Thorium dioxide thin films in the extreme ultraviolet

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ABSTRACT

We have measured the reflectance and transmittance of thorium dioxide thin films from 50-280 eV. We have developed several methods for fitting this data that gives the most reliable values for the complex index of refraction, $n = 1 - \delta + i\beta$. These fitting methods included fitting film thickness using interference fringes in highly transmissive areas of the spectrum and fitting reflectance and transmittance data simultaneously. These techniques give more consistent optical constants than solitary unconstrained fitting of reflectance as a function of angle. Using these techniques, we have found approximate optical constants for thorium dioxide in this energy range. We found that the absorption edges of thoria were shifted 4 eV and 2 eV to lower energies from those of thorium. We also found that the peak in δ was shifted by 3 eV to lower energy from that of thorium.

Keywords: Extreme ultraviolet, thorium dioxide, thin films, optical constants

1. INTRODUCTION

The extreme ultraviolet (EUV), 50−500 eV, has increased in the importance of its applications in a variety of areas in recent years. The most important commercial application of EUV light is in microelectronic photolithography. The microelectronics industry is rapidly approaching the diffraction limit of features that can be fabricated with the currently used ultraviolet wavelengths. Using EUV wavelengths of 11−13 nm would allow lithography of smaller, faster, and more efficient circuits that operate at lower temperatures. For biological and medical research, smaller images can be taken with EUV light than can be taken with visible optical wavelengths, due to the limits of optical diffraction. In addition, water is reasonably transparent in the range of 310-620 eV while carbon is opaque. Thus samples can be imaged without complex dehydration and staining procedures. EUV astronomy is also a blossoming field. Helium, the second most prevalent element in the universe, has both an atomic and an ionic emission line in the extreme ultraviolet and thus can be imaged in this wavelength region. For all of these developing applications, it is necessary not only to have well-designed and well-researched optics but highly reflective materials to make processes efficient. For this reason, the optical properties of new materials must be measured in this region for use in thin-film optics design. One of the main reasons why technology used for visible light optics has not become available in the extreme ultraviolet is the lack of reliable optical constants in this energy range.

For several years, Brigham Young University's Thin Films Research Group has been studying uranium as a possible candidate for high-reflectance in the EUV[1]. Theoretically, uranium's high density and large number of electrons would make it a good reflector in this region. Although uranium has been shown to be very reflective, its rapid oxidation in air limits its usefulness. When exposed to oxygen, the surface of pure uranium will oxidize in a matter of minutes to one of several compounds including UO_2, U_3O_8, UO_3, and often two or more of these coexist in the same sample. These different oxidation states have significantly different optical properties than elemental uranium. Because the exact state of the material in a uranium-based film is not known, it is nearly impossible to predict how it will reflect[2].

Thorium-based films provide a possible alternative to uranium films in that they also may be more reflective than commonly used EUV reflectors, but they avoid problems with multiple oxidation states because thorium only has one oxidation state, ThO_2 (thoria). Thorium and thoria should be good reflectors in the EUV because they also have large numbers of electrons. Although their respective densities (11.72 g/cm^3 for thorium and 9.86 g/cm^3 for thoria) are considerably less than that of pure uranium (19.3 g/cm^3), their densities are very similar to that of uranium dioxide

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(10.96 g/cm^3). The Center for X-ray Optics (CXRO) at Lawrence Berkeley National Lab[3] provides online a program where reflectance is calculated using tabulated optical constants obtained by interpolation between measured points and atomic scattering factor estimates. For compounds, CXRO weights elemental scattering factor estimates according to their relative densities in the compound. More about these methods is discussed in section 2. In Figure 1, CXRO reflectance data for thorium metal and thorium dioxide is plotted with data of three commonly used reflectance materials in this region: gold, nickel, and iridium. According to CXRO, thorium-based films may be more reflective than these materials for energies between 35 and 73 eV. In order to take advantage of its potentially high reflectance, it is necessary to accurately determine the optical constants for thorium-based films in the EUV. The purpose of this paper is to present measured optical constants for ThO_2 as determined by reflection and transmission measurements as a function of angle.

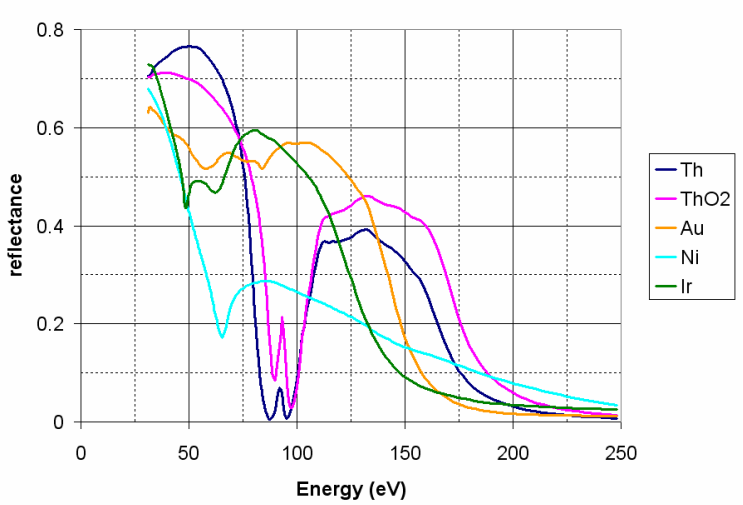


Figure 1: CXRO computed reflectance as a function of wavelength at 15 degrees from grazing. Although gold, nickel, and iridium are commonly used reflector materials in this region, thorium-based mirrors may be more reflective at energies between 35 and 73 eV.

In previous work[4 5] we attempted to find the optical constants of several materials by fitting reflection as a function of angle to the Parratt recursion relation[6]. We found that this is an unreliable technique. If left unconstrained, there are too many degrees of freedom, especially when the thickness of the film is not well characterized, and we were often left with non-physical solutions. For this reason, we adopted new procedures for finding optical constants that included taking several kinds of measurements. Each measurement constrained those parameters to which it was most sensitive. Some of these procedures included obtaining reflection and transmission data from the same sample, and then fitting the two sets of data simultaneously. Also, we have used interference fringes in reflectance to pinpoint an accurate film thickness since the spacing of these fringes is very sensitive to this thickness. The results of these methods are discussed further in this paper, and also in work by Allred et. al.[7] We found that we were unable to obtain reliable reflectance data from samples deposited on polyimide membranes because of the unstable structure of the membranes. However, by fitting transmission data from these samples, we were able to obtain approximate values for β in the complex index of refraction, $n = 1 - \delta + i\beta$. Using these values while fitting reflection data from a sample deposited on silicon, we were able to obtain approximate values for δ. We also deposited samples on AXUV100 silicon photodiodes[8]. We found that we were able to obtain good reflection and transmission data from these samples, and by fitting the two sets of data simultaneously, we were able to confirm the approximate optical constants we had found.

2. BACKGROUND

Optical constants for many materials in the EUV are not well known. The most comprehensive database of these constants is used by CXRO in their online programs. CXRO programs calculate reflectance and transmission for model structures using standard approaches (Fresnel equations) and archived optical constants[9]. These optical constants come from atomic scattering factor tables where data was compiled from a variety of sources. Where measured optical constants are not available, the program uses interpolation between measured data points. For compounds, CXRO takes the standard approach, computing effective indices from the tabulated atomic scattering factors by adding up the

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contributions from each element according to their relative abundance in the compound. While this would be extremely inaccurate at energies less than 10 eV where the effects of chemical bonds and scattering of electrons in a lattice are important, it can be argued that at very high energies, these effects become negligible. In the EUV this approach is a good approximation, but is not necessarily correct. To find correct optical constants for a material (especially a compound), they must be experimentally determined.

For thorium, the archived optical constant data tabulated in reference 9 that is used in CXRO reflectance calculations was obtained by private communication with N. Kerr Del Grande from Lawrence Livermore National Laboratory. We were not able to obtain this original data or find out the conditions under which it was acquired. For this reason, all discrepancies between data presented in this paper and CXRO data may be due to some dissimilarity between ours and the original data taken by Del Grande.

Experimentally determining the optical constants of a material requires taking measurements of reflectance or transmittance of a film as a function of angle and working backwards with the Fresnel equations[10] to find δ and β. Reflectance and transmittance measurements are taken using a monochromator. In a monochromator, one wavelength of light is isolated using gratings, filters, and pinholes and is then either reflected off of or transmitted through a film. For reflectance, the film and the detector are rotated through a theta-two theta scan, giving a data set of reflected intensity as a function of sample angle. For transmittance, the detector is held fixed while the film is rotated around 90 degrees. All angles are measured relative to grazing.

Once reflection measurements are taken, data is fit to the Parratt recursion relation[11 12]. For transmittance, we use a matrix formulation to calculate transmission at each interface[13]. Our fitting program uses a least squares fitting technique to find the set of parameters that were most likely to have created the data set. The fitting library we used to analyze our data, MINUIT[14], was developed at CERN in Switzerland. Our interface, a program called matfit, defines the function and supplies the data to this library.

The material we chose as discussed earlier was thorium because of its theoretically high reflectance in the EUV[15]. We originally tried to find the constants of thorium metal, but had problems fitting data when we found that the natural oxidation layer on the surface of the film created a gradual interface[16] with the thorium metal. This oxidation layer also may have been changing with time. For this reason, we moved to reactively-sputtered thorium dioxide. After obtaining reliable constants for this compound, it may be easier to find the constants for thorium metal.

Reflectance and transmittance measurements were taken at the Advanced Light Source at Lawrence Berkeley National Lab[17], beamline 6.3.2. Beamline 6.3.2 consists of a bend magnet source and an energy range of 50-1300 eV (0.95 − 25 nm).

3. METHOD AND ANALYSIS

In order to decrease degrees of freedom in our fit and obtain accurate optical constants for ThO_2, we simultaneously took both reflectance and transmittance data from thorium dioxide deposited by reactive RF sputtering on a thin membrane of polyimide provided by Moxtek[18] (sample ThO2-072604). Our intentions were to fit these two sets of data simultaneously in order to obtain accurate values for the optical constants of thoria. At the same time that we coated our sample on polyimide, we coated a witness sample of ThO_2 on a silicon substrate. X-ray Photoelectron Spectroscopy (XPS) data showed the witness sample to be thorium dioxide and uniform through the sample. Atomic Force Microscopy data showed that the witness sample was not significantly rough, with an RMS roughness of 1.47 nm on a 500x500 nm length scale[19].

Figures 2, 3, and 4 show plots of reflectance as a function of wavelength and transmittance as a function of wavelength for the sample deposited on polyimide and reflectance as a function of wavelength for the witness sample deposited on silicon respectively.

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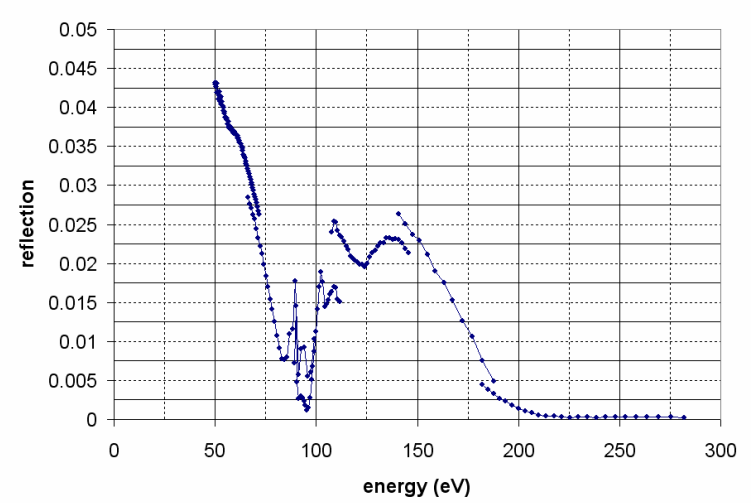


Figure 2: Measured reflection of ThO_2 on polyimide membrane at an angle of 14 degrees from grazing.

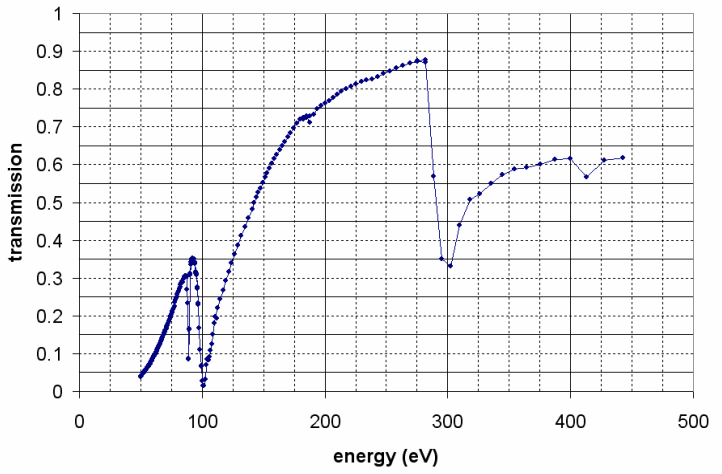


Figure 3: Measured transmission of ThO_2 on polyimide membrane at an angle of 93 degrees from grazing.

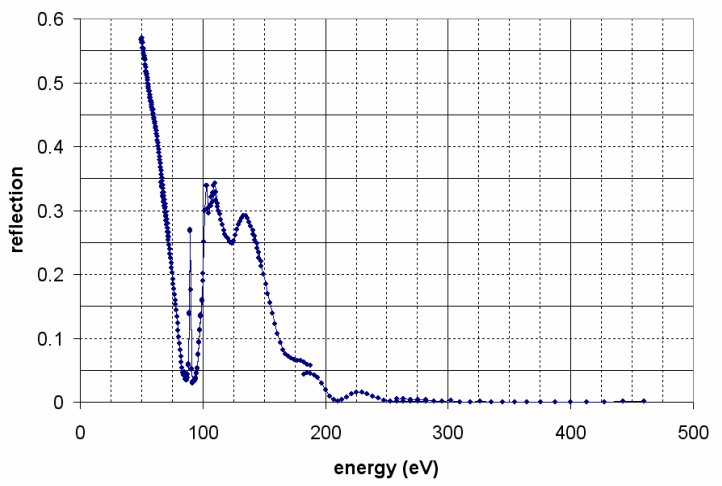


Figure 4: Measured reflection of ThO_2 on silicon at an angle of 14 degrees from grazing.

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One problem we had with our polyimide membrane samples was that the membranes were so thin that the polyimide buckled and warped after deposition. There were no places where the transmission looked uniform while doing a scan of the beam across the sample (see Figure 7). This non-uniformity was probably due to variations in the thickness of the polyimide membrane as well as variations in the thickness of the thoria film. Averaging over the film area, these variations gave us an uncertainty in absolute transmission of about 19%. Though the absolute transmission was uncertain at this level, the energy of the incident light was known to 0.012%[17]. Thus with this data we were able to determine edges in the optical constants of thoria even if the constants themselves are not accurate. The uncertainty in absolute reflection measurements was even more severe. The wavy nature of the film misdirected the reflected beam and small displacements of the beam on the sample created large discrepancies in reflected signal. We became aware of this problem when we found that the reflectance scans we took did not match up when we changed order sorter and filter sets (see Figure 8). These order sorter and filter sets displaced the beam by small amounts on the sample and affected reflectance measurements. We had never had this problem before on this scale. At some wavelengths, measurements disagreed by as much as 32% of total reflectance. Because of this significant mismatch, we did not know what measurements to believe. Our small signals in reflection measurements were also affected significantly by the dark current, which was on the order of 5% of our total signal. These large uncertainties and conflicting sets of data required us to discount the reflectance measurements of thoria deposited on a polyimide membrane.

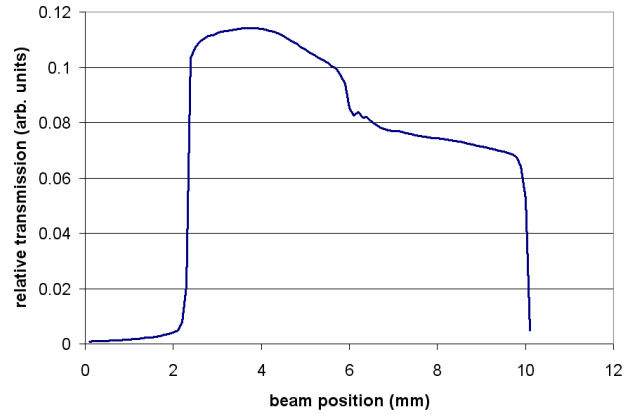


Figure 5: Transmission through our sample as a function of beam position. The high transmission on the left side of the plot is through the uncoated polyimide window. The lower transmission on the right side of the plot is through the polyimide membrane coated with thorium dioxide. There are no places on the plot where transmission is uniform as the beam position is varied.

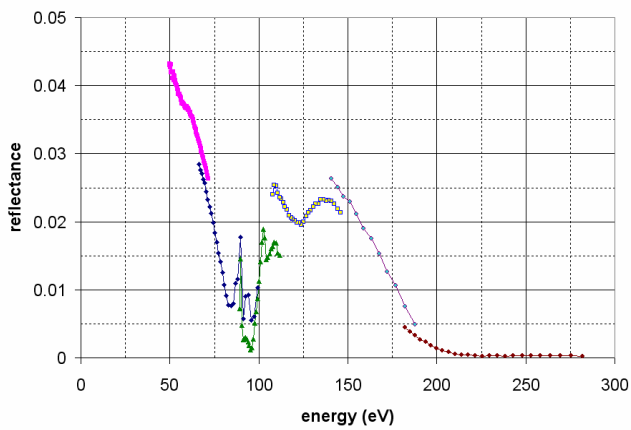


Figure 6: Reflection of ThO2 on polyimide at 20 degrees as a function of wavelength. Each data set was taken with a different set of filters and order sorters in order to get the desired wavelengths. In this plot, reflectance does not match up as the filter set was changed.

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Because of this problem with our sample deposited on polyimide, we were not able to fit reflection and transmission data simultaneously. By fitting transmission data from our polyimide sample and reflection data from our witness sample deposited on silicon, we obtained an approximate set of constants shown in Figure 7. β from this plot was obtained using transmission data from our sample deposited on a polyimide membrane. δ from this plot was obtained using β from transmission measurements and fitting reflection data from our witness sample deposited on silicon. The film thickness was chosen to minimize X^2 for all wavelengths.

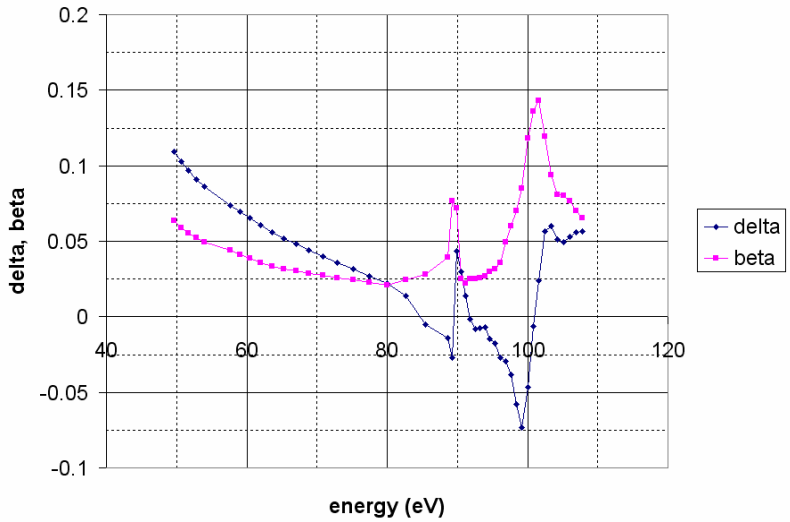


Figure 7: Preliminary optical constants of ThO_2. δ was obtained by fitting reflectance of a ThO_2-coated silicon wafer. β was obtained by fitting transmittance through a ThO_2-coated polyimide window.

Although depositing on polyimide membranes was not a viable option in order to concurrently measure reflectance and transmittance, we still believed fitting reflectance and transmittance simultaneously to be a good idea for obtaining accurate optical constants. Accordingly, we considered several alternative options to polyimide membranes in order to obtain these measurements. We decided to deposit our sample directly onto an AXUV100 silicon photodiode[8]. Measurements such as these were done by Uspenskii[20] et. al. in reference 20. We expanded the measurements made in this reference to include variable angle data and fits of reflectance and transmittance simultaneously. Transmittance measurements were made on the bare diode to determine the thickness of naturally oxidized SiO_2 on its surface. Because we could not obtain an incident intensity measurement for these fits, the incident intensity was made a parameter of the fit. Thus the data was fit by shape and not by amplitude. The fitted thicknesses were averaged, weighted by error. This average was determined to be 7.60nm +/- 0.06 nm. A representative fit is shown in Figure 8. This thickness for the SiO_2 layer was held fixed for the remainder of the fits.

Because of lack of knowledge of reactive sputtering rates, the thickness of our sample was only known to be 50 nm+/- 10 nm. For this reason, we chose three energies at which ThO_2 is very transmissive, 91.2 eV (13.6 nm), 87.3 eV (14.2 nm), and 82.7 eV (15 nm) and found that the three sets of reflectance data showed pronounced oscillations, presumably due to interference between the front and back surfaces of the ThO_2 film. First we performed a simple calculation using the interference fringes, $d = \lambda/(2 \sin(\theta_1-\theta_2))$, to find an estimate for the thickness. Using this calculation we found the thickness to be 53.5 nm +/- 3 nm. We used this as a preliminary guess when fitting these three data sets. Figures 11, 12, and 13 show the data and fit for each of these three energies. The interference fringes were fit very well, especially considering the low signal. The thicknesses given by the three fits and their uncertainties are given in Table 1.

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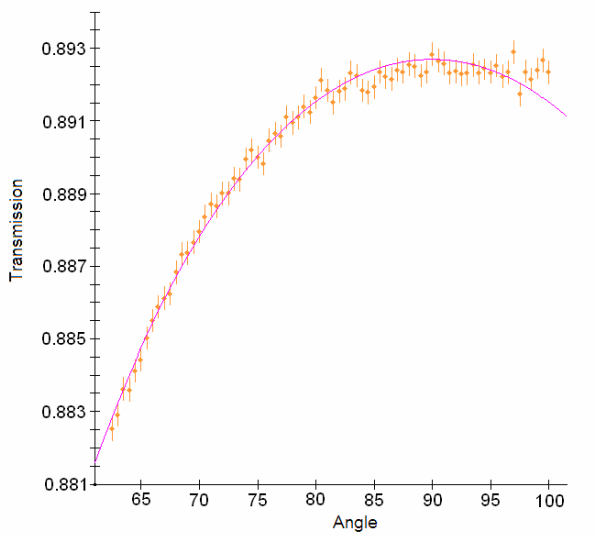


Figure 8: Transmission of a bare AXUV-100 diode at 91.9 eV (13.5 nm) as a function of angle fit to obtain a thickness of the SiO_2 layer.

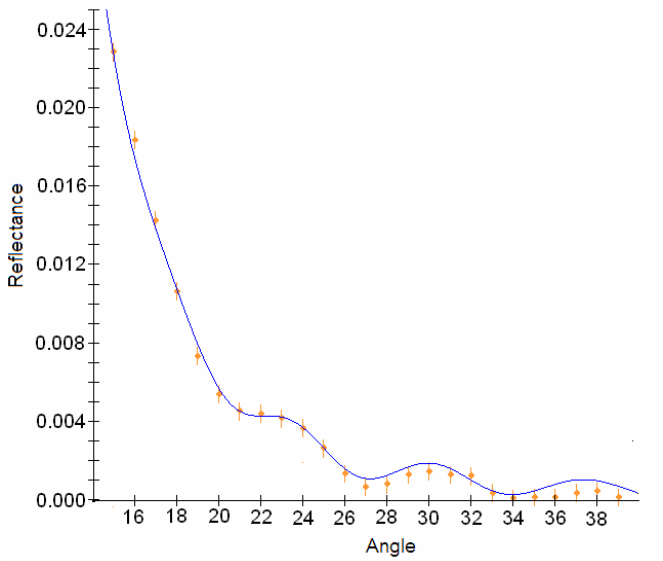


Figure 9: Reflectance of a ThO_2-coated diode at 91.2 eV (13.6 nm) fit to the interference fringes to obtain a thickness for the ThO_2 layer.

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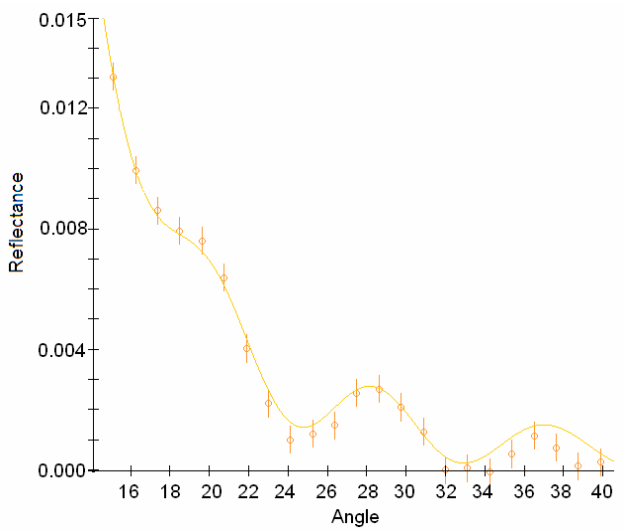


Figure 10: Reflectance of a ThO_2-coated diode at 87.3 eV (14.2 nm) fit to the interference fringes to obtain a thickness for the ThO_2 layer.

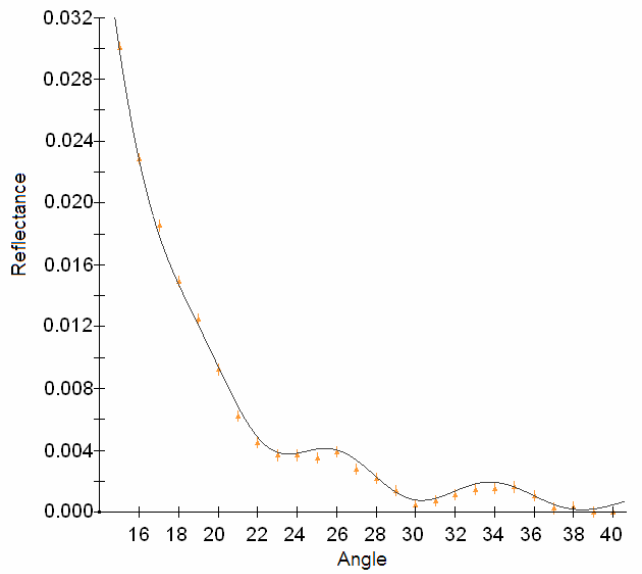


Figure 11: Reflectance of a ThO_2-coated diode at 82.7 eV (15.0 nm) fit to the interference fringes to obtain a thickness for the ThO_2 layer.

Table 1: Film thicknesses given by fitting interference fringes.

Energy (eV)	Thickness given by fit (nm)	Uncertainty in fit (nm)
91.2	56.53	1.08
87.3	54.58	0.78
82.7	55.34	0.48

A weighted average of these three thicknesses gives a thickness of 55.37 nm +/- 0.47 nm. After obtaining this value for the thickness of our film, we kept this value fixed for the remainder of our fits.

With a good thickness, we began fitting reflectance and transmittance simultaneously to determine optical constants. The majority of our fits looked very good. A representative fit is shown in Figure 12.

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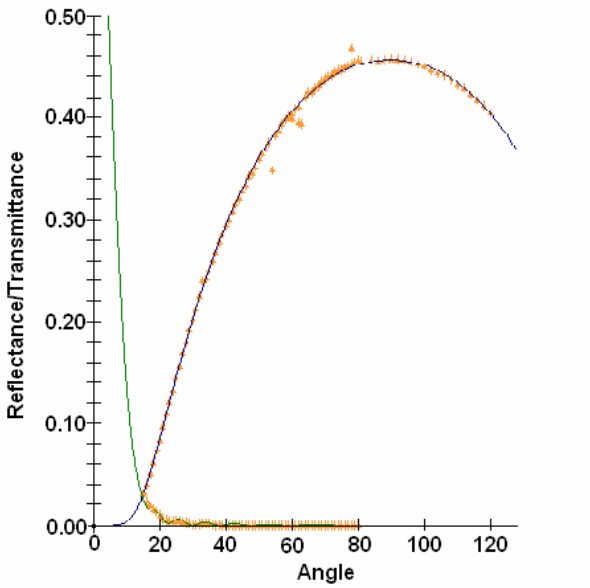


Figure 12: Reflectance and transmittance of a ThO_2-coated diode at 82.7 eV (15 nm) fitted simultaneously to obtain optical constants.

The constants we found with these fits are plotted in Figures 13 and 14 along with the preliminary constants we obtained using data from the ThO_2-coated polyimide window and witness sample. The calculated constants from the CXRO website are also plotted here.

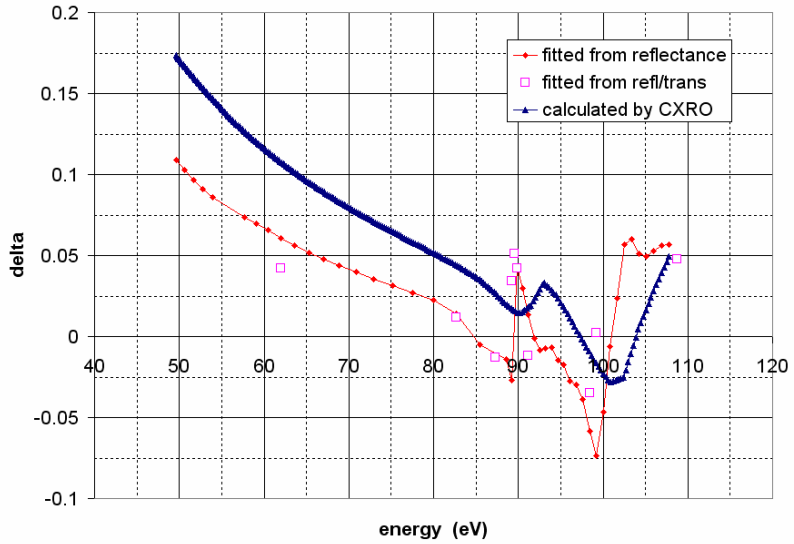


Figure 13: δ for ThO_2 obtained by three different methods: fitted from reflectance of a ThO_2-coated silicon wafer, fitted from reflectance and transmittance simultaneously from an AXUV-100 photodiode, and calculated by CXRO.

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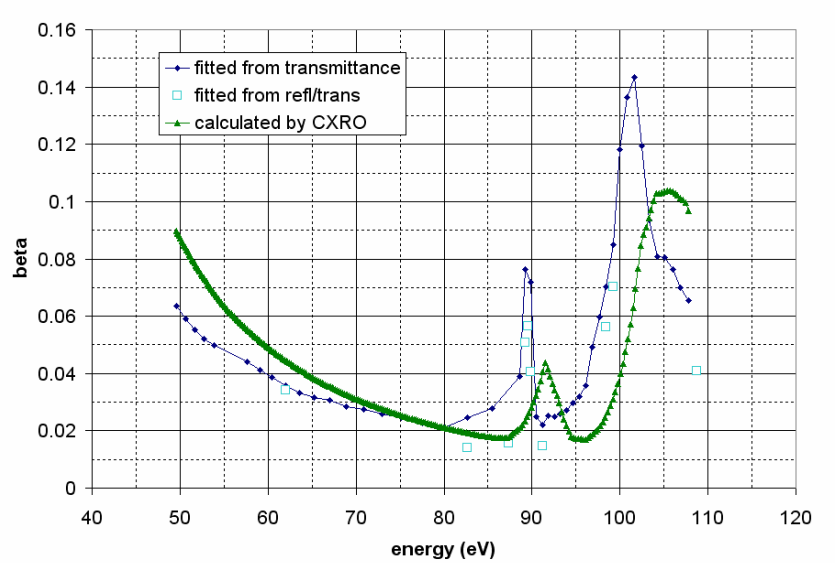


Figure 14: β for ThO_2 obtained by three different methods: fitted from transmittance through a ThO_2-coated polyimide window, fitted from reflectance and transmittance simultaneously from an AXUV-100 photodiode, and calculated by CXRO.

Table 2: Tabulated optical constants with δ fit from reflectance data and β fit from transmittance data.

Energy (eV)	δ	β
49.60	0.1091	0.0634
50.61	0.1031	0.0592
51.67	0.0966	0.0553
52.77	0.0912	0.0521
53.91	0.0859	0.0497
57.67	0.0737	0.0442
59.05	0.0698	0.0412
60.49	0.0655	0.0386
62.00	0.0608	0.0358
63.59	0.0562	0.0333
65.26	0.0518	0.0316
67.03	0.0480	0.0306
68.89	0.0438	0.0285
70.86	0.0401	0.0274
72.94	0.0356	0.0260
75.15	0.0316	0.0246
77.50	0.0271	0.0228
80.00	0.0223	0.0211
82.67	0.0140	0.0247
85.52	-0.0049	0.0279
88.57	-0.0140	0.0390
89.21	-0.0268	0.0764
89.86	0.0435	0.0719

Energy (eV)	δ	β
90.51	0.0297	0.0248
91.18	0.0137	0.0219
91.85	-0.0013	0.0252
92.54	-0.0082	0.0248
93.23	-0.0074	0.0257
93.94	-0.0070	0.0271
94.66	-0.0143	0.0297
95.38	-0.0173	0.0318
96.12	-0.0274	0.0357
96.88	-0.0296	0.0492
97.64	-0.0386	0.0599
98.41	-0.0582	0.0704
99.20	-0.0734	0.0849
100.00	-0.0468	0.1181
100.81	-0.0061	0.1362
101.64	0.0238	0.1433
102.48	0.0566	0.1193
103.33	0.0599	0.0936
104.20	0.0511	0.0808
105.08	0.0493	0.0805
105.98	0.0530	0.0765
106.90	0.0559	0.0701
107.83	0.0567	0.0653

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Table 3: Tabulated optical constants fit from reflectance and transmittance data simultaneously.

Energy (eV)	δ	β
62.00	0.0420	0.0343
82.67	0.0120	0.0141
87.32	-0.0129	0.0155
89.21	0.0340	0.0507
89.53	0.0510	0.0565
89.86	0.0420	0.0407
91.18	-0.0120	0.0146
98.41	-0.0350	0.0563
99.20	0.0020	0.0702
108.77	0.0480	0.0410

There are some significant differences between constants fit using data from the coated polyimide window and reference wafer and data from the coated diodes (See Tables 2 and 3). δ is different by as much as 0.0754 and β is different by as much as 0.018. However, the qualitative shape of the two sets of data is the same. On the other hand, it is clearly shown that the data calculated by the program on the CXRO website does not qualitatively match the data that we fit. The absorption edges for thorium dioxide are shifted to lower energies from those of thorium by 4 eV from 105.6 eV and 2 eV from 91.5 eV. The peak in δ is shifted to lower energies by 3 eV from 92.8 eV.

4. CONCLUSIONS

We have measured the reflectance and transmittance of thoria thin films from 50-270 eV. Using constraining techniques, including fitting film thickness using reflection interference fringes and simultaneously fitting reflectance and transmittance data, we have determined the index of refraction for thorium dioxide in this energy range. We have found that the absorption edges in thoria are shifted to lower energies from those of thorium by 4 eV from 105.6 eV and 2 eV from 91.5 eV. We have also found that peak in delta of thoria is shifted to lower energies from those of thorium by 3 eV from 92.8 eV. These shifts in edges from thorium to thoria may be due to compound effects such as bonding that are not accounted for in CXRO data calculations. These shifts may also be a result of inaccurate data of the optical constants of thorium metal used by CXRO in their calculations. A reliable measurement of the optical constants of thorium is needed to discount this possibility.

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