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Slits as Adjustable Pinholes for Coherent X-ray Scattering Experiments

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The combination of accurate translation stages with carefully polished slit blades leads to slits that have many advantages as pinholes for coherent X-ray scattering experiments. The size is adjustable and can be made as small as 0.5 μm. Setting up is easy, while the blade thickness (1 mm tungsten) also makes the slits useful for hard X-rays. A relation between the slit–sample distance and the minimum beam size, together with the corresponding slit size, is derived. This shows that a micrometer-sized beam can be achieved with this type of slits.

Keywords: pinholes; coherent X-rays; Fraunhofer diffraction.

1. Introduction

In recent years the use of coherent X-ray beams has rapidly gained popularity as a probe of the static and dynamic fluctuations in matter (Sutton *et al.*, 1991; Cai *et al.*, 1994; Brauer *et al.*, 1995; Dierker, Pindak, Fleming, Robinson & Berman, 1995). When a sample exhibiting density fluctuations is illuminated with a coherent beam a so-called speckle pattern is observed. When the fluctuations vary in time one may look at the time correlations in this speckle pattern and probe the dynamics down to millisecond time scales (or even less). This technique is called X-ray photon correlation spectroscopy (XPCS).

The X-radiation used for speckle and XPCS experiments comes from synchrotron radiation sources. A synchrotron source itself is incoherent, but by placing a small pinhole at a sufficiently large distance from the source, the beam after the pinhole can be made coherent. The pinhole has to be smaller than the so-called transverse coherence length, L_T (Hecht, 1987),

$$L_T = \lambda R_s / 2d_s, \tag{1}$$

where λ is the wavelength, R_s is the distance from the source and d_s is the source size. For a typical set-up at a synchrotron, L_T is several micrometers. A pinhole of similar dimensions is therefore required. To date, most experiments have used laser-drilled holes in metal foils. Such pinholes have a number of disadvantages: (i) several foils need to be aligned in order to obtain sufficient attenuation, (ii) the pinholes have a fixed size, and (iii) the smallest size possible is ~2.5 μm. Ferrer *et al.* (1995) used a mirror at grazing angles to obtain an effective one-dimensional slit, but it is not easy to change the slit size. Very recently, van der Veen, Riemersma, Schlatter, Abernathy & Grübel (1997) made pinholes in thick metal sheets, thus avoiding problem (i). A method of overcoming all three disadvantages is the use of slits.

2. Pinhole slits

In order to obtain a slit size in the micrometer range, the translation stage has to be very accurate *and* the slit blades have to be polished. We used Newport translation stages (Newport Precision Slits model PS10-X, with an MM4000 Motion Controller) with a minimum step size of 0.2 μm. The slit blades were made in-house of 1 mm-thick tungsten, and a sharp edge was achieved by polishing two sides of the slit (by René Koper at the Surface Preparation Laboratory) (see Fig. 1*a*). The polishing was done such that the roughness of the edges was only a few tenths of a micrometer. In order to avoid (small-angle) slit scattering, slit blades usually have an edge that is several degrees smaller than 90°. For the pinhole slits the deviation from a straight edge was chosen to be only 0.5° because only in that case is the X-ray beam sufficiently attenuated over a lateral distance of a few tenths of a micrometer. The slit

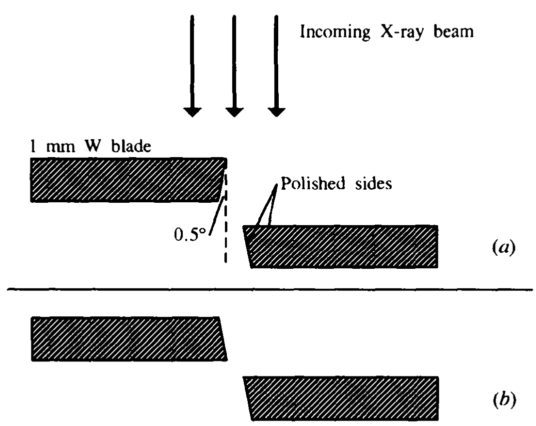

Figure 1
(*a*) A schematic diagram showing the geometry of the slit set-up and the two faces that were polished. (*b*) Mounting of the slit blades such that the longitudinal offset is minimum, leading to more symmetric Fraunhofer patterns.

blades were slightly offset along the longitudinal direction in order to avoid damaging the polished sides (see Fig. 1*a*). To make a pinhole, we used a crossed pair of slits.

3. Experiment

The slits were tested at the surface diffraction beamline ID3 of the ESRF. The slits were placed after the Si(111) monochromator at a distance of 35 m from the undulator source. The wavelength used was 0.96 Å and the source size was ~100 µm horizontally and ~10 µm vertically (FWHM). This leads to a transverse coherence length ≥10 µm, so for pinholes smaller than this, coherent effects should be observed. The pattern after the slit was observed using a CCD camera at a distance of 10 m.

For a perfect rectangular slit one simply expects the product of two Fraunhofer slit patterns generated along each direction (Hecht, 1987):

$$I(x,y) = Cs_x^2 s_y^2 \sin^2(\pi x s_x/\lambda R)/(\pi x s_x/\lambda R)^2 \times \sin^2(\pi y s_y/\lambda R)/(\pi y s_y/\lambda R)^2, \quad (2)$$

where x (y) is the horizontal (vertical) position on the detector, s_x and s_y are the slit openings, R is the distance to the detector and C is a scale factor (see Fig. 2). Fig. 3 shows an observed pattern along the horizontal direction. Rather than exhibiting the symmetric pattern predicted by (2), the observed Fraunhofer pattern is asymmetric. The reason for this is very simple when one considers Fig. 2. The blades have a small longitudinal offset, which means that when viewing the slits from an angle, the effective slit opening is smaller/bigger than the average one. The effective slit opening is given by

$$s_x = s_0 + x h_x/R, \quad (3)$$

where h_x is the longitudinal offset between the slit blades (see Fig. 2). An equivalent expression holds for s_y. When this expression is used in (2) the experimental data in Fig. 3 can be fitted as shown by the solid curve. The only additional fitting parameters are a linear background and a smearing width.

The asymmetry can be reduced when the blades are mounted in the way shown in Fig. 1(*b*). The scattering from the first slit blade is not a problem since it is blocked by the second blade for small slit openings. Fig. 4 shows a Fraunhofer pattern that was generated using the slits mounted in this way and with a horizontal slit setting of 0.72 µm and a vertical slit setting of 0.64 µm. In this case one generates a 'Fraunhofer cross'. Fig. 5 shows a number of horizontal and vertical traces through such patterns, together with the fitted curves using (2) and (3). In all cases we could fit the horizontal pattern better than the vertical one, a fact we cannot explain. The patterns are still asymmetric, *e.g.* the fit of the horizontal patterns requires a longitudinal offset of the slit blades of 0.13 mm. Generating symmetric Fraunhofer patterns is not a goal in itself since for practical applications one is normally only interested in the central cone. In fact, one may want to suppress the Fraunhofer fringes since they may lead to a distortion in the observed speckle pattern. Using a longitudinal offset is thus one way to suppress the fringes on one side of the profile.

4. Minimum beam size

The smallest pinhole we have made in this way measured $0.27 \times 0.51\ \mu m^2$ (see Fig. 5*a*), much smaller than the sizes typically available in laser-drilled holes. Of course, such small holes do not mean that the illuminated area of a sample placed behind the hole is also very small. The width of the beam at the detector position (where a sample can be placed instead) is determined by three factors:

(i) Fraunhofer diffraction. From (2) we find for the beam width, w_f, at the detector position due to the Fraunhofer diffraction

$$w_f \simeq \lambda R/s. \quad (4)$$

(ii) Source size. The slit acts as a pinhole camera of the source and thus produces an image of the source with the following size

$$w_s = d_s R/R_s. \quad (5)$$

(iii) Slit size. When the dimension of the beam is similar to the slit size, the Fraunhofer limit [as used to derive (2)] no longer holds, and the effective beam size is determined by the slit opening s.

In order to see coherent diffraction features we require that the contribution from the source size is less than that from the Fraunhofer diffraction. If we take a factor of two difference in width we find

$$2w_s < w_f \Leftrightarrow s < \lambda R_s/2d_s. \quad (6)$$

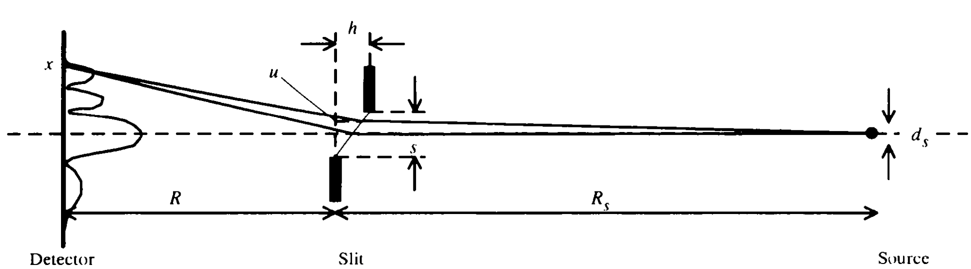

Figure 2
Schematic diagram of the experimental set-up. The dimensions are not to scale.

Comparing this with (1) we see that this is another way to state that the slit opening has to be less than the transverse coherence length. Assuming that (6) holds, the total width at the sample position is in a first approximation given by the square sum of contributions (i) and (iii):

$$w \simeq (\lambda^2 R^2/s^2 + s^2)^{1/2}. \quad (7)$$

This equation accurately describes the beam width for the case where either Fraunhofer diffraction or the slit size dominates. In the slit-size range where both contributions are important, and where the beam obtains its smallest dimensions, a more accurate expression is required. First note from (7) that the following proportionality holds for the minimum beam size:

$$w_{\min} \propto (\lambda R)^{1/2}. \quad (8)$$

Next we calculate the intensity at the detector position. It is straightforward to derive an expression for the path length difference, Δr, between a ray passing through the centre of the slit and through a point given by index u, that both arrive at a point x on the detector (see Fig. 2). Expanding this expression in the most important terms in x, u and h yields

$$\begin{aligned}\Delta r \simeq\ & (-x/R - hx^2/2R^2 s)u \\ & + (1/2R_s + 1/2R + hx/2R^2 s)u^2. \quad (9)\end{aligned}$$

The amplitude at a point x on the detector is found by integrating the contributions over the entire slit width

$$A(x) = C \int_{-s/2}^{s/2} \exp(-2\pi i \Delta r/\lambda)\, du. \quad (10)$$

In the Fraunhofer limit ($R > s^2/\lambda$) the xu term dominates and one arrives at (2). For distances closer than this (Fresnel case) the result of the integral is a (lengthy) expression involving error functions, that can, however, easily be evaluated using *e.g.* *Mathematica* (Wolfram Research, Inc.).

First consider the case when $h = 0$. We refer to Hecht (1987) for a visualization of the resulting diffraction patterns in the case. Of importance here is the minimum achievable beam size. Using the full width at half maximum as the relevant quantity we obtain the following result for the minimum beam size at the detector (or sample) position

$$w_{\min} = 0.62(\lambda R)^{1/2}, \quad (11)$$

while the corresponding optimum slit setting is

$$s_{\rm opt} = 1.68(\lambda R)^{1/2}. \quad (12)$$

[For this slit setting, (6) is usually satisfied for typical geometries at a synchrotron radiation source.] Fig. 6 shows

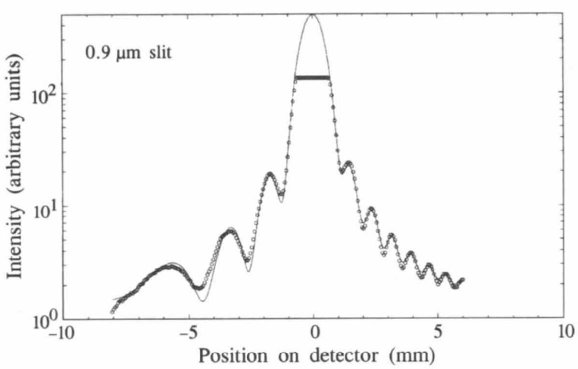

Figure 3
Fraunhofer pattern of a 0.9 μm horizontal slit (and 8 μm along the vertical direction). The pattern is asymmetric because of the longitudinal offset between the slit blades. Circles represent data points whereas the curve is a fit. The flat top of the experimental data is due to saturation of the detector.

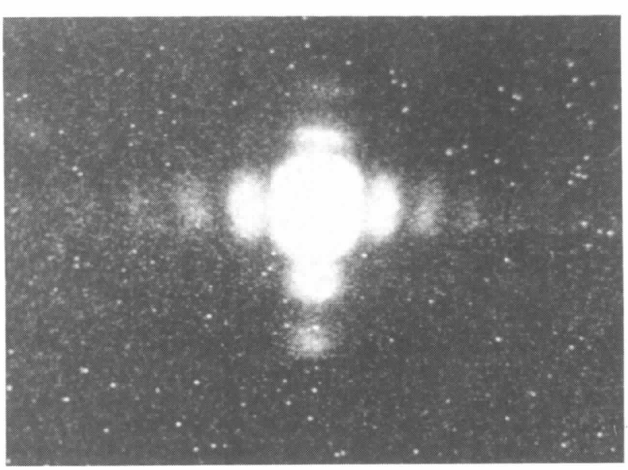

Figure 4
Fraunhofer pattern for a slit of 0.72 × 0.64 μm (horizontal × vertical) as observed using a CCD camera.

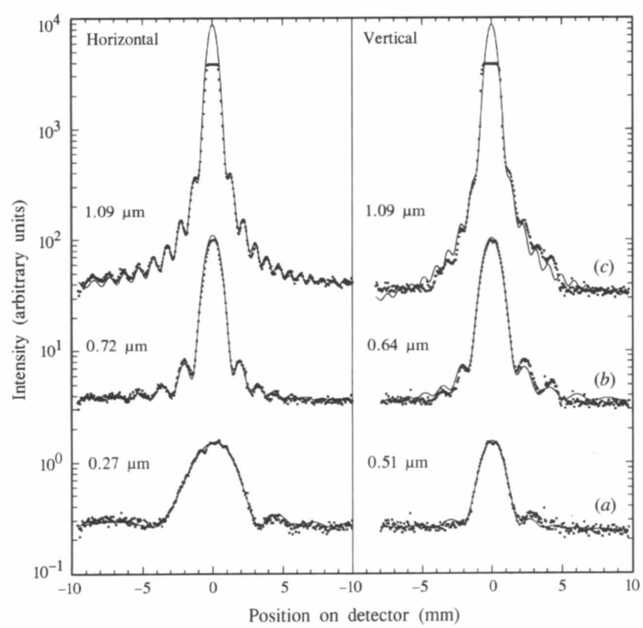

Figure 5
(*a*)–(*c*) Horizontal and vertical traces through Fraunhofer patterns from a square slit with the dimensions indicated (closed circles), together with the fitted curves. Data (*b*) are from the pattern shown in Fig. 4. The flat top of curves (*c*) arises from saturation of the detector. For clarity, the various curves were given an offset.

a plot of $w_{\min}$ and s_{opt} as a function of R. For $R \simeq 1$ m the minimum spot size is $\sim 6\,\mu$m. In order to obtain an illuminated sample area of $\sim 1\,\mu$m, R has to be ~ 3 cm. Only in special cases will it be possible to use smaller slit–sample distances, so in general the minimum useful slit size will be $\sim 1\,\mu$m.

Finally we need to consider the case where $h \neq 0$. In the Fraunhofer limit (2) and (3) give the correct answer, but near the condition of minimum beam size this is not valid. From (9) we see that the h-containing terms are not important when $hx/Rs \ll 1$. Since near the minimum beam size $x \simeq s$, we thus find that (11) and (12) are correct when $R \gg h$. This condition will be satisfied for many experimental set-ups. It is interesting to note, however, that when R is only a few times h, a second, narrow peak occurs. This happens because Δr is quadratic in x. Thus with a slit having a longitudinal offset one can generate two spatially separated coherent beams (see also Jark *et al.*, 1996). The width of each beam is still approximately given by (11).

5. Discussion and conclusions

The major advantage of the slits described here is their flexibility. In a coherent X-ray experiment one may start with a much wider and more intense beam during setting up. Without any adjustments the slits can then be closed to the required value. The main setting-up of the slits themselves consists of finding the value for zero opening. Once this is known, any value can be set by remote control. Even the very small slit sizes that we used proved to be stable within a few tenths of a micrometer over periods of hours. The use of a crossed pair of slits allows the beam dimensions/coherence to be tailored to the experimental requirements. The rectangular slits have a (small) advantage in that the intensity along the diagonal drops down faster than for a circular pinhole. This may be useful when scattered intensity is measured close to the direct beam.

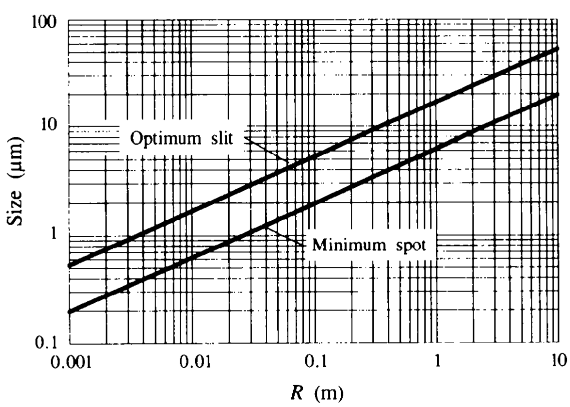

Figure 6
The minimum beam size and corresponding slit setting as a function of the slit–sample distance assuming a wavelength of 1 Å.

Very recently, Libbert, Pitney & Robinson (1997) obtained asymmetric Fraunhofer patterns using slits based on the rotation of a pair of polished rods. A potential disadvantage of such rods is the increased small-angle scattering from the edges compared with normal slit blades. This will be most important for small slit-to-sample distances. Lang, Kowalski, Makepeace, Moore & Clackson (1987) have used a similar set of polished rods to obtain Fraunhofer diffraction patterns corresponding to a minimum slit size of 2.8 μm.

In conclusion, slits of the type described here offer a flexible and simple way to produce micrometer-sized pinholes for coherent X-ray scattering experiments. Depending on the slit-to-sample distance it is possible to have an illuminated sample area as small as a micrometer.

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