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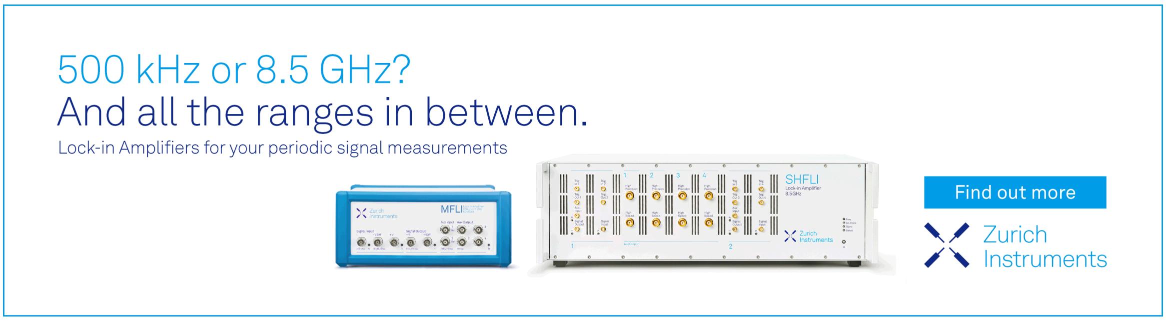

Innovative instruments based on cryogenically cooled silicon crystals for the CARNAÚBA beamline at Sirius-LNLS

Hélio C.N. Tolentino[1, 2, a)], Márcio M. Soares[1], Francisco M.C. Silva[1, 3], João H. Rezende[1], Denise Puglia[1], Anderson Bordin[1], Marlon S. Silva[1] and Renan R. Geraldes[1]

[1]*Laboratório Nacional de Luz Síncrotron, LNLS, CNPEM, CP 6192,13085-970 Campinas, Brazil.*
[2]*Université de Grenoble Alpes and CNRS, Institut Néel, BP 166, 38042 Grenoble, France.*
[3]*Instituto de Física Gleb Wataghin, UNICAMP, 13083-970 Campinas, Brazil*

a) Corresponding author: helio.tolentino@lnls.br

Abstract. The CARNAÚBA beamline is the tender-to-hard X-ray nanoprobe under construction for the new source Sirius at the Brazilian Synchrotron Light Laboratory (LNLS). The all achromatic optics relies on KB mirrors and a horizontal secondary source aperture (SSA) to reach beam size down to ~30x30 nm^2 at the sample position. To handle the power on the optical elements the choice has been to build instruments based on cryogenically cooled Si crystals. These optical elements – X-ray diagnostic, primary mirrors, secondary source aperture and monochromator – and expected performance are described here.

INTRODUCTION

The CARNAÚBA (Coherent X-Ray NAnoprobe Beamline) beamline [1] provides two separated experimental stations, one with sub-micrometer resolution and another with nanometer resolution, to cover various analysis techniques, including XRD, XAS, XRF, XEOL and CDI with 2D and 3D imaging capabilities exploring the coherent properties of the Sirius X-ray beam [2, 3]. An innovative modified-Delta undulator is optimized to provide photons with vertical polarization allowing for scattering in the horizontal plane from the source up to the end-stations, without spoiling flux in the tender X-ray range due to the polarization factor. An in-house developed horizontal deflection four-bounce crystal monochromator (4CM) covers continuously the energy range from 2.05 to 15 keV, with a resolution of $\Delta E/E=10^{-4}$. The all achromatic CARNAÚBA optics, based on a primary mirror that creates a horizontal secondary source and on state-of-the-art KB mirrors, delivers an unprecedent coherent flux @Si(111)BW close to 10^{+11} – 10^{+12} ph/s/100mA. The focus is nearly diffraction limited in the whole energy range: at the microprobe end-station the optics delivers a focus of 500 nm at 2.05 keV down to 100 nm above 8 keV, limited by a numerical aperture (2NA) of about 1.0 mrad; at the nanoprobe end-station the focus is around 120 nm at 2.05 keV and 30 nm above 8 keV, with 2NA≈5 mrad. The nanoprobe station has the KB optics and sample environment in vacuum with a working distance of about 50 mm. A cryogenic sample holder and transfer system is under development. The innovative high-dynamic actuation on the vertical and horizontal KB mirrors provides a fast-scanning capability in the 10 to 100 Hz range for fast fly-scan operation. We estimate collecting a full 100x100 ptychographic image in tens of seconds. On the other hand, the microprobe will have a more flexible sample environment and a much larger working distance, with nearly 350 mm from the end of the horizontal mirror to the sample stage. The UHV environment of the KB mirrors is separated from the sample environment, whose holder is not in vacuum. Both end-stations will cover a large variety of scientific areas ranging from environmental, geophysical, agricultural, biological research to energy and more condensed matter related areas.

In this contribution, the CARNAÚBA design and construction progress is reported. Many pieces of instrumentation have been developed at the LNLS and are under commissioning stage. Among the most relevant, a

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modified Delta undulator (DU21), an X-ray diagnostic for the undulator (XDU), a cylindrical fixed-curvature mirror (MH1) to create the secondary source at the horizontal, a secondary source aperture (SSA) working under the focused pink beam, a four-bounce crystal monochromator (4CM) scattering in the horizontal and finally a prototype sample manipulation system developed for the microprobe. The nanoprobe is at the conceptual design stage.

The beamline components detailed here are highlighted in the layout optics, represented schematically in Fig.1. From the primary optics to the monochromator the optical elements come in pairs along the beamline. The X-ray diagnostic (XDU) has been accommodated in the same chamber as the first mirror (MH1), at 27 m from the source, in the primary optic hutch. This side-bounce cylindrical mirror (MH1) creates the secondary source in the horizontal in a 1:1 ratio. In the second optical hutch, the SSA and the second side-bounce flat mirror (MH2) are also sharing a common chamber. The SSA, at 54 m, defines the source size horizontally and the MH2 deflects back the beam, completing the double-mirror set-up with MH1. Finally, the beam reaches the 4CM, at 130 m, composed by two side-bounce Si(111) channel-cut crystals, before being focused by KB mirrors at one of the two experimental stations. Up to the monochromator, all mentioned elements are cryogenically cooled to properly handle the power load. The KB mirrors receive monochromatic light and are not cooled, being the first mirror of each KB pair the only vertically deflecting optical component of the whole beamline (Fig.1).

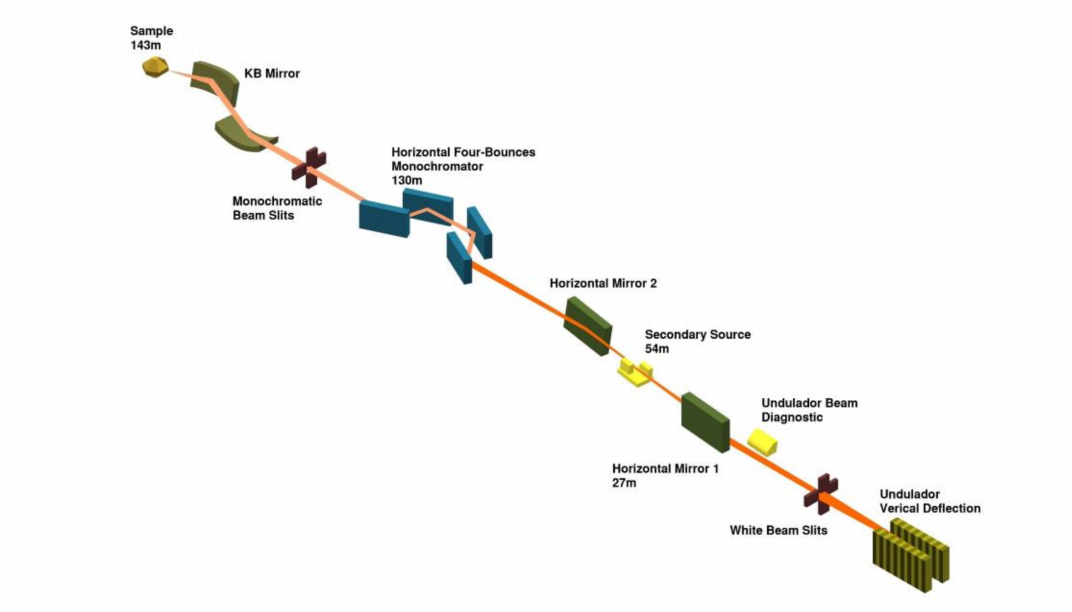


FIGURE 1. Schematic layout of the CARNAÚBA beamline, highlighting the cryogenically cooled optical elements: X-ray Diagnostic for the Undulator (XDU), Horizontal (cylindrically curved) Mirror (MH1), Secondary Source Aperture (SSA), Horizontal (flat) Mirror (MH2) and four-bounce crystal monochromator (4CM).

CRYOGENICALLY COOLED OPTICAL ELEMENTS

The power management on all optical elements, from the diagnostic to the monochromator, has been based on indirect cryogenically cooled silicon crystals. This power management strategy has been applied to almost all SIRIUS mirrors, whose detailed concept and mechanism, as well as preliminary characterization, is being presented elsewhere [4]. Cryogenically cooled silicon crystals are widely employed in CARNAÚBA optics because of their unusual thermal conduction and expansion properties. Moderate power conditions allow to keep temperatures around 125 K (or at least below 150 K), where the thermal expansion is close to zero and silicon is better than copper for heat dissipation. The optic design taking care of all vibrational and thermal conditions, as well as alignment and commissioning procedures, are presented in a separated paper with the preliminary metrology results [5]. Here, we focus on some specificities related to the CARNAÚBA mirrors and report on all cryocooled optical elements specifications and expected performance.

The CARNAÚBA X-ray source is a modified Delta undulator with 115 periods of 21 mm (DU21) and deflection parameter K=2.2 (B=1.2 T). A prototype of this undulator has been assembled and commissioned on its mechanical parts and control, but a fine magnet characterization has not been done, yet. The power it delivers, in cascade, on each optical element is given in Table 1, with the SIRIUS storage ring working at 3.0 GeV and 350 mA.

From the total emitted radiation, only 60x60 urad^2 are selected by the front-end slits, delivering a total power of 136 W to the beamline. White beam slits (WBS), just in front of the optics, as well as the XDU, are designed to stand such power when the WBS is completely closed or open, respectively. The expected standard beamline operation would select a divergence of 40x40 urad2 with the WBS, which corresponds to a total delivered power of 61 W to the optics downstream the WBS. The total power (delivered to the element and absorbed by it) and the power density is calculated for the worst case of angle of incidence (≈5 mrad) and mirror coating selection (Table 1). These power conditions are moderate but are impeditive for simply using water cooling. On the other hand, direct nitrogen cooling or high power cryo-coolers would lead to unnecessary costs and would introduce complications to the design, affecting performance in terms of stability and maintenance. After simulations, using finite element analysis on optical elements [5], the envisaged solution was to use standard cryostats and optimize indirect cryocooling using cooper braids connected to silicon crystals in all optical devices.

TABLE 1. Total power (delivered to the element/absorbed by the element) and power density on the CARNAÚBA optical elements. The figures are given for normal operating conditions of the beamline: at XDU the WBS is fully open to 60x60 urad2; at other elements WBS is open to 40x40 urad2.

Optical element	Total power (W) (delivered/max.absorbed)	Power density (W/mm2) (Max.absorbed)
WBS: White Beam Slits	136/136	47
XDU: X-ray Diagnostic (w/filter)	136/136	5.6
MH1: First mirror	61/45	0.17
SSA: Secondary Source Aperture	35/35	17
MH2: Second mirror	35/<5	< 0.1
4CM1: 4CM 1st Crystal	7/<10	< 3
4CM2: 4CM 2nd Crystal	< 0.001	< 0.001

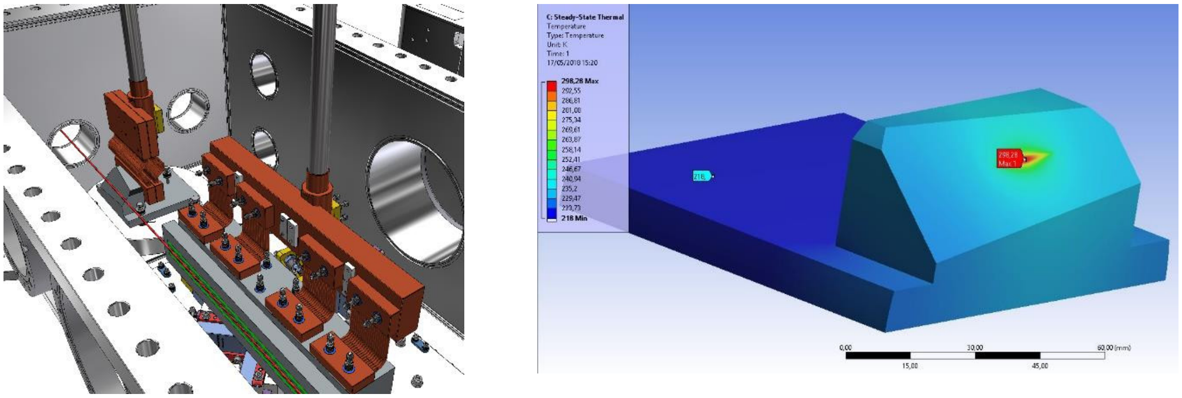
The X-ray Diagnostic and the First Mirror

The design parameters and power management strategy in mirrors is one of the most critical task in a beamline, especially when a nanoprobe is the final target. In the CARNAÚBA beamline, the primary optics employs a side-bounce fixed curvature cylindrical mirror, MH1, to deflect and focus the beam, creating a secondary source in the horizontal with a typical FWHM of 50 µm. MH1 has three stripes, Rh and Ni coatings separated by a stripe of pure silicon. These stripes, selected by displacing vertically the mirror chamber, are employed in different energy ranges to reject higher energy harmonics. The incidence angle is close to 5.0 mrad, but may be adjusted to optimize the focused beam at the SSA. The simple design concept, which has been employed in all SIRIUS side-bounce mirrors with fixed curvature [4], preserves the extreme quality of mirror figures [5] using a deterministic high resolution exactly-constrained flexure based support with a single degree of freedom for the pitch tuning. The adopted cooling strategy was indirect cryocooling via cryostats connected to the silicon crystals through copper braids (Fig.2-a).

The MH1 in CARNAÚBA receives a total power of 61 W in normal operation conditions. In the worst case, when the Si stripe is used, about 73% of the power remains in the substrate, i.e., 45 W (Table 1). Even with such a power condition, it is still possible to handle the power and dissipate it using Cu braids, whose conductivity and interfaces with silicon are the limiting factors. However, as shown in Fig.2-a, MH1 must be connected to the cryostat through four Cu braids, instead of one in the standard design [4]. This is needed because otherwise the Si temperature rises above 150 K, which is above the optimal temperature for thermal conductivity (125 K). Simulations show that with this four-braids design the same cooling strategy as for the other mirrors can be maintained.

The XDU has been inspired in a similar device developed by the instrumentation group at the SOLEIL synchrotron [Desjardins et al, DOI: 10.1109/NSSMIC.2008.4774883]. It is located downstream the white beam slits and accommodated in the same chamber as MH1, upstream to it (Fig.2-a). The device is based on highly asymmetric Si(220) (also Si(440)) reflections at scattering angles of 90° related to the beam direction. The main Si crystallographic axis, [100], is oriented along the beam propagation, while the [010] and [001] are pointing vertically and horizontally, respectively. The face exposed to the beam is oriented along the [011] axis with a small asymmetry of 6°, in such a way that the beam spread along the surface, reducing the power density by a factor of 10 (Fig.2-b). In this geometry, Si(-220) and Si(-202) highly asymmetric Bragg reflections diffract at 90° in relation to the incoming beam and along the vertical and horizontal planes, respectively. This device can then work as a polarimeter.

When the XDU absorbs the total delivered power, the crystal, after simulations, may reach temperatures as high as 298 K at the hot spot (Fig.2-b). Even if the silicon can stand such gradients, a 300-μm diamond filter is planned when performing beam diagnostics to reduce the power from 136 W down to about 90 W. The scattered beams will be recovered from the chamber through Be windows and collected by area detectors. This device is intended for beam diagnostics and is inserted to or taken out from the beam by sliding laterally the whole chamber.



(a) (b)

FIGURE 2. (a) Internal view of XDU and MH1 chamber, showing the cooling scheme, with cryostats connected to Cu blocks from above and Cu braids (4 for MH1 and 1 for XDU) connecting the Si crystals (light gray) to the Cu blocks. The red line represents the beam path and green stripes in the mirror corresponds to the coatings. (b) Thermal simulation of XDU when submitted to the total power of 136 W, with temperatures varying from 218 K near the extraction region to 298 K at the hot spot.

The Secondary Source Aperture and the Second Mirror

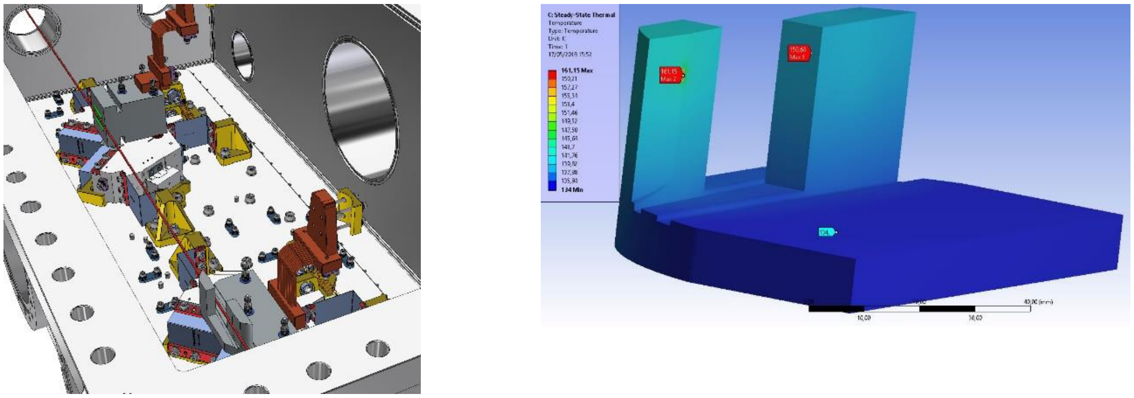
The secondary source aperture (SSA) and the second mirror (MH2) are also sharing a common chamber (Fig.3-a). The SSA selects a beam fraction, modulating the horizontal size of the source to define the probe conditions in terms of size, flux and coherent fraction, and the MH2 bounces back the beam into the original direction.

The SSA is one of the most critical components of the beamline optics, requiring extremely good quality in terms of beam definition and stability in position. The MH1 focus the beam in the horizontal at 54 m from the source, reproducing the size (≈50 μm) of the undulator source, leading to a high-power density (Table 1) which is difficult to handle. As the WBS cuts the vertical divergence to ≈40 μrad, the beam surface (FWHM) at the SSA will be ≈50x2160 μm^2. Then, a precise slit system with variable aperture is needed to select different apertures in the horizontal and control the horizontal nanoprobe size at the experimental station [1]. The SSA stability is then an issue and should be precisely monitored. If the beam moves laterally, one must steer the beam back to the aperture. This might be accomplished by the MH1 pitch control, as far as it has a feedback input. It is then compulsory to have an X-ray beam position monitor (XBPM) at the aperture, or the SSA itself be an XBPM. The SSA device is then a composition of two sub-systems: an accurate and stable slit aperture system and an XBPM in the horizontal. The vertical beam has not to be limited at this position.

In the innovative SSA presented here, both slit and monitoring function are coupled in the same device using a channel-cut silicon crystal (Fig.3-b) in which the crystal faces work as slit blades. The same mechanism adopted for mirrors [4] is employed to hold and rotate the SSA crystal, in such a way that the X-ray beam size is selected by the fine pitch rotation. Each blade of the crystal is apart from the center of rotation by the same amount, resulting in symmetric cutting of the beam. The channel-cut design (Fig.3-b) is shaped with the hexagonal [111] axis pointing along the vertical, while the [0-11] and [2-1-1] are pointing along and perpendicular to the beam propagation, respectively. In this geometry, one has in the horizontal plane the [1-21] axis pointing at 30°, the [-101] at 60° and the [2-1-1] at 90° related to the beam propagation. This gives rise to highly asymmetric Bragg diffraction peaks occurring at scattering angles of 60° and 120°, which corresponds to the energies of 4.57 keV and 9.13 keV, respectively. These diffracted beams can be collected by area detectors and used for XBPM purposes: any lateral position fluctuation will be sensed by the area detectors. The internal mechanism of the SSA provides an aperture from 0 to 300 μm with a resolution better than 0.1 μm, which is well suited for the device.

A prototype of the SSA was characterized at the LNLS and a sensitivity on lateral beam fluctuations of ±1 μm has been demonstrated. The internal crystal faces were coated with a 50-nm layer of Au to make the slit opaquer to hard X-rays. This solution improved significantly the beam sharpness. It may also be used to monitor the total flux through the total electron yield.

The internal faces of the channel-cut have an on-purpose miscut, of -3° in the first and +3° in the second slab, for grazing incidence purpose, spreading the beam along the exposed face by a factor of 20, and reducing the power density on each slab. Simulations for the worst possible situation (Table 1), with the SSA closed and/or the focused beam reaching one single slab, shows that the maximum temperature attained in the hot spot (Fig.3-b) is 161 K, with the base of the crystal kept at 134 K. These results demonstrate that the same strategy based on the mirrors indirect cooling is well suited to the SSA.



(a) (b)

FIGURE 3. (a) Internal view of SSA and MH2 chamber, showing similar cooling scheme for both components. The mechanism for holding and rotating both components have the same design and allows to tune the horizontal beam size at the SSA and deflect the beam in horizontal with MH2. (b) Thermal simulation of SSA channel-cut crystal when the beam is completely blocked with a single blade, reaching 161 K at the hot spot and 134 K at the base.

As far as the second mirror, MH2, is concerned, the total power is very low [Table 1] and different strategies could be adopted. However, we kept the same strategy for the sake of simplicity and maintenance. In fact, this was the main reason to associate SSA and MH2 together in the same chamber because they can share the same cryogenic set-up.

The First and the Second channel-cut crystals for the 4CM monochromator

The 4CM is a mirrored two-bounce system diffracting in the horizontal plane. The important point here is that the concept employs two channel-cut crystals, where at least the first and second bounces, and the third and fourth ones, are supposed to be perfectly aligned. Si single-crystals are required to cover the energy range from 2.05 up to 15 keV with an intrinsic energy resolution of $1.0\text{x}10^{-4}$. Each goniometer holding a channel-cut scans from 8° to 75° with a resolution better than 1 μrad. The 4CM is designed to reach the -5° and 90° angles for alignment purposes and, in addition, to be shifted laterally to let the pink beam go through.

The power and power density on the first slab have been limited cutting the divergence to 20x20 μrad^2. The beam reaches the first channel-cut crystal (4CM1) with total power below 7 W and size of 1.5x2.6 mm^2. For almost normal incidence, the power density is smaller than 2.5 W/mm2. To handle this power, the same strategy using cryostat has been adopted, where both crystals are connected to the same cold finger. Figure 4 shows the cryogenic set-up for the 4CM, with the channel-cut crystals connected by copper braids to the cryostat. A similar approach was adopted for handling 20 W in an artificial channel-cut monochromator at the XFEL [7]. Our thermal simulations have shown that lattice parameter gradients are not an issue, as far as the temperature stays around 125 K. Temperature gradients up to 10 K are acceptable within slabs and crystals because they would give rise to angular differences of less than 1 μrad.

Another important issue with the 4CM is that the crystal design should be easy to machine and polish, with internal faces are extremely accessible for polishing (Fig.4-b). The gap, set to be the nominal 8.0 mm, can be easily corrected to match the same value on both crystals. The crystals are firmly attached to the holder using a flexure-based solution that decouples thermally crystal and holder and helps to reduce stresses and deformations, like the adopted solution for the LNLS HD-DCM monochromator [8,9].

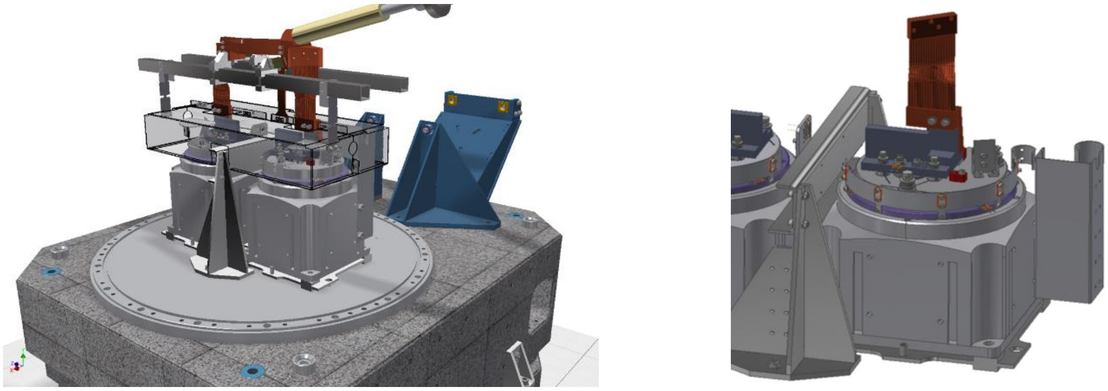

(a) (b)

FIGURE 4. General view of the 4CM setup including cooling scheme and rotation stages. (b) Detail of channel-cut crystal mounted on rotation stage with flexure holder optimized avoid thermal loses and to accommodate deformations.

FINAL REMARKS

The CARNAÚBA beamline has been designed based on all achromatic mirror optics and a four-bounces Si monochromator that allows for high resolution spectroscopy, scattering and imaging applications over the range of 2.05 to 15 keV. To handle the moderate power conditions on all optical elements, a unique solution based on indirect cryogenic cooling has been adopted. The adopted solution is simple and eliminate vibrations that could be introduced by liquid nitrogen or water flows through the mechanism. Most of the elements presented here have already been tested, at least separately, and should cope with the required stability of the nanoprobe.

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