DEVELOPMENT STUDY OF THE X-RAY SCATTERING PROPERTIES OF A GROUP OF OPTICALLY POLISHED FLAT SAMPLES

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# Development Study of the X-ray Scattering Properties of a Group of Optically Polished Flat Samples

**Abstract**

A group of twelve optically polished flat samples were used to study the scattering of X-rays. The X-ray beam reflected from the twelve optical flat samples was analyzed by means of a long vacuum system of special design for these tests. The scattering measurements were made at 8.34° and 0.92° angle of incidence. The results for ten of the samples are comparable, the two exceptions being the fire polished samples.

**Key Words**

X-ray Optics, Scattering
PREFACE

The objective of Phase I of this program was to study the X-ray scattering of 12 optically polished flat samples at 8.34Å and 0.92° angle of incidence. The tests indicate comparable results for ten samples with the two fire polished samples showing irregularities in the data.
INTRODUCTION

It has been shown\textsuperscript{1-4} that the limiting factor in the performance of X-ray telescopes is the scattering of soft X-rays by the optical surfaces.

One important point learned in previous tests of soft X-ray telescopes is that visible light tests do not predict the performance in the soft X-ray spectral region. The manufacturing techniques used were not sufficiently controlled to produce a telescope giving predictable performance. For future X-ray astronomy missions such as the HEAO, it is important to choose the optimum materials and manufacturing techniques to provide higher quality, lower cost telescopes. A previous study\textsuperscript{5} had made some of the required measurements and answered some questions regarding materials, but it also produced some questions which can only be answered by continued research into the problem.

The first phase of this program involved measuring the properties of an X-ray beam reflected by optical flat samples at one wavelength and one angle of incidence. In order to obtain data which are suitable for application to grazing incidence mirror construction, it is necessary to use a measurement system having angular resolution of the order of telescope resolution - one arc second or so. A total of one dozen optical flat samples were tested in the course of this work.
EXPERIMENTAL PROGRAM

During the first phase of this program the scattering of X-rays by polished optical flats was measured for 12 samples, at one wavelength (8Å), and at one angle of incidence (0.92°). The products of the first phase of this program were the data recorded in the various tests. The following paragraphs in this chapter describe these data.

Optical Flat Tests

The scattering of X-rays by polished optical flats was measured by means of a special vacuum line which was approximately 36 feet long. The experimental configuration of the X-ray scattering measurement equipment is shown in Figure 1. The vacuum system consisted of three stainless steel chambers, each 24 inches in diameter and 12 inches high. The three chambers were interconnected by 6 inch I.D. stainless steel tubing. The distance between the center line of each chamber was 17.2 feet. The middle chamber housed the sample and secondary detector with one end chamber enclosing the X-ray source and the other end chamber enclosing the detector used for measuring the reflected X-ray beam.

The X-ray source used in this phase of the program was aluminum (8.34Å), the anode voltage was 3.5 KV and the anode current was 20 - 30 ma. The filament was essentially a line source which was .005 inch diameter tungsten wire. The purpose
Figure 1 - Schematic of Scattering Equipment
of the slit in front of the source and the slits at the sample and detector were to yield a theoretical FWHM value of the scattering curves which, when compared to the experimental FWHM value, would give a value of the amount of scattering produced by the surface imperfections of the sample.

In the middle chamber, 17.2 feet away from the X-ray source, the sample was mounted in the sample holder which could be rotated in either direction in one arc minute steps about an axis parallel to the sample surface and perpendicular to the incoming beam. A secondary detector was placed adjacent to the sample holder to monitor the direct beam from the X-ray source so that any variations that occurred at the source could be taken into account in the final analysis of the scattering data. A beam stop with a one inch diameter hole was placed directly in front of the sample between the sample and the detector end of the system. The beam stop helped reduce the amount of X-rays scattered from the chamber walls or X-rays from the direct beam from reaching the primary detector. In the end chamber 17.2 feet from the sample the primary detector was placed on a mount that could be translated across the scattered X-ray beam in 0.1 arc second intervals. The detector was a flow type proportional counter with a 1/4 mil aluminum window. A .004 inch wide slit was placed directly in front of the window of the detector.
The system was aligned by means of a laser so that the angle of incidence between the sample and the X-ray source was 0.92°. After the sample and the primary detector were put in position, the chamber was closed and the pressure reduced to approximately 1 x 10⁻⁵ Torr. The high voltage for the X-ray source was then turned on and the scattered beam was located. After the peak of the scattered beam was located, the primary detector was positioned +30 arc seconds from the peak. A typical run consisted in translating the detector and slit across the scattered X-ray beam in one arc second intervals and counting for 100 seconds at each point from +30 arc seconds to -30 arc seconds.

Two types of data were collected for each sample. The first was a scan of intensity values as a function of angle as the detector was scanned across the reflected beam. Readings were taken every arc second so that a detailed shape of the scattering curve could be determined. These curves were expected to be symmetrical, but the results indicated that they were not always regular in shape. These curves were used to find the position of the peak reflected intensity, and the width of this peak was interpreted as a characteristic of the sample. The second type of data consisted of readings taken at nine selected points (0, ±5, ±10, ±20, ±30 arc seconds) about the peak. These points were used in making calculations of the percentage of radiation scattered away from the main reflected beam.
Data Format

The experimental configuration used in this phase of the program had the source slit set at .002 inch, the sample slit set at .002 inch (equivalent to .004 inch), and the detector slit set at .004 inch.

From geometrical considerations, assuming no scattering, the X-ray beam reflected from the sample should have a trapezoidal intensity profile that has an eight arc second FWHM. The 17.2 ft working distance was chosen so that one arc second was equal to .001 inch of linear motion. The value $S_w$ was defined as the difference between the observed curve width (FWHM) and the calculated width (FWHM) based on geometrical considerations. Negative values of $S_w$ are thought to be due to error in setting the slits or in the motion of the system during the course of a run. The error in $S_w$ is about ±2 arc seconds.

As shown in the graphs in Figures 2 - 13, the negative angles are in a direction away from the surface of the sample. The failure of the nine points data to be exactly symmetrical should be interpreted as an uncertainty in the position of the detector. This positional uncertainty in locating the peak was ±3 arc seconds, but the relative locations of the nine points were uncertain by less than ±1 arc second.

Scattering Curves

Figures 2 through 13 show the relative intensity curves obtained from the 61 point raw data. Each individual graph contains
Figure 2 - Scattering Curve

$S_W = 0.3$

\[ G_1 = 8 = 0.92 \]
Figure 3 - Scattering Curve

G 2 - 8 - 0.92

$S_w = -0.6$
Figure 4 - Scattering Curve

G 3 - 8 - 0.92

$S_w = 0.4$
Figure 5 - Scattering Curve

G 4 - 8 - 0.92
Sw = -0.4
Figure 6 - Scattering Curve

G 5 - 8 - 0.92
S_w = 0.3
Figure 7 - Scattering Curve

$S_W = 13.4$
Figure 8 - Scattering Curve

Relative Intensity

G 7 - 8 - 0.92

$S_{\text{W}} = -0.8$

Angle (Arc Seconds)

-13-
Figure 9 - Scattering Curve

G 8 - 8 - 0.92(2)

$S_W = -1.0$

ANGLE (ARC SECONDS)

RELATIVE INTENSITY
Figure 10 - Scattering Curve

\[ \theta_{\text{G}} = 0.9 \]

\[ S_W = 0.9 \]
Figure 11 - Scattering Curve

G 10 - 8 - 0.92

$S_W = 0.6$
Figure 12 - Scattering Curve

$G_{11} - 8 - 0.92$
$S_w = 0.6$

RELATIVE INTENSITY

ANGLE (ARC SECONDS)
Figure 13 - Scattering Curve

G 12 - 8 - 0.92
$S_W = 8.7$

Relative Intensity

Angle (Arc Seconds)
an identification number in the upper right hand corner. The first number is the sample number, the second number is the wavelength, and the last number is the angle of incidence at which the sample was positioned. Each graph also contains a value representative of the scattering width in arc seconds, designated as $S_w$. This value was defined as the difference between the observed curve width (FWHM) and the calculated width (FWHM).

The following summarized observations are made for samples which indicate obvious irregularities.

**Sample No. G6** showed a double peak and wide FWHM with a slight bump occurring at +30 arc seconds. **Sample No. G12** showed a slight bump near the peak and had a wide FWHM with slight irregularities near +30 arc seconds.

**Nine Points Data**

The nine points data was processed so that tables showing relative intensities could be prepared. The intensity values at the nine points were defined as:

$I_{-4} = \text{Intensity at } -30 \text{ arc seconds}$

$I_{-3} = \text{Intensity at } -20 \text{ arc seconds}$

$I_{-2} = \text{Intensity at } -10 \text{ arc seconds}$

$I_{-1} = \text{Intensity at } -5 \text{ arc seconds}$

$I_0 = \text{Intensity at peak value}$

$I_1 = \text{Intensity at } +5 \text{ arc seconds}$

$I_2 = \text{Intensity at } +10 \text{ arc seconds}$
\[ I_3 = \text{Intensity at +20 arc seconds} \]
\[ I_4 = \text{Intensity at +30 arc seconds} \]

The relative intensity \( R_I \) was then defined as:

\[ R_I_j = I_j / I_0 \]

This definition produced a set of relative numbers which would then be compared with one another and these values are listed in Table 1.

The percent scattered, \( S \), was defined as:

\[ S = \left( \frac{\sum_{j = -4}^{4} I_j - \sum_{j = -2}^{2} I_j}{\sum_{j = -4}^{4} I_j} \right) / \left( \sum_{j = -4}^{4} I_j \right) \]

This then is another number which can be used as an index of sample performance in comparisons. These values of percent scattered are tabulated in Table 2. The percent scattered values represent an intensity that must come only from scattered radiation. They provide a calculation method which is objective and does not depend on experimental uncertainties in slit width. The method of adding the area under the curve is subject to large uncertainties because of possible slit width errors.

**Reflecting Efficiency Measurements**

The "efficiency factor", see Table 2, is not an attempt at measuring reflecting efficiency, but, rather is used to normalize the data against fluctuations in source intensity. The secondary detector records an intensity (not flux) simultaneously with that obtained in the primary detector. The values recorded will vary
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Angle (Arc Sec)</th>
<th>Relative Intensities</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>-30</td>
<td>-20</td>
</tr>
<tr>
<td>1</td>
<td>0.004</td>
<td>0.010</td>
</tr>
<tr>
<td>2</td>
<td>0.001</td>
<td>0.003</td>
</tr>
<tr>
<td>3</td>
<td>0.002</td>
<td>0.006</td>
</tr>
<tr>
<td>4</td>
<td>0.001</td>
<td>0.005</td>
</tr>
<tr>
<td>5</td>
<td>0.005</td>
<td>0.009</td>
</tr>
<tr>
<td>6</td>
<td>0.017</td>
<td>0.048</td>
</tr>
<tr>
<td>7</td>
<td>0.003</td>
<td>0.006</td>
</tr>
<tr>
<td>8</td>
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<td>0.006</td>
</tr>
<tr>
<td>9</td>
<td>0.003</td>
<td>0.006</td>
</tr>
<tr>
<td>10</td>
<td>0.006</td>
<td>0.004</td>
</tr>
<tr>
<td>11</td>
<td>0.001</td>
<td>0.004</td>
</tr>
<tr>
<td>12</td>
<td>0.064</td>
<td>0.119</td>
</tr>
</tbody>
</table>

TABLE 1 - Nine Point Data Phase I Test
<table>
<thead>
<tr>
<th>Sample</th>
<th>Scattering Width ($S_w$)</th>
<th>Percent Scattered ($S$)</th>
<th>Efficiency Factor ($E$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.3</td>
<td>1.30</td>
<td>0.01077</td>
</tr>
<tr>
<td>2</td>
<td>-0.6</td>
<td>0.69</td>
<td>0.01230</td>
</tr>
<tr>
<td>3</td>
<td>0.4</td>
<td>0.75</td>
<td>0.01811</td>
</tr>
<tr>
<td>4</td>
<td>-0.4</td>
<td>0.76</td>
<td>0.01507</td>
</tr>
<tr>
<td>5</td>
<td>0.3</td>
<td>1.33</td>
<td>0.01207</td>
</tr>
<tr>
<td>6</td>
<td>13.4</td>
<td>12.77</td>
<td>0.00842</td>
</tr>
<tr>
<td>7</td>
<td>-0.8</td>
<td>1.00</td>
<td>0.01938</td>
</tr>
<tr>
<td>8</td>
<td>-1.0</td>
<td>1.10</td>
<td>0.00884</td>
</tr>
<tr>
<td>9</td>
<td>0.9</td>
<td>0.91</td>
<td>0.01274</td>
</tr>
<tr>
<td>10</td>
<td>0.4</td>
<td>0.83</td>
<td>0.01067</td>
</tr>
<tr>
<td>11</td>
<td>0.6</td>
<td>0.64</td>
<td>0.01144</td>
</tr>
<tr>
<td>12</td>
<td>8.7</td>
<td>11.77</td>
<td>0.01903</td>
</tr>
</tbody>
</table>

**TABLE 2 - Phase I Test Results 8Å - 0.92°**
as the source output varies, but their ratio will not vary (neglecting counting statistics). Thus, the shape of the curves is not influenced by fluctuations in the source intensity, but the absolute reflecting efficiency is not measured. The efficiency factor used in this report is defined as the ratio of secondary detector intensity to that recorded by the primary detector at the peak of the reflected intensity curve. This number can be used, however, to give some insight into the location of critical angles in reflecting efficiency.

Compilations of scattering widths, percent scattered, and the efficiency factor are given in Table 2.

STATISTICAL CONSIDERATIONS

There was no statistical requirement with respect to intensity in the case of the 61 point scattering curves. The time interval was fixed at 100 seconds, and the number of counts collected in this period was used.

The nine points data were taken so that the number of counts at the peak was greater than 10,000 for the one angle of incidence. These times ranged from 100 seconds to 500 seconds.

Twelve government furnished samples were tested in this phase of the program. These were numbered as follows:

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Material</th>
<th>Polishing Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>CER-VIT</td>
<td>Bowl Feed</td>
</tr>
<tr>
<td>G2</td>
<td>CER-VIT</td>
<td>Fresh Feed</td>
</tr>
<tr>
<td>Sample No.</td>
<td>Material</td>
<td>Polishing Process</td>
</tr>
<tr>
<td>-----------</td>
<td>---------------------------</td>
<td>------------------------------------</td>
</tr>
<tr>
<td>G3</td>
<td>CER-VIT</td>
<td>Ionic Bombardment</td>
</tr>
<tr>
<td>G4</td>
<td>CER-VIT</td>
<td>Ionic Bombardment</td>
</tr>
<tr>
<td>G5</td>
<td>ULE</td>
<td>Fresh Feed</td>
</tr>
<tr>
<td>G6</td>
<td>ULE</td>
<td>Fire Polish</td>
</tr>
<tr>
<td>G7</td>
<td>ULE</td>
<td>Ionic Bombardment</td>
</tr>
<tr>
<td>G8</td>
<td>CER-VIT with Vacuum</td>
<td>Bowl Feed</td>
</tr>
<tr>
<td></td>
<td>Deposited Gold Film</td>
<td></td>
</tr>
<tr>
<td>G9</td>
<td>CER-VIT with Vacuum</td>
<td>Bowl Feed</td>
</tr>
<tr>
<td></td>
<td>Deposited Aluminum Film</td>
<td></td>
</tr>
<tr>
<td>G10</td>
<td>Aluminum with Polished</td>
<td>Fresh Feed</td>
</tr>
<tr>
<td></td>
<td>Electroless Nickel Coating</td>
<td></td>
</tr>
<tr>
<td>G11</td>
<td>Optical Grade Quartz</td>
<td>Bowl Feed</td>
</tr>
<tr>
<td>G12</td>
<td>Optical Grade Quartz</td>
<td>Fire Polish</td>
</tr>
</tbody>
</table>

The only irregularity observed on almost all of the samples was a thin ring left by the teflon spacers between the samples. All samples appeared to have a good polished surface.

Throughout this phase of the program, all samples were placed in the sample holder at a definite orientation. This orientation was defined as 0° (angle with respect to X-ray beam) and determined for the sample by an arrow on the side of the sample.

CONCLUSIONS

The samples showed a low percent scatter for 8° and for 0.92° angle of incidence. There were two exceptions, samples No. G6 and No. G12, both of which show irregularities in the scattering
curve and a high percent scatter. Both of these samples were fire polished. The remaining ten samples show comparable results.

REFERENCES


