QEXAFS – Most Recent Developments and Applications

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Abstract

Time resolved XAFS experiments can be performed in the quick scanning EXAFS (QEXAFS) mode, where the spectrum is collected “on-the-fly” using a stable double-crystal monochromator [1]. This technique is e.g. well suited for in situ investigations of catalytic reactions. Piezo driven tilt tables oscillating the monochromator crystals allow the continuous recording of XANES scans with repetition rates of about 100 Hz [2]. The acquisition of full EXAFS spectra with a scan range of typically 1 keV at a repetition rate of 10 Hz, i.e. 50 ms/spectrum, was possible using a cam driven tilt table for rapid angular oscillations [3]. A significant improvement was made with the development of a fast readout system for the Bragg angle [4], enabling e.g. the remote controlled change of the angular range of the scans [5]. Such a system was successfully installed at the SLS (Villigen, Switzerland) for user operation in 2008 [6].

The high scan rate of current QEXAFS monochromators require fast detector systems with response times in the microsecond range. Conventional ion chambers suffer from the slow ion drift velocity, which limits the time response. Using a hard disk drive based microsecond chopper, the time response of ion chambers and photodiodes was evaluated [7]. A solution is the use of gridded ionization chambers, which suppress the influence of the ion movement nearly completely [8]. Since in those detectors essentially only electrons contribute to the ionization current, significantly improved response times of less than 5 μs were achieved with self-made fast current amplifiers.

For the 3rd generation 6 GeV storage ring PETRA III at DESY (Hamburg, Germany) a completely new monochromator was developed, which was optimized for the undulator source. Using cryogenic cooling, the silicon crystal can cope with the full heat load of the insertion device. The oscillatory movement of the crystal stage is realized by a unique open-loop driving scheme operating a direct drive torque motor. This motor is installed inside of a goniometer located on the atmospheric side of the vacuum chamber. This design allows remote adjustment of the oscillation frequency and spectral range, giving complete control of QEXAFS measurements. It also features a conventional step scanning mode, which operates without a feedback loop to prevent induced vibrations. Equipped with Si(111) and Si(311) crystals on a single stage an energy range from 4.0 keV to 43 keV will be accessible by remote exchange of the monochromator crystals. A fully functional prototype of this monochromator was installed in January 2015 at the SLS and is in user operation since. EXAFS spectra up to k = 14.4 Å⁻¹ have been acquired with excellent data quality within 17 ms (30 Hz), and XANES spectra covering more than 200 eV within 10 ms (50 Hz).

After a review of the QEXAFS technique its current state, most recent developments and challenging scientific results will be presented. Details about fast detection schemes and the usability of tapered undulators for QEXAFS will be discussed. A preview of submitted publications about the new monochromator and the setup at the SLS will conclude the presentation.

Keywords – QEXAFS, XAS, X-ray monochromator, time-resolved spectroscopy, synchrotron instrumentation

References