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Micrometry combined with profile mapping for the absolute measurement of Integrated Column Density (ICD) and for accurate X-ray mass attenuation coefficients using XERT

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ABSTRACT

Absolute values of the column densities $[\rho t]_c$ of four gold foils were measured using micrometry combined with the 2D X-ray attenuation profile. The absolute calibration of $[\rho t]_c$ was made with a reference foil and the $[\rho t]_c$ of other foils were determined following the thickness transfer method. By this method, we obtain absolute calibration to 0.1% or better which was not possible using only the X-ray map of a single foil over its central region.

1. Introduction

Previous measurements of X-ray mass attenuation coefficients [1–3] are usually performed using only a single foil. The X-ray Extended Range Technique (XERT) [4,5] uses multiple foils and measures their integrated column densities for the absolute measurements of X-ray mass attenuation coefficients. The use of multiple foils in absorption and attenuation measurements has been found to be important for measurement of mass attenuation coefficients [6,7]. Accurate measurements of X-ray mass attenuation coefficients depend on the determined integrated column density of the absorbing sample [8,9].

To determine the integrated column density, the foil is mapped with the X-ray beam over the surface. The average integrated column density $[\rho t]_{ave}$ of the foil is then determined from attenuation measurements made at each point across the foil surface. Relative measurements at different energies are then used to determine the X-ray mass attenuation coefficients on an absolute scale. This technique is referred to as the full-foil mapping technique [8].

In this analysis, we introduce a new technique which combines the attenuation measurements mapped across the central $8\times 8\,\mathrm{mm}^2$ region of the foil and thickness measurements across the entire foil using a micrometer. To obtain absolute measurements of $[\rho t]_{ff}$, we introduce a scaling factor by scaling the average thickness t_{ff} of the micrometer measurements to the average integrated column density $[\rho t]$ of the central region scanned by an X-ray beam. Both methods determine the absolute value of the integrated column density $[\rho t]_{ff}$ of the reference foil. The integrated column density $[\rho t]_{ff}$ determined by this technique makes it possible to achieve high accuracy in measured X-ray mass attenuation coefficients without applying the full foil mapping to the entire foil area. This enables us to reduce the X-ray beam time needed. This method is a useful development to the XERT.

The measurements were made at the Advanced Photon Source (APS) using the XERT. Four gold foils, supplied by Goodfellow, with nominal thicknesses varying from 9.3 to 275 μm, were used for the measurements (Table 1). The purity of all the foils was 99.99%. An X-ray beam scan of the thickest foil was made over the central $8\times 8\,\mathrm{mm}^2$ region of the foil. This was used to calibrate micrometer measurements made over the full foil area.

2. Attenuation measurements

To determine the attenuation $[\mu t]$ at each point of the foil, an X-ray raster scan of the foil was performed at 50 and 42 keV. The attenuations of the reference foil at 42 and 50 keV are shown in Fig. 1(a) and (b) respectively. The attenuation $[\mu t]$ at each point of the raster scan was obtained following the subtraction of dark current and using

$$[\mu t] = -\ln\left[\frac{(I/I_0)_s}{(I/I_0)_b}\right] \quad (1)$$

where the subscript s refers to measurements made with foil in the path of the beam and the subscript b refers to the measurements made with the foil removed. I and I_0 are the attenuated and unattenuated intensities respectively. The uncertainty of the measurements was determined from the uncertainties of the intensity measurements with and without the sample in the path of the beam.

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3. Determination of $[\rho t]_{ff}$ using of the micrometer measurements and X-ray map

Micrometer measurements were used to measure the average thickness profile of the foil, with 0.5 μm precision. There were 25 micrometer measurements performed over the full $25\times 25\,\text{mm}^2$ area of the foil. The uncertainty in $[\rho t]$ is weighted by the area of the corresponding micrometer measurements contributing to the X-ray profilometry as shown by the squares in Fig. 2. The weight of the central micrometer measurement is one and the area A_i of the other measurements are taken as the intersection of the micrometer footprint area with the region of the X-ray map.

The average thickness of the region mapped by the X-ray beam t_m is

$$t_m=\frac{\sum_i A_i t_i}{\sum_i A_i} \tag{2}$$

where A_i is the intersectional area of the ith micrometer measurements. The corresponding uncertainty was determined by

$$\sigma_{t_m}=\sqrt{\sum\left(\frac{A_i}{\sum A_i}\right)^2\sigma_{t_i}^2}. \tag{3}$$

The average of the n measurements was found to be $t_{ff}=269.68\pm 0.1\,\mu\text{m}$ (0.037%), while the corresponding regional average t_m was found to be $269.25\pm 0.19\,\mu\text{m}\,(\pm 0.074\%)$. Making use of the scaling factor $t_{ff}/t_m=1.0016(5)$, the attenuation over the full foil $[\mu t]_{ff}$ is

$$[\mu t]_{ff}=\frac{t_{ff}}{t_m}\times[\mu t]_m \tag{4}$$

where $[\mu t]_{ff}$ is the average attenuation over the full-foil and $[\mu t]_m$ the average attenuation of the micrometer intersection X-ray map. The corresponding uncertainty was then

$$\sigma_{[\mu t]_{ff}}=\sqrt{\left(\frac{\sigma_{t_{ff}}}{t_{ff}}\right)^2+\left(\frac{\sigma_{t_m}}{t_m}\right)^2+\left(\frac{\sigma_{[\mu t]_m}}{[\mu t]_m}\right)^2}. \tag{5}$$

Making use of Eqs. (4) and (5), $[\mu t]_{ff}$ was found to be $5.7866\pm 0.10\%$ at 42 keV and $3.6683\pm 0.11\%$ at 50 keV. The attenuation at the central point $[\mu t]_c$ of the reference foil and the average attenuation $[\mu t]_m$ were 5.7868(7) (i.e. $\pm 0.01\%$) and 5.7754(37) ($\pm 0.06\%$) at 42 keV and 3.6575(8) ($\pm 0.02\%$) and 3.6605(7) ($\pm 0.07\%$) at 50 keV.

Table 1
The determined integrated column densities of the four gold foils.

t_{nom} (μm)	$[\rho t]_{ff}$ (g/cm^2) (%)
275.0	0.5115 ± 0.10
116.5	0.2240 ± 0.11
100.6	0.1972 ± 0.12
9.3	0.0177 ± 0.17

The weighted mean value of the column density of the reference foil determined at the two energies was $0.5115\,\text{g/cm}^2\pm 0.1\%$. This value was used for determining the integrated column densities of the other foils by a measurement relative to the reference foil at 42 keV.

4. Conclusion

This method makes it possible to determine the $[\rho t]_{ff}$ accurately by scanning the central region of the foil. This technique enables us to save experimental time spent scanning

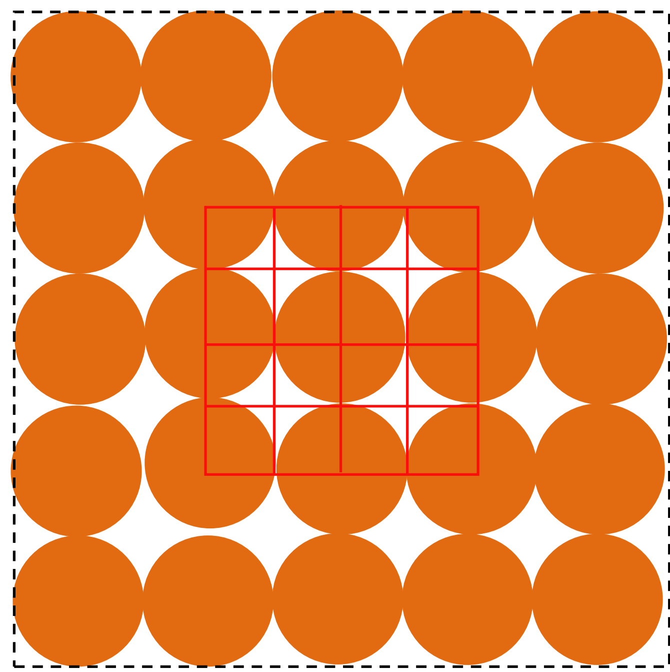

Fig. 2. Micrometer measurements at 25 points across the foil are represented by the circles. The central area of the foil ($8\times 8\,\text{mm}^2$) was scanned by an X-ray beam. These measurements are represented by the squares. The central region intersects nine of the micrometer measurements.

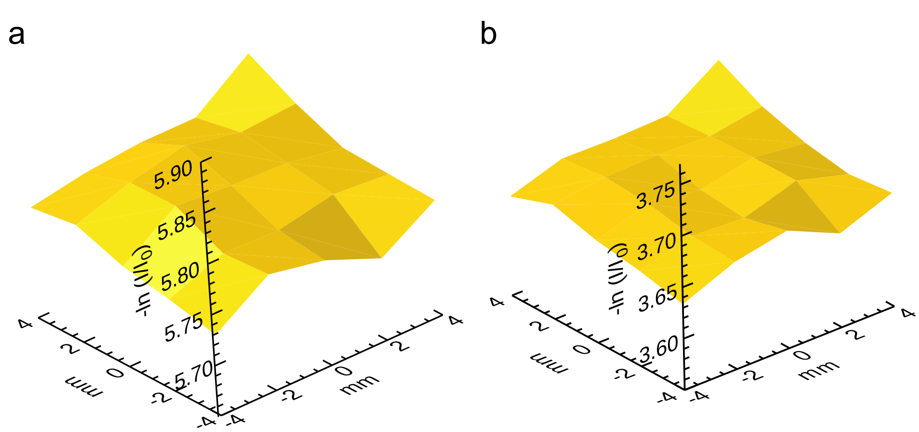

Fig. 1. The two-dimensional X-ray maps of the attenuation $[\mu t]$ of the reference foil obtained from raster scans at 42 keV (a) and 50 keV (b). The variations of foil attenuation on the maps were found to be within the range of $-0.64\%\rightarrow+0.74\%$ at 42 keV, and $-0.72\%\rightarrow+1.02\%$ at 50 keV respectively. For most prior determination of absorption coefficients, this variation of 1–2% yields a systematic error of the same magnitudes. In the current work, residual uncertainties lie at the 0.1% level.

the full foil. Instead of a variance or systematic uncertainty corresponding to the variation of thickness of perhaps 2% or more for the measured point compared to the average thickness, we obtain an absolute calibration to 0.1% or better. This method thus provides an important method for identifying and correcting uncertainty contributions from the foil metrology to achieve highly accurate results.

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