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## Low-resolution small-angle scattering using neutron focusing optics

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abstract

Experiments at low values of scattering vector require tight collimation in the primary beam, as well as the ability to make measurements with good spatial resolution on the detector. Because neutron sources are not as intense as X-ray sources, large source and sample areas are used, and long instruments are necessary for adequate resolution. Increased count rates may be obtained using collimated beams which converge to a point on the detector. Further increases may be obtained with converging guides in the form of a focusing lens to increase the effective flux. Narrow guides enable the spectrometer dimensions to collapse for the same resolution. The critical angle of the fibers forming the focusing lens dominates the resolution, and such an instrument is useful only for low resolution measurements. However the greatly reduced length is only valuable if there is a high resolution detector to match the dimensions of the guide.

### INTRODUCTION

Small-angle neutron scattering instruments with large area position-sensitive detectors usually have long flight paths for adequate resolution, together with large source and sample areas for good counting statistics. In general, it is desirable to have greater neutron flux densities at the effective source position to allow both narrower beams and shorter distances for a higher detector count rate for the same resolution conditions. Increased count rates can be obtained using converging collimation [1], using collimating elements whose sizes are not much different from those on current spectrometers, so that the lengths of these instruments are of the same magnitude. The idea is to bring various small beams together such that they converge to a point at the center of the detector plane, and so increase the intensity at the sample.

Recent developments in both the use of capillary optical fibers for a neutron focusing lens [2,3] and high resolution solid state detectors [4] suggest the possibility of reducing both the transverse and longitudinal distances in such an instrument. The use of capillary optics as converging guides enables the transverse dimensions of the spectrometer to be reduced by an order of magnitude, so that the length of the instrument may be reduced by the same magnitude for measurements of comparable resolution. A necessary condition is the use of a solid state detector with very high spatial resolution which can enable the experimental arrangement to be placed in miniature.

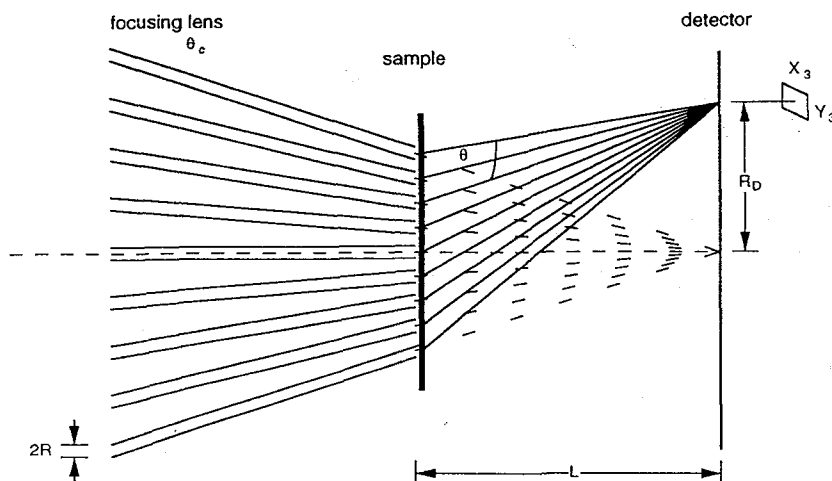


Fig. 1. The experimental arrangement of the focusing guide small-angle neutron scattering.

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### FOCUSING SPECTROMETER

The concept is depicted in Figure 1. A large number of guides of critical angle  $\theta_c$  and radius  $R$  point to the center of a detector with pixel sizes of  $X_3$  and  $Y_3$ . The sample is placed at the exit of the guides which is at a distance  $L$  from the detector. Scattering at an angle  $\theta$  is measured in a pixel at a distance  $R_D$  from the axis at a scattering vector  $Q = (2\pi/\lambda)(R_D/L)$ , where  $\lambda$  is the neutron wavelength. This is only useful for a detector whose resolution element is of the same magnitude as the radius of the fiber, and whose size is many orders of magnitude greater than this, so that a reasonably large range of scattering vectors may be measured simultaneously. The collimation affects the resolution characteristics for small-angle scattering measurements, and cross correlation in angle and position for points on the sample are minimized by placing the sample as close as possible to the exit of the collimation.

We have reported measurements [5] on a neutron focusing lens [6] composed of 721 capillary fibers, each of radius  $R = 0.18$  mm, which converges a quasi-parallel beam to a point with a half-divergence angle of  $3^\circ$ . Figure 2 shows an electron micrograph of the cross section of the lead glass polycapillary fiber. Note that the narrow capillary fibers have cross sections which are close to hexagonal. Each fiber contains nominally 1261 hollow channels, each of which has a  $6 \mu\text{m}$  diameter. The open area of the fiber is about 36% of its cross section. The transmission characteristics of the capillaries depend on the diameter of the channels and their radius of curvature [7]. However, the fiber contains over a thousand tiny capillaries, each of which are parallel to each other, to comprise the fiber guide. It is the radius  $R$  of the fiber which is important when the fiber is placed in a device, since most rays emerge from the fiber within a solid angle  $\pi\theta_c^2$  and from the area  $\pi R^2$ .

We have been using a video radiation detector [4] consisting of a charge injection device, with a pixel size of  $13.7 \times 12.5 \mu\text{m}^2$ , with a thin converter of fine  $^6\text{LiF}$  powder in epoxy. This device has enabled the imaging of the output (Figure 3) of neutrons transmitted by individual fibers of the capillary lens. The Figure only shows a small number of the transmitting fibers. It also shows the neutron spatial distribution at the focal plane of the lens (at a distance of 100 mm from the lens exit), indicating the focusing action of the lens. The focus has a full width at half maximum of about 1 mm, with a base width of about 2 mm. The mean neutron wavelength is about 0.3 nm, indicating that the alignment of individual fibers within the lens is not perfect.

We consider the design of a focusing guide small-angle scattering spectrometer, with the detector placed at the focal point of the focusing lens. The two dimensional detector is required for measuring anisotropic inhomogeneities. Optimization of the count rate in the detector element for a given value of the

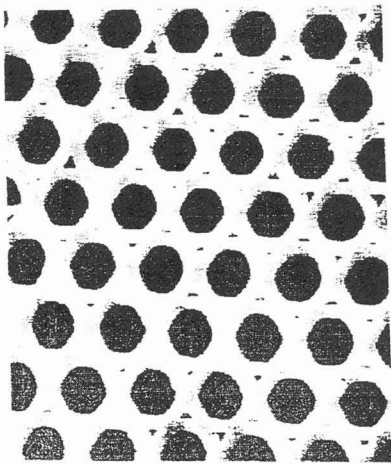


Fig. 2. An electron micrograph of the cross section of a capillary fiber showing the arrangement of the hollow capillaries.

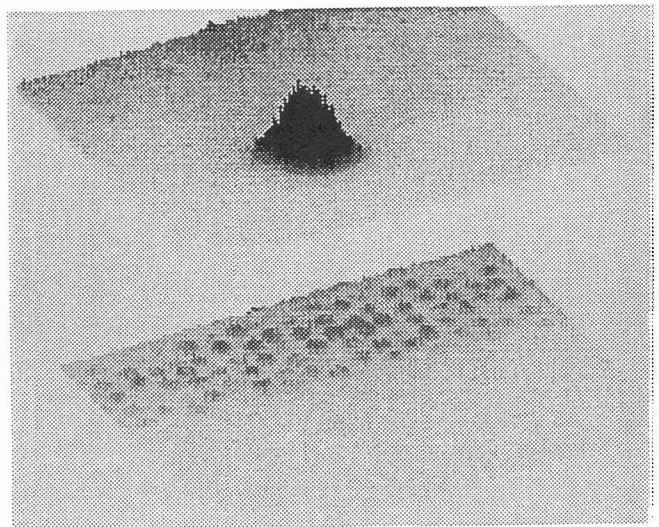


Fig. 3. The output of neutrons transmitted by individual fibers of the capillary lens, and (above) the neutron spatial distribution at the focal plane of the lens.

experimental resolution [8] results in a focusing distance  $L = (\sqrt{3}/2)R/\theta_c$ . The critical angle  $\theta_c$  depends both on the material of the capillaries and on the neutron wavelength  $\lambda$  used. The lead-glass multicapillary fibers (with a critical angle of 11 mrad nm<sup>-1</sup>) in the lens we have used [2] give an optimum focal distance  $L$  of only 28 mm for a beam of neutrons of wavelength 0.5 nm. In practice, the focusing lens used in our measurements has a focal length of 100 mm, which is optimum for SANS at a wavelength of 0.14 nm.

## MEASUREMENTS

We have tested this concept in an attempt to understand some of the magnitudes involved, using a sample of a layered block copolymer (kindly provided by K. Koppi, Univ. of Minnesota) such that the scattering pattern gives two equivalent (equatorial) peaks at  $Q = 0.216 \text{ nm}^{-1}$ . Figure 4 shows the peak profiles taken on NG7 small-angle scattering instrument, with the detector of pixel size 5.5 mm at a distance of 4 m, and using a neutron wavelength  $\lambda = 0.5 \text{ nm}$  with a resolution  $\Delta\lambda/\lambda = 15\%$ . The area between the two peaks has been masked by the beam stop (equivalent to  $Q < 0.104 \text{ nm}^{-1}$ ). From separation between peaks on SANS detector, we determine that the peaks occur at  $Q = 0.216 \text{ nm}^{-1}$ . For the SANS instrument in this normal configuration the resolution is  $\Delta Q_x = 0.081 \text{ nm}^{-1}$ . Using a pinhole of size 0.7 mm before the sample and a shorter source-sample distance, the data (also shown in Figure 4) now have a resolution  $\Delta Q_x = 0.112 \text{ nm}^{-1}$  (with the source term dominant in the resolution expression [9]).

When the VRD detector is placed a short distance ( $\sim 100 \text{ mm}$ ) behind sample, and with a pinhole at the sample, the source term remains large but now the sample term is dominant, with a resolution  $\Delta Q_x = 0.110 \text{ nm}^{-1}$ . Now the two peaks are not completely separated from the incident beam. This can be observed in the data in Figure 5 showing the profile through the incident beam which was not blocked. It is clear that the resolution is insufficient to resolve the peaks from the incident beam.

For the capillary lens, the guide critical angle defines the incident beam collimation, and the capillary term in the resolution is dominant, with a resolution of  $\Delta Q_x = 0.099 \text{ nm}^{-1}$ . This assumes that every capillary fiber points exactly to the same point on the detector plane, so that the two peaks should be resolved from the incident beam, if the lens is perfect. (This gives a slight improvement in resolution, though still less than for the SANS normal configuration, but a great decrease in intensity.)

The parameters for the various configurations are given in Table 1, with the relative intensities in Table 2. for the various configurations. We include a detector efficiency  $\eta$  of 70% for the SANS detector, and only 2% for the VRD. Note that the pixel count rate for the VRD detector with the focusing lens is about  $4 \cdot 10^{-5}$  of that for the SANS detector in a normal configuration. It should be possible in the future to improve the efficiency of the VRD by an order of magnitude.

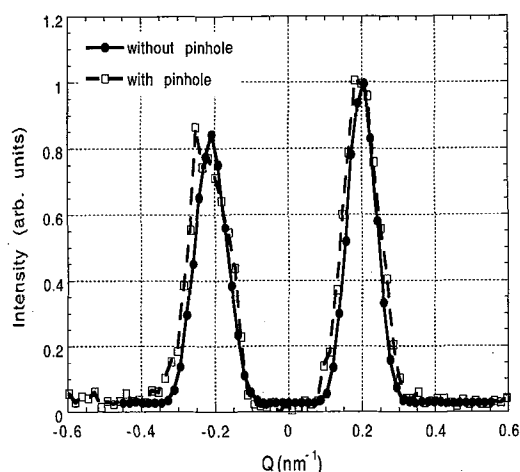


Fig. 4. The peak profiles of a block copolymer taken on NG7 small-angle scattering instrument showing two equivalent peaks at  $Q = 0.216 \text{ nm}^{-1}$ .

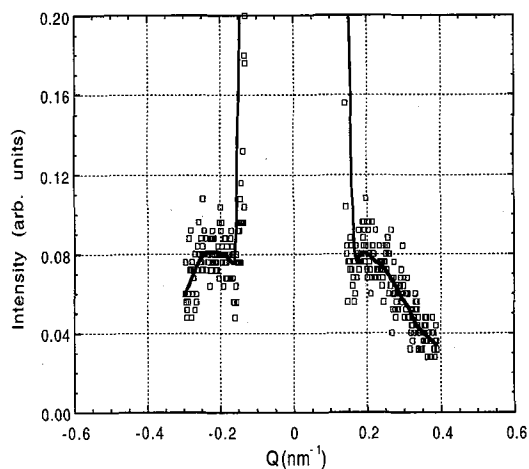


Fig. 5. The profile through the beam using the pinhole before the sample and the VRD camera for the block copolymer.

Table 1 The transverse and longitudinal dimensions of the various configurations

	$R_1$ (mm)	$R_2$ (mm)	$L_1$ (mm)	$L_2$ (mm)	$X_3$ (mm)
ordinary SANS	25	8.5	6400	4000	5.5
pinhole SANS	25	0.7	3400	4000	5.5
pinhole VRD	25	0.7	6400	115	0.0136
lens VRD	*	0.18	*	115	0.0136

\*equivalent  $R_1/L_1 \sim \theta_c = \gamma_c \lambda = 5.5 \cdot 10^{-3}$

Table 2 Relative intensities: count rate within a detector element is proportional to  $\eta A_1 A_2 A_3 / (L_1^2 L_2^2)$ , where  $\eta$  is the efficiency of detector: 70% for SANS and 2% for VRD

	scattering per unit solid angle $R_1^2 R_2^2 / L_1^2$	count rate per unit solid angle $\eta R_1^2 R_2^2 / L_1^2$	count rate per pixel $\eta R_1^2 R_2^2 X_3 Y_3 / (L_1^2 L_2^2)$
ordinary SANS	1	1	1
pinhole SANS	$2.40 \cdot 10^{-2}$	$2.40 \cdot 10^{-2}$	$2.40 \cdot 10^{-2}$
pinhole VRD	$6.78 \cdot 10^{-3}$	$1.94 \cdot 10^{-4}$	$1.43 \cdot 10^{-6}$
†lens VRD	$5.78 \cdot 10^{-1}$	$1.65 \cdot 10^{-2}$	$1.22 \cdot 10^{-4}$

†721 capillary fibers (note: in practice, there is a  $7 \times 9 \text{ mm}^2$  mask in front of the lens to shield the detector from the direct beam, so that the effective number of capillaries is reduced to about 650)

## CONCLUSIONS

We have confirmed experimentally that the use of a focusing lens using capillary fiber optics with a high resolution area detector is possible for low resolution small-angle scattering measurements. The major contribution to the resolution is from the critical angle of the capillary fibers. It would be helpful to have fibers with a lower critical angle, though this would reduce the transmission. In fact, the critical angle  $\gamma_c$  per unit wavelength of the fiber material defines a limiting value to the resolution, whatever the length of the instrument. In addition, fibers with a larger radius would enable the lens to be closer to optimum. (In practice, it is probably advantageous to have focal distances of perhaps up to 500 mm, which for the same material and the same wavelength implies fibers of diameter 4 mm, an order of magnitude greater than those that we have studied.) The poor resolution is a major limitation of the use of focusing lenses using capillary optics for small-angle scattering. The technique requires a detector with much higher efficiency, while keeping uniformity of response over the detector area; the detector spatial resolution is adequate. While this configuration will obviously not replace the traditional SANS configuration, it should eventually be useful for survey or characterization measurements, in order to determine the exact experimental conditions before using the long flight paths of a specialized SANS instrument.

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