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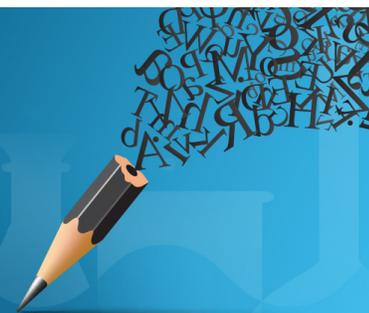


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A New Generation of X-ray Spectrometry UHV Instruments at the SR Facilities BESSY II, ELETTRA and SOLEIL

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Abstract. A novel type of ultra-high vacuum instrument for X-ray reflectometry and spectrometry-related techniques for nanoanalytics by means of synchrotron radiation (SR) has been constructed and commissioned at BESSY II. This versatile instrument was developed by the PTB, Germany's national metrology institute, and includes a 9-axis manipulator that allows for an independent alignment of the samples with respect to all degrees of freedom. In addition, it integrates a rotational and translational movement of several photodiodes as well as a translational movement of a beam-geometry-defining aperture system. Thus, the new instrument enables various analytical techniques based on energy dispersive X-ray detectors such as reference-free X-Ray Fluorescence (XRF) analysis, total-reflection XRF, grazing-incidence XRF, in addition to optional X-Ray Reflectometry (XRR) measurements or polarization-dependent X-ray absorption fine structure analyses (XAFS). Samples having a size of up to (100 x 100) mm² can be analyzed with respect to their mass deposition, elemental, spatial or species composition. Surface contamination, nanolayer composition and thickness, depth profile of matrix elements or implants, nanoparticles or buried interfaces as well as molecular orientation of bonds can be accessed. Three technology transfer projects of adapted instruments have enhanced X-Ray Spectrometry (XRS) research activities within Europe at the synchrotron radiation facilities ELETTRA (IAEA) and SOLEIL (CEA/LNE-LNHB) as well as at the X-ray innovation laboratory BLiX (TU Berlin) where different laboratory sources are used. Here, smaller chamber requirements led PTB in cooperation with TU Berlin to develop a modified instrument equipped with a 7-axis manipulator: reduced freedom in the choice of experimental geometry modifications (absence of out-of-SR-plane and reference-free XRS options) has been compensated by encoder-enhanced angular accuracy for GIXRF and XRR.

1. INTRODUCTION

The controlled development of nano-scaled materials calls for correlating the materials' functionality with their chemical and physical properties requiring sensitive and selective analytical methods. The reliability of most methods is based on reference materials or calibration samples with spatial compositions similar to the specimens of interest. However, there is a lack of reference materials at the nanoscale¹. PTB addresses this challenge by XRS techniques where all instrumental and experimental parameters are determined with a known contribution to the uncertainty budget of the analytical results. The functionality of nano-scaled materials is driven by optimized spatial elemental and chemical distributions involving layered structures², interfacial passivation³, and elemental depth profiles⁴, or 3D nanostructures⁵ calling for adapted analytical sensitivity and information depth as can be provided

by XRS, even in a reference-free quantification⁶ mode, e.g. without using any calibration samples⁷, but using well-known excitation radiation and calibrated X-ray detectors. To fully explore these analytical capabilities, a flexible UHV instrument is required, e.g. for varying the angle of incidence of the exciting radiation ranging from total reflection- (TXRF)⁸, grazing incidence- (GI) to conventional XRF. While the sensitivity increases with decreasing angle of incidence, the quantification reliability meets challenges regarding the knowledge of the X-ray standing wave (XSW) field and the solid angle of detection. The XRS reliability can be improved by complementary XRR studies providing access to optical constants, thickness, and density values of nanostructures. XAFS can determine the electronic structure, the chemical binding state, and the orientation of the molecular bond when changing the SR polarization vector. An optimized UHV instrument implements all above XRS and XRR methods by means of an appropriate manipulator enabling alignments of nano-scaled specimen⁹ such as SAM⁵ and atomic layer depositions¹⁰.

2. DESIGN OF THE INSTRUMENT AT THE PTB

The requirements of the instrument related to the various XRS methods as well as to the reference-free XRF approach call for a unique design including high flexibility as well as very precise sample manipulation under UHV conditions. The instrument is operated at the plane grating monochromator (PGM, 78 eV to 1860 eV) and the four crystal monochromator (FCM, 1.75 keV to 10 keV) beamlines in the PTB laboratory at the electron storage ring BESSY II¹¹ that provide high spectral purity and high photon flux. The UHV chamber can be aligned at the focal plane at both beamlines. Through the use of a load-lock with a sample transfer system, the main UHV chamber remains evacuated at all times, which maintains the UHV conditions in the chamber. It can be equipped with two energy-dispersive X-ray detectors being oriented perpendicularly to the incident beam and to each other. One of these detectors is positioned in the plane of the storage ring to take advantage of the p polarization geometry with respect to the incoming SR, thus reducing scattered radiation. The other detector can be installed on the top cover of the chamber and enables polarization-dependent measurements when turning the sample towards a horizontal position. The various experimental techniques require the sample stage to be moveable in all degrees of freedom. This is ensured by means of a 9-axis manipulator inside the UHV chamber. A schematic view of the sample manipulator is shown in Fig. 1. The *X-Y-Z* table is used for the translational positioning of the sample. In addition, three rotational axes allow for aligning the sample: The *sample_χ* axis realizes the upright positioning of the sample as well as polarization-dependent XAFS. In general, measurements are performed with the sample in this upright position. The angle of incidence of the incoming beam with respect to the surface of the sample is realized by *sample_θ* and can be varied from 0° to 90°. *Sample_φ* allows for the sample rotation in the XY-plane of the sample surface. XRR measurements can be performed simultaneously with GIXRF measurements by using the rotational axis of the second manipulator arm for the photodiodes in a $\theta/2\theta$ mode. For reference-free XRF a calibrated aperture system can be moved in by means of the *aperture_Y* axis to define well the beam geometry and the solid angle of detection. The whole system is designed for a maximum sample size of (100 x 100) mm², where each point on the surface can be analyzed. All axes are equipped with stepper motors. The base frame allows for the alignment of the instrument with respect to the incident X-ray beam by means of two translational axes and one rotational axis.

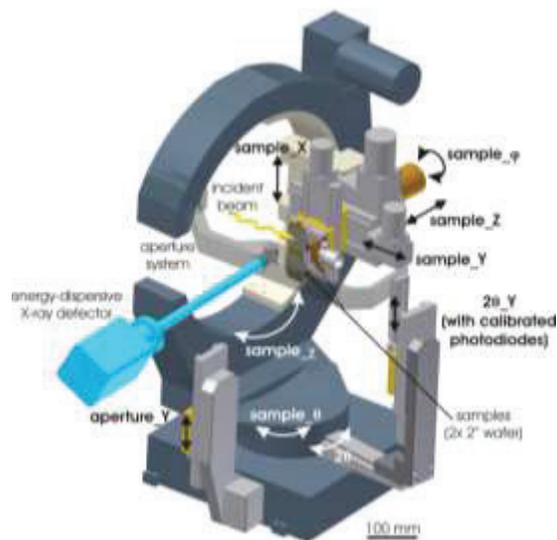


FIGURE 1: Schematic view of the 9-axis sample manipulator in TXRF geometry. The samples are mounted on *sample_X*, which is supported by *sample_Y*, *sample_Z*, *sample_φ*, *sample_χ*, and *sample_θ* (order of the axes of the sample movement).

3. TECHNOLOGY TRANSFER PROJECTS

The instrumental concept of PTB merging several XRS techniques in only one versatile instrument met the requirements of three institutes. A first technology transfer project was performed with the TU Berlin (TUB). To-

gether with TUB two transfer projects to the IAEA and to the French National Metrology Institute LNE-LNHB were completed. The UHV configurations at the three partner sites are mainly aiming at reference-sample based XRF analysis⁷, however, may be employed for reference-free quantification modes by introducing calibrated detection systems at well defined distances⁶. Key differences in the manipulator design of the three technology transfer projects as against the PTB template involve the reduction of axes: The rotational *sample_χ* axis is not realized for different reasons such as a smaller chamber diameter of 500 mm instead of 700 mm, the use of laboratory sources or the waiving of polarization-dependent XAFS options. Furthermore, the linear axis allowing for positioning the fixed aperture system in the PTB UHV chamber had been also omitted due to priority for reference-sample based XRS methods. The maximum sample size of all systems is 100 x100 mm² with each point on the surface being accessible.

A. Instrument of the Technische Universität Berlin

The XRS instrument of the TUB had to fulfill slightly different requirements as this instrument is operated in the Berlin Laboratory for innovative X-ray Technologies (BLiX) with laboratory sources¹². A smaller chamber size of 500 mm in diameter with a more flexible sample manipulator with a *sample_θ* range of $\pm 175^\circ$ was aspired. The 7-axis manipulator employed for TU Berlin is shown in Fig. 2. The higher flexibility also appears in the design of the UHV chamber due to variable slots for different types of X-ray sources as an X-ray tube for the hard X-ray range and a laser-produced plasma source for the soft X-ray range. The current X-ray tube has a Cr anode and is operated up to 50 kV high voltage. A polycapillary lens focuses the beam to the Pivot point of the 7-axis manipulator. The laser-produced plasma source has various options for its targets which deliver photon energies from 100 eV up to 1200 eV. Furthermore, energy-dispersive detectors as well as wavelength-dispersive equipment are available. The whole instrument was assembled, aligned, characterized and commissioned at PTB. The commissioning of this instrument was successfully performed at PTB's PGM beamline at BESSY II in Nov. 2012. After verification of the functionality of the whole instrument at PTB, it was sent to the BLiX premises at the TUB where it is in operation since Feb. 2013.

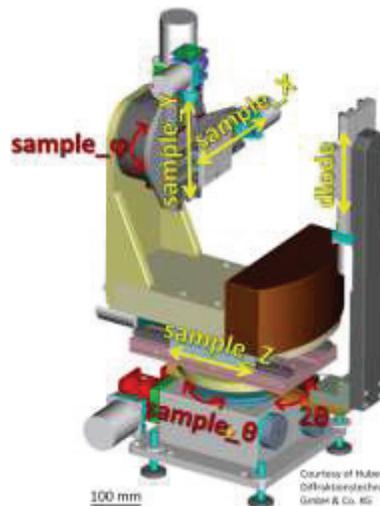


FIGURE 2: Schematic view of the 7-axis sample manipulator of the TUB. The samples can be mounted on *sample_X*, which is supported by *sample_Y*, *sample_Z*, *sample_φ*, and *sample_θ* (order of the axes of the sample movement).

B. Instrument of the International Atomic Energy Agency

In cooperation with TUB another XRS instrument with its own specification was developed for the IAEA. Here, the more compact version of manipulator with 7 axes (Fig. 2) with a *sample_θ* range of $\pm 175^\circ$ inside a 500 mm chamber size in diameter is employed for the usage of either tunable SR at the electron storage ring ELETTRA in the energy range of 3.7 keV to 14 keV or of a Mo anode X-ray tube in the laboratory¹³. To ensure the accuracy of the angle of incidence at high energies the axis *sample_θ* is additionally equipped with an encoder. After assembling, aligning and the characterization of the instrument at PTB the first commissioning was successfully performed at PTB's PGM beamline in July 2013. It is in operation at ELETTRA at the XRF beamline¹⁴ since Mar. 2014.

C. Instrument of the French National Metrology Institute (CEA/LNE-LNHB)

Yet another technology transfer project in collaboration with the TUB was performed with the French National Metrology Institute LNE-LNHB. Again the 7-axis manipulator and its smaller chamber size of 500 mm in diameter with a *sample_θ* range of $\pm 95^\circ$ was chosen but with a slightly different design but the same functionality. Additionally all three rotational axes are equipped with encoders. This instrument is used at the SR facility SOLEIL at the metrology beamlines. After assembling and aligning the manipulator with the vacuum chamber the autocollimator characterization measurements were performed at PTB. The union of the chamber and the base frame was realized at the final destination SOLEIL. The commissioning was successfully performed at SOLEIL in Aug. 2014. Ever

since that point this instrument is in operation at the PGM (45 eV to 1850 eV) and DCM¹⁵ (2.95 keV to 45 keV) metrology beamlines used by CEA/LNE-LNHB.

D. Commissioning of all Instruments

To operate those new instruments at high precision and to evaluate the results of measurements accurately, the characteristics of the axes of the sample manipulator have to be well known. Due to increasingly complex samples, motion artifacts of the sample manipulator may lead to misinterpretations of, e.g., spatial scanning measurements at grazing incidence conditions. To determine the respective instrumental uncertainties, the sample manipulator was commissioned by autocollimator and SR measurements, which delivered satisfying results¹⁶. As a representative example of all instruments the autocollimator characterization measurement of the *sample_X* axis of the manipulators was chosen. By means of the autocollimator measurements (Möller-Wedel Optical GmbH, ELCOMAT 2000), the translational axes were reviewed regarding all three rotational degrees of freedom (yaw, pitch, and roll) with the aid of two standardized mirrors (a plane and a polygon mirror). All measurements were performed with an evacuated instrument to evaluate effects of the heat emitted by the stepper motors. The axes were moved the full range of 110 mm back and forth at least three times. Table 1 shows the results of these autocollimator measurements of *sample_X*, whereas the specifications are fulfilled for each system by itself.

TABLE 1: Autocollimator measurements of *sample_X* of all instruments with an error of $\pm 0.7^\circ$

Instrument of	yaw / °	pitch / °	roll / °
PTB	14	11	9
TUB	6	21	10
IAEA	16	30	11
CEA/LNE-LNHB	15	48	7

4. CONCLUSION AND OUTLOOK

The new instruments were successfully commissioned and are in full operation at three different SR facilities and in a university laboratory, strengthening the XRS community by measurement capacities at different sites for nanotechnology development and quality control. The requested specifications of the technical equipment have been checked very carefully, which is a benefit for both manufacturers e.g. regarding their suppliers and users when experimental results cannot be affected by any artifact related to the sample movements. These instruments allow for accurate XRF measurements of various types of samples with sizes ranging from a few square millimeters up to 100 mm wafers and shapes from small pieces and thin foils to complex micro- or nanostructures or even curved samples.

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