Progress toward high-performance astronomical coatings

Andrew C. Phillips*[a], William E. Brown[a], Brian Dupraw[a], David F. Hilyard[a], David J. Cowley[a]

[a]University of California Observatories, Santa Cruz, CA, USA 95064

ABSTRACT

We present an update on efforts at University of California Observatories to develop improved optical coatings for astronomical telescopes and instruments. The main thrust has been in the areas of protected silver mirror coatings and sol-gel based anti-reflection coatings. We report on the performance of silver coatings used for several years in Keck and Lick instruments, as well as that on the Lick 1-m telescope. We discuss process improvements, including use of reactive ion-assisted deposition of oxides. Sol-gel based AR coatings have been exposed to cryogenic environments to test their suitability for IR instruments, with encouraging results. Finally, we describe our plans for future work.

Keywords: Optical coatings, reflective coatings, anti-reflection coatings, mirror coatings, silver coatings, sol-gel

1. INTRODUCTION

University of California Observatories (UCO) has undertaken a program to develop more efficient coatings for astronomical optics. The requirements for such coatings are rigorous, and in some cases fairly unique to astronomy. The efficiency of observations generally scales with the rate at which photons are collected, so improvements in reflectance for mirrors and better anti-reflection (AR) coatings for transmissive optics has a direct benefit to observational astronomy. Furthermore, if mirror coatings can maintain their performance for longer periods of time, operational costs involved in periodic mirror recoating can be significantly reduced.

The requirements and challenges for astronomical optics are discussed in some detail by Phillips et al. (2008)[1], and are briefly reviewed here. First, the coatings must be "high-performance", that is, highly reflective for mirrors or transmissive for lenses and windows. For telescope mirror coatings in the thermal IR, high reflectivity is particularly important to reduce emissivity. Second, the coatings must be durable, cleanable and stable in their performance for as long as possible, realistically for 4 years or longer. Finally, if the coating lifetime is less than that of the optic (as is usually the case in astronomy), we must be able to strip the coating and deposit a new coating without damage to the underlying surface. We use the term "efficient" to include all the criteria above, meaning the coatings are both high-performance (science efficient), and do not need much time or labor for maintenance (operation efficient). For observing time lost to maintenance, operation efficiency also equates to science efficiency.

Most astronomical coatings have two additional challenges. The first challenge is that they generally must have high-performance over broad ranges in wavelength. For example, many telescopes are required to operate from the atmospheric cutoff to at least the mid-IR ($0.31\ \mu m \leq \lambda \leq 12\ \mu m$). AR coatings usually must cover either the optical ($0.31\ \mu m \leq \lambda \leq 1.1\ \mu m$) or near-IR ($0.8\ \mu m \leq \lambda \leq 2.5\ \mu m$) ranges. The second challenge is that many astronomical substrates are large. Several current-generation large telescopes have ~8-m monolithic mirrors, and next-generation telescopes will have ~1-m segments and also secondary and tertiary mirrors in the ~3-4 m range. Next-generation wide-field cameras will need AR coatings for lenses with diameters of order 1-m.

In Phillips et al. (2008), we presented our initial efforts at improving coatings for astronomy; this effort focused on protected silver reflective coatings and solgel based AR coatings. The current short paper provides an update to those activities, and a discussion of planned future work.

2. REFLECTIVE COATINGS

Most of our work has concentrated on protect silver coatings, as silver has the highest reflectivity of any metal at wavelengths $\lambda \geq 370$ nm. However, silver is soft, it adheres poorly to glass, and it is subject to tarnishing (mostly in reaction to sulfur compounds) and it reacts with halogens. Therefore, it must have both an adhesor layer underneath it

*phillips@ucolick.org; phone 1 831 459-2476; fax 1 831 459-5717; www.ucolick.org

and barrier/protective layers above it. The protective layers must be transparent to maintain the reflectivity, but they may also be used to produce an interference boost in the UV where the reflectance of silver falls rapidly. Protected-Ag coating designs and their quirks, advantages and disadvantages have been discussed previously[1]. Here we address three specific areas of study over the past two years: suitable adhesor layer(s); a scattering problem; and field testing of the "HG" coating on the Lick 1-m telescope. In addition, we are starting to explore some new materials made possible by reactive ion-assisted deposition (IAD).

2.1 Adhesor Layers

The original "HG" coating design[1] has a base layer of Al intended as an adhesor layer. We had already reported that the original Al+Cu layers under the Ag led to corrosion, and had proposed Al+Ti instead. To study if further improvements could be made, we coated a number of witness samples with six different underlayers and then coated them with Ag and protective layers simultaneously. These were then subjected to high-temperature (~60C) and high-relatively humidity (R.H. ~95%) for 48 hours.

The results were immediately apparent: Al+Ti, Al+ Al_2O_3 and Al+ Al_2O_3+Ti all showed areas of corrosion, with the former being the worst. Y_2O_3+Ti showed a few small spots where the coating was damaged. NiCr, Ti and NiCr+Ti showed no damage. These samples were periodically examined for a long period of time after the test, and all were stable except the Al+Ti sample: about a year following stressing, it developed many small spots ("measles") that scattered light badly.

We conclude that any base layers with Al will likely corrode on short timescales, so the standard adhesor layers for silver (Cr, NiCr) are strongly preferred, and can be removed chemically with chrome etchant. Ti and Ni are also stable, but cannot be easily removed. The results with Y_2O_3, which strips very easily in acid, are less promising but should be explored further.

2.2 Scattering in silver

We noticed that our coatings sometimes had a high density of small scattering centers, and eventually we able to confirm that this was arising in the silver layer itself. Silver is known to "spit" due to the fact that oxygen is easily dissolved in silver; bubbles of gas being released throw small particles of metal onto the substrate[2]. The problem is aggravated by the high deposition rates. An example of a larger "spit" is shown in Figure 1. These small silver particles are problematic not just because of the light scattering; they adhere only weakly to the surface, and are easily brushed off leaving pinholes through the protective layers. We found that the use of a molybdenum liner to hold the silver inventory was crucial to reducing these "spits".
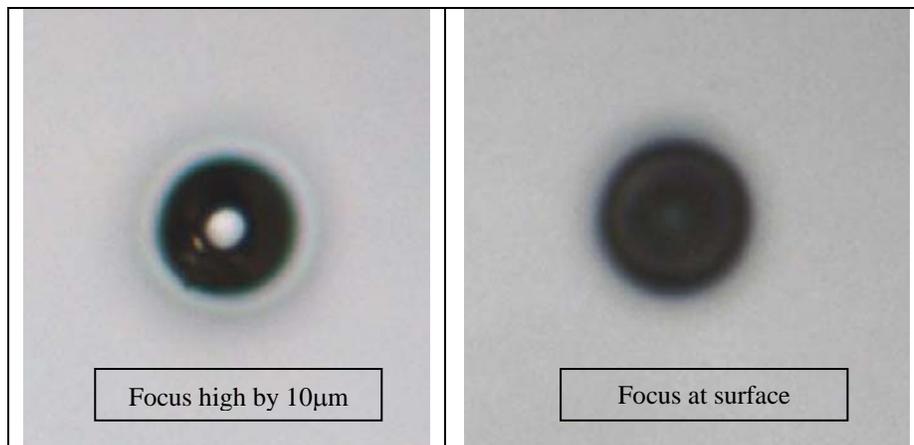

Figure 1. An example of a relatively large silver "spit" at two different microscope focii. The width of each image is about 40 μm. The appearance is roughly consistent with a hemisphere of metal (the bright central spot in the left image is actually an image of the light-filled microscope objective; the small spot just below and left of it is the image of a ceiling light).

2.3 Status of the HG coating on the Lick 1-m telescope

The Lick 1-m primary and secondary mirrors were coated with a modified HG coating[1] in the fall of 2007. This coating had Al+Ti as the adhesor layer and an additional (non-reactive) Al_2O_3 overcoat. After about 4 months, a few small spots of corrosion had developed, but these seemed to stabilize. However, after about 1.5 years in service, AO observers noted that the halos of PSFs had suddenly increased dramatically. Therefore, we stripped both mirrors and recoated them with aluminum. At this point, the secondary mirror had a fair amount of corrosion in a limited zone around the edge of the mirror but was otherwise in good condition. The primary's coating had degraded in numerous small spots (a few millimeters in size), in addition to the corrosion seen earlier. The degraded spots occupied about 3-4% of the total surface area. Adhesion was checked with a tape test, and was found to be good except where the tape crossed a spot of degradation. We measured reflectance in several locations, and found it reduced by about 4%, consistent with the total area of degradation (and overall, still higher than bare Al). We conclude that the performance was generally unchanged except in the numerous small spots, which were presumably also the cause of the increased light scatter.

The behavior of the primary was almost identical to that of the witness sample with the Al+Ti underlayers discussed above. It also developed corrosion spots when exposed to moisture, which then seemed to stabilize; then over the course of about a year it developed numerous small defects which increased light scatter dramatically. We conclude that this underlayer was probably responsible for most of the coating degradation seen in the 1-m primary mirror. We expect that a similar coating, put down with a Cr or NiCr adhesor layer (and without the silver "spits"), would last for at least two years and perhaps much longer.

2.4 New materials

We are starting a program to evaluate oxides and nitrides deposited reactively. Such reactively deposited coatings are generally found to have superior film structures. Primarily we have focused on oxides of Hf, Y, Al and Si. In addition, we have started to produce nitrides of Si, Al and Hf for evaluation. This is still very much a work-in-progress and most of our studies have been to characterize the optical performance of the resulting compounds. Testing with high-temperature and high relative humidity shows good results, but we have not yet conducted environmental tests in the H_2S atmosphere.

Another material we have been evaluating is YF_3, which is a promising low-index material for the IR. It is very easy to e-beam and appears to be moisture-resistant in our tests. We are currently evaluating protected Ag coatings using YF_3 and an oxide (HfO_2, Zr-Ti oxide or Zr-Ta oxide). These coatings have reflectance R > 90% for λ > 340 nm, R > 95.5% for λ > 400 nm, and R ~ 98.5% at 2 μm. They show the performance needed for next-generation telescopes, but their durability is still to be determined.

3. AR COATINGS

For the Keck Cassegrain ADC[3], we developed an efficient broad-band AR coating consisting of a base layer of MgF_2, followed by a thin bond layer of Al_2O_3, and a final top layer silica sol-gel hardened in an ammonia atmosphere. We usually refer to this as the "three-layer" coating. We have continued to explore these coatings with different fluoride and oxide materials.

Sol-gel is usually applied to round optics by spin coating, and the actual application is quite straight forward. We always start with coating a calibration sample, and from interference patterns can easily determine the optical thickness. It is extremely easy to "zero-in" on the correct rotation rate to deposit the desired thickness. However, a pure sol-gel coating is extremely fragile, so we always "harden" the sol-gel in a moist ammonia atmosphere for about 40 hours. The resulting sol-gel layer easily withstands gentle wiping without sleeking. During the hardening process, the nano-spheres of silica fuse somewhat, leading to both a thinner and denser layer. There is always a degree of uncertainty about the magnitude of this effect on optical thickness; we have seen changes in the optical thickness of 8-to-22%. We speculate that this variation has to do with the concentration, and perhaps age, of the sol-gel; it does not seem to correlate with temperature and/or humidity. Also, some sol-gel coatings seem to emerge from the hardening process with a small increase in scattering, which is highly undesirable.

One concern with the three-layer coating is how to remove it should the hardened coating not have the desired properties. Sol-gel itself is traditionally removed by a weak solution of hydrofluoric acid, which breaks the bonds

between the silica nanospheres without seriously attacking the substrate. However, stripping the coating down through the MgF_2 is often problematic. Therefore, we have looked into other materials that could be easily removed. AlF_3 would be a good substitute for MgF_2 optically, but we found that the AlF_3 layer clouds during the hardening process. An alternative approach is to put a thin removable bond layer between the substrate and the MgF_2 (making it a four-layer coating); an obvious choice for this material is Y_2O_3. This four-layer coating appears to withstand high-temperature/high-humidity tests well, and it strips easily in hydrochloric acid within a few minutes.

As reported earlier[1], the three-layer coating withstands cryogenic temperatures. In fact, the protected vacuum environment of cryogenic IR instruments is ideal for these porous coatings. As a result, we have started testing actual AR coatings designed for the NIR. Our first result is shown in Figure 2, demonstrating that these simple coatings can produce good NIR performance.
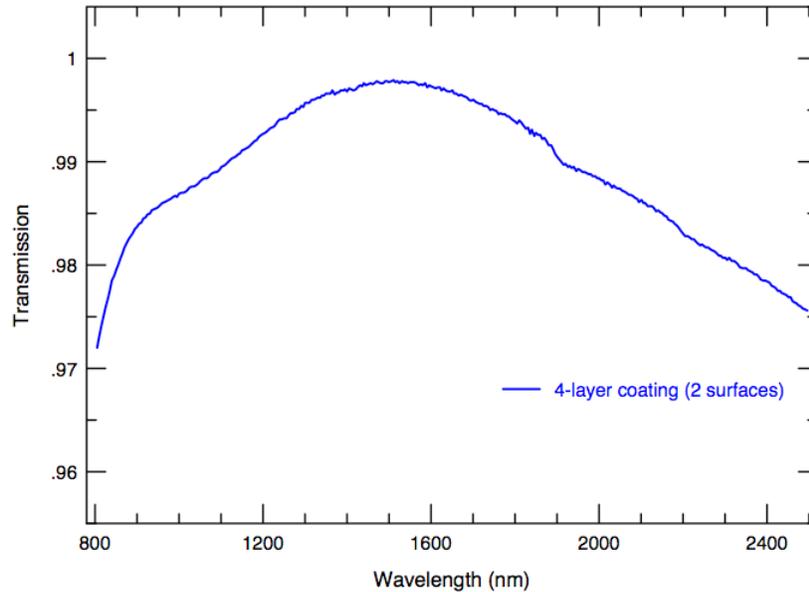

Figure 2. Broad-band AR coating for the NIR. This figure shows measured transmission through a silica witness sample where both sides were coated with the "four-layer" recipe. This coating uses silica sol-gel and MgF_2, plus two thin bond layers of Y_2O_3. The yttria dissolves in acid, so the coating is easily stripped.

4. MISCELLANEOUS

In collaboration with Christian Schwab, we have done some testing of a hydrophobic, oleophobic coating developed primarily for the ophthalmic industry. Such coatings show promise for improving abrasion resistance and cleanability, especially for coatings with a top layer of SiO2. This work is reported separately[4].

5. PLANS FOR FUTURE WORK

We plan major upgrades to our vacuum chamber to allow development in three areas: the vacuum system, the chamber geometry, and addition of magnetrons. We have just been awarded funding from the NSF ATI program, with additional funding already secured from TMT and the UCO Director to cover the upgrades. These upgrades will allow us to execute a series of experiments both to characterize new materials, and to develop and directly compare coating processes.

5.1 Improved vacuum system

We plan to replace our existing diffusion pumps with a large cryopump. The increased pumping speeds, particularly for water, will allow us to reach the $\sim 10^{-7}$ torr level necessary to produce good nitride layers. While nitrides can be deposited

with our current system, the pumpdown times needed to remove residual water are excessively long and our current films are probably oxynitrides.

5.2 Swing-arm stage

Coincident with the installation of the cryopump, we plan to install a "swing-arm" stage to carry our evaporation sources and ion-source. This technique was pioneered by Surface Optics Corporation (SOC), who built a radially-moving stage under a rotating substrate in order to cover large areas with uniform thin films[5]. The stage carries both e-gun and ion source, so that during IAD the substrate/e-gun/ion source geometry are all fixed, and both ion and adatom fluxes remain constant across the substrate. This technique is scaleable to arbitrarily large optics.

Our design replaces the linear stage with a stage that moves from center to edge by pivoting about a point near the outer wall of the chamber. This is shown schematically in Figure 3a,b. The advantage of this design is that all gas, cooling and electrical lines are fixed inside the vacuum; all flexing of lines takes place on the atmosphere side of the chamber. We expect that once this system is working it will be very stable to vacuum leaks and electrical cross-talk.

5.3 Magnetrons for Sputtering

The swing-arm stage will also be capable of carrying magnetrons, and we plan to install these following a period of e-beam IAD experiments. The swing-arm stage should allow us to coat large optics with relatively small magnetrons.

5.4 Coating experiments

The improved vacuum system will allow us to produce reliably good nitrides, and our immediate goal is to both replicate the Gemini-style coating[6,7] with silicon and NiCr (nichrome alloy) nitrides. We especially wish to explore whether other metal nitrides with better optical properties can take the place of NiCr nitride. If successful, we will test if oxynitrides (which tend to be more transmissive than pure nitrides) are also suitable; here we will need to determine how much oxide can be tolerated before durability is compromised.

We expect that calibration of the swing-arm for uniformity may present challenges. When operational, we should be able to coat optics such as the Lick 1-m primary without reconfiguring the chamber, and we will be able to deposit our most promising coatings (developed on small samples) onto optics that will immediately go into operation for actual field testing. The risk that a new coating unexpectedly fails over a short timescale is ameliorated by the fact that it can be quickly replaced if necessary.

After the installation of the magnetrons, we plan to reproduce the most promising coatings developed with e-beam IAD, allowing us to directly compare the two deposition processes. We believe this is an essential test of whether one or the other process produces superior films for our use. We will be in an excellent position to perform this comparison, as the chamber "environment" will be identical between the two processes.
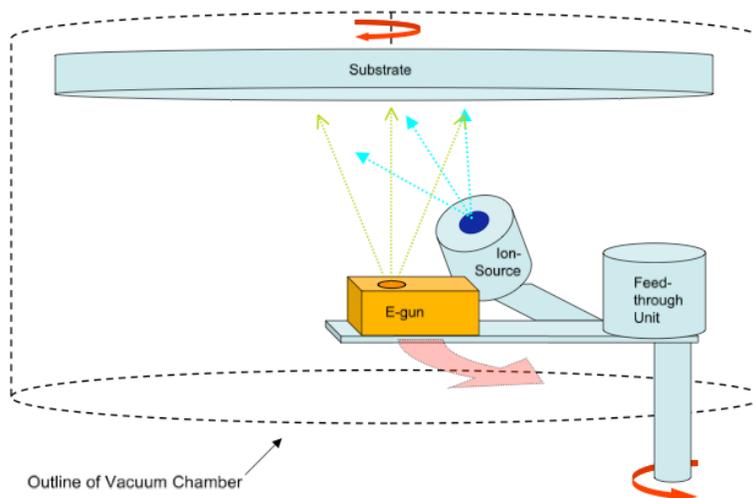

Figure 3a. A side-view schematic of the vacuum chamber with swing-arm. The rotating substrate is downward facing. The stream of evaporated atoms or molecules from the e-gun (green), mixed with high-energy ions from

the broad-beam ion source (cyan), strikes the substrate and adatoms are deposited. As the swing-arm slowly pivots near the edge of the chamber, the e-gun and ion source are slowly moved radially and the coating is "painted" across the entire surface of the rotating substrate. The rate of radial motion is variable and must be calibrated to maintain thickness uniformity in the deposited layer. All service lines (electrical, gas and cooling) pass into the chamber through the rotating shaft of the swing-arm, which is supported by a ferro-fluidic feed-through on the chamber floor. The shaft terminates in a "feed-though unit" which rotates as part of the swing-arm. The inside of the feed-through unit is at atmospheric pressure; the actual vacuum feed-throughs for all lines are located in this feed-though unit. This means that all flexible lines are located outside the vacuum, and the geometry of all components (e-gun, ion source, vacuum feed-throughs and lines) within the vacuum are fixed at all radii on the substrate. The fixed configuration of e-gun, ion source and substrate height permits coating process uniformity for all locations on the substrate; the mechanically fixed lines means the system should be robust to vacuum leaks. (Note that shutter, thickness monitors, baffles and service lines are not shown in this schematic.) During the later phases of the upgrade, the e-gun will be replaced by a bank of magnetrons.
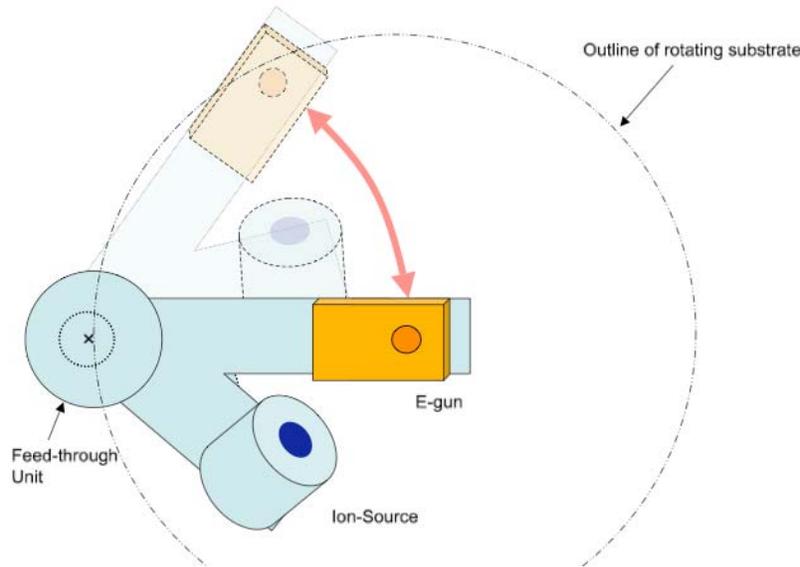

Figure 3b. Top-view schematic of the swing-arm assembly. By moving the swing-arm though relatively small angles (<60-80°) around its pivot point (marked ×), the e-gun/ion-source may be positioned at all points under the rotating substrate.

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