Measurement of Refractive Indices of Materials for Broadband mirrors-UVOIR Space-Based Observatories

Next-generation, space-based telescopes could profit from broadband & dual-band mirror coatings. The top reflective layer - necessarily aluminum- can give high IR-to-far-UV reflectance & the appropriate multilayer below it can provide relatively high VUV-EUV reflectance tailored to the application. There is a need for the optical constants of promising compounds to be measured (beamline 6.3.2) for the lower-energy portion (10-100 eV) of the EUV range. Such compounds include fluorides (e.g. AlF3, MgF2, LiF, HfF4 etc.) since the Al mirror may be protected by thin fluoride layers.

Experiment Leader / Person Completing Form

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Structural Biology  ☐ Yes  ☐ No

Beamline & Shifts Requested

<table>
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<tr>
<th>Beamline</th>
<th>Endstation</th>
<th>Rollup</th>
<th>Shifts (total program)</th>
<th>Shifts (first cycle)</th>
<th>2-Bunch</th>
<th>Wavelength/Energy range</th>
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<tr>
<td>6.3.2</td>
<td>6.3.2 (EUV/Soft X-Ray Reflectometer/Calibrations)</td>
<td>☐</td>
<td>16</td>
<td>4</td>
<td>☐</td>
<td>25-100 eV</td>
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Industrial Group  ☐ Yes  ☐ No  Industry Funded  ☐ Yes  ☐ No
Productivity of previous beamtime at the ALS for the Principal Investigators

Beamtime Allocated in the last 3 years

<table>
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<tr>
<th>Proposal Cycle</th>
<th>Beamline/Endstation</th>
<th>Shifts Allocated</th>
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<tr>
<td>2013-2 August-December</td>
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<td>2014-1 January-July</td>
<td>6.3.2</td>
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<tr>
<td>2015-2 Jul - Jan</td>
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Comments on Productivity at the ALS
We are in the process of preparing a paper on optical properties of yttrium oxide as prepared by a number of different methods. It should be submitted in the next six weeks.

Refereed journal articles resulting from previous beamtime published in the last 3 years
None
**Measurement of Refractive Indices of Materials Potentially Useful in Broadband mirrors for Next Generation, Large UV-Optical-IR (LUVOIR) Space-Based Observatories-Beam line 6.3.2 ALS.**

**ABS:** Soon NASA will hold decadal review panels, refocusing its scientific priorities for the 2020 decade and beyond. The measurements proposed here could provide vital information to planners. Next-generation, space-based telescopes could profit from extended broadband (IR- far UV) or dual-band mirror IR-UV + EUV) coatings. The top reflective layer - necessarily aluminum- can give high IR-to-far-UV reflectance and the appropriate single layer or multilayer below it could provide relatively high vacuum ultraviolet (VUV) to extreme UV (EUV) reflectance above aluminum’s plasma frequency at about 15 eV. (Above this energy aluminum becomes somewhat transparent so that the mirror’s EUV performance could be controlled by the layers underneath which could be tailored to match the desired application.) We are doing calculations using a genetic algorithm and require a complete set optical constants of various materials to see the impact of various layers over the whole potential range of interests (0.1 to 100 eV). Through the use of spectroscopic ellipsometry and our VUV monochromator we can measure the range from 0.4-12 eV well and can make measurements at a few selected lines over the range of 12- 40 eV. Beamline 6.3.2 will allow us to better understand our laboratory measurements from 25-40 eV and to make the measurements from 40-100 eV which cannot be obtained in any other way. We contemplate making measurements on 3 to 4 previously uncharacterized materials over the next two years. The first would be aluminum fluoride because of its high bandgap and its ability to block the oxidation of aluminum in conjunction with other fluoride films. At the presence its optical constants have only been measured below 21 eV. (Bridou, et al. 2010) Other materials will probably include additional fluorides used as barrier layers for aluminum (e.g., MgF2, LiF, will etc.) and some promising compounds that may have overlooked by past investigators for the 15 to 25 eV range, such as TaS2. (G. Vacher 1978) We have the facilities and experience to prepare and characterize these samples. Beamline 6.3.2 is the right system to characterize these films and we have experience using the beamline for such purposes.

**Scientific Merit**

**Background:** wherein we hope to persuade you as to the importance of what we propose.

Basically, our goal is to provide VUV and EUV options in the form of basic science knowledge to those who will be planning next decade’s space-based observatories that will use broadband mirrors. We think that a few carefully measurements to determine the optical constants of selected samples on beamline 6.3.2 could make a significant impact on the telescopes used to advance humanities knowledge about exoplanets and astrophysics in general.

In the next 2-3 years NASA will hold several decadal review panels, refocusing its scientific priorities for the 2020 decade and beyond. The current flagship observatories: James Webb and WFIRST are primarily IR observatories. Since the end of the Hubble mission there has been no visible-UV large US space telescope. There have been no broad-spectrum US observatories since the end of EUV Explorer and FUSE. NASA contemplates 4 kinds of missions for the next decade. Two of these missions- astrophysics & exoplanet characterization- would profit if their mirror and grating coatings could have high reflectance beyond the current limit of 10 to 11 eV provided by fluoride-coated aluminum mirrors. We believe that there are improvements that could approximately double the range of broadband mirrors. These improvements depend, however, on knowing the optical constants of some materials in the 1-100 eV range that are presently not well over this whole range.

Our aim is to contribute basic science mirror fabrication/design information to the engineers and astrophysicists who will be making decisions how much of the vacuum and extreme ultraviolet can be added broadband IR, optical, UV mission by making adjustments to the aluminum mirror coatings that are currently being contemplate for space-based observatories. We hope to make these mirrors multiwavelength. We imagine two scenarios: first, the “extended broadband” mirror. We have
computational evidence that putting layers of the appropriate materials and thicknesses under the aluminum mirrors could extend their range 5-10eV beyond where aluminum’s reflectance falls, albeit at reduced reflectance. The second scenario is a “dual-band, broadband” mirror concept. The aluminum layer needed for IR and visible reflectance would be the cap layer on more standard EUV mirrors-for example, Mo/Si for wavelengths below 30.4 nm. That is, the kind currently used for Helios-observing space-telescopes like the Solar Ultraviolet Imager. (Martínez-Galarce. 2013)

We are using a genetic algorithm for calculations and some unexpected combinations have turned up for realizing this “extended broad band” mirror concept. This is why we need the option to measure materials like TaS₂ at the ALS.

Reason indicates that the IR to EUV broadband mirror with highest reflectance would have no fluoride barrier layers on top. And while we would like engineers to consider how aluminum could be deposited at the point of use in space so that it would be bare, we recognize that at the present, evaporated aluminum, overcoated in situ by various fluoride salts, has been the only option for obtaining high far-UV reflectance, thus the community will need the EUV optical constants of those which are not characterized, such as aluminum fluoride. See below.

We are looking at how far high reflectance of broadband mirrors can be extended into the VUV, what obstacles must be dealt with, and the best solutions. Independent of this proposal, we are currently involved in several activities which show our commitment to these the materials we are discussing. These activities include depositing an aluminum mirror inside our vacuum monochromator and measuring in situ the changes in its VUV reflectance as a function of time. We have also determined that spectroscopic ellipsometry is a good method for measuring subnm changes, such as oxidation of MgF₂-coated Al mirrors with time. For example, we evaporated in rapid succession onto a Si wafer substrate coated with 700nm of silicon nitride, 33nm of Al then 7.7nm of MgF₂; which protects the aluminum from immediate oxidation. We recently took ellipsometric measurements first at 0.25hr postdeposition, then for successively longer time intervals. A transparent layer under the MgF₂ presumed to be alumina was observed to form & slowly thickened, from 0.09nm to 0.95nm at 234 hours.


What we propose (in brief):
Aluminum fluoride will be the first material we propose studying. It has recently shown considerable promise as a protective layer for aluminum mirrors. Particularly, in conjunction with more commonly known fluorides like magnesium fluoride and lithium fluoride. (Moore 2014) (Manuel A. Quijada, Enhanced Far-Ultraviolet Reflectance of MgF₂ and LiF Over-coated Al Mirrors 2014) (J. T. Cox 1968) (Aznarez, Larruquert and Mendez 1996) The optical constants have been measured in vacuum-ultraviolet wavelength greater than 60 nm (21 eV). (Bridou et al. 2010) We plan on measuring reflectance of aluminum fluoride thin films for wavelengths between 25 and greater than 100 eV at the ALS. We will confirm Bridou’s data with our VUV monochromators equipment at
BYU and extend the measurements above 21 eV. And use both beamline 6.3.2 and our own measurements to determine optical constants up to 100 eV.

We wish measure reflectance and transmission (R&T) of these materials deposited on International Radiation Devices (IRD) PIN diodes on beam line 6.3.2 at the ALS. We will focus mainly on near edge over the energy range of 25 to about 100 eV. The data obtained from this will be used to compute the real and imaginary compounds of these materials. Usually these are referred to as n and k respectively. For high energies these are usually written in the form of delta (=1-n) and beta (k). Traditionally the most reliable method of obtaining optical constants in the x-ray range has been to measure the transmission of a foil or film. When the thickness of the absorber is known the absorption constant of the materials can be computed, and from this beta, $\beta = \alpha \lambda / (4\pi)$, where $\lambda$ is the wavelength of the light. Delta is often calculated then from beta using Kramers-Kronig equation. We will calculate delta using both R & T data taking into account sample roughness and any barrier/oxide/contamination layers, and the oxide and dead layers of the detector. Safety aspects of the experiments are fully addressed elsewhere. Only a small amount of chemicals are used typically in the nanogram range. None of the materials listed above is inherently dangerous and they are carried in sealed containers until they need to be opened and placed into the vacuum chamber. None of the materials mentioned above are volatile at room temperature and all are well adhered to the substrate so they do not represent a danger to the beamline or other experimenters.

**Detailed Description of Experimental Program:**

**Capability of the Experimental Group**

The members of the group include at BYU: professors Turley, Vanfleet and Linford and at the ALS, Erik Gullikson. We referenced our group’s previous work above.

**Availability of Resources required:** the standard set up at beam line 6.3.2 is adequate for our needs.

**Justification for beam line and the amount of beam time requested:**

“Beamline 6.3.2 at the ALS is ideally suited for our studies. We have done in the past all of the kind of measurements which will be required for these experiments. It is the proper beamline for the measurement of optical constants. As the website says it is a “bend magnet beamline dedicated to EUV and soft x-ray reflectometry and scattering…” In addition to its extremely accurate reflectometry there are leads that can be used to measure transmission when an XUV diode is the sample. It has good brightness, resolution and well suited for accurate measurements in both angle and energy. Calibration and alignment procedures are straightforward and rapid.

We are requesting the minimum about of time that experience has shown us is required we require based on the familiarity which Professor Turley, Allred and some of the senior students have. We will study between 2-4 samples each time we come, dedicating one to two shifts per sample. The samples will have been characterized at BYU previously so that the results will mostly likely be useable in obtaining $\delta$ and $\beta$ as a function of energy for the desired materials. It is our goal to model the R & T vs. angle data while at the ALS so that we can fill in any missing data or remeasure any suspect data before the end of our allotted time. The high-harmonic EUV measurement system at BYU is not able to do all of these at the present.

**Outcomes:**

We expect to publish the optical constants obtained in these studies. In addition we hope to be able to add to an online data base (probably associated with CXRO) which supplies optical constants for compounds.

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1 The use of IRD detector to obtain transmission data is well known in this field. See Uspenskii. ( for example Uspenskii, Yu. A., "Efficient method for the determination of extreme-ultraviolet optical constants in reactive materials: application to scandium and titanium" J. Opt. Soc. Am. A 21 #2 (Feb 2004)) We are adding reflectance data
References


Sandberg, Richard L., and David D Allred. n.d.
