

## Aluminum oxide growth rate as a function of partial pressure of oxygen

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*Abstract: Aluminum oxide growth rate is needed to properly design an aluminum mirror for reflection of far ultraviolet radiation. An apparatus has been designed for evaporation of aluminum mirrors within a vacuum chamber where their reflectance can be tested without exposure to the atmosphere. The mirrors will then be exposed to different partial pressures of oxygen. Aluminum oxide growth rate over time will be determined as a function of oxygen partial pressure.*

### I. Introduction and Background

A new generation of telescopes has emerged in the last couple of decades that gather light from the ultraviolet and x-ray portions of the electromagnetic spectrum. Such telescopes can potentially glean information from highly energetic objects and events in the universe. These include protostars, supernovae, and black holes immersed in dust-and-gas-filled environments. Information from the far ultraviolet can also play a role in understanding the transfer of energy within highly dynamical systems. Examples include the centers of galaxies and their surrounding environments (see Figure 1), the atmospheres of hot stars, and shock propagation in gamma ray bursts.

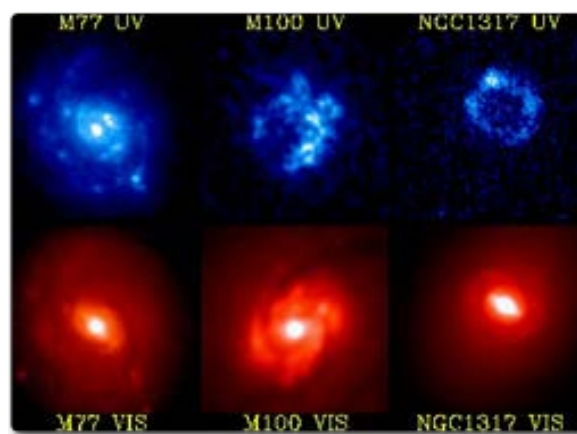


Figure 1: Galaxies shown in visible light (bottom row) and ultraviolet radiation (top row). The ultraviolet images highlight newly formed stars while the visible light images mostly show older red and yellow stars. (Credit: NASA)

Since Earth's atmosphere blocks most far ultraviolet radiation, these observations require telescopes in space (see Figure 2).

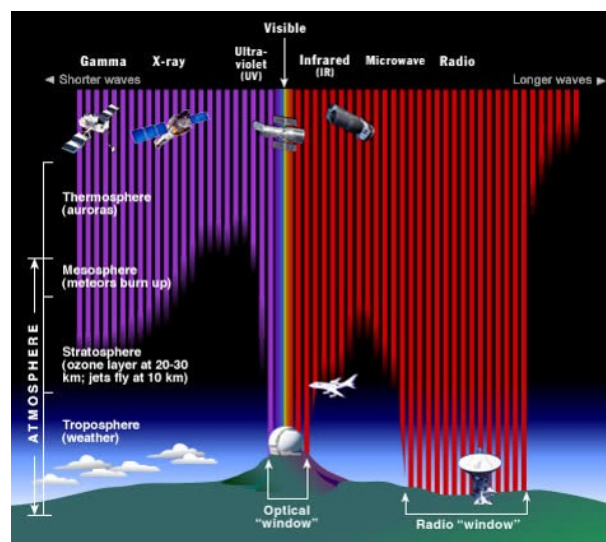
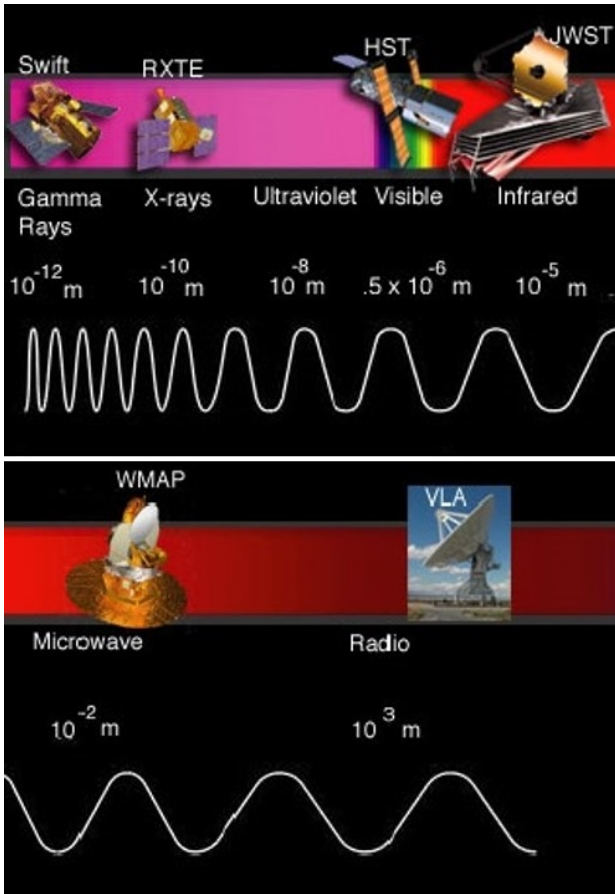


Figure 2: Far Ultraviolet Radiation Blocked by Earth's Atmosphere (Credit: STScI/JHU/NASA)

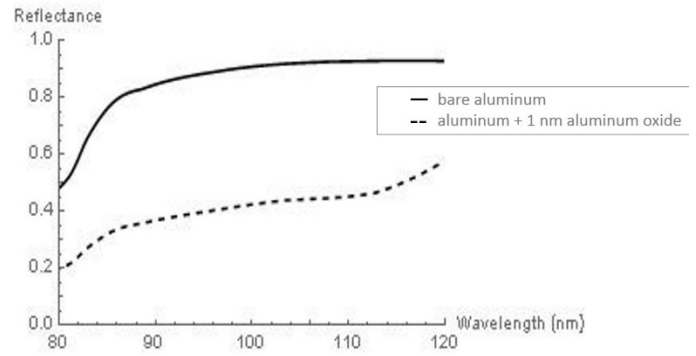
Therefore, the need for telescope mirrors that reflect well in the ultraviolet range has grown in recent years (see Figure 3).



**Figure 3: The Need for Telescopes in the Ultraviolet Range**  
 This figure shows the Swift observatory, Rossi X-Ray Timing Explorer, Hubble Space Telescope, James Webb Space Telescope, Wilkinson Microwave Anisotropy Probe, and Very Large Array. (Credit: NASA)

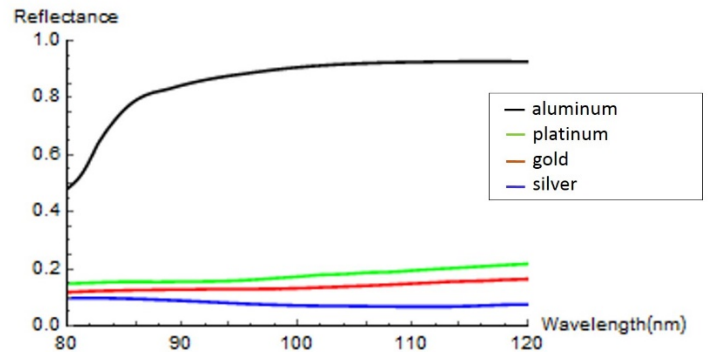
Indeed, NASA’s Cosmic Origins Program calls for broadband telescopes that can collect radiation from the infrared to the far ultraviolet<sup>1</sup>.

Aluminum is an excellent candidate for reflection at ultraviolet wavelengths; however, aluminum mirrors made on Earth oxidize upon contact with Earth’s atmosphere. This is a problem because a layer of aluminum oxide significantly decreases reflectance in the ultraviolet<sup>2</sup> (see Figure 4).



**Figure 4: Reflectance vs. Wavelength for Aluminum and Aluminum Oxide**

To circumvent this problem, one possible approach is to coat the aluminum mirror in space (where oxidation won’t take place). While this solution has been suggested previously, a proposed method has not yet been delineated<sup>3</sup>. A second solution is to use a material other than aluminum for the mirror. However, the reflectance of other materials in this wavelength range is far less than that of aluminum (see Figure 5 below)<sup>4</sup>.



**Figure 5: Reflectance for Various Materials in the Ultraviolet**

A third possibility is to evaporate the aluminum mirror on Earth and then coat it with a substance that prevents the aluminum from oxidizing. This idea is being actively pursued by multiple research groups<sup>5-7</sup>. A fourth plan is to evaporate the aluminum mirror in vacuum

and then keep it in vacuum at a certain pressure until it is placed in space.

In considering each of these possible solutions, it is important to understand how fast aluminum oxidizes at different partial pressures of oxygen. Even if the mirror is coated in orbit, it will still come in contact with some oxygen. If the oxide growth rate is too high, it may be necessary to choose a material other than aluminum. If the aluminum is coated with another material, scientists need to know if aluminum oxidation will occur between the aluminum and the coating, and if the aluminum is evaporated and preserved in vacuum, scientists need to know what reflected wavelengths they can expect if the mirror is preserved at a certain vacuum pressure.

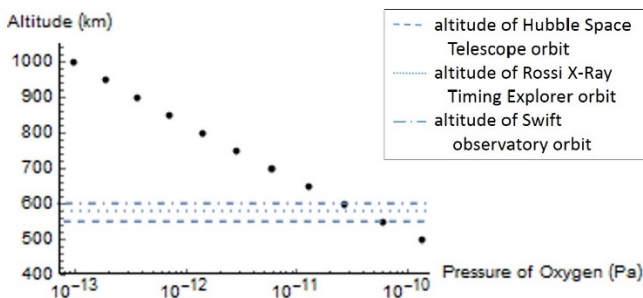


Figure 6: Pressure of Oxygen as a Function of Altitude (Pressures of Oxygen an Orbiting Telescope Would Likely Encounter)

Researchers have looked at some aspects of aluminum oxidation in the past. Madden and Hass tested reflectance of aluminum over time as it oxidized for wavelengths greater than 102 nm and showed that reflectance does decrease with oxidation<sup>8,9</sup>. Grande and Burton showed a decrease in reflectivity for 102 nm radiation incident on thin film aluminum as a function of exposure to H<sub>2</sub>O, air, and O<sub>2</sub><sup>10</sup>. Larruquet exposed aluminum thin film samples to different partial pressures of oxygen at very low pressures and immediately measured reflectances<sup>11</sup>.

## II. Objective

Data is needed for a wider range of far ultraviolet wavelengths over a range of partial pressures of oxygen. To gather this data, we will evaporate an aluminum mirror in vacuum, measure the changes in its reflectance as oxidation takes place, and use those measurements to calculate oxide growth rate as a function of oxygen partial pressure.

## III. Methodology

We designed an apparatus that will allow aluminum deposition within the same vacuum chamber where reflectance measurements will take place. Previously, we had deposited films in an electron-beam evaporation system and then transported the samples (exposing them to the atmosphere on the way) to the vacuum chamber where their reflectance was tested. This new apparatus will evaporate aluminum in situ without allowing aluminum gas to coat the reflectance equipment already located in the vacuum chamber.

It's important to deposit the aluminum mirror in situ because aluminum oxidizes so quickly (see Figure 7).

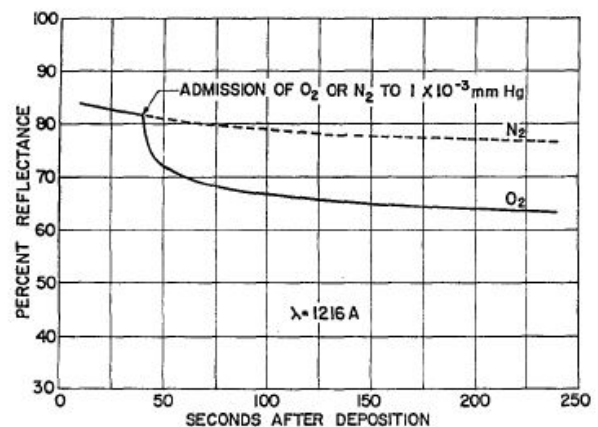


Figure 7: "Effect on the reflectance of freshly deposited aluminum films at  $\lambda 1216 \text{ \AA}$  of introducing oxygen and nitrogen into the vacuum system." (Credit: Madden, Canfield, and Hass<sup>12</sup>)

In her reflectance measurements of copper and copper oxide, Nicole Brimhall found that in some cases other researchers thought their thin film surface was copper when in fact it was copper oxide: Brimhall's reflectance data for copper oxide matched their data for bare copper<sup>13</sup>. In order to avoid this happening with our aluminum films, we will deposit the mirror in vacuum and test its reflectance there as well.

An aluminum wire will be placed in contact with a tungsten filament and then wire and filament will be placed inside a cup (see Figure 8).

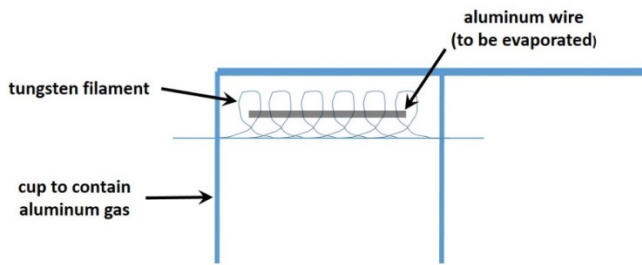


Figure 8: Aluminum Wire with Tungsten Filament in Deposition Chamber

A servo motor will move the cup so that it covers a silicon substrate (see Figure 9), and

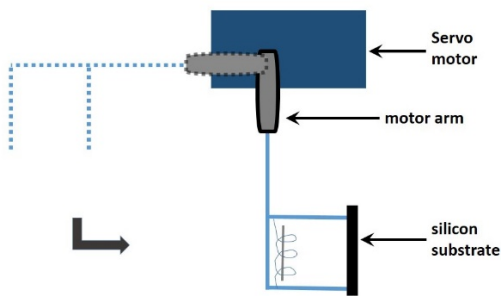


Figure 9: Moving Deposition Chamber to Cover Substrate

the tungsten will be heated to the point that the aluminum evaporates. Aluminum will then be deposited on the substrate and will coat the inside of the cup – but will not be deposited on the reflectance instruments inside the vacuum chamber. The motor will then remove the cup

so that the mirror's reflectance can be tested (see Figure 10).

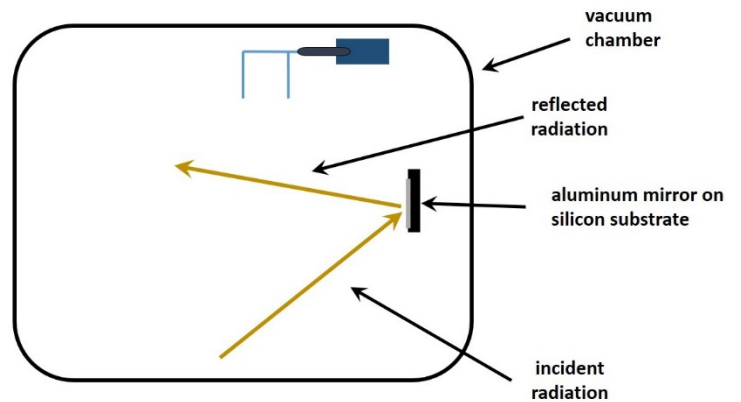


Figure 10: Ready to Test Reflectance

Next, we will use a hollow cathode plasma discharge light source, a monochromator (which selects the desired wavelength from the spectrum produced by the plasma source), and a Channeltron detector (which registers the reflected radiation) (see Figure 11),

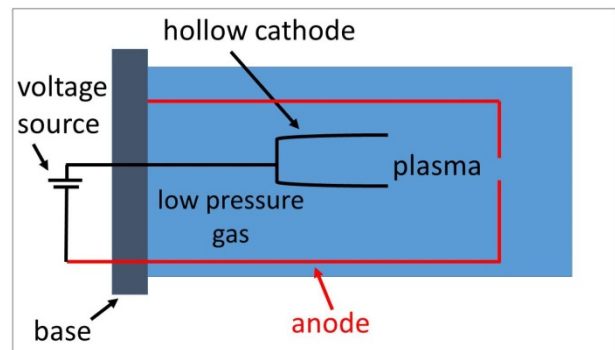


Figure 11a: Hollow Cathode Plasma Discharge Light Source

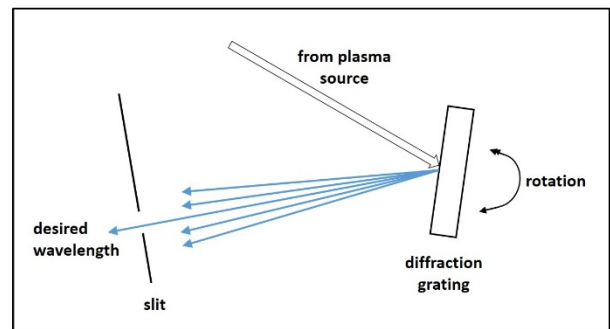


Figure 11b: Monochromator



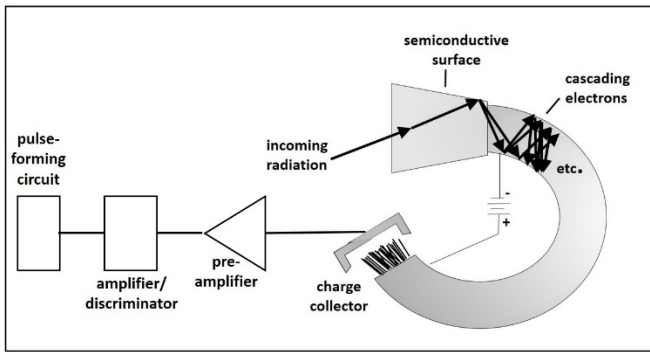


Figure 11c: Channeltron detector

to test the aluminum mirror's reflectance for selected wavelengths between 80 and 120 nm (see Figure 12).

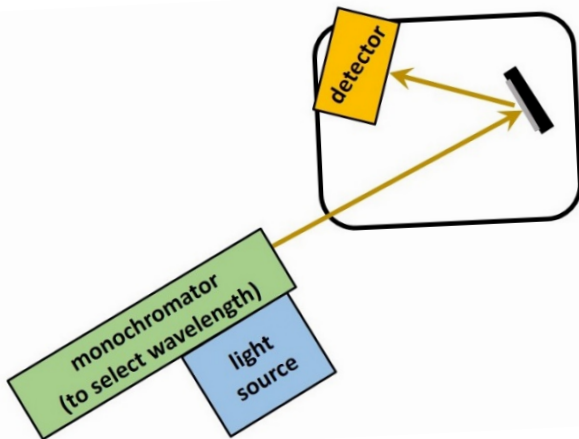


Figure 12: Setup for Reflectance Measurements

Reflectance is simply measured by dividing the amount of radiation arriving at the detector by the amount of incident radiation from the monochromator.

Third, we will allow the aluminum to oxidize at a specific partial pressure of oxygen (measured by a mass spectrometer in the vacuum chamber) by venting air from the atmosphere into the vacuum chamber. We will measure the mirror's reflectance over time as the mirror oxidizes.

Fourth, we will use these measurements of reflectance over time to determine the rate of oxide growth on the mirror. We will repeat steps three and four for different partial

pressures of oxygen. This will give us oxide growth rate as a function of partial pressure of oxygen and as a function of time.

Using these results, NASA will be able to select wavelengths in the far ultraviolet and determine what level of vacuum (what partial pressure of oxygen) will be necessary to preserve reflectance for an aluminum mirror at those wavelengths.

#### IV. Progress

We already have the plasma light source, monochromator, and Channeltron detector on site, but the monochromator's diffraction grating was damaged and needed to be replaced. We bought an aluminum diffraction grating, and will now coat it (to prevent oxidation) before it will be ready for use. We also purchased a mass spectrometer that is operable over a wide pressure range (since we will be venting air into the vacuum chamber).

The cup for aluminum deposition has been made. It is designed to allow air molecules to escape (so the cup can remain under vacuum during deposition) while containing the aluminum gas. Its design allows aluminum wire to easily be inserted for deposition and allows tungsten filaments to be removed and replaced once they are burned out. We have also developed a preliminary program for the servo motor to move the cup. Feedthroughs allowing the tungsten filament to be attached to high voltage outside the vacuum chamber and connecting the servo motor to the power supply have been designed and installed.

#### VI. Future Work

The deposition cup and servo motor will be installed in the vacuum chamber and connected to their respective power supplies.

Deposition of aluminum thin films can then take place.

Once the new diffraction grating has been coated, monochromator alignment will take place. The system will then be ready to test reflectance of bare aluminum and aluminum with oxide growth.

The new mass spectrometer will also be installed in the vacuum chamber – enabling measurement of partial pressures of oxygen as the aluminum oxidizes. Analysis can then be performed to determine oxide growth rates at different partial pressures of oxygen.

Determining the oxide growth rate will provide essential data for the design of aluminum mirrors for space. It will allow telescope mirrors to be made that will reflect well in the far ultraviolet. Such telescope mirrors will assist in many areas of astrophysics research – including studying protostars, supernovae, black holes, exoplanet atmospheres, and energy transfer to and from galaxies. This information will aid in examining exoplanet atmospheres for biomarkers – to search for life outside our solar system. It will help us draw conclusions about the origins of the universe by enabling us to see emissions at the start of supernovae explosions. In sum: it will allow us to investigate and analyze previously unseen features of the universe.

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<sup>5</sup> A. Karim. "Thin Film Heater for Removable Volatile Protecting Coatings". *The Scientific World Journal*, vol. 2013, 2013.

<sup>6</sup> C.S. Moore *et al.* "Current progress in the characterization of atomic layer deposited AlF<sub>3</sub> for future astronomical ultraviolet mirror coatings". *Proceedings of the SPIE*, vol. 9601, 2015.

<sup>7</sup> J. Hennessy *et al.* "Ultraviolet optical properties of aluminum fluoride thin films deposited by atomic layer deposition". *Journal of Vacuum Science and Technology*, vol. 34, no. 1, Jan/Feb 2016.

<sup>8</sup> R.P. Madden, L.R. Canfield, and G. Hass. "On the Vacuum-Ultraviolet Reflectance of Evaporated Aluminum before and during Oxidation". *Journal of the Optical Society of America*, vol. 53, number 5, 1963.

<sup>9</sup> G. Hass. "Reflectance of Evaporated Aluminum in the Vacuum Ultraviolet". *Journal of the Optical Society of America*, vol. 46, number 12, 1956.

<sup>10</sup> M. Grande and W.M. Burton. "Aluminum Mirror Coatings in Space: a Study of the Decrease in Ultraviolet Normal Incidence Reflectance Produced by Controlled Oxidation of Evaporated Aluminum Mirror Surfaces". *Surface and Interface Analysis*. PA: Heyden and Son, 1986, p. 518.

<sup>11</sup> J. L. Larruquert. "Degradation of far ultraviolet reflectance of aluminum films exposed to atomic oxygen. In-orbit coating application." *Optics Communications*, 124, pp 208-215, 1996.

<sup>12</sup> R.P. Madden, L.R. Canfield, and G. Hass. "On the Vacuum-Ultraviolet Reflectance of Evaporated Aluminum before and during Oxidation". *Journal of the Optical Society of America*, vol. 53, number 5, 1963.

<sup>13</sup> N. Brimhall, "Extreme Ultraviolet Polarimetry with Laser-Generated High-Order Harmonics: Characterization of Uranium," Ph.D. dissertation, Dept. Physics and Astronomy, Brigham Young Univ., Provo, UT, 2009.

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<sup>1</sup> P. Tyler, "Cosmic Origins," (2016, Mar. 11). [Online.] Available: <http://cor.gsfc.nasa.gov/>.

<sup>2</sup> K. Balasubramanian, *et al.* "Aluminum Mirror Coatings for UVOIR Telescope Optics including the Far UV". *Proceedings of the SPIE*, vol. 9602, 2015.

<sup>3</sup> I. Appenzeller, "UV, X-Ray, and Gamma Spectroscopy," in *Introduction to Astronomical Spectroscopy*. New York: Cambridge University Press, 2013, ch.7, sec. 1, p. 181.

<sup>4</sup> B. Wang and L Gallais. "A theoretical investigation of the laser damage threshold of metal multi-dielectric mirrors for high power ultrashort application". *Optics Express*, vol. 21, issue 12, p. 14698-14711, 2013.