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A scattering filter for energy-dispersive optics

Michael Hagelstein,[a]*† Ulrich Lienert,[a] Thorsten Ressler,[a] Alfonso San Miguel,[a] Andreas Freund,[a] Sabine Cunis,[b] Clemens Schulze,[c] Alain Fontaine[d] and Jean-Louis Hodeau[d]

[a]European Synchrotron Radiation Facility, BP 220, F-38043 Grenoble CEDEX, France, [b]University of Ulm, D-89081 Ulm, Germany, [c]Paul Scherrer Institut, CH-5232 Villingen, Switzerland, and [d]CNRS, BP 166, F-38042 Grenoble CEDEX, France. E-mail: hagel@anka.fzk.de

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A filtering technique to remove parasitic scattering from X-ray absorption spectra that are acquired in energy-dispersive mode has been developed and tested at the European Synchrotron Radiation Facility. The improved set-up removes small-angle scattering of the sample or the windows of sample cells which may spoil the energy resolution or reduce the intensity of prominent features in the absorption spectrum, such as the white line at the Pt L_{III} edge. The sample is placed behind the curved monochromator and between two plane perfect crystals in the Bonse–Hart configuration. The dispersion of the Bonse–Hart double-crystal camera is matched to the dispersion of the curved monochromator by inclining the scattering planes of the two optical elements against each other.

Keywords: energy-dispersive XAFS; Bonse–Hart camera; SAXS filters.

1. Introduction

Energy-dispersive X-ray absorption fine structure (XAFS) spectroscopy is a powerful method for studying fast time-dependent processes such as the activation of a catalyst. The atoms under study are usually dispersed on an inorganic support. Small metal loading below 1 wt% requires the preparation of thick samples in order to optimize the signal-to-noise ratio. The energy-dispersive XAFS technique is also used for the study of matter under high pressure, for which, apart from diamond, boron nitride is a common anvil material for large-volume cells.

The standard energy-dispersive optics with a single curved monochromator crystal and a linear position-sensitive detector are sensitive to X-ray absorption and small-angle X-ray scattering (SAXS) to about 20 mrad scattering angle simultaneously. Small-angle scattering of a sample or the boron nitride anvils of a high-pressure cell can seriously affect the absorption measurement in two ways. First, a sensible enhancement of the background signal can be observed, since the total elastically scattered intensity is proportional to the thickness of the sample (Guinier, 1963). Second, the small-angle scattering can affect the width and the amplitude of high spatial frequency intensity variations of the incident radiation. Two consecutive transmission measurements have to be taken, one with the sample in the beam path and an incident-intensity reference measurement. For the reference measurement, a sample that does not contain the element under study may be used. Nevertheless, both measurements can be affected by scattering in a different way. The intensity variations are caused by multiple beam diffraction of the monochromator, variations of the reflectivity of the monochromator, and streaks on windows, mirrors or attenuators due to inappropriate polishing. The XAFS itself may contain strong variations of the absorption coefficient close to the absorption threshold.

† New address: Forschungszentrum Karlsruhe, D-76021 Karlsruhe, Germany.

2. Optical principle

The optical design of the small-angle X-ray scattering filter is derived from a set-up that has been proposed in order to increase the flux of a neutron small-angle scattering station (Freund, 1983). The incident white beam, monochromated by a bent monochromator, converges on the sample after reflection by a plane perfect silicon crystal (Fig. 1). The second plane perfect crystal is in the non-dispersive setting, satisfying the angle photon energy correlation of the first crystal. The pair of plane perfect crystals is called the Bonse–Hart camera (Bonse & Hart, 1965, 1966). The rocking curve widths of the employed reflections of the plane perfect crystals are narrower than the acceptance of a single pixel of the detector so that the small-angle scattering is effectively filtered.

The whole photon energy range diffracted by the bent monochromator ($\Delta E/E = 5\%$) must be transmitted by the two plane perfect crystals. The bent monochromator and the pair of plane perfect crystals of the Bonse–Hart camera will in general have different dispersions for a certain energy which is prescribed by the absorption edge under study. The dispersions can be matched by selecting a proper set of crystals and inclining the scattering plane of the crystal pair by an angle ψ against the scattering plane of the bent monochromator (Fig. 1). The inclination angle, ψ, can be determined by considering the main geometrical parameters of the energy-dispersive set-up,

$$\cos\psi = \tan\theta_{\rm perf}(1 - bq/p)/2\tan\theta_{\rm bent}, \qquad (1)$$

where p is the source to monochromator distance, q is the monochromator to polychromatic focus point distance, $\theta_{\rm perf}$ is the Bragg angle of the plane perfect crystals, $\theta_{\rm bent}$ is the Bragg angle of the bent monochromator, and b is the asymmetry factor of the plane perfect crystals ($b = \sin\theta_{\rm in}/\sin\theta_{\rm out}$, $\theta_{\rm in}$ is the angle between

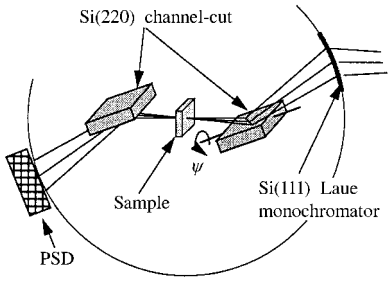

Figure 1
Top view of the energy-dispersive XAFS set-up using a filter for small-angle scattering. The two Si(220) channel-cut plane perfect crystals arranged in the Bonse–Hart configuration are rotated by an angle ψ in order to match the dispersion of the curved Laue monochromator. PSD: position-sensitive detector.

the crystal surface and incoming beam, θ_{out} is the angle between the crystal surface and outgoing beam). The sign of b is defined as always positive, q is always positive, and p is positive for a Bragg bent monochromator and negative for the Laue monochromator.

3. Experimental

XAFS at the Pt L_{III} edge (E = 11.564 keV) was recorded at the European Synchrotron Radiation Facility on bending-magnet beamline D5 and undulator beamline ID24 (Hagelstein *et al.*, 1997) in energy-dispersive mode. An Si(111) curved Laue monochromator (Hagelstein *et al.*, 1995) of 0.12 mm thickness and a Bonse–Hart camera with two Si(220) plane perfect crystals were employed. The dispersion was matched over the whole photon energy range with the Bonse–Hart camera inclined by $\psi = 22.4°$. The intensity of the prominent white line of a Pt metal foil was strongly damped due to scattering of a BN sheet fixed on the foil (Fig. 2). A much stronger white-line intensity was measured with the Bonse–Hart double crystal inserted into the beam path, proving the effective filtering of small-angle scattering. Similarly, the correct white-line intensity could be recovered at the Pt L_{III} edge for an H_2PtCl_6 powder sample using a Bonse–Hart channel-cut filter (Fig. 3).

An experiment to measure XAFS/anomalous SAXS successively using the energy-dispersive set-up with the Bonse–Hart filter has been performed. The small-angle scattering of a Pt–BN

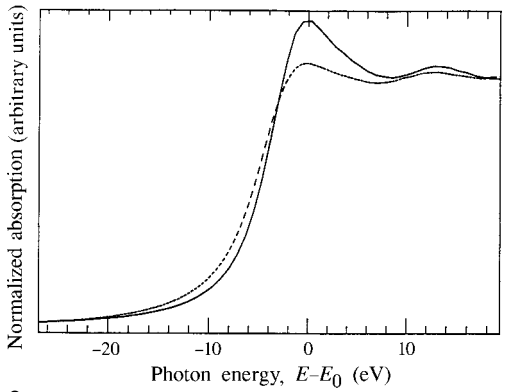

Figure 2
A curved Laue Si(111) monochromator and a set of two Si(220) flat crystals were used for filter scattering of a Pt metal foil/BN sheets sandwich at the Pt L_{III} edge (E_0 = 11564 eV). Correcting the data for resolution losses, the white-line intensity was damped due to BN scattering by 50% without filtering (dashed line) and by only 5% using the filter (continuous line).

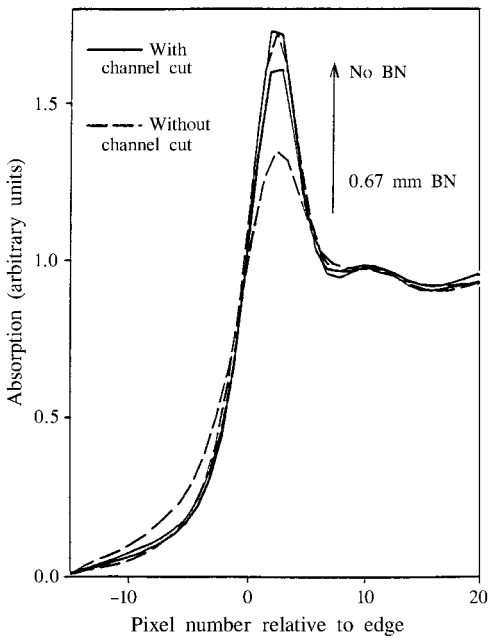

Figure 3
The near-edge structure obtained for an H_2PtCl_6 powder sample using an energy-dispersive Si(111) Laue monochromator and an Si(220) channel-cut Bonse–Hart camera on beamline D5. The addition of a 0.67 mm-thick BN sheet leads to stronger damping of the white-line intensity due to enhanced SAXS. The dashed and solid curves, which display the highest and narrowest white lines, were recorded without BN.

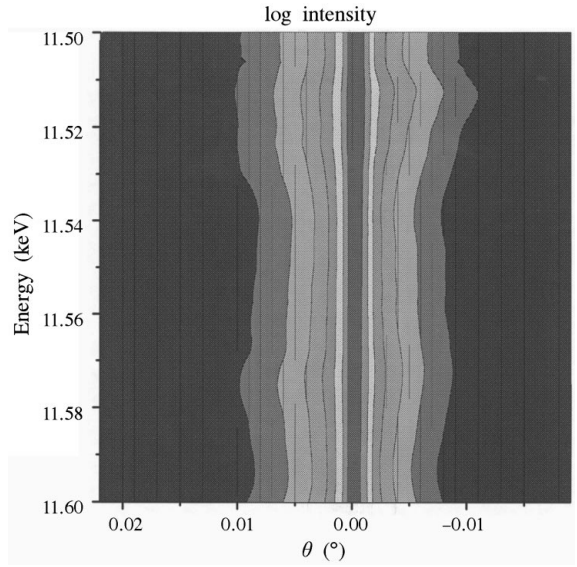

Figure 4
Surface plot of the small-angle scattering signal of an Al metal foil. It was measured rocking the second crystal of the double-crystal Bonse–Hart arrangement, representing mainly the instrument function.

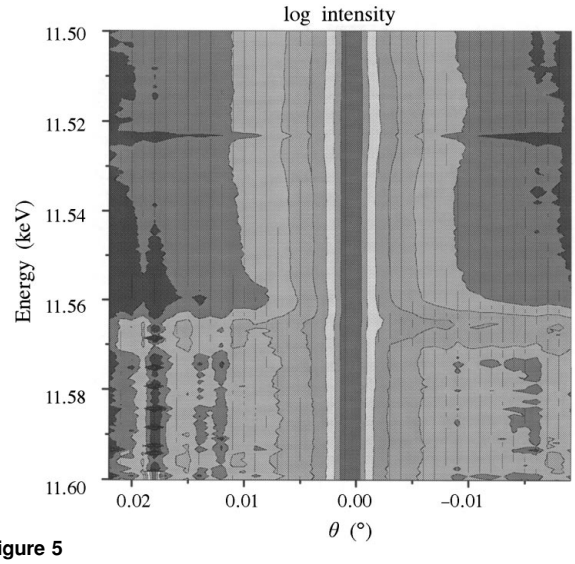

Figure 5
The same measurement as in Fig. 4 but with a sandwich of a Pt metal foil and a 1 mm-thick sheet of BN between crystals of the Bonse–Hart camera. Anomalous scattering at the threshold (11.568 keV) and enhanced glitches (11.523 keV) due to uncompensated scattering background are discernible.

sandwich sample has been measured, rocking the second crystal of the Bonse–Hart camera against the first. It is compared with the scattering of an Al foil with low scattering efficiency, representing mainly the instrument function (Fig. 4). The anomalous small-angle scattering signal above the absorption edge at 11.564 keV with a particularly strong and well resolved white line and smooth near-edge structure is clearly discerned in Fig. 5.

4. Conclusions

It has been experimentally verified that the small-angle scattering of the sample or the windows of sample cells can lead to damping of the amplitude of XAFS spectra if measured in the energy-dispersive mode. The signal quality can efficiently be improved by applying the Bonse–Hart filter. The proposed geometry permits one to acquire energy-dispersive XAFS spectra that are hardly affected by small-angle X-ray scattering. The set-up may be used as well for the combined measurement of XAFS spectra and anomalous SAXS.

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