A Novel Technique for the Simultaneous Collection of Reflection and Transmission Data from Thin Films in the Extreme Ultraviolet

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Abstract
Studies of thin films in the Extreme Ultraviolet (EUV) are difficult given that most materials readily absorb photons of these energies. By depositing a thin film of the material of interest on a silicon photodiode, transmission measurements can be made throughout the EUV. If the measurements are made in a range of low absorption, the extinction coefficient, k, can be found with relative ease. However, if the material’s absorption is considerable, reflection measurements are needed to supplement the transmission data in order to find the optical constants n and k. The technique developed allows for reflection and transmission measurements to be taken simultaneously, which combined, account for all of the measurable photons from the original beam: (those which cannot be counted are photons absorbed into the thin film material). Also, the technique presented allows for data to be collected from practically all angles of incidence. This technique has been applied to a thin film of scandium oxide (d=65 nm), with measurements taken over wavelengths from 2.5-25 nm, and at angles of incidence 12 degrees from grazing to normal.

Introduction
Since most materials have yet to be definitively studied in the Extreme Ultraviolet (EUV), experiments which minimize the number of unknowns present in the model describing the data are preferred. For studying thin films in the EUV, transmission measurements are an excellent first step in developing an optical understanding of a material. Subsequent experiments build upon the information found from the transmission experiments.

Thin film samples are prepared by depositing thin films directly on the face of photodiodes. This process greatly facilitates the collection of transmission data, as all photons transmitted through the film are guaranteed to be incident upon the detector face. The transmission data can then be used to determine the extinction coefficient k (which is the imaginary portion of the complex index of refraction, \( N = n + ik \)) of the deposited film. Successive reflection measurements of the same sample, can be used to calculate the refractive index using the newly determined extinction coefficient. A schematic illustrating the use of a photodiode with a thin film on its detection surface is offered in Figure 1.

The optical properties of thin films in the EUV are sensitive to a variety of parameters. Among these are: film thickness, presence of impurities, interface quality, film density, and grain size and orientation. Each of the listed items are nanoscopic in scale, such that even subtle variations in of one of the variables will effect the optical performance of the sample. Also, since it is difficult to ensure the uniformity of a thin film with regard to any of these physical parameters, the correlation between the reflection and transmission data may not be optimal if the
The detection face of a photodiode is coated with a thin film to be studied. In doing so, the signal reported by the photodiode corresponds to photons that have traveled through the thin film. With the use of a second, auxiliary detector, reflection measurements of the thin film can be made. At the bottom of the figure, distribution of the photons is illustrated—the energy from the incident will be present in the reflected and transmitted beams, or lost as photons are absorbed by the thin film (suggested by the dashed line representing the transmission beam).
measurements are taken on different regions of the thin film. Thus, ensuring that a single description of the physical qualities of a thin film is appropriate for use in a model of both reflection and transmission data is not a trivial matter, since the beam probing the sample is likely to have encountered the film at different positions across the sample surface.

**Design**
A technique was developed to make possible the *simultaneous* collection of transmission and reflection data. This was achieved by designing and machining a new sample stage, which allows both the photons reflected and transmitted by a thin film sample to be recorded—each of which are originated from the same incident radiation *and* correspond to the exactly the same region of the film. Also, data can be collected over a wide range of angles, each of which has a different “effective path length” over which the EUV photons interact with the material of interest (illustrated in Figure 3, and described in Table 1). This enables a single sample to be provide data that corresponds to several film thicknesses, while only requiring the physical characterization of one film.

**Figure 2**
Openings in the stage face have a lip which contacts the diode support perimeter only. Detectors are held in place from behind by a securing plate. The channel in the stage face ensures that the beam of incident light (shown in red), as well as reflected light, encounter no obstruction. The curved arrows indicate the freedom available for changing the angle with which the beam is incident upon the sample.
Figure 3
This cross sectional view of the photodiode and the stage reveals the limitation to the angle nearest grazing which can be measured is due to the diode support frame. The red vectors show beam paths at various angles of incidence.

<table>
<thead>
<tr>
<th>Effective Film Thickness</th>
<th>Angle of Incidence (from grazing)</th>
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<tbody>
<tr>
<td>1d</td>
<td>90°</td>
</tr>
<tr>
<td>2d</td>
<td>30°</td>
</tr>
<tr>
<td>3d</td>
<td>19.3°</td>
</tr>
<tr>
<td>4d</td>
<td>14.4°</td>
</tr>
<tr>
<td>5d</td>
<td>11.5°</td>
</tr>
</tbody>
</table>

Table 1
Experiment

A thin film of reactively sputtered scandium oxide was deposited on a silicon photodiode (manufactured by International Radiation Detectors, model XUV 100), as well as a fused quartz slide and a quarter of a 4-inch silicon wafer. All three substrates were coated during the same deposition, such that each of the three samples are as identical as possible. Ellipsometry on the coated silicon and through the coated quartz reported the film thickness to be 65 nm, which matches the thickness found through X-ray diffraction on the coated silicon. The coated photodiode was measured at the Advanced Light Source, Beamline 6.3.2, at the Lawrence Berkeley National Laboratory. Measurements were taken over photon wavelengths of 2-200 nm, among which select wavelengths were used for theta-2theta reflection measurements. With each of these, transmission and reflection data were taken simultaneously. Unfortunately, the model for analyzing the data has only been recently been completed—a majority of the data collected has not been analyzed at the time of this writing.

Despite analysis of the theta-2theta data being incomplete, it is worth noting that this study has provided new insight into the understanding of scandium thin films. Scandium, being a transition metal, readily oxidizes upon exposure to atmosphere. The presence of an oxide would alter the performance of a scandium thin film, perhaps enough that further study of the oxide would be warranted. Indeed, the measurements made of the scandium oxide thin film show that the L_{23} edge appears in a position different than that which standard calculations would predict (see Figure 4). The predicted position of the edge is 3.10 nm (400 eV), while the measured thin film of scandium oxide shows the peak at 3.05 nm (406.7 eV). There is no published work concerning the measured optical properties of scandium oxide thin films, as most researchers studying scandium thin films rely on generating constants for the oxide that is a weighted combination of the optical constants of scandium and oxygen. However, the 6 eV difference shown with these measurements warrants separate study of scandium oxide, in order to properly describe the performance of scandium thin films.
Figure 4
The measured data displayed is raw, in that some of the features in the measured line can be attributed to either the response of the detector, or the presence of the filters in the ALS beam. Despite not having reduced the data to show only the properties of the scandium oxide thin film, the peak seen at wavelengths just larger than 3 nm is the $L_{23}$ peak, and it can be seen that it is at a noticeably different position that the corresponding peak in the line representing the calculated scandium oxide peak.
Conclusions

A new technique has been developed to allow for the simultaneous collection of transmission and reflection data from thin films. Analysis of data collected using the new technique should be greatly simplified by the strong correlation between the two sets of data. This technique has been used to study thin films of scandium oxide, for which information regarding its optical properties is inappropriate for study of films less than 100 nm thick, in the EUV.