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Bare Aluminum Oxidation

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1 Introduction

The proposed LUVOIR mission calls for a large aperture mirror capable of reflecting light from the deep ultraviolet to the near infrared. A strong candidate for that mirror would be aluminum, which has a high reflectance over this entire range. Unfortunately, aluminum readily oxidizes in the atmosphere. A thin layer of aluminum oxide, Al_2O_3 , is transparent in the visible and infrared, but is strongly absorbing in the ultraviolet, significantly limiting the usefulness of such a mirror in the deep ultraviolet. In her Master's Thesis[1], Margaret Miles showed that a thin layer of AlF on top of the aluminum immediately after deposition provides an effective barrier to slow the aluminum oxidation and still all good light reflection in the deep ultraviolet. It is instructive to examine the oxidation rate of bare aluminum to compare it to the rates Miles measured in her thesis. A good point of comparison is the study made by Madden, et al. in which they measured the reflectance of evaporated aluminum immediately after deposition without exposing the sample to atmosphere[2]. This report is a review of the Madden work and a computation of oxidation rates derived from that.

2 Madden's Approach

Madden evaporated 70-90 nm aluminum films onto a substrate in a vacuum system with a base pressure of 2×10^{-7} torr. Immediately after deposition, the chamber pressure would rise to $3\text{-}6 \times 10^{-6}$ torr and then drop before 1×10^{-6} torr with 5 sec. Madden attributes

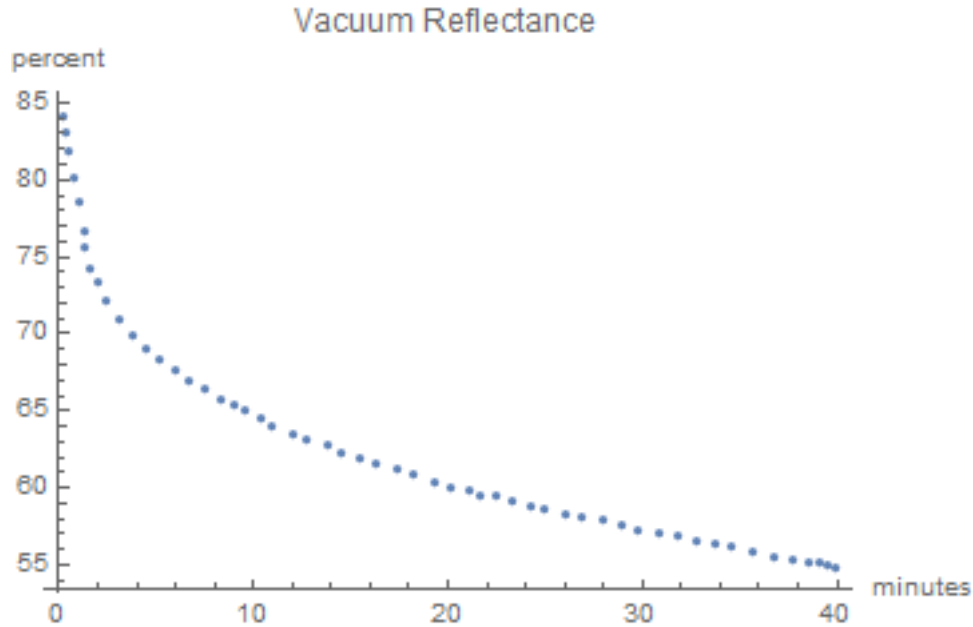


Figure 1: Reflectance of Al film under high vacuum conditions

the rise in chamber pressure to residual gas generated by the high temperatures of the materials near his heating filaments. They used a hollow cathode source to generate the vacuum ultraviolet (VUV) light for their measurements limiting them to spectral lines from hydrogen, helium, and argon.

Madden used a photomultiplier tube imaging the fluorescence from a phosphored plate to detect the VUV light reflecting from the sample. Since his detector was sensitive to residual light from the remaining glow from the heating elements used for evaporation, the light from the monochromator was chopped to allow for use of phase-sensitive detection to reject residual light from the filaments and other background sources. This allowed them to begin measuring the mirror reflectance within 10 seconds of evaporation.

3 Madden's Measurements

Madden measured the reflectance of the Al film at a number of wavelengths between 102.5 nm and 200 nm. For this analysis, I focussed on the measurements at 102.5 nm, the shortest wavelength he studied and the wavelength at which the light attenuation from oxide formation should be the greatest. I utilized two figures from his paper which I digitized by hand:

- Madden's Figure 4 which shows the reflectance of the mirrors kept in a high vacuum environment as a function of time up to 40 minutes from the time of deposition. My digitized data is shown in my Figure 1 and in Tables 1 and 2 .

time(min.)	percent reflectance
0.273	84.06
0.395	83.13
0.546	81.87
0.789	80.09
1.00	78.49
1.24	76.72
1.31	75.63
1.64	74.19
1.94	73.35
2.43	72.08
3.04	70.99
3.73	69.89
4.52	68.96
5.19	68.37
5.98	67.62
6.71	66.94
7.53	66.43
8.29	65.76
9.05	65.42
9.56	65.00
10.41	64.58
10.99	64.07
11.96	63.57
12.72	63.06
13.75	62.72
14.51	62.30
15.51	61.88
16.27	61.63
17.40	61.20
18.25	60.87
19.34	60.36
20.16	59.94
21.10	59.77
21.71	59.52
22.47	59.43
23.28	59.18
24.22	58.84
24.92	58.59
26.08	58.34
26.87	58.08

Table 1: Reflectance of Al film under high vacuum conditions

time(min.)	percent reflectance
28.02	57.92
28.87	57.58
29.69	57.24
30.90	57.07
31.88	56.90
32.76	56.57
33.73	56.31
34.55	56.14
35.64	56.14
36.73	55.55
37.74	55.30
38.49	55.22
39.04	55.13
39.53	54.96
39.92	54.88

Table 2: Reflectance of Al film under high vacuum conditions (continued).

- Madden’s Figure 12 which includes data on the reflectance of the aluminum films in air as a function of time. My digitized data is shown in my Figure 2 and Table 3.

4 Computed Oxidation Rates

Madden didn’t specifically state the angle of incidence for the reflectances reported in Tables 4 and 12 in his paper. I’ve assumed normal incidence. Since the theoretical reflectance increases as the angle approaches the grazing angle, this would make my computed oxide thicknesses a minimum possible thickness. I used interpolated data from CXRO[3] and Palik[4] to get the index of refraction of Al and Al₂O₃ to compute the thickness of the oxide assuming a planar surface with no roughness. The calculation was done using the Fresnel coefficients at each interface and then the Parratt[5] formula to combine the effects for each interface in the stack. The thickness of the oxide layer was adjusted for time until the computed reflectance matched the measured reflectance.

Figure 3 is a plot of the oxide thickness as a function of time for the sample kept in a high vacuum. Figure 4 is a plot of the same data using a logarithmic time axis. This illustrates the almost logarithmic growth of the sample. Here is a copy of the exported data from the calculation. The first column is the time in minutes and the second column the sample thickness in nm.

```
0.273224044      0.137445732
0.394656952      0.153933787
0.546448087      0.176631528
```

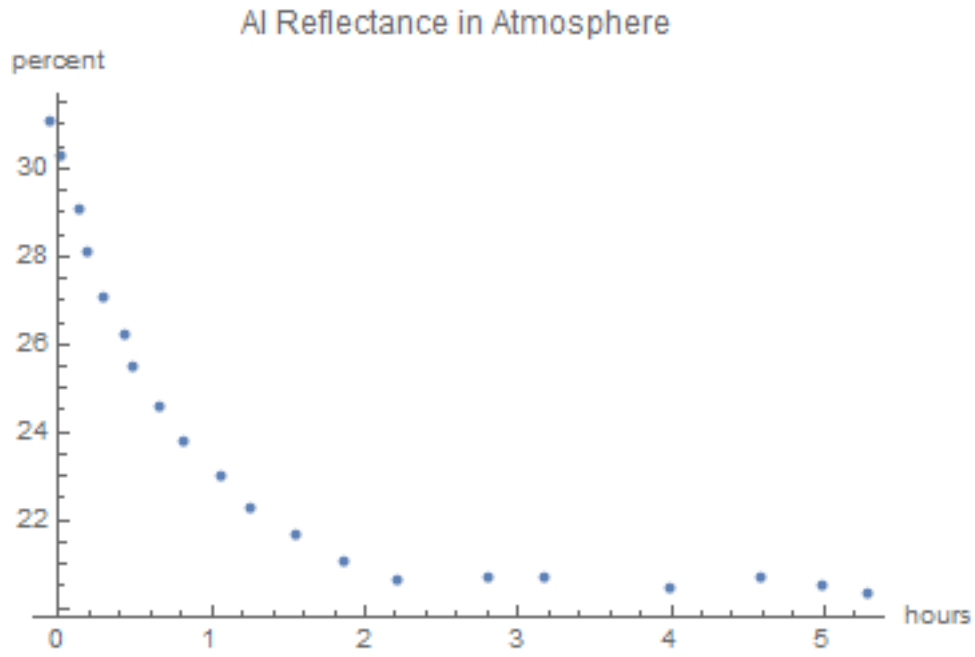


Figure 2: Reflectance of aluminum film exposed to atmosphere.

time (hours)	reflectance (percent)
-0.06	31.10
0.0	30.27
0.12	29.10
0.29	27.11
0.42	26.25
0.47	25.50
0.66	24.56
0.81	23.78
1.06	23.00
1.24	22.26
1.54	21.67
1.86	21.08
2.21	20.61
2.81	20.69
3.18	20.69
3.98	20.46
4.58	20.73
4.99	20.50
5.28	20.34

Table 3: Reflectance of aluminum film exposed to atmosphere.

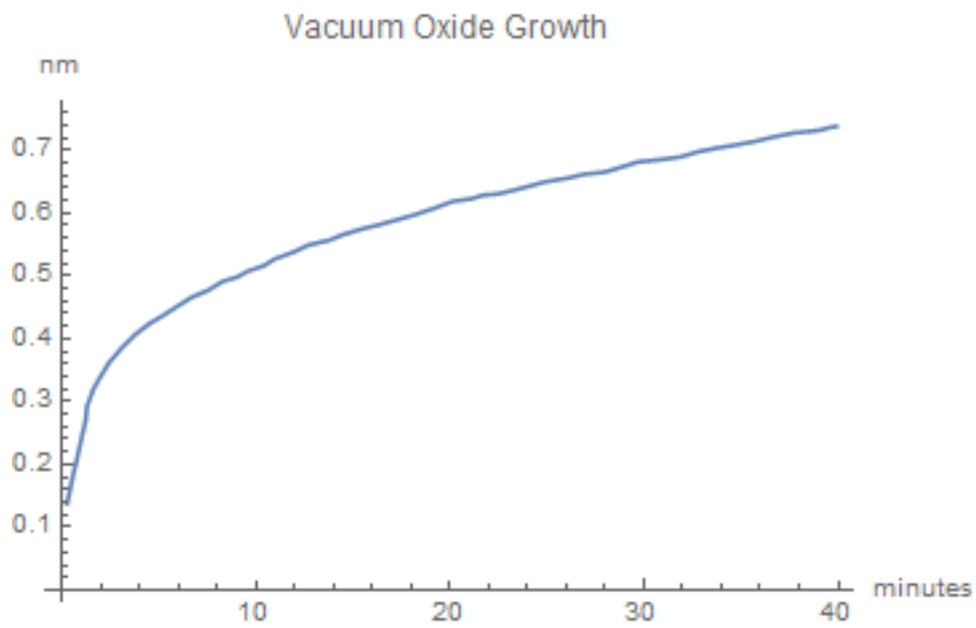


Figure 3: Oxide thickness of sample in high vacuum.

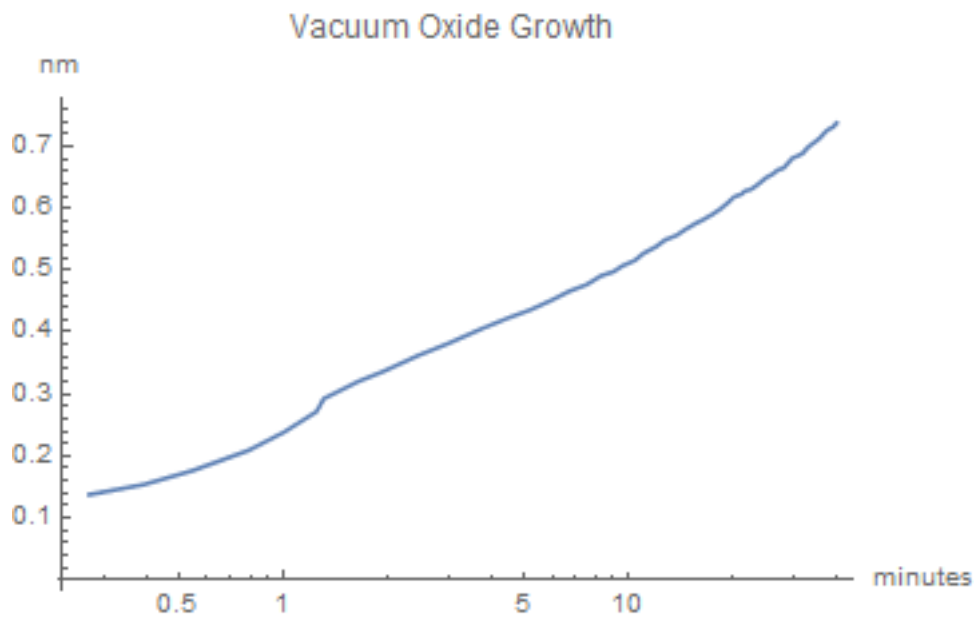


Figure 4: Oxide thickness of sample in high vacuum with a logarithmic time axis.

0.789313904	0.208836809
1.001821494	0.238422343
1.24468731	0.271638319
1.305403764	0.292482654
1.639344262	0.320078332
1.942926533	0.336495195
2.428658166	0.361384311
3.035822708	0.383218406
3.734061931	0.405304979
4.523375835	0.424196538
5.191256831	0.43631766
5.980570735	0.452017796
6.709168185	0.466084988
7.528840316	0.476705542
8.287795993	0.490961509
9.04675167	0.498130909
9.56284153	0.507132031
10.41287189	0.51617741
10.9896782	0.527091065
11.96114147	0.538070226
12.72009715	0.549115893
13.75227687	0.556517122
14.51123254	0.565811334
15.51305404	0.575153534
16.27200971	0.580782148
17.39526412	0.590202434
18.24529447	0.59777437
19.33819065	0.609192573
20.15786278	0.618763752
21.09896782	0.622606645
21.70613236	0.628386575
22.46508804	0.630317398
23.28476017	0.636122484
24.22586521	0.643892254
24.92410443	0.649742022
26.07771706	0.655611202
26.86703097	0.661499951
28.02064359	0.665436736
28.87067395	0.673336808
29.69034608	0.681272544
30.90467517	0.685253909
31.87613843	0.68924434
32.75652702	0.697252601
33.72799029	0.703282974

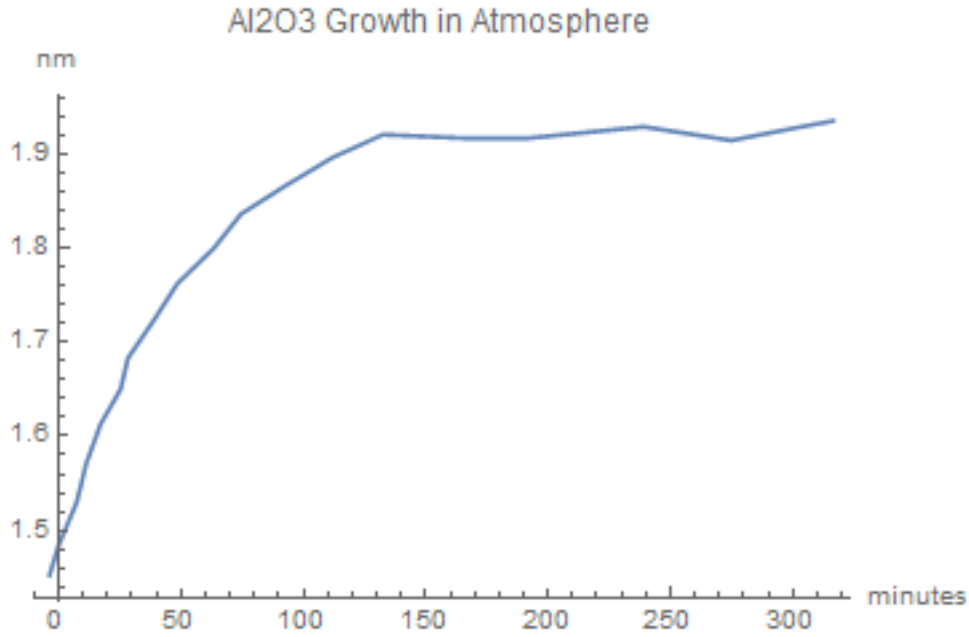


Figure 5: Oxide thickness of the sample in air.

34.54766242	0.707314833
35.64055859	0.713380169
36.73345477	0.721500319
37.73527626	0.727615444
38.49423194	0.729658619
39.04068002	0.731704207
39.52641166	0.735802645
39.92106861	0.73785551

Figure 5 is a plot of the oxide thickness as a function of time for the sample exposed to air. Figure 6 is a plot of the same data using a logarithmic time axis. They show logarithmic growth of a sample up to about 100 hours and then a saturation in the oxide growth. The exported data from the computation follows. The first column is the time in hours and the second column is the computed thickness in nm.

-3.723816912	1.45249865
0	1.4841463
7.447633825	1.530570104
11.17145074	1.570415356
17.1295578	1.613049715
25.321955	1.650138651
28.30100853	1.682963747
39.47245927	1.725543537
48.40961986	1.762037687

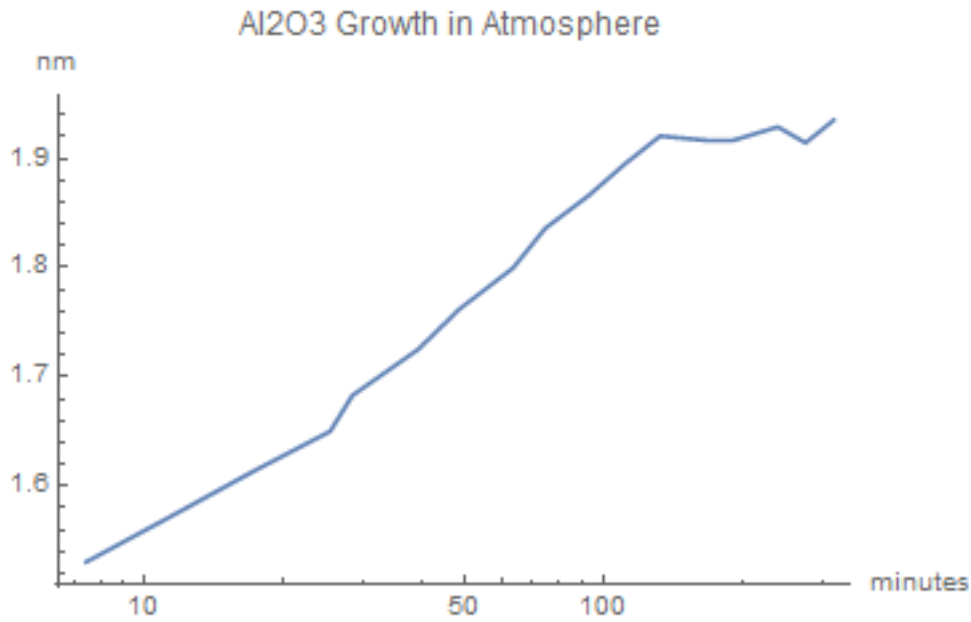


Figure 6: Oxide thickness of the sample in air with a logarithmic time axis.

63.30488751	1.799510692
74.47633825	1.836072945
92.35065943	1.865639342
111.7145074	1.89585848
132.5678821	1.920526328
168.3165244	1.916383743
190.6594259	1.916383743
239.0690458	1.928849731
274.8176881	1.914317191
299.3948798	1.926764073
316.5244375	1.935126089

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