# Interim Report on UCO/Lick Efforts for Protected Silver Coatings

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## Introduction

UCO/Lick has been attempting to develop robust protected-Silver coatings for astronomical optics, based on Bill Brown's "Holy Grail" coating that has been successfully used in HIRES for over 11 years. This short report gives the status of this project as of November 2004, with the goal of helping to decide how to proceed with recoating the DEIMOS tent mirror and collimator.

Progress has been significantly slower than anticipated. However, we feel confident that we will be ready to coat DEIMOS optics in February 2005, should the decision be made to follow that route.

## Equipment

We have in place a 48-inch vacuum chamber, just capable of holding the DEIMOS collimator. A dummy of the collimator has been installed and removed. The optics are held in a fixture which rotates at up to 40 rpm.

For evaporating the coating materials, we have 2 sets of resistance filaments (for Al), a large resistance boat (for $MgF_2$) and a 4-pocket e-gun. The e-gun has just been completely serviced (mechanics and high-voltage supply) and is being re-characterized now. Several sources of potential process variability were identified, and it is expected the system will be vastly more stable from now on.

All oxides except for silica and sapphire are subject to oxygen-depletion when evaporated, and must be supplied with a low partial pressure of oxygen to re-oxidize them during deposition; failure to do so results in significant losses in transparency. While air can be used for the bleed gas, it is highly desirable for several reasons to be able to bleed pure $O_2$. This involves having both roughing and diffusion pumps with fluids suitable for handling pure $O_2$ or other reactive gases. We have just purchased a new roughing pump with this capability, and will be installing it and rebuilding the diffusion pumps for oxygen service shortly.

Currently, the deposition sources are located off-center under a pie-shaped opening in an aluminum mask. Thickness uniformity is achieved with a petal-shaped mask located approximately over the source. The shape of this mask has been determined via a computer program and yields thicknesses uniform to +/- 5% for both e-gun and resistance boat.

Plans call for placing the sources on center and adding a spool to increase the height of the tank. This allows us to replace the existing uniformity masks with two petals. The advantage is lower angle of deposition and a somewhat simpler geometry. Increases in the deposition rates due to less masking are offset by the additional source-to-target length, so the deposition rates remain almost unchanged. (This modification is unnecessary for the coating of DEIMOS optics, but is needed for ADC optics which must be coated through their centers.)

## Materials

The original "Holy Grail" coating consisted of 6 layers described in Table 1. Briefly, Ag does not adhere well to glass, so the Al acts as a glue layer. The Cu layer is of questionable functionality – it was expected to act as a passivation layer to help chemically protect the Ag, but there is little or no evidence that it actually has any effect. The Ag is the effective reflecting layer. The thin layer of sapphire ($Al_2O_3$) is another glue layer. The $MgF_2$ and $ZrO_2$ layers form a single high-low index pair which boosts the reflectance via interference effects; the top $ZrO_2$ layer also provides mechanical protection of the surface. In practice, the top layer has a small quantity of $TiO_2$ mixed into it to help relieve the internal stresses of pure $ZrO_2$.

Table 1

| | |
|---|---|
| $ZrO_2$ / $TiO_2$ | 25 nm |
| $MgF_2$ | 25 nm |
| $Al_2O_3$ | 2.5 nm |
| Ag | 120 nm |
| Cu | 50 nm |
| Al | 50 nm |

We have has no success in depositing the $ZrO_2$ layer, particularly because of poor thickness uniformity, and therefore have investigated two other promising materials. One, Cerac's M-1126, is a medium-index ternary compound of MgO, $Al_2O_3$ and $ZrO_2$ and has the desirable characteristic of producing amorphous films under e-gun deposition. Oxygen depletion is negligible for this compound. The other is $Ta_2O_5$, a high-index material that has somewhat less hardness than $ZrO_2$.

## Theoretical Results

Coating models were produced by the "Concise MacLeod" program, and are shown in Figure 1 for bare Ag, and a modified "Holy Grail" formula with $Ta_2O_5$ instead of $ZrO_2$ as the top layer. Two thicknesses of $Ta_2O_5$ (40 and 62 nm) are shown.

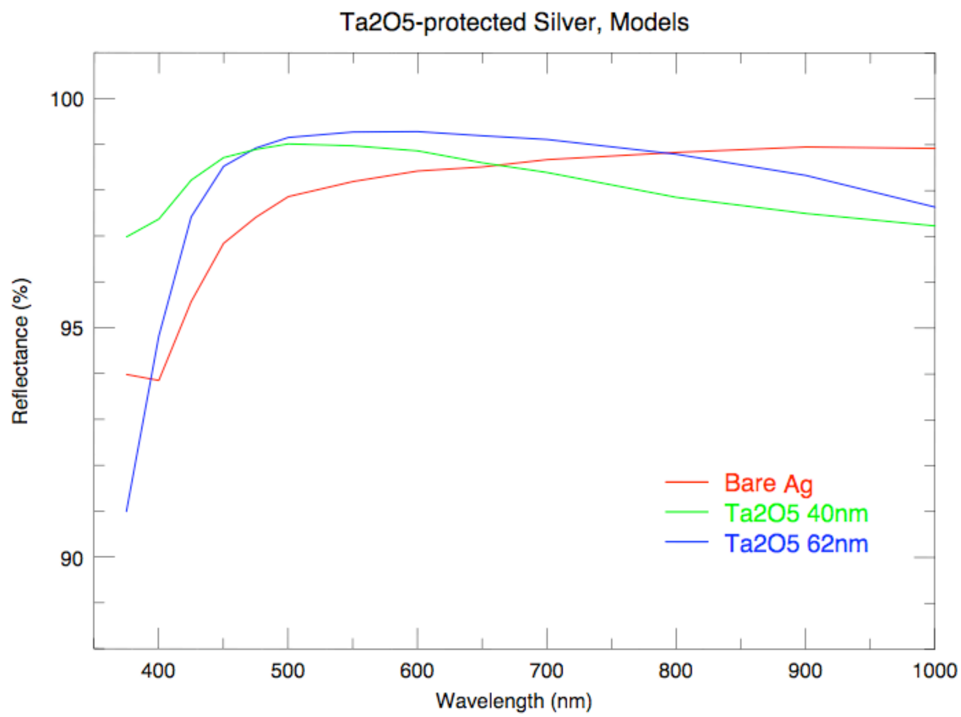


*Figure 1*

## Measured Results

Reflectance measurements of the coating samples were measured with the Varian/Cary 5000 spectrophotometer over the range 200-1100 nm. While DEIMOS optics do not need to perform blueward of 380 nm, the UV measurements allow us to verify the thickness of the top layers by examining the interference fringes below the Ag cutoff (320 nm).

Results for the DEIMOS spectral range are shown in Figure 2. Note that the performance falls steeply at the blue end. There is currently an unexplained absorption feature at ~370 nm; this feature is absent in earlier runs and thus we believe it is caused by something in our current process that can be modified to prevent it. Thus, we expect performance in the 370-400 nm range to increase.

Included in Figure 2 are the measured points for the Gemini protected-Ag coating. This coating is optimized for low IR emissivity, and the NiCr nitride and Si nitride layers both contribute to the lower performance in the visual-blue range. This is the range where the interference effects in the "holy grail" high-low pair actually gives it superior performance compared to bare Ag.

Figure 3 compares the modeled and measured reflectance for the $Ta_2O_5$-top layer coating. The theoretical performance for both bare and protected Ag are about 2% higher than the measured reflectance, but in general the behavior of the overcoated Ag relative to bare Ag agree between the models and actual measurements, with the exception of the steep fall-off below 400 nm noted above.

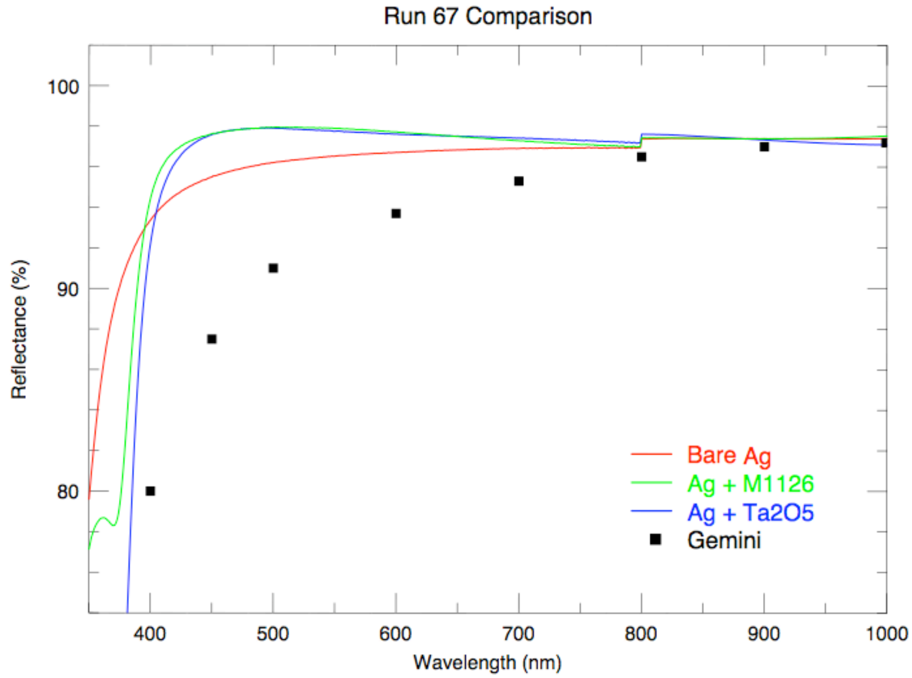


*Figure 2*

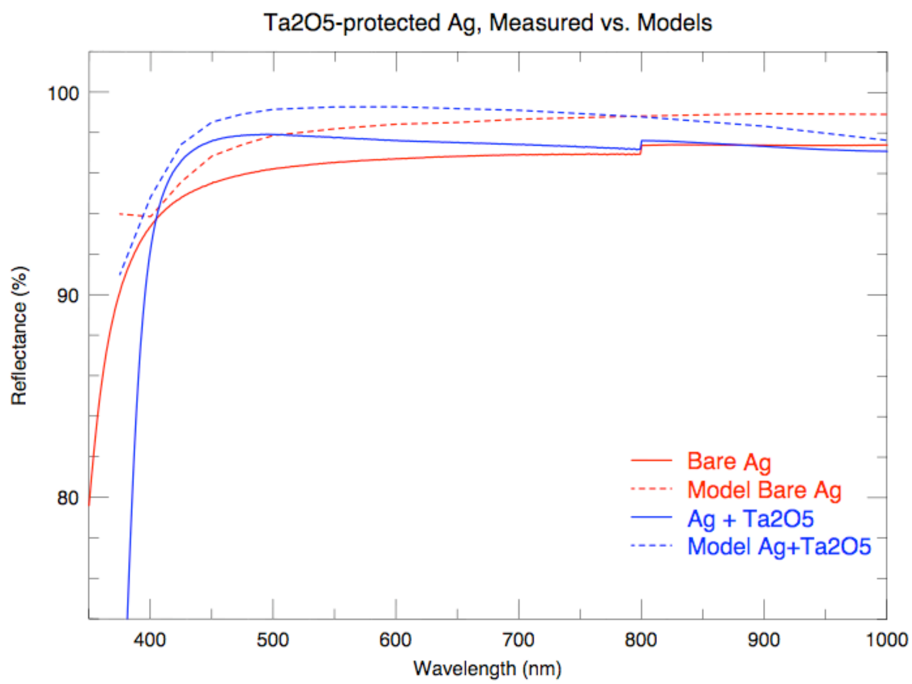


*Figure 3*

## Environmental Testing

So far, we have performed only limited environmental testing with qualitative results. These tests were fairly aggressive. The first was to expose samples to an

$H_2S$ atmosphere for about 3 hours. The second involved enclosing the samples in Petri dishes with several drops of bleach. The third was a high temperature and humidity test, heating the samples to ~90 C in an oven with an open container of water for about 24 hours.

The coatings tested were (a) bare Ag; (b) Ag over Al and Cu; (c) M-1126 over the lower 5 layers in Table 1; (d) same, except no Cu; (e) same as (d) with $Ta_2O_5$ instead of M-1126; and (f) same without top layer, ie, coating stops at $MgF_2$ layer.

$H_2S$ Test (equipment vented after 3 hours):

(a) Bare Ag started obvious tarnishing within 30s; gone within about 10m;
(b) (ditto)
(c) Ag failing after 3 hours;
(d) filmy after 3 hours;
(e) filmy after 3 hours;
(f) filmy after 3 hours.

The nature of the film is uncertain, and may have had to do with a reaction between residual ammonia and the polycarbonate plastics of the test setup. The film could be easily wiped off. However, by several days after the test, all overcoated surfaces had clearly failed from stress even though the Ag remained bright. The best-lasting coating was the one with the $Ta_2O_5$ top layer. Results with M-1126 were mixed: (d) held up reasonably well, whereas the Ag in (c) failed.

Bleach Test:

(a), (b) showed degradation of the Ag;
(c) through (f) all failed from surface stress.

It is clear that bleach will be a good chemical for stripping these coatings. (Bleach is used to strip the LLNL protected-Ag coatings).

Temperature-Humidity Test:

(a) Bare Ag "fogged" after 12-24 hrs;
(b) Al+Cu+Ag developed a gold cast (Cu diffusing into Ag??) after 12 hr;
(c) Slight fogging after 24 hr;
(d) Slight surface failure (possibly in regions of glove prints);
(e) Slight surface failure particularly along edges;
(f) Massive surface failure.

As with the $H_2S$ test, the $Ta_2O_5$ top layer seems to have stood up best.

We do not consider any of these tests to be conclusive; they were actually designed primarily as proof-of-concept processes of accelerated aging. The $H_2S$ test in particular was probably compromised by the presence of contaminants. Furthermore, we have not compared reflectance before and after the tests in order to quantify loss of performance. We intend to address these shortcomings in future tests. In spite of this, the modified

“Holy Grail” coatings are clearly more robust than bare Ag, and it appears $Ta_2O_5$ is more effective than M-1126.

## Future Considerations

One area we have considered but not yet investigated concerns pinholes, which are typically produced by residual dust particles on the cleaned substrate. Our chamber is equipped with a back-fill manifold located close to the substrate. It is believe that by blowing clean $N_2$ on the substrate between deposition of individual layers during a coating run, we should be able to redistribute these particles and thus prevent pinholes from going through all the protective layers and into the Ag. We plan to quantify the efficacy of this procedure soon.

When DEIMOS is recoated, we believe it is imperative to install sulfur “gettering” material (and possibly dessicants?) inside the instrument. We also urge that a set of witness samples be installed inside the instrument, and samples occasionally removed for measurement.