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High-resolution 3D scanning X-ray microscopes at the Swiss Light Source

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Ptychography is a promising method to overcome limitations of traditional X-ray imaging schemes in resolution and depth-of-field. A sample is scanned at overlapping positions through a coherent beam of X-rays and for each spot a far-field diffraction pattern is recorded. A real-space image is reconstructed using iterative algorithms. At multi-keV photon energies the penetration depth allows imaging thick samples where 3D reconstructions are crucial for understanding the internal structure of the sample measured. This can be achieved by measuring the sample at various orientations and combining these measurements via computed tomography.

As a scanning technique ptychography remains sensitive to inaccuracies in sample positioning, which deteriorates the resolution and signal-to-noise ratio. Additionally, many sample systems suffer from radiation induced changes that alter the sample's structure during the measurement, which can be alleviated by cryogenic sample conditions. The latter is also essential for the measurement of cryogenically fixed biological samples in a near to native state.

In 2010 the OMNY (tOMography Nano crYo)[1] project started at the Paul Scherrer Institut with the goal to develop a dedicated instrument to perform tomography at the nano-scale on biological samples and condensed matter physics samples using ptychography in a controlled sample environment.

To achieve high position accuracy, dedicated laser interferometry[2,3] was developed which is compatible with scanning (for ptychography) and rotation (for tomography). This metrology measures the relative position between sample and a Fresnel zone plate, which is used to define the illumination on the sample. This scheme is now implemented in two instruments, one called flOMNI (flexible tOMography Nano Imaging)[4] for measurements at room temperature and atmospheric pressure, and OMNY for measurements in ultra-high vacuum at cryogenic conditions.

In the presentation both instruments will be explained in detail. Both use the same custom-developed sample holder, the OMNY pin[5], which has proven to work well at both, room-temperature and cryogenic conditions. The setups are operated at the cSAXS beamline. We will show the routinely used procedures for setting up and operating the instruments. Latest improvements but also current and future limitations will be discussed.

While flOMNI was initially intended as feasibility study for the cryogenic instrument, it became clear that limitations created by an in-vacuum cryo instrument are unfavorable for many experiments. flOMNI is much more easily adapted to non-standard measurement geometries, for example for in-situ experiments, and it is also simpler to set up and operate. Therefore flOMNI remains in regular use for measuring sample systems that are insensitive to radiation and the setup is continuously improved.

In high contrast samples, such as integrated circuits, a 3D resolution of 14.6 nm was demonstrated in a 10 µm diameter sample extracted from an Intel CPU[6]. Another example demonstrating the routine operation and thus throughput of the setup is the visualization of a complete redox cycle in a solid oxide cell (SOC) electrode, which required several measurements of the same sample after several ex-situ treating steps[7]. An example of the flexibility of the setup is its modification for tomography with magnetic contrast[8]. Here X-ray magnetic dichroism is used to obtain magnetic contrast. The magnetization parallel to the axis of rotation cannot be determined. Therefore, a dual-axis tomography had to be realized and two measurements of the same sample at different orientations were combined in the data analysis.

The design of OMNY[1] started in 2012 and the concepts were presented at the XRM conference 2014 in Melbourne. In 2015 OMNY was in operation for the first time and at liquid nitrogen temperature. Already then it demonstrated sub-30 nm 3D imaging on a high-contrast biological sample, a beetle wing scale[9]. Other examples include the measurement of polymer structures[10] and the imaging of unstained and cryogenically fixed tissue[11]. With a few years of experience with OMNY, limitations and current difficulties will be discussed.

The two instruments are currently complemented by a third one measuring in the laminography geometry. Here the axis of rotation is not perpendicular to the x-ray beam which makes the setup better suited for flat samples, such as integrated circuits. The setup is not compatible with the interferometry scheme used in flOMNI and OMNY and thus a different strategy is used here. The concept and status of the LAMNI (lAMinography Nano Imaging) will be briefly presented.

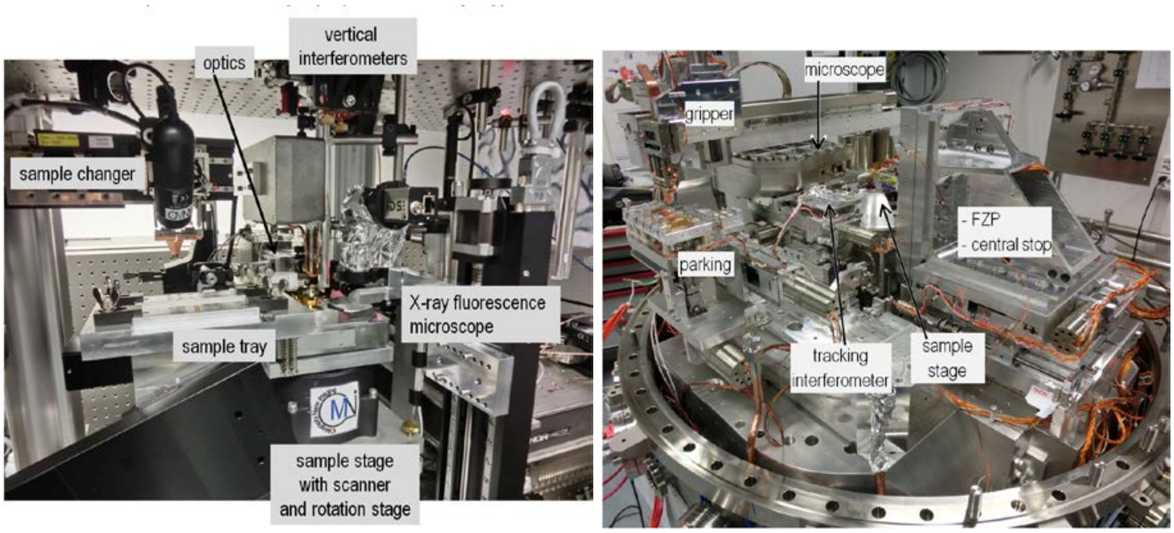

Figure. 1 (left) Photograph of the flOMNI instrument. (right) Photograph of the open vacuum vessel of OMNY.

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