J. Synchrotron Rad. (1998). **5**, 986–988

On a spatially resolving USAXS instrument for operation at a third-generation synchrotron radiation source

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(*Received 4 August 1997; accepted 6 November 1997*)

A point-collimation USAXS system based on the Bonse–Hart-diffractometer concept is proposed which takes advantage of a CCD detector to rapidly obtain two-dimensional SAXS data at both high spatial and angular resolution for a line of points on the sample. The method might typically be employed using Bonse–Hart crystal optics in the horizontal plane, encompassing the high-angular-resolution scan direction, and a condensing monochromator in the out-of-diffraction-plane direction as the basis for a pinhole SAXS camera in that plane. The new system is well suited to operation at an undulator beamline on a third-generation synchrotron radiation source. Other applications of the instrument include (i) both absorption and phase-contrast imaging/tomography, (ii) polycrystalline topography, (iii) high-resolution triple-crystal measurements and (iv) diffuse scattering measurements.

Keywords: USAXS; small-angle scattering (SAXS); Bonse–Hart; phase-contrast; tomography.

1. Introduction

The Bonse–Hart (1965) method (BH) for small-angle X-ray scattering (SAXS) offers the ultimate resolution in q-space and also offers a very low value of $q_{\min}$ of the order of 3×10^{-4} Å^{-1}. In common practice both in the laboratory context and also at synchrotron sources, the BH method suffers from poor resolution in the out-of-diffraction-plane direction (essentially infinite slit height) and poor data throughput due to single-point (in q-space) counting (see, for example, Siddons *et al.*, 1990; Long *et al.*, 1991; Chu *et al.*, 1994; Koga *et al.*, 1996; Diat *et al.*, 1996; Riekel *et al.*, 1997). Improvement in collimation in the out-of-diffraction-plane direction by introduction of a crystal collimator is an option (Bonse & Hart 1966) but further significantly reduces the data-collection rate even at a third-generation synchrotron radiation source (see, for example, Pahl *et al.*, 1991; Diat *et al.*, 1996).

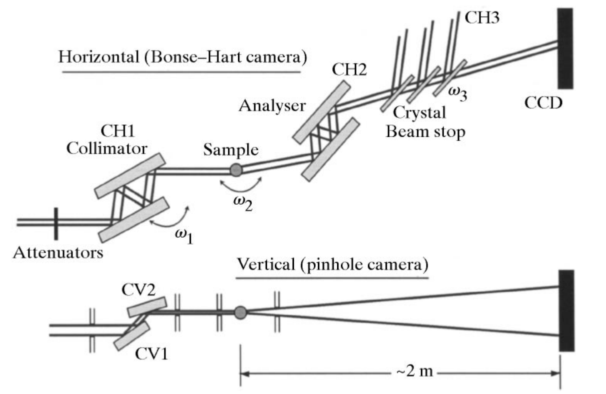

Figure 1
Schematic outline of the proposed spatially resolving USAXS system with a Bonse–Hart configuration in the horizontal plane and a crystal monochromator followed by pinhole camera in the vertical plane. CH1 and CH2 are multiple-reflection grooved monochromator/analyser crystals while CH3 is a set of ultrathin wafers appropriately aligned to act as a 'crystal beam stop'. CV1/CV2 is typically a double-crystal monochromator capable of narrow bandpass and harmonic rejection. The beam in the horizontal plane may be quite wide, *e.g.* 20 mm, because monochromatization is achieved *via* CV1 and CV2.

Given the very low emittance of a third-generation synchrotron radiation source, especially at an undulator beamline, and the high speed and spatial resolution of two-dimensional electronic detectors such as those based on CCDs, it would appear that the introduction of a CCD detector not only enables one to overcome the apparent limitations of the usual BH method, but also yields exciting new opportunities for greatly increasing the types of information obtained about the sample at no extra overhead in data-collection time (see Fig. 1). In particular, it becomes practical to measure the full two-dimensional distribution of small-angle X-ray scattering at high resolution, implying that the intensity in the out-of-diffraction-plane direction (*e.g.* vertical) is obtained and so, *a fortiori*, the slit-height effect is minimized. This arises because a line of SAXS data in q-space can be collected for each setting of the analyser crystal, since the camera operates as a pinhole camera in the out-of-diffraction-plane direction (Fig. 1*b*). In the in-diffraction-plane (BH) direction (*e.g.* horizontal), the two-dimensional detector may be viewed as collecting data with each column of data yielding a pinhole-camera SAXS profile for each corresponding point on the sample (see Fig. 2). Looking along a row, each data point on a row provides *one point* of a BH USAXS profile for the corresponding point on the sample. Such a system would open up exciting new possibilities for SAXS studies of interfaces in *e.g.* metal, colloid and membrane systems.

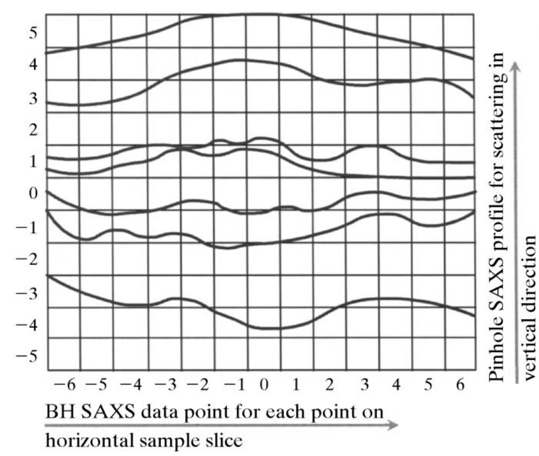

Figure 2
Schematic illustration of the generic information content of a single CCD data frame for the present instrument. It may be viewed as a linear array of pinhole SAXS patterns for a pinhole camera collected as a function of position on the sample. Spatial resolution at the sample is determined by crystal optics of CH1 and CH2, and by the spatial resolution of the CCD. Each point in a row of data corresponds to a BH SAXS data point for each corresponding point on the horizontal slice of the incident beam through the sample. Each column of data corresponds to a pinhole camera SAXS profile in the vertical direction for the corresponding point (horizontal slice through, *i.e.* column position) on the sample.

Given the high degree of collimation possible with third-generation sources, which is well matched to perfect-crystal optics, and the high spatial resolution possible with two-dimensional electronic detectors (*e.g.* <10 μm), it would be possible to collect of the order of 1024 pinhole SAXS profiles simultaneously, each one corresponding with high spatial resolution to a different point on the sample taken as a slice through the sample. This therefore leads to the possibility of *SAXS imaging* by collecting data for different slices through the sample (*e.g.* by vertically translating the sample through the beam or, alternatively, the beam on the sample).

The proposed system would appear to not only be useful for USAXS imaging but also for more general types of imaging such as (i) conventional (absorption-contrast) tomography (Bonse *et al.*, 1991), (ii) phase-contrast imaging and tomography in various modes (see, for example, Davis *et al.*, 1995), (iii) polycrystalline topography (see, for example, Chikaura *et al.*, 1982), (iv) analysis of strain profiles in semiconductor crystals (see, for example, Nikulin *et al.*, 1997, and references therein).

2. On some technical aspects of the conceptual design

Looking at Fig. 1, the function of the instrument can be simply considered by separately looking at the horizontal- and vertical-scattering geometries.

2.1. *Vertical-scattering geometry*

In the vertical plane the intrinsic collimation of the beam is greatest and the primary function of the monochromator crystals CV1 and CV2 is to monochromatize the beam (including harmonic suppression). They might also be used to spatially condense the beam in order to produce a thinner horizontal slice at the sample. Viewed in the vertical plane, the instrument operates as a pinhole SAXS camera with monochromatic radiation. The angular resolution in this plane with camera lengths proposed (*e.g.* 2 m) could be about an order of magnitude worse than that for the direction in which the BH mode is operative.

2.2. *Horizontal-scattering geometry*

In the horizontal plane the system corresponds to a Bonse–Hart camera when the analyser crystal (CH2) is scanned (*i.e.* ω_2 is varied) and is essentially *non-dispersive.* Because of the large working distance between the source and the collimator crystal (CH1), the beam incident at CH1 is highly parallel and a wide (highly parallel) beam can be produced at the sample (especially after monochromatization of the beam using CV1 and CV2; see §2.1 above). Since the beam has been monochromated, the analyser crystal only significantly transmits X-rays which are parallel to a given direction (the Bragg condition for the given X-ray energy) and thus the one-dimensional signal in the horizontal plane corresponds to a BH-SAXS data point for each point on the sample slice. As the analyser crystal is swept around the Bragg condition for the transmitted beam, the profile for each pixel corresponds to a BH-SAXS profile. When the analyser crystal is at the exact Bragg condition for the direct beam from the collimator crystal, the danger of damaging the two-dimensional detector exists and some protection system is required. One approach would be to shutter/attenuate the beam when the analyser is approximately aligned with the straight-through beam. However, this would lead to a missing band of SAXS data in the two-dimensional SAXS plot around the condition $\omega_2 = 0$ for each point in the sample as depicted by the shaded band in Fig. 3.

2.2.1. *Crystal beamstop system.* A second possible approach to this problem of the direct beam hitting the detector is to use a set of appropriately oriented thin (*e.g.* ~2 μm-thick silicon wafers) crystals (CH3) to transmit small-angle scattered radiation but to be aligned such that they act as a *beam stop in reciprocal space* operating *via* Bragg diffraction of the direct beam from the collimator crystal in the case where this passes through the analyser crystal (*i.e.* $\omega_2 \simeq 0$). Thus only 'one point' of data is lost from the two-dimensional plot shown in Fig. 2 rather than a whole column. SAXS scattered radiation will essentially pass through the crystal beamstop with only normal attenuation of the beam due to normal absorption. This crystal beamstop might be operative only for $\omega_2 \simeq 0$ and removed otherwise. Because the BH mode, involving scanning in the q_x direction, allows very close approach of ω_2 to zero, the excluded region of (q_x, q_y)-space in Fig. 3 is quite small and may be readily amenable to interpolation of data throughout that region.

2.3. *Estimated resolution and other performance characteristics*

The resolution of the system in the (horizontal) direction where the Bonse–Hart configuration is operative is independent of photon energy and is of the order of 3×10^{-4} Å^{-1}. $q_{\rm min}$ is also of the order of the same value for the BH direction. On the other hand, for the (vertical) direction in which the pinhole mode is operative, the resolution depends on beam geometry and detector resolution, but with a 20 μm beam height at the sample, it might be roughly of the same order of magnitude as for the BH direction. However, with a physical beamstop of the order of 0.5 mm at 2 m distance from the sample, $q_{\rm min}$ would be an order of magnitude worse than for the BH direction. The 'crystal beam stop' proposed above would appear to be much more efficacious as it could give a smaller $q_{\rm min}$. Spatial resolution in the horizontal plane might be further improved by use of more specialized crystal optics (see, for example, Wilkins & Stevenson, 1988).

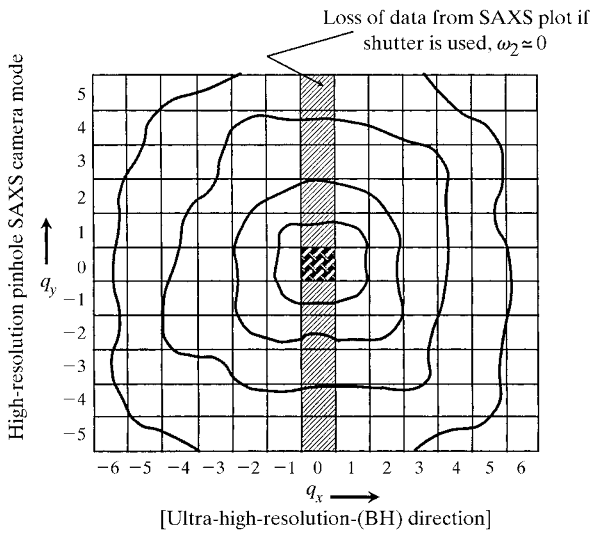

Figure 3
By collecting a full set of data for a scan range of the BH analyser crystal, CH2, a set of two-dimensional SAXS patterns can be collected as a function of position on the sample for a horizontal slice through the sample.

3. Discussion

The presently proposed system could readily be extended to encompass the following enhanced features:

(i) operation of the system entirely in vacuum (highly desirable) with the possible exception of the sample stage (see also Barnea *et al.*, 1989, 1992);

(ii) extension to SAXS/WAXS mode encompassing high-resolution diffractometry with a CCD detector (see also Barnea *et al.*, 1989, 1992).

The present conceptual outline forms the basis of a proposal to ChemMatCARS for operation on an undulator beamline at the APS.

Helpful discussions over a long period on the general topic of this work with Thomas Zemb, Andrew Stevenson, Andrzej Radlinski, Phil McMahon, John White, Ian Gentle, Dudley Creagh, David Cookson, and Richard Garrett are acknowledged.

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