The signal of sample environment as a way to measure sample transmission

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Abstract. In a recent article, the combined effects of the sample environment and instrument collimation have been described for the dedicated instrument for amorphous systems at ILL (D4) [1]. This undesirable effects on the diffractograms force to make complex and careful absorption corrections, which not always produce good enough results. In such cases, parts of the detector banks must be discarded with the consequent lose of statistics. This experimental problem can be used to obtain the transmission of the sample by an indirect way.

Considering the most generalized case of a cylindrical sample with a cylindrical sample environment (furnace, cryostat, etc), the diffractogram can be described as coming from three different sources: the sample itself, the upstream and downstream sample environment. These sources produce three well-differenced regions on the individual diffractograms observed by one particular detector. These three different measured signals are linear combination of the sample, upstream and downstream intensities produced by each source. In this way, upstream and downstream intensities can be determined, and the experimental transmission of the sample can be obtained. The experimental results corresponding to the standard cryostat are compared with the theoretical calculation of the transmission for a series of samples measured at D4 over the past years. This comparison shows that an undesirable effect observed on the diffractograms could even be useful under some circumstances.

1. Introduction

The transmission of a sample is useful to perform attenuation corrections in scattering experiments. This magnitude gives also the possibility of determining the macroscopic cross section of the sample, which contains the global density of the system.

It is not always easy or possible to perform a direct measurement of the transmission. When it is possible the intensity of the incident beam (no sample in the beam) and the transmitted intensity through the sample should be registered by a detector placed behind the sample position in the beam direction. If the intensity is to high, an attenuator should be placed in front of the detector to avoid any damage. The transmission of the sample is simply calculated as the ratio of the transmitted intensity I and the incident intensity I_0

$$T = I/I_0, \tag{1}$$

where a slab geometry is assumed.

In the case where a sample environment is necessary to perform the experiment, the walls of this device (cryostat, furnace, etc) will be additional sources of radiation being registered by the detector. Usually these walls can not be neither too far from nor too close to the sample position, *i.e.* at some centimeters of the sample. In this way, the intensity scattered by the walls placed upstream and downstream are useful to determine the transmission of the sample [1], without placing a detector in the direct beam, just a detector located at a wide angle position is necessary. The problem is that without collimation in front of the detector, the signals coming from the three sources will be registered together, avoiding in this way any determination of the transmission. The D4 collimation allows this kind of measurements, as it will be shown below.

2. Geometrical effects

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 (see Fig. 1 taken from [1]).

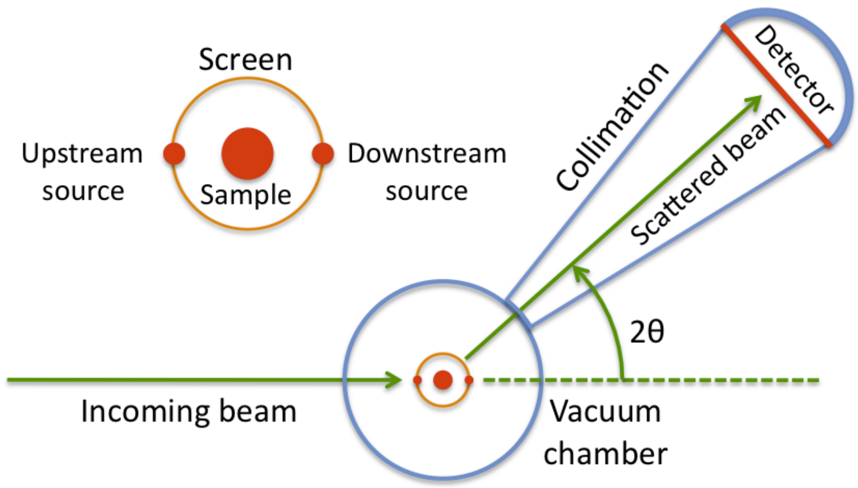

Figure 1. Sketch of the diffractometer device.

Now we focus the discussion on a particular neutron diffractometer having a series of detector banks, each one with a collimation [2]. This collimation is designed in order to register the sample signal with the whole surface of the detector, but tight tight to sufficiently reduce the background. What happens with this collimation is that the central part of the detector sees the sample plus the two extra sources, upstream and downstream, coming from the sample environment. The low-angle part of the detector can only see the upstream signal plus the sample, while the high-angle part can see the sample plus the downstream signal. Figure 2 shows a typical diffractogram, where we can clearly identify the observed signal in the high-, mid- and low-angle regions as I_1, I_2 and I_3, respectively (where $I_3 < I_1 < I_2$). I_3 is smaller than the others due to the attenuation of the sample. In this way, the relationship between these observed intensities and the point sources is

$$\left\{ \begin{array}{lcl} I_1 & = & I_\mathrm{u} + I_\mathrm{s} \\ I_2 & = & I_\mathrm{u} + I_\mathrm{s} + I_\mathrm{d} \\ I_3 & = & I_\mathrm{s} + I_\mathrm{d} \end{array} \right. , \tag{2}$$

or

$$\left\{ \begin{array}{lcl} I_\mathrm{u} & = & I_2 - I_3 \\ I_\mathrm{s} & = & I_1 - I_2 + I_3 \\ I_\mathrm{d} & = & I_2 - I_1 \end{array} \right. , \tag{3}$$

where I_s, I_u and I_d are the intensities scattered by the sample, upstream and downstream sources, respectively. These expressions allow the calculation of the experimental sample transmission T_s simply using the Eq. (1) where I_d and I_u are I and I_0, respectively.

We can also obtain a theoretical expression for the transmission of a cylindrical sample of radius R,

$$T_\mathrm{c} = \exp(-\frac{\pi}{2}\, n\, \sigma\, R), \tag{4}$$

where n is the atomic density of the sample (number of scatterers per unit volume) and σ is the total (scattering plus absorption) neutron cross section. In the evaluation of the absorption cross section, we use the wavelength-dependent expression

$$\sigma_{\mathrm{abs},\lambda} = \frac{\lambda}{\lambda_\mathrm{th}}\, \sigma_\mathrm{abs}, \tag{5}$$

where σ_abs and λ_th are the absorption cross-section and wavelength for thermal neutrons, respectively.

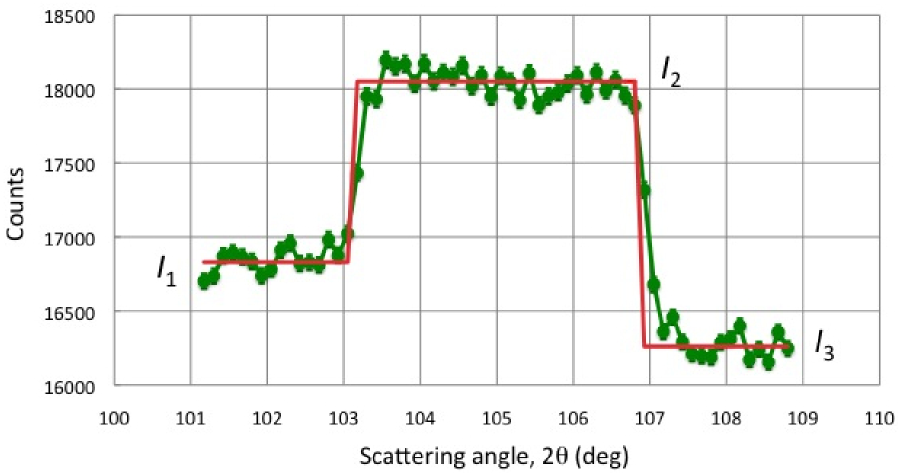

Figure 2. Typical shape of the registered signal on a given detector. The three levels of intensity corresponding to the low- (I_1), mid- (I_2) and high-angle (I_3) regions are shown.

3. Experiments

The diffraction experiments were performed at the two-axis diffractometer D4, ILL, Grenoble, France [2]. We analyzed two independent sets of experiments on a cylindrical vanadium sample of 6.37 mm diameter placed at the center of a cryostat tail also made of vanadium (50 mm inner-diameter and 0.45 mm thick). The incident neutron wavelength was of 0.5 Å and both experiments were performed at room temperature.

Data sets for each experiment correspond to different positions of the detection ensemble constituted of 9 banks of detectors, each one with 64 detection cells. The signal of the 9 banks are independently registered, but only those detectors clearly affected by the shadow effect were used in the evaluation of the three levels of intensity (I_1, I_2 and I_3). This shadow effect (Fig. 2) is maximum for those detectors placed near a scattering angle of 90°. In this way, for the first experiment we could collect 49 independent set of data, while for the second we collected 102 independent data.

For each set of data, constants were fitted to the three observed levels of intensity I_1, I_2 and I_3. These three constants are the input values for Eq. (3), allowing the obtention of the experimental upstream and downstream intensities.

4. Results and discussion

For vanadium, the scattering cross section is 5.1 b and the absorption cross section for thermal neutrons (1.8 Å) is 5.08 b [3]; the absorption cross section corresponding to the wavelength of 0.5 Å is 1.41 b, giving a total cross section of 6.51 b. The vanadium density is $\rho = 6.11$ g/cm^3, which corresponds to an atomic density of $n = 0.07223$ Å^{-3} ($n = \rho/M_{\text{mol}}\, N_{\text{A}}$, where N_{A} is the Avogadro's number and $M_{\text{mol}} = 50.9415$ g/mol is the molar mass). Using these values and the Eq. (4), the theoretical value for the sample transmission is 0.791 ± 0.003.

From the two sets of experimental data, and using Eqs. (1) and (3), we obtain the experimental transmission of the sample 0.75 ± 0.06 and 0.73 ± 0.13. These values compares very well with the theoretical one, confirming the validity of the proposed method.

It is worth noticing that the experimental values are slightly smaller than the theoretical one. This could be explained by the approximations of point sources, made to deduce the relationship between the sources and the observed intensities (Eq. (2)). In fact, the neutron beam is bigger than the sample diameter; in this case, the beam width was of 11 mm meaning that only 55% of the incident beam will pass through the sample. Consequently, part of the beam scattered by the downstream source has not been attenuated by the sample. Taking into account the focalization of the neutron beam on the sample position, the scattered intensity by the downstream source should be reduced by 43%. Thus, the new transmission can be obtained multiplying the previous experimental values by a factor of $0.45/0.43 = 1.05$, which yields to the following values for the experimental transmissions: 0.79 ± 0.06 and 0.77 ± 0.13, much closer to the theoretical value.

There is an experimental way to take into account this background effect. Replacing the sample by an absorbent, the scattered beam not attenuated by the sample can be directly measured. The absorbent sample depends on the energy of the incident neutrons, being Boron the best one for hot neutrons and Cadmium for thermal neutrons. This background could be subtracted from the experimental data and the effect will be more important for the I_{u} intensity than for the I_{d}, which naturally will produce a higher transmission value.

This method could be applied for all the working wavelengths on the instrument, because no influence of the neutron wavelength is expected, except by the known dependence of the absorption cross section with the neutron energy (Eq. (5)).

5. Conclusions

The transmission of a known cylindrical sample has been theoretically calculated using the its density and macroscopic total cross section. This value is in a very good agreement with two independent experimental values obtained applying the method proposed in this article. The method consists in using the scattered signals from the upstream and downstream sample environment as incident and transmitted intensities. Thanks to the instrument collimation, these signals are easily observed on the detection banks as a double step function (Fig. 2). A priori this is an undesirable (but correctable) effect observed on the diffractograms; however it has been demonstrated that it could be useful under some circumstances.

References

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