AN INNOVATION IN TRANSMISSION COEFFICIENT MEASUREMENT

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ABSTRACT

An innovative device is developed to measure the diffraction pattern and transmission coefficient simultaneously. In a diffraction system using a two-dimensional (2D) detector, a beam-stop is normally used in the transmission mode to prevent the direct beam from striking the detector. In the present system, the beamstop is modified to allow the direct beam to pass through the center of the beamstop. A pinhole and an attenuator are used to control the size and intensity of the direct beam passing through the beamstop.

INTRODUCTION

In transmission mode x-ray diffraction, such as small angle x-ray scattering (SAXS), the transmission coefficient ($T$) of the sample is an important parameter in data collection and data analysis. The transmission coefficient ($T$) is defined as the ratio of direct beam intensity observed behind the sample ($I$) to that observed without sample ($I_0$).

$$T = \frac{I}{I_0} = \exp(-\mu \cdot t)$$ (1)

Where $\mu$ is the linear absorption coefficient of the sample and $t$ is the sample thickness. The transmission coefficient can be calculated if the absorption coefficient ($\mu$) and the sample thickness ($t$) are given. $T$ ranges from 0 to 1 (0-100%), $T=0$ corresponds to a complete attenuation, while $T=1$ (100%) represents complete passage. The transmission coefficient is an important factor in determining diffraction results. For instance, the maximum scattered intensity occurs when $T=\exp(-1)=0.37$ [1]. It is also necessary to have the transmission coefficient measured when doing SAXS scanning over an inhomogeneous sample, so the SAXS data can be normalized against density variation [2,3].

However, in most cases, it is difficult or impossible to calculate the transmission coefficient of a sample due to its inhomogeneous structure or dimension variation. The transmission coefficient can be measured by collecting data with and without sample according to the definition. In many cases, such as in SAXS systems, the measurement of transmission coefficient is either tedious or very difficult. For example, in a suggested method [1], one has to move the direct beam away from the beamstop, turn the generator to its minimum power and attenuate the beam intensity by a factor of $10^3$. Another indirect method measures the scattering from an amorphous carbon film positioned behind the sample as is shown in Figure 1. The transmitted beam through the sample is scattered by the glassy carbon foil. The integrated intensity in a defined region on the 2D detector represents the transmitted beam intensity. For scanning SAXS, the sample is first scanned with the GC to measure the transmission coefficients at each scanning position. The scanning SAXS data is then collected after removing the glassy carbon.
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With the new method the diffraction pattern and the transmission coefficient can be measured simultaneously. The design has a patent pending with the trade name of SAXSEye. Figure 2 shows the schematic of a scanning SAXS (Bruker NanoStar) system with the modified beamstop. It is a small angle scattering system using a two-dimensional (2D) detector. A beam-stop is used to prevent the direct beam striking the detector. It is necessary sometimes to measure the beam transmission so the diffraction data can be normalized against the sample thickness (or density) variation. It is especially important to measure the transmission coefficient variation at each scanning position when scanning the sample in the X-Y directions. In a diffraction system with SAXSEye the direct beam passes through a modified beamstop, so that the direct beam is attenuated and limited in the center region.
BEAMSTOP DESIGN

Figure 3 is an illustration of the modified beamstop for SAXSEye. The beamstop has a through hole with the diameter of \( BL \) to allow the direct beam pass through the center of beamstop. The pinhole and the attenuator are used to control the size and intensity of the direct beam passing though the beamstop. The pinhole aperture diameter is \( d \) and the attenuator thickness is \( t_0 \).

![Diagram of beamstop](image)

Figure 2. The modified beamstop for SAXSEye in which the transmitted direct beam from the sample passes the beamstop through an attenuator and the transmission size is limited by the pinhole.

The integrated intensity of the measured primary beam passing through the above SAXSEye beamstop is approximately given by

\[
I_0 = kP_G \frac{d^2}{s^2} \exp(-t_0 \cdot \mu) \tag{2}
\]

Where \( k \) is a scaling factor determined by the SAXS system, \( P_G \) is the power of the x-ray generator, \( s \) is the size of the primary beam spot on the detector if the beamstop is removed. Although both the pinhole size \( d \) and the attenuator thickness \( t_0 \) determine the integrated intensity \( I_0 \), the two parameters play different roles. The beam leak pinhole \( (d) \) allows a small portion of the direct beam passing through the beam-stop. The attenuator with an appropriate thickness \( (t_0) \) attenuates the transmitted beam below the detector saturation level. A good combination of \( d \) and \( t_0 \) should limit the intensity below the maximum count rate per pixel when \( I_0 \) is measured, but enough counting statistics when a relative thick sample is loaded. The ideal situation is that the \( I_0 \) is measured near the maximum counting rate.

TRANSMISSION COEFFICIENT MEASUREMENT

The measurement was done on the Bruker D8-Discover GADDS system with the following parameters: Cu-tube, 40kV/40mA power, 0.3mm collimator, 150 mm detector distance, the beamstop size BS=4mm and BL=2mm, the pinhole diameter \( d=30\mu m \), and the Al foil attenuator \( t_0=60\mu m \). The latter two parameters were determined by gradually increasing the power and
reducing the absorber in front of the 2D detector. The SAXSEye makes the beamstop positioning easier and more accurate for one can adjust the beamstop to the beam center by maximizing the transmission intensity. With the above configurations, the $I_0$ was measured at the maximum counting rate of 125c/s/p (counts per second per pixel). The previous experiment shows that the Bruker HI-STAR area detector can measure up to 200 counts/second/pixel with good linearity.

The transmission coefficients of various layers of the oriented polyester film of 0.2mm thick are measured. The intensity ($I_0$) was taken as the average of the measurements before and after the sample measurements. The result shows that the intensity fluctuation within 24 hours is less than 3% so the one measurement before the sample measurement can be used as $I_0$ without introducing much error. Figure 4 is a magnified frame collected with 6 layers of the oriented polyester films. The majority of the transmitted direct beam is blocked by the beam-stop. The bright center spot bounded by the beamstop shadow represents transmission intensity. It can easily be read from the total counts within a “box” cursor. In the future, the GADDS and SAXS programs will be able to calculate the integrated intensity from a circular region user-defined by its center pixel position and radius. The results of the transmission measurement collected in 60 seconds for 0 to 14 layers of the oriented polyester films are listed in Table 1. The intensity ($I$) is the total counts of the transmitted beam though the SAXSEye. $\Delta T$ is the transmission coefficient of each individual layer calculated from the intensity ratio of the consecutive measurement.

$$\Delta T_n = \frac{I_n}{I_{n-1}}$$

(3)

The average transmission coefficient of each layer is 87.3% with the standard deviation of 1.3%. $T$ is the transmission coefficient of the total layers.

Figure 4. The center portion of the frame collected with 6 layers of the oriented polyester films with magnification of 8X. The bright spot inside the shadow of the beam-stop represents the transmission.
Table 1. The total counts of the transmission and transmission coefficients for 0 to 14 layers of the oriented polyester plates.

<table>
<thead>
<tr>
<th>Number of layers</th>
<th>Frame filename</th>
<th>Intensity $I$ (counts)</th>
<th>$\Delta T$</th>
<th>$T$</th>
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<tbody>
<tr>
<td>0 (before)</td>
<td>SAXINou.060</td>
<td>335,997</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0 (after)</td>
<td>SAXIPL00u.060</td>
<td>327,413</td>
<td></td>
<td></td>
</tr>
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<td>0 (average)</td>
<td>&lt;Average&gt; $I_0$</td>
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<td>100.0%</td>
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</tbody>
</table>

Average transmission coefficient of one layer: $87.3\% \pm 1.3\%$

The transmission coefficient of a single layer $T_1$ is given as

$$T_1 = \exp(-\mu \cdot t_1)$$

(4)

Where $t_1$ is the thickness of one layer. Then the transmission coefficient for a stack of $n$ layers is given as

$$T_n = \exp(-\mu \cdot n t_1) = T_1^n$$

(5)

or

$$\ln T_n = n \ln T_1$$

(6)

The equation shows that the logarithm of total transmission of $n$ layer is proportional to the number of layers. Figure 5 is the measured transmission coefficient for various layers in logarithm scale plotted against the number of layers. A linear correlation is observed with an almost perfect correlation with coefficient of determination of 0.999.

**CONCLUSIONS**

- The SAXSEye allows a SAXS system to measure the diffraction pattern and the transmission coefficient simultaneously. In the Scanning SAXS system, it is possible to take radiographs from transmitted direct beam and scattering pattern, analogous to the bright field and dark field images of the transmission electron microscope (TEM).
• The SAXSEye can measure the transmission coefficient with high accuracy. The transmitted beam size and intensity should be properly controlled by the pinhole and attenuator so that the transmitted beam is isolated from the diffracted pattern and the maximum transmission intensity is below the detector local count rate.

• The SAXSEye feature can be used to position the beamstop more precisely. It is normally easy to adjust the beamstop to block the direct beam, but difficult to set the beamstop to the true center of the x-ray beam. Since most beam profiles have the maximal intensity at the center of beam, the beamstop can be precisely positioned by maximizing the passing through intensity.

• The SAXSEye makes it possible to normalize diffraction patterns collected with fluctuating sources, such as synchrotron radiation. Since the passing through intensity is proportional to the direct beam intensity, for a intensity fluctuating source, the diffraction data can either be normalized by the total passing though counts, or be collected with the total exposure controlled by a given passing tough counts.

Figure 4. The transmission coefficient as a function of sample thickness (number of 0.2mm polyester layers).

REFERENCE

[1] John D. Barnes, SAXS From Polymers, APS-DHPP Short Course, National Institute of Standard and Technology, Gaithersburg, MD 20899, USA, 19 March 1994