Effects of the sample environment and collimation in the background measurement

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Abstract. Most of diffractometers have collimators in front of detectors either to reduce the background contribution or to increase the angular resolution. These collimation devices are designed to collect only the neutrons coming from the sample position. When a sample environment is required, its walls are close to the sample position and produce some unwanted effects on the diffractograms. A simple geometrical model allows a description of the observed patterns in the two-axis diffractometer D4 (ILL, Grenoble, France). The calculated effects are compared with experimental data for a vanadium bar as measured in the standard cryostat of that instrument. This kind of measurements allows the experimental determination of the sample transmission.

1. Introduction

In a scattering experiment, the design of the instrument is conceived in a way allowing the detector to see the whole sample. In order to reduce the background signal, the majority of instruments has a collimation system focused at the sample position, as shown in Fig. 1. Because the instruments must adapt to different sample sizes, usually the collimators are able to see a volume larger than the sample size, which is not a problem for ambient condition experiments, i.e. without a sample environment around the sample position. When a sample environment is required, if it is large enough to be far from the sample, its signal will stop by the collimator, as happens with the signal of the vacuum chamber in Fig. 1. But there is a region near the sample position where any material present will produce a scattered beam reaching partially the detector, producing a shadow effect on the observed diffractogram.

The cylindrical symmetry of a two axis diffractometer advice the use of cylindrical samples and environments. Thus the sample environment (a cryostat, a furnace or others) can be represented by a cylindrical screen around and centered at the sample position (Fig. 1).

We will consider the problem of an incoming neutron beam diffracted at three points, namely the sample and the two boundaries of the sample environment (Fig. 1). The scattered beam is registered by the detector placed at the end of the collimator. The shadow effects observed in the diffractograms are determined by the following set of parameters (Fig. 2): (i) the scattering angle ($2\theta$), (ii) the window length ($2w$), (iii) the detector length ($2f$), (iv) the sample-detector distance ($D$), (v) the sample-window distance ($d$), (vi) the sample diameter ($2s$), and (vii) the screen diameter ($2f$).

The geometrical relationship of all these parameters will describe the shadow effects. In particular, we will consider the case of neutron scattering and the D4 instrument at ILL [1],
but it is worth noticing that these effects could also be observed for other kinds of radiation or instruments with collimation tubes in front of the detectors. The aim of this work is to provide a geometrical interpretation to the effects observed in diffraction experiments performed at D4.

2. Geometrical calculation

We will consider the problem of an incoming beam of neutrons onto a cylindrical sample surrounded by a cylindrical screen. At this point, for the sake of simplicity, we make two assumptions: (i) we consider just only one screen around the sample, and (ii) we assume the three hitting points are punctual sources of scattered radiation. The former assumption is reasonably because considering a more complex situation with several concentric screens could hinder the understanding of a quite simple effect; the generalization to a more complex case can be easily made. The latter is also a reasonable hypothesis, because the non-punctual character of these sources will only contribute to produce smooth shapes instead of sharp features, but the rational behind is exactly the same.

In Fig. 2 we show the notation adopted for the description of the problem. The sample is located at the origin and the segment $AB$ represents the detection surface; the segment $PQ$ is the window of the collimator, which in fact defines the view of any point on the detector. Apart the sample itself, two other sources ($s_u$ and $s_d$) are defined by the intersection of the incoming beam (the $x$–axis) with the cylindrical sample environment. Only using some elementary geometrical considerations the coordinates of the points lying on the detector can be calculated as follows:

\[
\begin{align*}
    x &= D \cos 2\theta + (\ell - z) \sin 2\theta \\
    y &= D \sin 2\theta - (\ell - z) \cos 2\theta
\end{align*}
\]  

(1)

where $z$ is a variable increasing from A to B: $z = 0$ is A, $z = \ell$ is C and $z = 2\ell$ is B. The same geometrical considerations are valid for the points lying on the window, whose coordinates can be obtained just replacing $D$ by $D - d$, $\ell$ by $w$ and $z$ by $u$ in Eq. (1):

\[
\begin{align*}
    x &= (D - d) \cos 2\theta + (w - u) \sin 2\theta \\
    y &= (D - d) \sin 2\theta - (w - u) \cos 2\theta
\end{align*}
\]  

(2)

where $u$ is a variable increasing from P to Q: $u = 0$ is P, $u = w$ is R and $u = 2w$ is Q.

Tracing the straight lines from the upstream and the downstream sources located a $(−f,0)$ and $(+f,0)$ to the window edges Q and P, we obtain the light cone of both sources. At this point we identify two critical points ($\alpha$ and $\alpha'$) defining two special segments $\alpha B$ and $\alpha'A$. These
regions see only two of the three sources: $s_d$ and O, $s_u$ and O, respectively. Using Eqs. (1) and (2) we can compute the coordinates of $\alpha$ and $\alpha'$

\[
\begin{align*}
\alpha &= \frac{y_P f (x_B-x_A) + (x_P-f) (y_B-y_A)}{y_P (x_B-x_A) - (x_P-f) (y_B-y_A)} \quad \text{(3)} \\
\alpha' &= \frac{y_Q f (x_B-x_A) + (x_Q-f) (y_B-y_A)}{y_Q (x_B-x_A) - (x_Q-f) (y_B-y_A)} \quad \text{(4)}
\end{align*}
\]

Using the explicit expressions for points A, B, P and Q we obtain the following formulae showing the explicit dependence of the coordinates with the instrumental parameters:

\[
\begin{align*}
\alpha &= \frac{fD - (D-d) f (\sin 2\theta)^2 + D \cos 2\theta - w [f \cos 2\theta \sin 2\theta + D \sin 2\theta]}{(D-d) \cos 2\theta + w \sin 2\theta} \quad \text{(5)} \\
\alpha' &= \frac{fD - (D-d) f (\sin 2\theta)^2 + D \cos 2\theta - w [f \cos 2\theta \sin 2\theta - D \sin 2\theta]}{(D-d) \cos 2\theta + w \cos 2\theta} \quad \text{(6)}
\end{align*}
\]

These equations allow the calculation of the geometrical pattern shown in Fig. 3, where the instrumental parameters of D4 were used as example. As expected, because of the symmetry of the problem, the effect is maximum at $90^\circ$ and null at $0^\circ$ or $180^\circ$.
Figure 3. Calculation of the shadow effect for a given position of the detector ensemble, where we observe its angular dependence. The maximum effect is observed around 90°.

3. Comparison with D4 data
In previous section we have identified three different regions on the detector surface. A central region (αα′) that can see the three sources, and two side regions (α′A, α′B). For a flat diffraction pattern we should expect to observe step features on the detector with more intensity in the middle and less near the borders. In fact, taking into account the beam attenuation produced by the sample we should expect the low-angle step having more intensity than the high-angle one.

Figure 4 shows a comparison of our calculation with a real experiment performed at the two-axis neutron diffractometer D4. The experiment was performed on a vanadium sample (6.37 mm diameter), which is ideal to compare with our calculation because of its almost incoherent flat diffractogram [2]. The sample was contained in a standard orange cryostat, which here is represented by a vanadium cylinder of 50 mm of diameter surrounding the sample position (the screen in Fig. (Fig. 2)). The sample-detector and sample-window distances are 1146 mm and 280 mm, respectively. The example shown in Fig. 4 corresponds to two scattering angles near 90 degrees, where the effect is expected to be more important; the sample-window distance was left as a free parameter in the model. The results shown in Fig. 4 correspond to a final value of 770 mm.

In Fig. 4 we observe three levels of intensities for a given detector: at low-angles ($I_1$), at central-angles ($I_2$) and at high-angles ($I_3$). These intensities are related to the scattered intensities at the three sources ($I_u$, $I_s$ and $I_d$, for upstream, sample and downstream, respectively) by the following expressions: $I_1 = I_u + I_s$, $I_2 = I_u + I_s + I_d$, and $I_3 = I_s + I_d$. The three levels were fitted with constants and from them the intensities of the sources were obtained using the following relationships: $I_u = I_2 - I_3$, $I_d = I_2 - I_1$ and $I_s = I_1 + I_3 - I_2$.

In this case, for the cylindrical vanadium sample the observed intensities around 2θ = 90° (in counts per million of monitor counts) are $I_1 = 16860 \pm 30$, $I_2 = 18090 \pm 30$ and $I_3 = 16420 \pm 30$ (see Fig. 4), which yield to $I_u = 15190 \pm 50$, $I_s = 1670 \pm 40$ and $I_d = 1230 \pm 40$. Now we are able
to calculate the transmission of the sample as the ratio $T = I_d/I_u$, which gives for our vanadium cylinder $T = 0.74 \pm 0.03$. This transmission can also be calculated independently from tabulated values using the expression

$$T = \exp(-\sigma n x), \quad (7)$$

where $\sigma$ is the total cross section (absorption plus scattering), $n$ is the atomic density and $x$ is the thickness of the sample. For vanadium the scattering and absorption (for neutrons of 0.5 Å) cross sections are 5.1 b and 1.41 b, respectively [3]; the density is 6.11 g/cm$^3$ [4], which represents an atomic density of 0.07223 Å$^{-3}$. The thickness, in the punctual approximation, is actually the diameter, i.e. 6.37 mm. Using these values the calculated transmission is 0.741 in a very good agreement with the measured value.

![Figure 4](image.png)

Figure 4. Comparison of experimental data (points with error bars) and the calculation.

4. Conclusions

The effect of the sample environment can be qualitatively described with a very simple geometrical model. The small differences with the experimental data likely stem from the rough assumptions made in the model, mainly the fact of considering only punctual sources. The main objective of this article is to show that this geometrical effect exists and to give an interpretation. A more detailed analysis could provide a quantitative description without changing the essence of the observed effect. It is worth noticing that this a priori unwanted effect opens the possibility of measuring the sample transmission as the ratio between the downstream and upstream intensities.

References

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