Improved EUV Filter Transmission with Plasma Cleaning

Bruce M. Lairson^{*a}, Dave Grove^a, Ryan Smith^a, Heidi Lopez^a, Travis Ayers^a, Brennan L. Gantner^b, and Matthew N. Beasley^b ^aLuxel Corporation, 515 Tucker Ave., Friday Harbor, WA, USA 98250; ^bCASA, 389 UCB, University of Colorado, Boulder, CO, USA 80309

ABSTRACT

As-fabricated free-standing indium foils were found to have transmission in the 90nm to 120nm band ranging from 10% to 70% of modeled values based on pure indium. Auger depth profiling of the as-deposited indium showed little surface contamination and high purity. However, final free-standing filters were found to have heavy contamination, particularly on the surface. An argon/hydrogen plasma bombardment was developed which improved EUV transmission by 50% to 500% in the finished filters without causing significant pinholes to develop in the foils or appreciably affecting blocking characteristics.

Keywords: hydrogen, plasma, cleaning, contamination

1. INTRODUCTION

Indium is occasionally used as a spectral filter in the EUV wavelength range, especially in the range 50nm to 120nm, where relatively few materials have useful transparency[1-4]. For the present project, the Far Ultraviolet Imaging Rocket Experiment (FIRE) [5], the filter is required to have to have transparency in the 90nm-110nm range, while blocking Lyman α at 121.5nm. Figures of merit for the filters are transmission at 91nm, blocking at 121.5nm, and visible light leakage through pinholes.

Previous studies have shown strong degradation of indium filter transmission in the passband region from 77nm to 90nm, as well as high variability between filters[1,2]. Such degradation occurred even under clean dry nitrogen storage, and has generally been attributed to accumulation of impurities and on the surfaces of the freestanding films. Discrepancies between theoretical and measured transmission as large as 6.5X were reported for ostensibly good filters after storage in dry nitrogen[1]. For the FIRE project, it is necessary to have film transmissions within about 2X of the transmission of pure indium. Generally, the FIRE requirements correspond to a required indium thickness of between 200nm and 250nm.

2. EXPERIMENTAL

Indium films were evaporated onto semiconductor-grade silicon wafers coated with a release agent. As reported previously, dynamic deposition rates of >100nm/sec were maintained to improve various aspects of film formation and purity[1]. Base vacuum levels of about 1e-6 Torr were maintained during deposition. After deposition, the films were released from their substrates and attached to an 80% open mesh using a space-qualified epoxy. Films were air freighted from Friday Harbor, WA, to Boulder, CO, and typically measured within a few days of fabrication. As-

fabricated filters had transmissions that varied from 10% to 70% of theoretical transmission at 91 nm.

Transmission was measured in vacuum using argon and argon-hydrogen plasmas to generate spectral lines from 91 nm to 121.5nm. Transmission values at 91 nm were typically measured to be in the range from 1% to 15%, while transmission at 121.5nm was generally below 1E-4[5].

Typical transmission at 91nm versus film thickness for the as-fabricated filters are shown in Figure 1. Adjustments in thickness were made by changing the evaporation rate for a fixed deposition time of a few seconds. Figure 1 also shows calculated transmission using reference data for pure indium (8.1/micron), and assuming an 80% mesh transparency[3,4]. For the as-fabricated filters, maximum transparency at 91nm of about 10% is obtained, with considerable (3X) variability. The maximum transmission occurs for film thicknesses between 100nm to 150nm, with lower transmission observed at higher and lower thickness. The inset shows the calculated absorption coefficient versus film thickness for the same data. The absorption coefficient increases rapidly with decreasing film thickness, reaching about 50/micron for the 50nm thick films. For the thinnest films, the very high absorption coefficients are presumably due to an increase in contaminant content. For the 295nm film, the lowest absorption coefficient was observed, 10/micron, which is near the bulk absorption coefficient of 8.1/micron.



Figure 1. Filter transmission at 91 nm versus indium thickness. Solid line is the expected filter transparency based on reference data and opaque mesh transmission loss. Inset: Absorption coefficient versus indium thickness.

Contamination was investigated as the source for the low transmission and high variability. Various aspects of vacuum system cleaning, filter assembly, shipping container materials, etc. were measured and varied, with no apparent change in the filter transmission or variability.

Contamination was characterized using Auger depth profiling. Typical depth profiles for a film on wafer, and a free-standing film, are shown in Figure 2. For the freestanding film, the depth profile was concentrated at the center of a 340micron mesh cell aperture, minimizing the contribution from the mesh and epoxy. For the freestanding film, the carbon concentration at the surface is approximately 50%, compared with approximately 20% on wafer. Auger measurements from indium samples from Alfa Aesar showed similar levels of carbon surface contamination. At a depth of 20nm into the film, the carbon content on wafer is about 1-2%, compared with 20% for the freestanding film. Alternately, at a depth of 20nm, the indium concentration is about 97% on wafer, compared with 30% for the freestanding film. For the freestanding film, the principal remaining impurity was oxygen, while for the film on wafer the oxygen contamination was below 1%. For the freestanding film, the transmission loss due to the presence of the measured amount of carbon was estimated to be about 70%[3,4]. Thus, remediation of carbon contamination appeared to be a significant issue for achieving higher filter transmission.



Figure 2: Depth profile density of indium and carbon contaminant for freestanding indium filter and film on wafer.

Plasma cleaning was investigated as a method for improving the film transmission at 91nm. The plasma chamber was equipped with a RF Services, Inc. plasma chamber source, driven by a 13MHz generator and matching network. To reduce the potential for pinholes and mitigate against EMI damage, a stainless steel mesh with 6mm apertures was installed between the RF chamber and the sample chamber. The sputter gas was chosen to be 95% Ar/5% H to selectively attack hydrocarbons.

Figure 3 shows the measured transmission of indium filters before and after hydrocarbon cleaning for nominally 210nm thick films. Transmission at 91nm increased from an average of 2% to an

average of about 10%, compared with a modeled transmission of 15% for a pure film on mesh. The remaining discrepancy is presumably due to the residual surface oxidation of the indium. For instance, a 1nm thick oxidation layer on each surface would be expected to reduce the transmission by about 20% [3,4].



Figure 3. Transmission at 91 nm for three 200-220 nm thick indium thin film filter before and after plasma cleaning.

The plasma excitation in the sample chamber was measured using an optical probe. The emission spectrum is shown in Figure 4. Given that an EMI shield was present between the plasma generator and the sample, it is uncertain whether the activated species in the sample chamber are ions or activated neutrals. The minimum observed hydrogen wavelength at 486nm in Figure 4 corresponds to an excitation energy of 2.4eV, which is insufficient to directly break the 4.0eV InO bond heat of formation[6]. In principle the activated hydrogen molecules H₂ are energetic enough to drive the formation of H₂O via InO+H₂ +1.5eV \Rightarrow In+H₂O, which requires a net 1.5eV per oxygen atom to be added via the activation. However, it does not appear that the plasma clean removes oxides, either due to low rates of reaction or due to residual oxygen in the chamber. For instance, plasma treatment of a freestanding Zr filter, where the primary contaminant was oxygen, resulted in a transmission improvement of only 1-2%. Subsequent plasma cleaning treatments on the filters shown in Figure 3 altered their transmission by less than 1%. The hydrocarbon removal rate was estimated to be about 1nm/minute based color changes of hydrocarbon films such as polyimide, though this appeared to vary greatly from location to location within the chamber.

Damage to the filters was characterized by measuring changes in visible light leakage before and after plasma cleaning. Cleaning using a reducing plasma has been shown to remove tarnish without damage to the underlying iron and silver artifacts[7]. Typical results are shown in Table 1. Small increases in the light leakage were observed, but remained low compared to the 1E-4 requirement for blocking at 121.5nm. Measurements at 121.5nm showed the cleaned films to have acceptable blocking. The increased visible light leakage is likely due to pinhole formation, either due to handling or due to plasma-induced defects. The small increase in light leakage indicates negligible

thinning of the metal. For instance the average optical density decrease in Table 1 after plasma cleaning is about 5%.

	Thickness	Pre-plasma Clean Visible	Post-plasma Clean Visible
Sample ID	(nm)	Transmittance	Transmittance
1178	235	3.10E-06	3.20E-06
1081	206	3.25E-06	7.68E-06
911	206	2.01E-06	2.63E-06
1718	215	2.08E-06	5.15E-06

Table 1. Visible transmittance of freestanding indium filters prior to and after plasma cleaning.



Figure 4: Emission spectrum for the Ar/H plasma used for cleaning freestanding filters.

3. CONCLUSIONS

Plasma cleaning with an activated Ar/H gas mixture was found to improve transmission at EUV wavelengths from about 10% of model expectations to about 70% of model expectations based on pure indium. No significant damage or thinning of the filters occurred from the plasma treatment. The plasma cleaning improved the apparent variability in filter transmission, and resulted in filters

acceptable for the FIRE program. The remaining differences in measured versus model are likely to be due to surface oxides and other contaminants which are less affected by the reducing plasma.

REFERENCES

- [1] R. Henneck et al., "Transmission of Thin Indium Filters in the EUV and Lifetime Tests", SPIE 3114, p648 (1997).
- [2] D. Hunter and R. Tousey, "Thin films and their Uses for the Extreme UV", Applied Optics v. 4, p891 (1965).
- [3] CXRO website http://henke.lbl.gov/optical_constants/
- [4] B.L. Henke, E.M. Gullikson, and J.C. Davis. "X-ray interactions: photoabsorption, scattering, transmission, and reflection at E=50-30000 eV, Z=1-92", Atomic Data and Nuclear Data Tables Vol. 54 (no.2), 181-342 (July 1993) and updates.
- [5] B. Gantner, et al., in press.

[6] CRC Handbook 75th Edition, D. R. Lide ed., "CODATA Key Values for Thermodynamics" (1975).

[7] K. Schmidt-Ott, "Plasma-reduction: Its potential for Use in the Conservation of Metals", Proceedings of Metal 2004, p235.