SPHINX

Structure probing by holographic imaging at nanometer scale with X-ray lasers

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1. SUMMARY

The SPHINX project aims generating femtosecond-exposure X-ray holograms of microscopic samples and of their internal parts with nanometer resolution. The application is based on a new implementation of the phase-contrast holography that overcomes the main limitations encountered in the current systems (most based on absorption-contrast), namely the low energy range, the limited detector granularity and the weak illumination. The combination of polycapillary lenses, large X-Ray CCD arrays with small pixel size and XFEL sources allows splitting the beam, focusing, magnification and phase-contrast imaging in the keV energy range. Due to the wavelength-dependent optical behavior of the samples, the refractive diffraction reduces the diffraction limit together with a fast drop of the characteristic angles, both essential for the resolving power (given the limited X-ray detector pitch), while also eliminating the shadow effect and giving access to full structure probing. The key parameters are defined by the focusing optics, which could be, according to the beam and sample sizes, a polycapillary micro semi-lens or a combination of the former with a parabolic monocapillary. The advantage of "non-perfect" optics (not providing a point-like focus) is their divergence, driven mostly by the single waveguide. This allows sending on the same detector area the diverging object and reference beams (Fresnel configuration), both provided by a single optical element, condition unreachable with a Fresnel lens or a crystal mirror. Moreover, the femtosecond exposure time on X-FELs permits holographic reconstruction of in-vivo cell elements, viruses and nanorobotic devices during ultrafast molecular processes, yet unexplored by imaging techniques. The first tests, targeting the proof-of-concept phase, where resolution and stability constraints are relaxed, will be performed on coherent synchrotron lines.

2. ACTIVITY

The SPHINX group activity in 2025 was centered on two objectives: the detector and optics setup completion and the preparation of the assigned run at DIAMOND synchrotron, in UK.

2.1 SPHINX setup

In order to reconstruct a holographic image acquired by an array of 16 X-ray CCD sensors, all the pixels belonging to different detectors (mounted with an interspace required by ceramics and cryogenics), have to be assigned to an unique coordinate system, with a precision better than the pixel size (24 um).

Therefore, two positioning systems have been developed: a visible light LASER setup at 450 nm, equipped with a Diffractive Optical Element (DOE), creating a pattern of 100 spots (10x10), to be used in the laboratory alignment procedure and an X-ray precision shadow grid, installed in front of the CCD array and equipped with a gliding mechanism driven by an actuator, which allows the insertion and the removal of the grid during synchrotron exposure. The grid was machined with high precision CNC and a robust low thermal coefficient steel structure was designed in order to grant deformations below 10 um under mechanical and thermal stress due to the cryogenic regime. The radiation transfer between the CCD array and the grid was measured in the lab and the operational range was confirmed. The design of the positioning system is presented in Fig. 1.

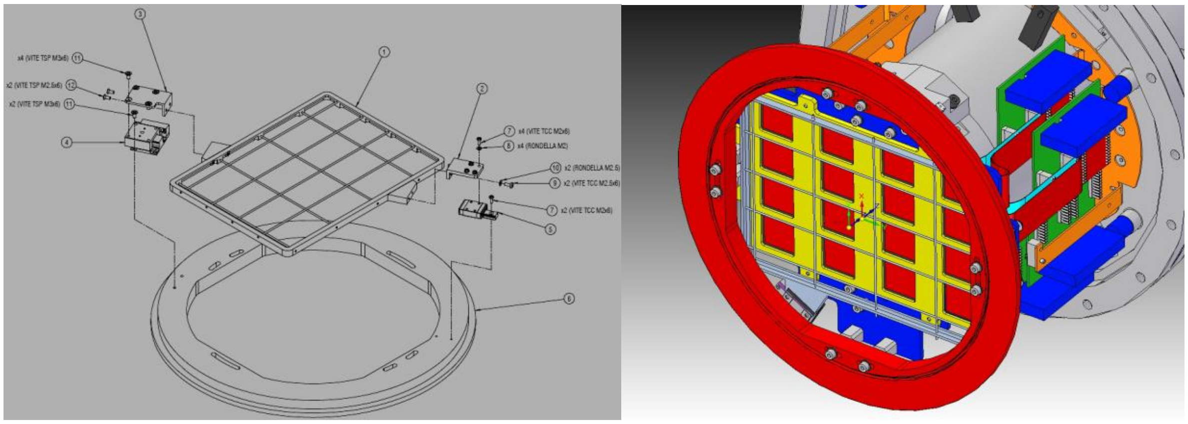

Fig.1 Left: The X-ray alignment grid with the actuator mechanics. Right: Grid placement in front of the CCD array.

For what concerns the optics, two interchangeable polycapillary lenses, realized by the INFN-LNF XLab, were installed, with custom design mountings. The optics is tuned by a five degree of freedom (DOF) mechanism (3 translations and 2 rotations), responsible for the lens positioning in the beam. The optical bench, shown in Fig.2, is controlled by a custom LabView code driving the actuator's interfaces.

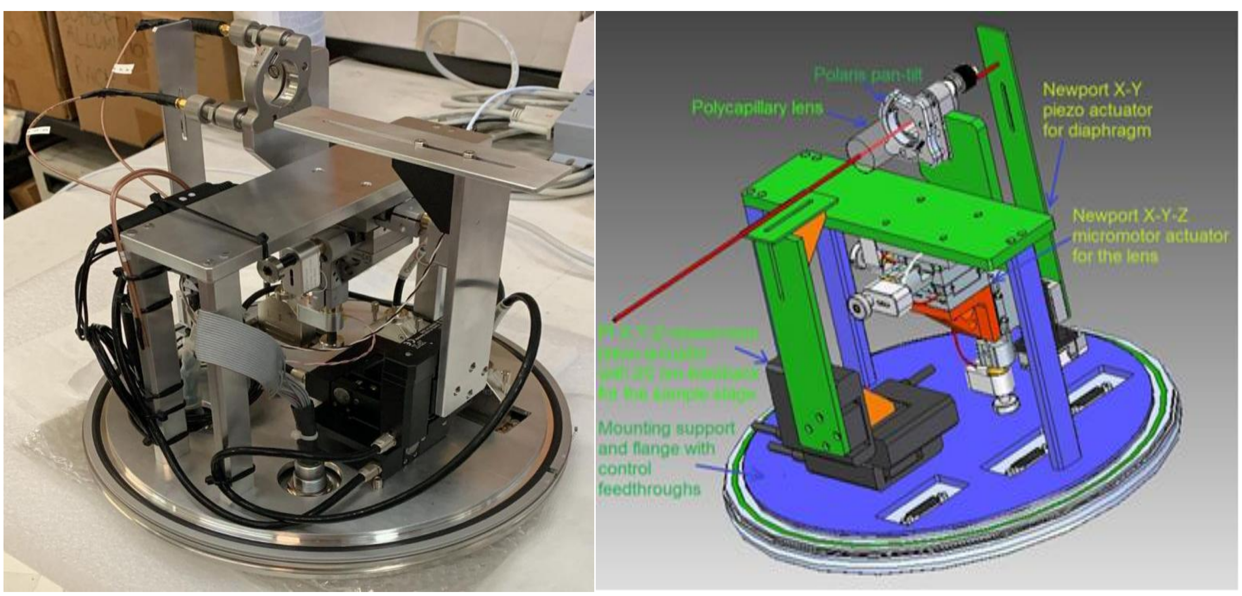

Fig.2. SPHINX optical bench featuring 5 DOF for the lens, 3 DOF for the sample and 2 DOF for diaphragms.

The diaphragm stage, included as auxiliary system, proved to be critical in matching the beam size and the optics. In order to mount a set of three pinholes (1 um for investigating the single capillary, 10 um for selecting a single channel and 50 um for selecting the illumination field) in the limited space of the vacuum chamber and to allow their movement (pinholes supports available were quite big, 12.2 mm diameter), a 10 um precision double face mount was designed and executed (Fig.3).

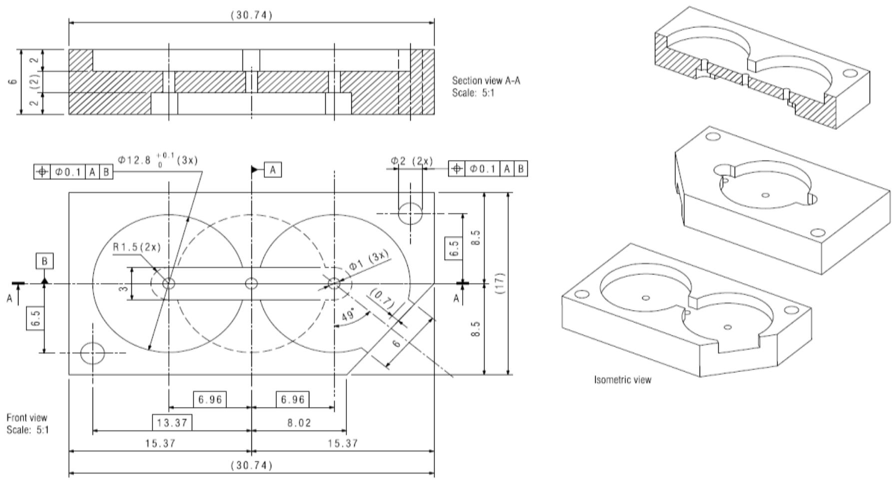

Fig. 3 Diaphragms mount

Monitoring tools: optical camera and sample adjustment X-ray microscope. Even though DIAMOND is well equipped with optical stage imaging tools, two cameras included in the setup proved to be useful. An 8 Mpixel pan-tilt-zoom camera allows near stage placement and fast check of actuators response. An X-ray microscope was custom-made, by installing a 150 um CdWO4 scintillating polished crystal plate in the focal plane of a 5 Mpixel 200x RS-Pro optical microscope, covering the entire field of view. The later was intensively used in the alignment procedures for all the configurations where the beamline X-ray microscope (better quality but bulky) was not possible to insert, which proved to be about half of the cases (Fig 4). The image acquisition software was also written in LabView.

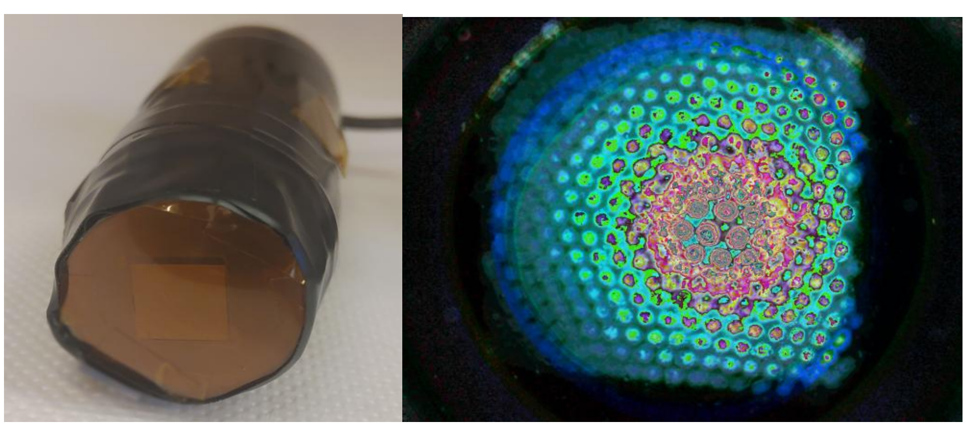

Fig. 4 Left: the X-ray "homemade" microscope. The CdWO4 crystal (10x10x0.15 mm) is visible in the center of the kapton window; right: polycapillary optics, X-ray image obtained with the microscope.

Other hardware upgrade: a new set of CCD drivers (pulse generators with adjustable ramps up-down and amplitude, triggered by TTL control signals) has been produced, to replace the obsolete ones. After the first tests of the setup under cryogenics stressed an unreliable behavior of the CCD connectors at low temperature, new kapton cables with more tight connectors were built, as well. The vacuum pumping system (scroll + turbo), 25 years old, was upgraded, too.

2.2 First DIAMOND synchrotron run

The SPHINX experiment was approved at DIAMOND UK for the fall 2025, receiving a long run period of 5 days. Due to the increased cost of the transportation, it was decided with the DIAMOND team to perform the first tests with the optical bench and controls built by LNF and to use the I13-1 X-Ray detectors instead of the LNF CCD array. This allowed the team to ship to Didcot a single box of ~1 m^3, thus avoiding sending the CCD vaccum chamber and cryogenics, requiring a prohibitive budget. The area and resolution reduction implied was considered a minor issue in the current phase of the experiment, when the proof of concept is aimed for.

Technical challenges and solutions emerging from the run:

-The lens pan-tilt actuator (Polaris) was a mixed manual and piezo controlled mechanism. The piezo actuator, the only available for the given mount at the time, wasn't mobile enough to fully perform the alignment in remote mode, thus requiring a large number of hall entries for manual adjustments. The procedure, repeated about 50 times for each of the two optics installed, took out a significant fraction of beam time (more than 1 day). As a consequence, at the return, the group decided to completely replace the mount with a newer one, now available on the market in the vacuum variant, granting wider remote range.

-The optics, despite having a good efficiency and correct focusing and channel divergence, presented too large capillaries (150 um instead of 15 um), creating a strong, direct central multi-spot, without wall reflections (needed for reaching the divergence), that altered significantly the image SNR. Therefore, the exposures had to be done off-axis, to work under the design parameters. As the focusing follows the optical axis of the lens, tilting it was not an applicable solution and the entire output synchrotron line (14 m) had to be realigned. The solution for the next run will be a new optics with thinner capillaries, agreed with the X-Lab.

-During the realignment attempt, the beamline actuators were found in a tripped state and during a whole weekend (13-14 Sept) the operation was not possible; therefore, the time was dedicated to samples preparation, their checking and positioning in beam using the microscopes and other optics tuning. The actuators were fixed in the morning of Monday, Sept 15 (last morning of run), as soon as the DIAMOND motor team became available.

The remaining exposure time after having aligned the beam, the detector and the beamline under He was very short (~ 4 hours); still, the obtained results were encouraging. The interference pattern radius matched perfectly the capillary X-ray divergence and direction and, more importantly, changed with the insertion of a transparent sample. As the pattern was obtained by making interfere output capillaries, 14 meter away from the stage, shining both directly on the detector and through the sample, the result is a phase contrast hologram (Fig.5).

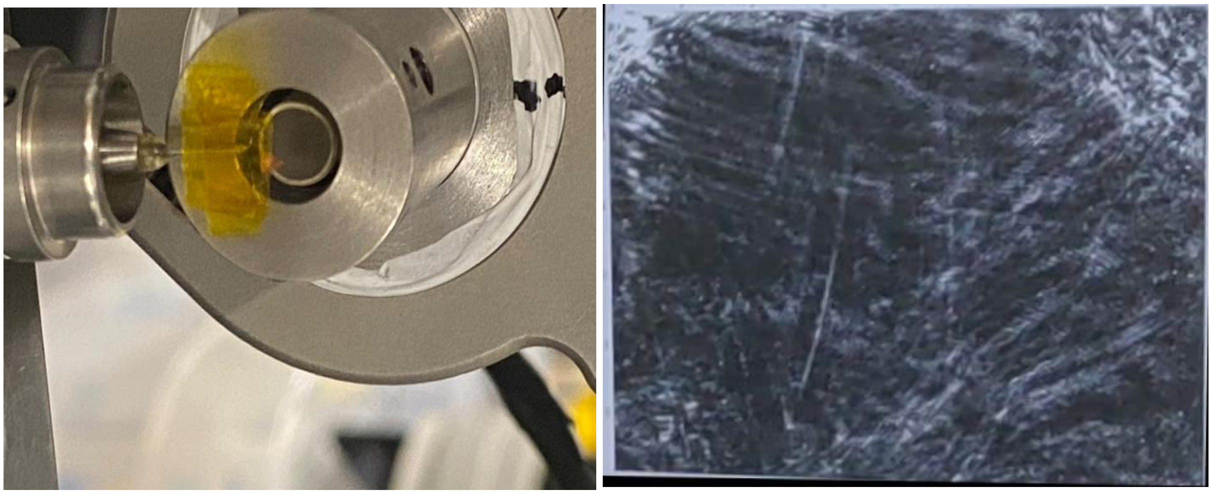

Fig.5. Left: thin transparent triangular mesh (sample) inserted upstream the lens focal plane in front of a fraction of capillaries shining on the detector. Right: interference pattern obtained.

-The beam time left was not sufficient to perform a thorough ptychographic scan of the beam not passing through the sample (the reference beam), mandatory for creating the phase-amplitude map and reconstructing the image. Nevertheless, a rough scan was performed, but the steps were large (15 um) and the detector wasn't shifted to cover entirely the interference image area.

As a conclusion, the run produced very important information on the setup operation and, given this was the first attempt ever to implement the new technique, the results exceeded the expectation. An interference image was obtained by mixing coherent X-ray beams passing through and aside a sample, produced with polycapillary optics and the pattern changed with the sample insertion. The major technical difficulties were identified and solutions were found and implemented for future experimentation. Therefore, the collaboration is looking forward to performing a second data acquisition at DIAMOND in 2026.

3. Pubblications

-M.A. Iliescu, C. Curceanu, F. C. Sirghi, A. Spallone, Structure probing by holographic imaging at nanometer scale with X-ray lasers (SPHINX), Nuclear Instruments and Methods in Physics Research A 1061 (2024) 169083, https://doi.org/10.1016/j.nima.2024.169083

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