Double von Hamos spectrometer for in-house X-ray absorption and X-ray emission spectroscopy studies

W. Błachucki[(1)], J. Czapla-Masztafiak[(2)], J. Szlachetko[(1,2)], J. Sá[(1,3)]

[(1)] Institute of Physical Chemistry, Polish Academy of Sciences, 01-224 Warsaw, Poland
[(2)] Institute of Nuclear Physics, Polish Academy of Sciences, 31-342 Kraków, Poland
[(3)] Department of Chemistry, Uppsala University, 75120 Uppsala, Sweden

E-mail: wojciech.blachucki@ichf.edu.pl

A few laboratory experimental setups for X-ray absorption spectroscopy (XAS) and X-ray emission spectroscopy (XES) were developed so far. It was shown that they allow synchrotron-quality spectra acquisition within few hours of measurement. Laboratory X-ray setups, unlike synchrotrons and X-ray free-electron lasers (XFELs) where the access is infrequent and not guaranteed, can be fully employed to dedicated scientific projects and used on a daily basis over entire year delivering the sought data as well as approaches that will be useful in experiments at 3rd and 4th radiation sources. At the Institute of Physical Chemistry of the Polish Academy of Sciences in Warsaw a new experimental setup for in-house simultaneous XAS and XES measurements has been constructed.

The setup is composed of one X-ray source, sample and two von Hamos geometry-based X-ray spectrometers: one for the measurement of the radiation transmitted through the sample and another for the emitted fluorescence detection. The X-ray source is XOS X-Beam Superflux PF X-ray tube with integrated focusing optics. The maximum voltage and the maximum current at which it can operate are 50 kV and 1 mA, respectively, and the emitted beam divergence is about 3º. The crystals used are two cylindrically bent Si(110) of 25 cm-radius of curvature. The diffracted radiation is registered by two Andor Newton DO920P cameras equipped with 250 μm-thick Be windows and front-illuminated CCD sensors of 1024×256 26 μm-sized pixels. The cameras are connected through flanges to vacuum pumping system decreasing the pressure in the sensors' proximity down to 10^{-7} mbar. This allows to safely cool down the sensors down to -70 ºC by means of the thermoelectric coolers integrated in the cameras. The whole setup is operated in air.

The developed XAS unit has been tested by measurement of the Ni *K* edge X-ray absorption spectrum for 5 μm-thick Ni foil. The experiment was performed at X-ray tube voltage and current of 40 kV and 0.9 mA, respectively. The radiation was detected at the 4th order of diffraction at the Bragg angle of 65.2º. The detector was operated at the temperature of -40 ºC. The registered spectral intensity was about 1 photon/(s×eV) and the total measurement took about 19 hours. The obtained spectrum is in excellent agreement with the data acquired with synchrotron radiation.

The XES unit is currently being commissioned. The built X-ray setup, however intended for probe of the lowest unoccupied and the highest occupied electronic orbitals of Ni atoms in different Ni containing compounds, can be easily adapted for detection of any X-ray radiation in the energy range 5 – 10 keV thus covering, e.g., *K*-edge binding energies of elements from vanadium to zinc.

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