ESRF	**Experiment title:** Light-element EXAFS using X-ray Raman Scattering	**Experiment number:** MI-879
Beamline: ID16	**Date of experiment:** from: 16 May 2007 to: 25 May 2007	**Date of report:** 31 August 2007
Shifts: 23	**Local contact(s):** Dr. Simo Huotari	*Received at ESRF:*

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Report:
X-ray Raman Scattering (XRS) is rapidly becoming a real alternative for x-ray absorption spectroscopy (XAS) in measuring x-ray absorption near-edge fine structure [1]. In principle, XRS is also readily applicable for studying the EXAFS oscillations in a variety of systems, where the possibility of studying soft x-ray absorption edges using hard x-rays is very promising. However, the requirements on the statistical and systematic accuracy of data for EXAFS analysis are very strict from the point of view of XRS measurements.

We thus optimized an experimental procedure for measuring XRS over an energy range of 1 keV with high accuracy, and applied the setup to measure the carbon K-edge EXAFS oscillations in diamond. The sample was a 2-mm thick pressed pellet of diamond powder (grain size < 1 μm) inside a vacuum chamber at room temperature. The measurements were performed utilizing the new multianalyzer spectrometer at ID16, in which the scattered radiation is focused on a 2D photon-counting Maxipix detector by 9 spherically bent Si(110) crystal analyzers. To optimize the use of measuring time, the energy scans were divided into suitable regions with different counting times, and in the true EXAFS region the scans were performed in equal steps in k-space. Spectra were gathered scanning the incident energy while analyzing the scattered radiation using (a) the Si(660) reflection near backscattering giving an elastic energy $E_0 = 9.68$ keV and energy resolution of $\Delta E = 1.8$ eV (FWHM) (b) the Si(880) reflection ($E_0 = 12.91$ keV, $\Delta E = 2.7$ eV). A total of five spectrometer setups (combinations of E_0 and scattering angle) were used, spanning a momentum transfer range of $q = 2.6 \ldots 12.6$ Å^{-1}.

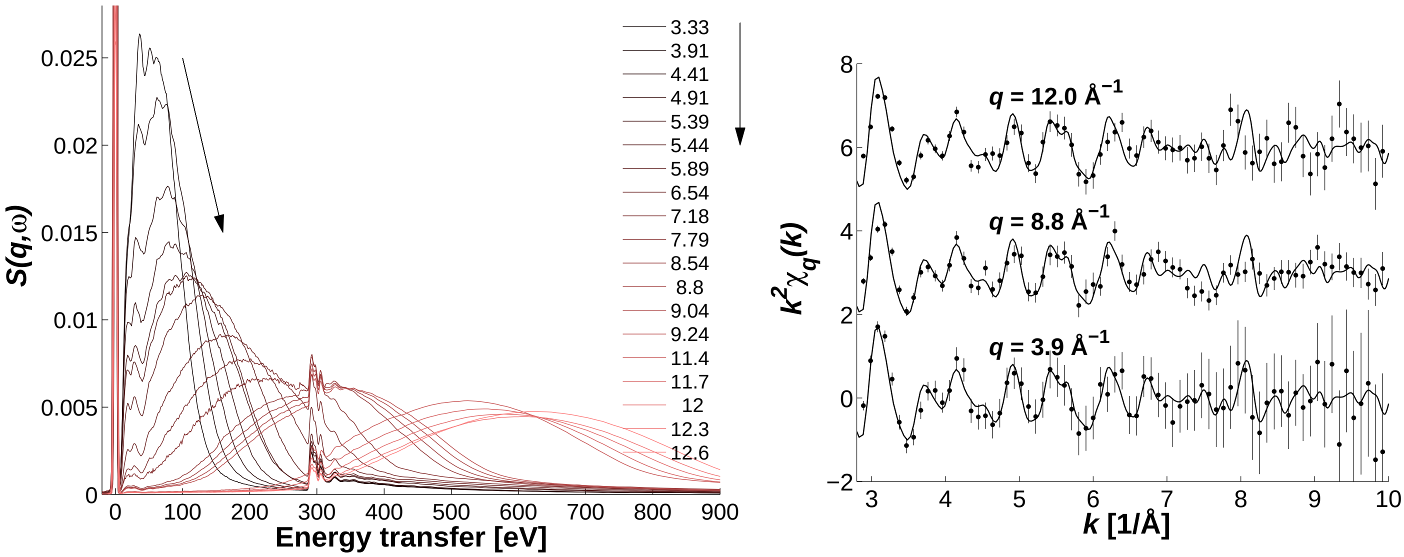

Figure 1. (a) (Left) The measured $S(q,\omega)$ of diamond for selected momentum-transfer values (in Å^{-1}). The spectra are normalized to same area as the computational results between $\omega = 200\ldots280$ eV. The broad peak moves to higher energies with increasing q as shown by the arrow. (b) (Right) The extracted oscillations of the core-electron $S(q,\omega)$ weighted with k^2 for selected momentum-transfer values with statistical error bars. The total measurement times were 2 h ($q = 3.9$ Å^{-1}) and 10 h ($q = 8.8$–12.0 Å^{-1}). The solid line is the x-ray absorption result of Comelli *et al.* [4].

To assure proper normalization, the incident intensity was monitored with various detectors: a Si pin diode detecting the scattered radiation from a kapton foil in the beam tube after slits; an avalanche photodiode looking at air scattering after the beam tube; and a Si pin diode measuring the transmitted intensity. The spectra were converted into $S(q,\omega)$ apart from an overall normalizing factor, which accounts for the different reflectivities of the analyzers. This allows the amplitude of the EXAFS oscillations to be determined accurately, once the background is correctly removed.

Figure 1(a) shows the extracted dynamic structure factors $S(q,\omega)$, which have been normalized to absolute units. The measured $S(q,\omega)$ are compared with computational results utilizing previously semiempirically determined core and valence Compton profiles for diamond powder [2], and a real-space multiple scattering calculation for the core electron $S(q,\omega)$ based on a modification of the FEFF code [3]. The valence contribution to $S(q,\omega)$ could thus be accurately removed. Figure 1(b) shows the obtained K-edge EXAFS oscillations $k^2\chi_q(\omega)$ of the core-electron $S(q,\omega)$ for selected q-values, compared with the soft x-ray result of Comelli *et al.* [4]. Figure 1(b) clearly demonstrates that we can reliably extract the oscillations in all cases, regardless of the underlying shape of the $S(q,\omega)$. Further studies into the subtle q-dependence of the oscillations are underway.

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