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Nanopositioning for the ESRF ID16A Nano-Imaging beamline

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Introduction

New scientific frontiers in biomedicine, materials science and nanotechnology make increasingly use of characterization methods at the mesoscopic and nanometer scale. These studies of heterogeneous samples are most often three-dimensional and require multiple and stable acquisitions, where the spatial and/or angular position of the sample is changed in a well-controlled way. The high average brightness of synchrotron sources, which will be further enhanced by the future diffraction limited storage rings [1], is invaluable in this context. Several nanoscale characterization methods have become available, using highly coherent probes, nanofocused probes, or both [2-8]. However, the required nanopositioning and stability (absence of vibrations and long term drifts) remain huge challenges, especially when the environment of the specimen needs to be controlled. The European Synchrotron has developed a number of specialised nanoprobe endstations covering energies from the tender X-ray regime (the X-ray microscopy beamline ID21 under refurbishment [9]), over moderate X-ray energies (the micro-diffraction imaging beamline ID01 [10], the microfocus beamline ID13 [11], the nano-imaging beamline ID16A [12] and the nano-analysis beamline ID16B [13]), to the high energy regime (the materials science beamline ID11 [14] and the future endstation of the high energy beamline ID31 [15]). Here we develop the specific case of the Nano-Imaging beamline ID16A that provides routine imaging on the nanoscale with a sub 13 nm X-ray beam with a very high flux. At the expense of flexibility, it exploits one of the most advanced nanopositioning endstations. We introduce its specifications, discuss the specific design choices and illustrate the performance with metrology and X-ray experimental results.

Experimental techniques and nanopositioning specifications

Designed for quantitative three dimensional characterization of the morphology and the elemental composition of specimens at the nanoscale, the ID16A beamline of the ESRF produces currently the world's brightest nanofocus. With the endstation located at 185 m from the source, the beamline is optimized for coherent hard X-ray imaging and X-ray fluorescence microscopy. It offers extreme focusing down to 13 nm with a very high photon flux (up to 10^{12} photons/s at $\Delta E/E \sim 1\%$) [12]. The selected energies, 17 keV and 33.6 keV, are well suited for applications in biomedicine, materials science and nanotechnology. Two coherent imaging techniques, X-ray holographic [16] and ptychographic [17] tomography, provide the distribution of the electron density through phase contrast at length scales ranging from ~ 130 nm down to ~ 10 nm, while keeping a relatively large field of view. Complementary, X-ray fluorescence microscopy delivers label-free, highly efficient trace element quantification with detection limits down to subppm level [18]. Additionally, correlative imaging is routinely performed by combining phase contrast and fluorescence microscopy [19].

The instrument attains its unique properties by combining highly performing nanofocusing optics (multilayer coated Kirkpatrick-Baez mirrors [20]) with a carefully designed mechanical device for stable sample positioning and accurate scanning. The fixed curvature KB optics provides a diffraction limited focus of less than 13 nm, a world-record at high energy [12]. Focusing X-rays down to this size requires

the last focusing element to be very close to the sample - in our case, the last optical face of the KB mirrors is at 25 mm upstream the sample. Both the fluorescence and phase contrast measurements can be performed under cryogenic conditions to preserve the biological samples close to their native hydrated state and slow down radiation damage effects [21]. Furthermore, free space is required around the sample to accommodate X-ray fluorescence detectors with a large solid angle and a light microscope to select the scanning region in X-ray fluorescence mode.

Concerning the requirements on the positioning, two groups of positioners can be distinguished: dedicated to alignment and dedicated to scanning. The alignment positioners are used to align the instrument, the orthogonality in tomography for example, and align the relevant position of the specimen with respect to the X-ray beam. The alignment translations typically have a large stroke: ± 5 mm transverse to the beam and $-2/+85$ mm along the beam for holographic phase contrast tomography. The accuracy requirements of the order of 500 nm are moderate, but the short and long term stability during the acquisition are critical. The scanning positioners include the rotation stage for tomography and small range translations transverse to the X-ray beam for mapping experiments. Tomography experiments require the parasitic translations at the level of the sample to be smaller than 50 nm over a rotation of at least 180 degrees. A continuous, 'vibration free' rotation mode is preferable. For scanning experiments (X-ray fluorescence analysis and ptychography), fast lateral positioning with an accuracy better than 5 nm over a scanning range of $50\text{ }\mu\text{m} \times 50\text{ }\mu\text{m}$ is needed. The axis perpendicular to the rotation axis, horizontal in our case, should be compatible with continuous scanning to avoid acquisition overheads. In some cases, software corrections on the acquired data can compensate for positioning errors. We prefer to have positioners with intrinsic reasonably optimal performance, because the software corrections are a bottleneck for routine operation and they often fail in the case of weak contrast.

Design choices of the endstation

The endstation is composed of long range stages (the alignment positioners) that integrate the fine movement stages (the scanning positioners). The fine movement stages include the tomography rotation stage and a piezoelectric stage for the scanning experiments. In addition to scanning the sample, the piezo-stage is also used to compensate the parasitic motion of the rotation stage. Movements of the piezo-stage is made under the control of capacitive sensors attached to a specific metrology frame. This active correction technique is implemented in the continuation of the work made by Leleu and David at the Laboratoire National de Métrologie et d'Essais [22]. The whole endstation is placed in a vacuum enclosure reaching 10^{-8} mbar in order to operate at cryogenic temperature and to limit contamination of the KB mirrors. Figure 1 shows the open vacuum enclosure revealing the long range stages and the fine movement stages.

The ID16A endstation aims at keeping the relative position of the sample with respect to the focal spot within a few tens of nanometers during experiments that can last a few hours. Main disturbances that can affect this performance have been identified as being the amplification of the ground vibration and geometric deformation due to variation in the temperature gradient of the endstation. We describe here the general design principles we have implemented to limit these disturbances and mitigate their effects on the positioning performance. To limit the amplification of ground vibration, we have decided to maximise the stiffness of the stages and support. However, due to the proximity of the nanofocusing optics, the space is limited to design stages and supporting structures with sufficient stiffness. To overcome this problem, the half-space below the X-ray horizontal beam plane has been left free to fit in

the sample stages, while the KB system and the light microscope are supported above this plane. This implies to hold the KB system from the top with a support structure that spans the sample stages. Space is then available to extend the width of stages and supports in the horizontal plane in order to increase stiffness and stability.

The whole endstation is placed in an experimental hutch where the temperature is controlled to ± 0.05 K. The vacuum enclosure acts as thermal shell that smoothens remaining temperature variations. To further limit the effect of the temperature changes on the geometry on the endstation, the materials have been carefully chosen and a specific design has been implemented. Most of the endstation is made of aluminum alloy and in some specific cases, Invar and stainless steel have been used. Aluminum has been chosen because of its high thermal conductivity, compared to Invar or steel, which helps in limiting the temperature gradients that can affect the geometry of the station. However, the thermal expansion is higher and the design must be adapted to overcome this disadvantage. One possibility is to make the design symmetric, for both geometry and thermal sources. This is the case for the endstation where we tried to satisfy as much as practically possible a reflectional symmetry. Sample and X-ray beam are placed in the plane of symmetry so that parasitic displacements outside of this plane are very limited. The geometrical stability of the station in the transverse direction illustrated in the Results section reflects this design choice. However, as the station is supported on the ground, a design having a top/bottom symmetry was impractical to implement. The relative position of the sample with respect to the focal spot is then much more sensitive to temperature changes in the vertical direction than in the horizontal plane. To limit this thermal drift in the vertical direction, Invar has been used for the specific parts of the structural chain that control the vertical position of the sample. The use of different materials requires a specific design to accommodate the differential thermal expansion. A typical solution is to use kinematic constraints between the parts of the station having different materials. For the ID16A endstation this is the case for example for the connection between the concrete block that supports the whole station and its main frame made of an aluminum alloy. However, kinematic constraints can sometimes be difficult to design with sufficient stiffness in the case of long and slender parts with no rotational symmetry. This is the case for example for the translation stage which provides a 100 mm stroke to swap between the two KB systems. The dimension of the stage is 80 mm x 260 mm, it is made of stainless steel and it is attached to a frame made of an aluminum alloy. Attaching this stage on three points with exact constraints would give too much bending compliance, so we decided to overconstrain the design while still leaving free the differential thermal expansion between steel and aluminum.

The piezo actuated hexapod

We have chosen to use a vacuum compatible rotation stage made of roller bearing elements and whose typical guiding errors are a few micrometers in translation and a few hundreds of microradians in rotation. In order to reach the tomography specification of 50 nm parasitic sample displacement, a correction of the parasitic motions is thus required. This correction is made by a feedback system that measures the errors with capacitive sensors and compensates them with a piezo-actuated stage producing small displacements of the rotation stage. The piezo-stage is a custom made hexapod composed of piezo-stack actuators having an individual stroke of 60 μm . Total displacement of the hexapod is about 50 μm in each direction and 500 μrad for tilts. These strokes are much larger than necessary to correct the rotation stage parasitic motions. This is because the piezo-stage is also used to perform the X-ray scanning experiments, with the rotation stage switched off.

Measurement of the rotation stage errors (3 translations and 2 rotations) is made thanks to capacitive sensors looking at a metrology reference attached to the rotating part of the stage as shown schematically in Figure 2a. This reference is a diamond turned aluminum ring whose shape errors have been previously calibrated so that only the rotation stage parasitic motions are measured by the capacitive sensors. In order to limit the measurement noise, we use fast averaging of the individual sensors and we use a total of 12 redundant sensors. With an integration time of 10 ms, the typical standard deviation obtained on the determination of the displacement of the ring is then well below 1 nm. Increasing the number of sensors allows also to measure the thermal expansion of the ring or to continue to operate the endstation in the case a sensor fails.

The principle of the control of the fine movement stages is illustrated in Figure 2b. From a user point of view, the feedback loop runs autonomously on a dedicated Debian Linux computer. Access to the fine stage control is done through specific Tango device servers [23]. They allow to change the sample stage setpoints: all zero during a tomography scan or a continuous ramp on the Ty setpoint for an X-ray fluorescence scan. When needed, the servers also provide access to the metrology of the capacitive sensors as raw values or transformed in Cartesian coordinates. Basic parameters of the feedback loop can be set and the correction can be turned on and off, for example on changing the sample.

Results

Extensive characterisation of the piezo and rotation stages has been conducted at the Precision Engineering Laboratory of the ESRF. First the shape errors of the reference ring were calibrated. To do this, an independent system used to measure the guiding errors of rotation stages was used in conjunction with the capacitive sensors of the station. By combining the measurements performed at the same time by these two systems, it is possible to separate the shape errors of the ring from the stage guiding errors. Results show that axial, radial and tilt errors of the rotation stage are about 3 μm , 6 μm and 120 μrad respectively. The shape error of the reference ring is below 1 μm . Assuming the shape errors do not change over time, they are used as a look-up table for the measurement of the stage errors by the capacitive sensors only. The piezo-stage is then actuated so as to compensate these errors. After having implemented the feedback loop, the residual errors are measured by the independent system of the Precision Engineering Laboratory. As shown in Figure 3 the axial error is decreased from 2 μm down to less than 20 nm when the control loop is active. Similar results have been obtained for the radial and tilt errors.

The described system is in user operation since late 2014, while the capabilities, such as cryogenic operation, have been gradually enriched. The X-ray characterisation of the ID16A nanofocusing and nanopositioning performance is illustrated in Figure 4. Operated at an X-ray energy of 33.6 keV, a fluorescence knife-edge scan shows that the focus is smaller than 20 nm. A careful analysis of the focus size using farfield ptychography demonstrated the focus to be about 13 nm in both directions [12]. The reproducibility of the scanning is excellent, while performed with the piezo actuated hexapod. The stability of the focus position of 12 nm over a period of 10 hours, shows that the symmetry and materials design choices were very appropriate. After an initial settling the horizontal stability reaches amazing values as low as 2-3 nm over a period of six hours. The excellent everyday performance of the instrument is illustrated with two representative scientific results in Figure 5, obtained respectively in X-ray fluorescence mode and in holographic nanotomography mode.

Summary and outlook

We presented the specifications, design choices and some selected results of the nanopositioning for the ESRF ID16A beamline. The key solution to the requirements imposed by the scientific needs are fine movements stages with an active correction scheme. The symmetry and materials design choices result furthermore in excellent short and long term stability. The full control of the sample position has opened unexpected possibilities such as applying a random motion during tomography acquisition to avoid artefacts related to X-ray optics imperfections [25]. On the downside, the very integrated design makes the maintenance of the system cumbersome. However, this integration is the key to an advanced system that has to reach its excellent performance routinely.

Highly specialized solutions, at the cost of flexibility, are expected to be further developed in the future for true nanoprobe operation. On the current system, three laser interferometry is being implemented to measure the vertical drift of the sample with respect to the KB system. The feedback system can integrate the compensation of this drift. Finally, the new generation of diffraction-limited storage-ring-based light sources will offer an increase of about two orders of magnitude in source brilliance and coherent fraction of the X-ray beams. The ESRF has started its upgrade to such an 'Extremely Brilliant Source' [26-27] that will be ideally suited for nanoprobe imaging applications. A major challenge will be the development of mandatory faster nanopositioning and detection schemes.

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Figure captions

Figure 1 Pictures of the ID16A endstation with open vacuum enclosure. (a) Large stroke actuators along the beam (Tx), and transverse to the beam (Ty and Tz). The Tz motion consists of a tripod that provides as well the Rx and Ry alignment degrees of freedom. The tomography scan is performed with the main rotation stage Rz, (b) Close-up of the endstation showing one of the KB nanofocusing optics (KB), the sample (S), the capacitive sensors (C) of the feedback metrology for the piezo actuated hexapod and the long working distance light microscope (LM).

Figure 2 Schematic of the piezo actuated hexapod and its control. (a) Principle of the short range piezo hexapod for scanning the sample and correcting the rotation stage parasitic movement through feedback with respect to a metrology frame, (b) principle of the feedback loop including the sample stage setpoints, the capacitive sensor metrology, the correction of the ring shape errors and the control of the short range hexapod.

Figure 3 Characterisation of the parasitic movements of the main rotation stage. (a) Axial error without compensation and (b) axial error with compensation by the piezo actuated hexapod. The axial error is reduced from 2 μm to less than 20 nm.

Figure 4 X-ray characterisation of the ID16A nanofocusing and nanopositioning performance. (a) Piezo actuated hexapod scan through the edge of a Ni lithography structure revealing a vertical focus size below 20 nm, (b) evaluation of the long term stability of the endstation through repetitive scanning of the structure. A stability of 12 nm over a 10 hours period is demonstrated in the horizontal direction.

Figure 5 Selected scientific results of the ID16A instrument. (a) X-ray fluorescence maps of ovarian cancer cells treated with Os containing metallodrugs, showing the cellular distribution of Os (red), Zn (green) and Ca (blue). The step size is 50 nm \times 50 nm (left panel) and 20 nm \times 20nm (right panel) [18]. (b) Volume rendering of a glass spicule of a marine sponge *G. cydonium* revealing simultaneously the outer structure and the internal axial filament. The data was obtained by X-ray holographic nanotomography [24].