEDSI2020-WEPB13

for higher-resolution fluorescence data. Finally, an experimental gantry is placed over the experimental bench, holding heavier or noisier instrumentation, and allowing for additional accessibility. Among these items are two fluorescence detectors, which have fan-based cooling systems, a cryojet system, and a pick-and-place gripper.

In addition to multiple techniques, TARUMÃ is open to a variety of setups on the sample stage, ranging from macroscopic holders for fossils and plants to microscopy holders, such as grids, silicon nitrite membranes and chips. Many of these setups bring embedded functionalities, such as infrastructure for liquids, gases or vacuum, as well as heating and cooling. More details on an open-atmosphere cryogenic setup can be found in [3], whereas the setups developed for microfluidics and electrochemistry in [4].

SYSTEM ARCHITECTURE

TARUMÃ specifications had the ambitious target of offering a rich infrastructure for multiple techniques and scientific cases, together with tomography and fast positioning for flyscan, while preserving the stability levels within a few tens of nanometers. Thus, the project has been fully developed in-house, according to a systemic perspective for the broadest possible range of present and future experiments, while following a predictive design approach in applying precision engineering mechatronic solutions.

The core concepts in TARUMÃ can be extracted from the schematic of its experimental bench in Fig. 2. The main aspect driving its mechanical architecture is the position sensitivity of the components in multiple degrees of freedom (DoFs) regarding keeping the beam variation at the sample within a fraction of its size, or better, for ptychography. Indeed, as detailed in [5], the numbers between the KB mirrors and both the source and the sample are in the order of 10 nm and 10 nrad only. On the one hand, part of the relative stability between the KB mirrors and the source (or virtual source) depends both on the motion of the floor along the multiple slabs in such a long beamline and on the stability of the intermediate optical components. All these aspects are carefully assessed in [6]. On the other, in what concerns the station itself, the critical stability of the KB mirrors with respect both to the floor in the station and to the sample is addressed precisely within the experimental bench, while complementary components are pushed as much as possible to the auxiliary separate structures, i.e., the experimental gantry and the area detectors table.

Firstly, the design of bench followed the concepts developed for Sirius optical systems [7]. The *bottom granite* is directly grouted to the floor, reaching measured suspension frequencies beyond 300 Hz. Above it, a *top granite* sits on four high-stiffness levellers for positioning in the *y*, *Rx* and *Rz* axes with respect to the beam. Its measured suspension frequencies exceed 150 Hz, which is sufficient to prevent the amplification of most of the content of floor vibrations. Moreover, considering that direct metrology between the KB mirrors in-vacuum and the sample in its complex setups would be practically impossible, the top granite effectively works as a sturdy common reference frame between the mirrors and the sample.

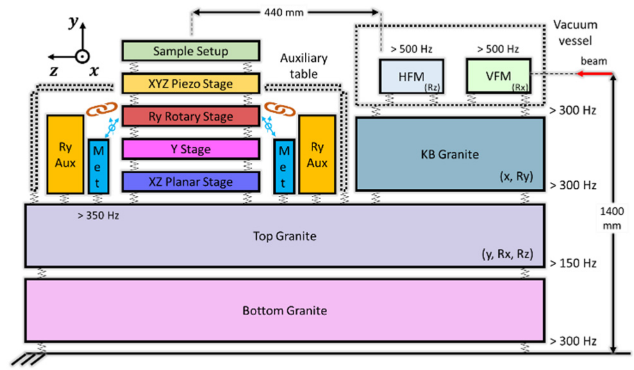

Figure 2: Schematic of experimental bench of TARUMÃ, highlighting some of the links and the dynamic characteristics of the main components, including: the granite parts, the KB mirrors (VFM and HFM), the sample stage, the auxiliary rotary stage and the metrology frame.

Regarding the KB mirrors, the *KB granite* is moveable in the *x* and *Ry* axes with respect to the top granite to provide the remaining DoFs for positioning the mirrors with respect to the beam. This follows a combination of embedded and commercial air-bearing [7], such that suspension above 300 Hz is also obtained once the air is off. Finally, the mirror mechanics is stiffly connected to the vacuum vessel bottom, which, in turn, is stiffly mounted to the KB granite, concluding the stiffness chain for the optics.

As described in [5], the design of Sirius mirror systems [8] has been further improved for the KB mirrors, such that the extreme figure quality can be preserved together with mechanical resonances beyond 500 Hz to reduce the sensitivity to disturbance sources. A minimum set of alignment fixtures, namely, one DoF in pitch (Rx) in the *vertical-focusing mirror* (VFM) and one DoF in roll (Rz) in the *horizontal-focusing mirror* (HFM), is implemented for fine tuning at the beamline. The remaining alignment targets between mirrors are achieved with novel offline alignment strategies (see [5]). With this concept, the dynamics of the mirrors with respect to the floor and, particularly, to the top granite can be kept within a few nanometers, whereas thermal drifts are minimized due to the thermal properties of granite, thermal management in vacuum, and the temperature stability of the air-conditioning system in the hutch.

Regarding the *sample stage*, shown in Fig. 3, it consists of a stack of commercial stages in air to position the sample with respect to the focal spot. It is formed by: an Aerotech's PlanarDL300XY with linear motors for the *x* and *z*; a Newport's IDL280-Z20 wedge stage with a DC motor for *y*; an Aerotech's ABRS-250MP air-bearing rotary stage with a direct drive for *Ry*; and a PI's P-563 piezo stage for fast and fine position in the *x*, *y* and *z*. Optionally, for ptycho-Bragg-CDI, Attocube's ANGp101 and ANGt101 piezo cradles can be mounted to the XYZ stage. The dynamics of the commercial stages have been measured and modelled to validate their consonance with the application, whereas all the interfaces were carefully designed. Moreover, except for the cradles, all the driving systems were selected for smooth actuation, i.e., preventing steppers, to minimize disturbances in flyscan operation.

ISBN: 978-3-95450-229-5 ISSN: 2673-5520 doi:10.18429/JACoW-MEDSI2020-WEPB13

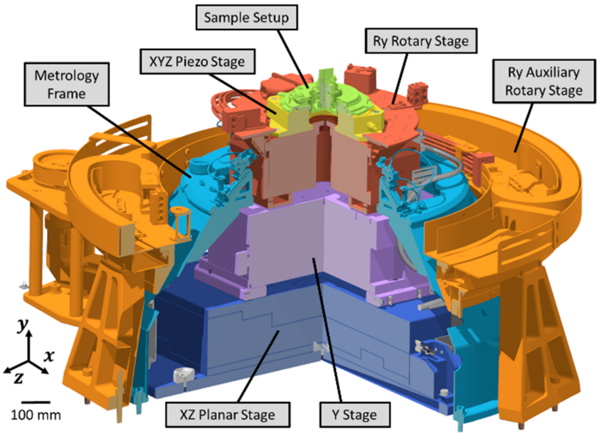

Figure 3: Quarter section view with the sample stage, the metrology frame and the auxiliary rotary stage in the experimental bench of TARUMÃ.

Still, as the position accuracy of the sample with respect to the top granite is limited to the basic metrology in the stages and the passive stability of the non-controlled DoFs over a large stiffness chain, a complementary metrology frame was implemented. With measured dynamics above 350 Hz, it has capacitive probes pointing to the nominal sample position in Abbé, over a conical target fixed to the rotor of the rotary stage, such that information in the nanometer level in the low- and mid-frequency ranges can be used in post-processing algorithms if desired. The sample setups must be designed for maximum dynamics.

Finally, to preserve the accuracy of the stages and the dynamics of the sample, while allowing for the infrastructure of electrical signals and fluids to the sample setups, an auxiliary rotary stage is electronically coupled with the air-bearing rotary stage over a functional range of $\pm$ 110°. Thus, the auxiliary stage takes the burden of carrying the cables and tubes, whereas only a pre-determined compliant link exists between the stages. At the top of the rotary stage, electrical panels and manifolds are used for distribution.

Naturally, the dynamics of the auxiliary table, with the microscopes, the XEOL set and the crystal analyzer, are also of concern. Yet, this partly filtered by the inertia of granite bench, with negligible expected impact in the sample position. Due to the limited space here, more details on the gantry, the detector table and complementary resources will be left for a future publication. Similarly, the control architecture that allows for the integration and synchronization of the several stages and detectors in the sub-millisecond range and the strategies for efficient flyscan experiments will be addressed in [9].

COMMISSIONING

Since first light, commissioning at CARNAÚBA has been ongoing for about six months. In addition to the TARUMÃ station, this includes first use, validation and optimization of several instruments that were designed by Sirius engineering teams and built in collaboration with partners, including: the cryogenic mirror systems [8,10], a four-bounce monochromator [11], innovative instruments for diagnostics [12], and even the area detectors.

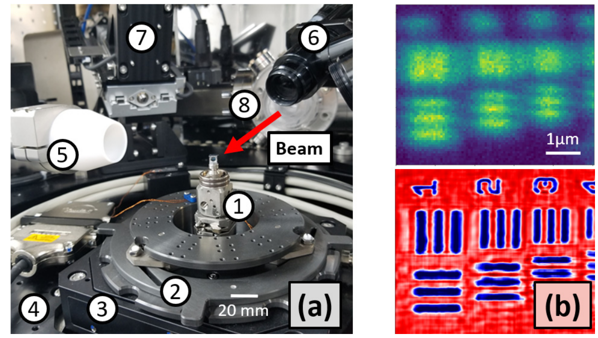

Figure 4: (a) Sample region at TARUMÃ, with: sample holder (1); interface plate (2) on the XYZ stage (3), rotary stage with electrical and fluid lines (4); fluorescence detector (5); optical microscope (6); luminescence stage (7); and KB vessel (8). (b) Simultaneous 3-minute 5x5µm^2 flyscan measurements of X-ray fluorescence (top) and ptychography (bottom).

At TARUMÃ, all the functionalities have been gradually implemented and validated. There are more than 50 motion axes and a dozen detectors. They are integrated in EPICS, but many also linked via hardware for flyscans. Figure 4 shows the setup and preliminary 3-minute flyscan results for 5x5µm^2 of a standard sample, comparing Au fluorescence and ptychography, with spatial resolutions around 800 x 300 nm^2 and 200 x 70 nm^2, respectively. Now, significant improvements are still expected for the near future, once: the fast orbit feedback correction is implemented in the storage ring; further optimization in alignment and residual instabilities in the primary optics is achieved; and the control in the station and the reconstruction algorithms reach maturity. Finally, preliminary tomography experiments have already been realized, and most of the special environments and sample setups are in commissioning.

CONCLUSIONS

TARUMÃ has been fully designed and built in-house, according to the same precision engineering concepts and predictive design principles that have led to a whole series of innovative instruments for Sirius beamlines. A complex sceneario results from the achromatic optics, with large KB mirrors and working distances, vacuum-air separation, and multiple techniques and setups, but the successful initial results build confidence in the current workflow, at the same time that bring valuable lessons for the ongoing and forthcoming projects. The unique features of TARUMÃ are expected to be open for users in the second half of 2021.

ACKNOWLEDGEMENTS

The authors would like to gratefully acknowledge the funding by the Brazilian Ministry of Science, Technology and Innovation, the contributions of the LNLS team and partners, and the participation of the MI-Partners in the SAPOTI project, with fruitful feedback to TARUMÃ.

ISBN: 978-3-95450-229-5 ISSN: 2673-5520 doi:10.18429/JACoW-MEDSI2020-WEPB13

REFERENCES

[1] H.C.N. Tolentino *et al.*, "CARNAÚBA: The coherent x-ray nanoprobe beamline for the Brazilian Synchrotron SIRIUS/LNLS", *J. Phys. Conf. Ser.*, vol. 849, p. 012057, 2017. https://doi.org/10.1088/1742-6596/849/1/012057

[2] Sirius Project, https://www.lnls.cnpem.br/sirius-en/

[3] F.R. Lena *et al.*, "A cryogenic sample environment for the TARUMÃ station at the CARNAÚBA beamline at Sirius/LNLS", presented at the 11th Int. Conf. on Mechanical Engineering Design of Synchrotron Radiation Equipment and Instrumentation (MEDSI2020), Chicago, IL, USA, virtual conference, July 2021, paper WEPC02, this conference.

[4] W.H. Wilendorf *et al.*, "Electrochemistry and microfluidic environments for the TARUMÃ station at the CARNAÚBA beamline at Sirius/LNLS", presented at the 11th Int. Conf. on Mechanical Engineering Design of Synchrotron Radiation Equipment and Instrumentation (MEDSI2020), Chicago, IL, USA, virtual conference, July 2021, paper WEPC03, this conference.

[5] 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 the 11th Int. Conf. on Mechanical Engineering Design of Synchrotron Radiation Equipment and Instrumentation (MEDSI2020), Chicago, IL, USA, virtual conference, July 2021, paper TUOB01, this conference.

[6] C.S.N.C. Bueno *et al.*, "Vibration assessment at the CARNAÚBA beamline at Sirius/LNLS", presented at the 11th Int. Conf. on Mechanical Engineering Design of Synchrotron Radiation Equipment and Instrumentation (MEDSI2020), Chicago, IL, USA, virtual conference, July 2021, paper MOPB08, this conference.

[7] R.R. Geraldes *et al.*, "Granite benches for Sirius x-ray optical systems", in *Proc. of MEDSI'18*, Paris, France, Jun. 2018, pp. 361-364. doi:10.18429/JACoW-MEDSI2018-THPH12

[8] R.R. Geraldes, *et al.*, "The design of exactly-constrained x-ray mirrors systems for Sirius", in *Proc. of MEDSI'18*, Paris, France, Jun. 2018, pp. 173-178. doi:10.18429/JACoW-MEDSI2018-WEOAMA04

[9] C.S.N.C. Bueno *et al.*, "Positioning Scanning Solutions at the TARUMÃ Station at the CARNAÚBA Beamline at Sirius/LNLS," to be presented at ICALEPCS 2021, Shanghai, China, 2021.

[10] L.M. Volpe *et al.*, "Installation and commissioning of the exactly-constrained x-ray mirror systems for Sirius/LNLS," presented at the 11th Int. Conf. on Mechanical Engineering Design of Synchrotron Radiation Equipment and Instrumentation (MEDSI2020), Chicago, IL, USA, virtual conference, July 2021, paper MOPB06, this conference.

[11] M. Saveri Silve *et al.*, "Four-bounce crystal monochromators for the Sirius/LNLS beamlines," presented at the 11th Int. Conf. on Mechanical Engineering Design of Synchrotron Radiation Equipment and Instrumentation (MEDSI2020), Chicago, IL, USA, virtual conference, July 2021, paper MOPB04, this conference.

[12] H.C.N. Tolentino *et al.*, "Innovative instruments based on cryogenically cooled silicon crystals for the CARNAÚBA beamline at Sirius-LNLS," *AIP Conf. Proc.*, vol. 2054, p. 060026, 2019. https://doi.org/10.1063/1.5084657