Coating the 8-m Gemini telescopes with protected silver

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ABSTRACT

The Gemini telescopes were designed to be infrared-optimized. Among the features specified for optimal performance is the use of silver-based coatings on the mirrors. The feasibility study contracted by Gemini in 1994-1995 provided both techniques and recipes to apply these high-reflectivity and low-emissivity films. All this effort is now being implemented in our coating plants. At the time of the study, sputtering experiments showed that a reflectivity of 99.1% at 10μm was achievable. We have now produced bare and protected silver sputtered films in our coating plants and conducted environmental testing, both accelerated and in real-life conditions, to assess the durability. We have also already applied, for the first time ever, protected-silver coatings on the main optical elements (M1, M2 and M3) of an 8-m telescope. We report here the progress to date, the performance of the films, and our long-term plans for mirror coatings and maintenance.

Keywords: silver coating, sputtering, high-reflectivity, low-emissivity, durability

1. INTRODUCTION

1.1 Coatings in astronomy and use of silver

Since Strong perfected the aluminum evaporation technique in the 1930s, it has been the standard coating solution[1] for large astronomical mirrors. Only in the 1990's did projects like Gemini and VLT decide to move to magnetron sputtering. Silver is the metal having the highest reflectivity for wavelength > 400nm, and some attempts[2] of producing, by physical vapor deposition, durable silver-based films for astronomy started in the 1980s. For near- and especially mid-infrared optimization, low emissivity is a key factor to increase telescope sensitivity. Besides obvious telescope design considerations (optical stop, obstructions, etc…), low emissivity (ε) is obtained by the use of high reflectivity (R) coatings. Figure 1 recalls data[3] obtained for freshly evaporated films. At 10μm, reflectivity is respectively 99.5% and 98.7% for Ag and Al, so if we assume that ε = 1-R, we find that Ag has an emissivity 0.38 times that of Al.

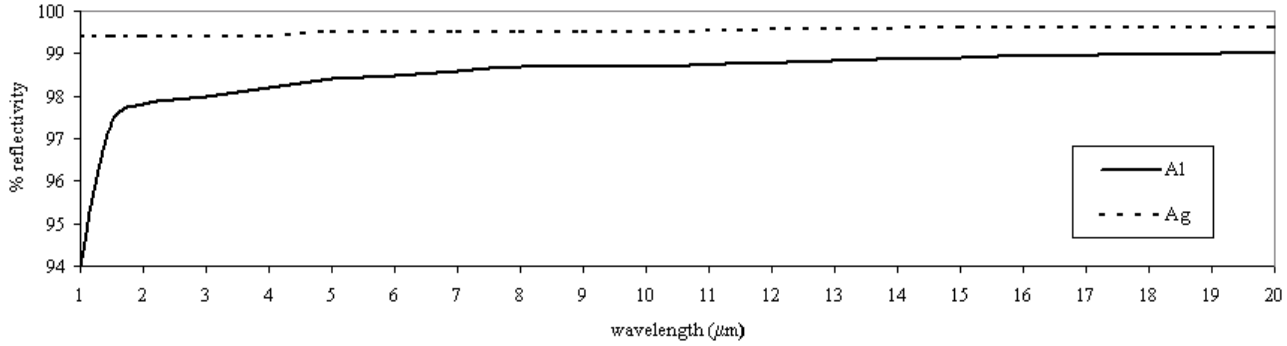

Figure 1: comparison of evaporated silver and aluminum reflectivity

In 1992, Gemini contracted an initial study[4] that reviewed tarnishing mechanisms and identified multi-layer recipes and sputtering as the most appropriate techniques to deposit durable silver films on large optics. In 1998, a progress report[5] summarizing the feasibility study and demonstration phase was published.

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More recently, progress has been reported on durable silver-based coatings[6-7] but no large mirror had yet been coated with protected silver. In this paper, we report on the performance of the protected silver coatings applied on the 1m-secondary (M2) and 8m-primary (M1) mirrors of our southern telescope applied respectively in March and May 2004 for the first time.

1.2 Gemini's science requirements for coatings

The Observatory science requirement document (1996 version) details the performance expected for coatings both in terms of reflectivity and emissivity. The visible reflectivity of freshly coated surfaces shall be:

	0.3 - 0.4 μm	**0.4 - 0.7 μm**	**0.7 - 1.1 μm**
Requirement	0.88	0.88	0.84
Goal	0.92	0.98	0.98

For infrared wavelengths > 2.2 μm, the throughput at the Ritchey-Chretien (RC) focus is calculated as the fraction of photons transmitted by the IR configuration (including stop size, M1 central obstruction, …) compared to that transmitted by an 8.00 m diameter telescope with no obscuration and with perfectly reflecting mirror surfaces:

$$\text{Throughput} = (1-\varepsilon) = 0.91 \text{ (0.93 goal)}$$

The fully-optimized IR configuration will have a telescope emissivity, including scattering and diffraction, of 4% with a goal of 2% immediately after coating or recoating optics, with 0.5% maximum degradation during operations, at any single wavelength beyond 2.2μm. The later requirement is very stringent and will determine our strategies for coating maintenance. We are acquiring a handheld 2.2μm reflectometer to monitor this requirement.

1.3 Gemini's feasibility and demonstration study for low-emissivity and durable coatings

The final report of this study[8] from Optical Data Associates (ODA) was delivered in 1995 and the main conclusions are summarized here. ODA selected two subcontractors for the demonstration phase: Airco Coating Technologies (ACT) produced silicon nitride (SiN_x) protected films whereas Deposition Sciences Inc. (DSI) made hafnium oxide (HfO_x) protected films using a microwave energy supported plasma. Both technologies and recipes consistently reached the same reflectivity $R_{10\mu m} = 99.1\%$ in production but the SiN_x protection proved to be slightly superior for durability test to tarnishing environment. Because the deposition of the SiN_x film was also made under classical sputtering, it is the path that Gemini selected for specifying the coating plant hardware.

It is worth noting that although the $\varepsilon_{2.2\mu m} = 2\%$ goal is achievable according to evaporated Ag results, the reference wavelength chosen for the feasibility study was 10μm and the demonstration phase reached 'only' $R_{2.2\mu m} = 98.6\%$ (i.e. $\varepsilon_{2.2\mu m} = 1.4\%$ per surface).

A very important phase of the study was obtaining accurate reflectivity measurements in order to reach the goal of $R_{10\mu m} = 99.2\%$. ODA and its subcontractors used similar and/or directly comparable spectrophotometers: for the UV-Vis-NIR range, absolute instruments like the Hitachi 4001 and Cary 5E in V-W configuration which yield the square of R without comparison to a reference mirror; and relative device like the PE 983G for the MIR range where the measurements were compared to NIST standards (Al and Au) analyzed with the Cary and with a 10.6μm absolute reflectometer (using a CO_2 laser source) built by Helios Inc. The accuracy obtained at 10μm was ±0.01%. Other consistent measurements were also performed at NOAO with an emissometer (called 'blue toad') working at $\lambda = 4\mu m$.

The last critical phase of the study was the environmental testing of the samples to assess their durability. Four different tests were performed: weathering (cycle through high temperature and humidity), delamination (scotch tape pull), abrasion and tarnishing (exposure to hydrogen sulfide fog). However, no real-life exposure tests were conducted at an observatory.

The final optimal coating recipe was a stack with the following layers:

Material	**Function**	**Thickness**
SiN_x	protector	15nm
$NiCrN_x$	adhesor	0.8nm
Ag	reflector	200nm
$NiCrN_x$	adhesor	5nm
substrate		

To avoid the absorption caused by the SiN_x layer at wavelength < 500nm, an alternative 'minimal' design omitted the top protective layer and proved to have promising durability (passed adhesion, T/RH and salt fog tests but was scratched under abrasion testing; no H_2S test done).

2. HARDWARE CONFIGURATION

2.1 Vacuum system

During the construction phase of the Gemini Observatory, the coating plant package was contracted to Royal Greenwich Observatory. The first system was delivered to Gemini North (GN) in 1998 and the second one to Gemini South (GS) in 2000. The vacuum vessel is a $150m^3$ stainless-steel chamber, formed by two parabolic-like shells with an overall size approximately 9m in diameter and 6m high. Sputtering magnetrons are mounted on several radial support structures attached to the upper vessel while the mirror resting on a whiffle tree rotates underneath facing up. The high-vacuum pumping is accomplished by two Leybold RPK30,000 cryopumps, each equipped with one single stage cold head and two dual-stage cold heads reaching 8-10K in operation. The cryopumps are located behind VAT gate valves on a side wall of the upper vessel, which is not the ideal central location to provide vacuum uniformity in such a large tank. The magnetron nearest to the cryopumps, and thus with an expected slightly better vacuum, is usually operated with the reflective material target to optimize film purity. Two additional single-stage cold heads are used as water traps in the upper half of the vessel. After a long period of troubleshooting various air and water leaks, the vessel is now capable of reaching the high vacuum threshold point ($5x10^{-3}$ Torr) typically 1h20min after the start of the roughing pumps and low 10^{-6} Torr after another 6h of run time. The cold environment of the mountain top has caused several operational and maintenance difficulties for the vacuum equipment.

2.2 Magnetron sputtering

A first generation of planar DC magnetrons, built by Gencoa Ltd., was used to make the first Aluminum coating of M1 in 1999. Because of operational difficulties, commissioning with these magnetrons was never fully completed. Another vendor, Teer Coatings Ltd, was contracted to build two new magnetrons. With these new units and marginally higher reliability, the GN and GS primary mirrors were successively coated with Aluminum at their respective sites in April and November 2000. At this time RGO's involvement was terminated due to the closing of this institution. In mid-2001, an internal development program was started. This effort aimed at improving the reliability of the coating plants and installing upgrades for the deposition of low-emissivity silver-based coatings. During that effort, we first repaired the Teer units to improve reliability (considerable work was needed on cathode, dark space, electrical insulation, seals, etc… in order to prevent electrical arcing). We then purchased two new units from Angstrom Sciences Inc., and finally refurbished two Gencoa units. The result was a vacuum plant at each site with a family of three permanently mounted magnetrons capable of producing multi-layer coatings.

These magnetrons have balanced magnetic fields and are of the direct-cooling type with an effective target length of 1.15m and width varying between 0.15 and 0.25m. The power requirement was originally set to operate Aluminum targets at 40kW in order to obtain the required thickness, typically 800Å, in a reasonable amount of time (target to glass distance is typically 110mm). The power module consists of a stack of three Advanced Energy 20kW Pinnacle supplies in master/slave configuration. Because the radius of glass to cover on M1 is 3.5m, the coating is done as three concentric rings by moving the magnetron radially. At a given radial position, the mirror is rotated over a complete revolution before going to the radial position to make the next ring. A specific rotation speed is calculated for each ring in order to maintain uniform film thickness (on average, it takes about 1h to coat one layer on M1). In addition, a thickness uniformity mask, consisting of two stepper motor driven blades, acts as a variable pie-shaped aperture. The aperture is placed below the deposition target in order to compensate for the radial variation in linear speed of the magnetron above the substrate. The proper combinations of speed and mask aperture for each ring are calculated geometrically and confirmed experimentally. The thickness uniformity requirement is +/-5% (that is +/-1nm for a substrate polished to a surface figure of 20nm RMS). Our measurements with quartz crystal sensors (repeatability of 1Å) located at various locations along the target length and radius to be coated indicate that we meet this requirement. An open/close pneumatic shutter is activated between the target and the mask in order to define precisely the coated areas on the substrate. At the joints between rings and where the shutter operates, we have localized thickness defects that we estimate to about 25% of total thickness over areas of 15mm in width. Both shutter and mask are also internally cooled with water to prevent thermal deformation.

For deposition of the SiN_x dielectric layer, we acquired an Advanced Energy Starburst 20KHz pulser which we use in Self-Run mode (continuous pulsing). We have also had good results with Self-Run mode for Ag and Active-Arc mode

(pulses when detection of arcs) for NiCr. All the motions and controls are made through software written under LabView. When applying our standard recipe of four layers (50Å NiCr / 1300Å Ag / 6Å NiCr / 80Å SiN_x), we use the following parameters for the outer ring:

	Adhesion layer	**Reflective layer**	**(adhesion) layer**	**Protective layer**
Target and Gas	NiCr (80/20) and N	Ag and Ar	NiCr and N	Si (B-doped) and N
Power (kW)	7	7	1	7
Pressure (mTorr)	1.6	3.2	1.6	3.3
Rotation speed (rph)	4	1.6	6.9	1.5

The milestones of our multi-layer development are summarized in the following table (R data taken with the Iris 908RS reflectometer) and performance will be detailed in the next section.

Sample	**Date**	**R @ 530nm**
First bare Silver (1120Å Ag)	25-Sept-02	98.8%
First 3-layer Silver-based (50Å NiCr / 1400Å Ag / 8Å NiCr)	16-Oct-02	93.4%
First 4-layer Silver-based (50Å NiCr / 1400Å Ag / 6Å NiCr / 75Å Si)	12-Aug-03	93.8%

The 3-layer recipe was first deposited on the GS M2 in October 2003, and recently in March 2004 we recoated that same mirror with the final 4-layer recipe. The GS M1 and M3 were coated with the 4-layer recipe in May 2004.

3. REFLECTIVITY AND EMISSIVITY DATA

3.1 Measurement devices

We have been using a combination of the following instruments:

- Iris 908RS scattero-reflectometer at Gemini : this is a handheld unit made by DMO. It measures the reflectivity at 470, 530, 650 and 880nm (with LEDs) over an area of Ø10mm. Repeatability and accuracy claimed by the vendor are respectively 0.1% and 0.6% and in agreement with our observation in the practice. The reflectometer has its own calibration gauge (a protected Aluminum mirror) which is a stable absolute reference (typically within 1% of the data obtained with the Cary) and allows excellent relative calibration over time (thus precise determination of loss rate for example). Reflectivity quoted is for normal incidence. The scatterometer measures the BRDF at 3 angles at 670nm (with a laser diode).
- Cary 500 spectrophotometer (in VW absolute mode) at Gemini (0.3-3µm).
- Emissivity Measuring Unit (EMU) at 3.8µm at Gemini: this is an emissometer similar to the one used at NOAO prior and during the demonstration phase in 1995. It is an InSb detector cooled with liquid nitrogen and fitted with a filter wheel (open, blank and 3.8µm L-filter). A LN_2-cooled gold mirror and a black-painted metal sample at ambient temperature are successively located on the dewar window and serve respectively as the white body ($\varepsilon_0 = 0$) and black body ($\varepsilon_1 = 1$) references. Next the sample is measured (x) and its emissivity is simply:

$$\varepsilon = (x - \varepsilon_0) / (\varepsilon_1 - \varepsilon_0)$$

- Cary 5E (VW absolute mode) and PE983G at ODA for complete reflectivity coverage over 0.3-3µm and 2-56µm respectively. The PE983G sample compartment is not purged so the data show strong atmospheric absorption features (no attempt to smooth them out in our figures). ODA uses a NPL standard to calibrate the PE983G with the Cary 5E.

3.2 Reflectivity results

Figures 2a, 2b and 2c show data obtained at ODA between 0.3 and 20µm comparing samples coated with Al, bare Ag and protected Ag. The SiN_x layer is transparent over the IR wavelength range (1.5 to 20µm) but causes increased absorption toward bluer wavelengths (3% at 500nm and 8% at 400nm). This absorption is constant for thickness from 50 to 100Å but for a thicker layer of 230Å, the absorption increases another 5% between 330 and 500nm. In the reflectivity optimization of the Ag layer, we have not seen any clear trend with base vacuum quality (monitored with RGA) or throw distance (distance target to substrate) but rather with power levels (variations of 0.8% in the visible over 2-10kW). For

reference, we plotted data obtained by ACT that shows that we might still have some margin to improve. The overlap region (2-3µm) between Cary and PE983G measurements verifies the absolute calibration.

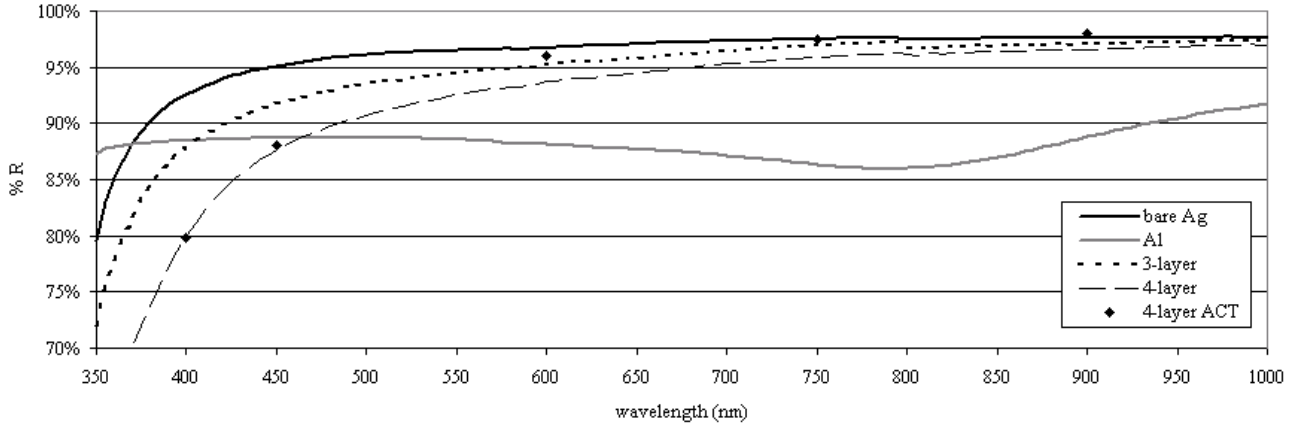

Figure 2a: comparison of Al, bare Ag and protected Ag in the visible (data with Cary 5 at ODA)

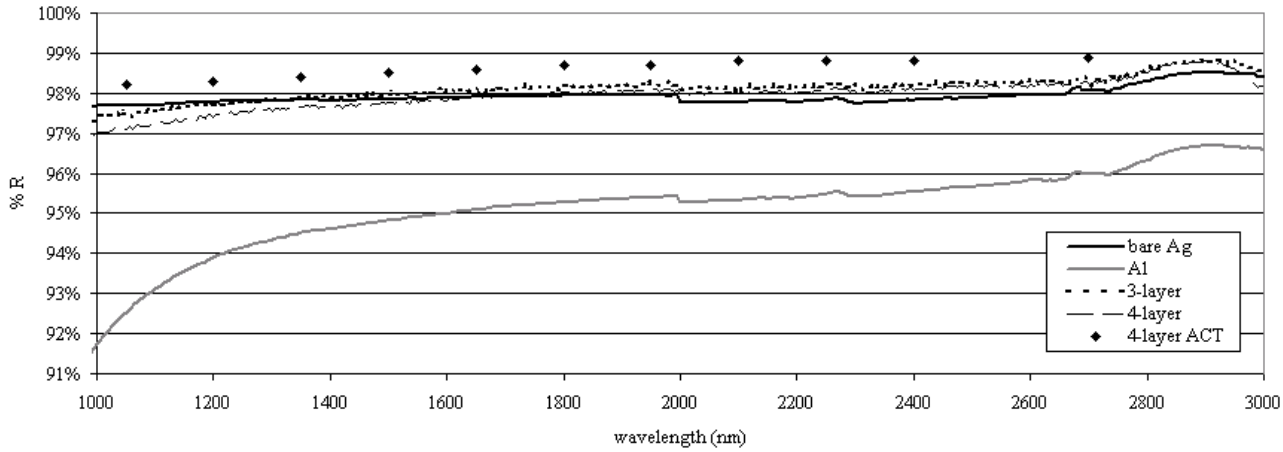

Figure 2b: comparison of Al, bare Ag and protected Ag in the NIR (data with Cary 5 at ODA)

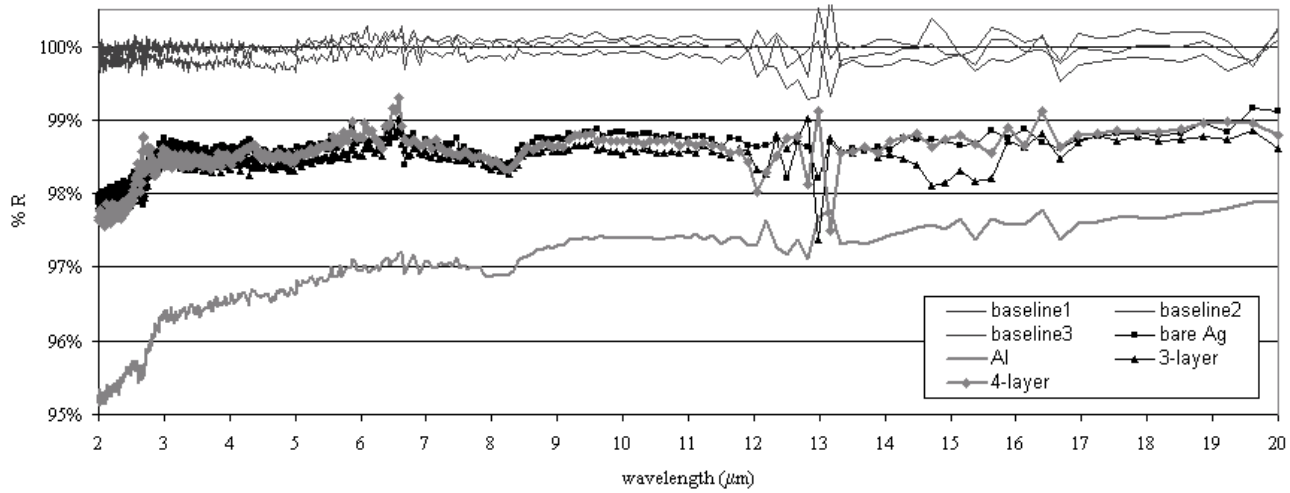

Figure 2c: comparison of Al, bare Ag and protected Ag in the MIR (data with PE983 at ODA)

We found that the $R_{10\mu m}$ values obtained are inferior to the ones mentioned previously for fresh evaporation (-0.7% and -1.3% respectively for Ag and Al), and also to the ones obtained in the demonstration phase (-0.4% for protected Ag). More than a limitation of our setup, this is likely due to film purity and micro-structure, and we think that better base vacuum and an optimal combination of parameters (throw distance, power, etc…) should lead to improved performance in future testing.

Figure 3 shows the influence of the NiCr layer thickness on top of Ag: while maintaining a minimum thickness for adhesion purposes, it clearly needs to be as thin as possible to limit the absorption in the visible. We usually apply 5 to 6Å. The +/-1Å repeatability makes difficult the precise control of blue reflectivity and we typically see R_{470nm} oscillates between 90 and 93%.

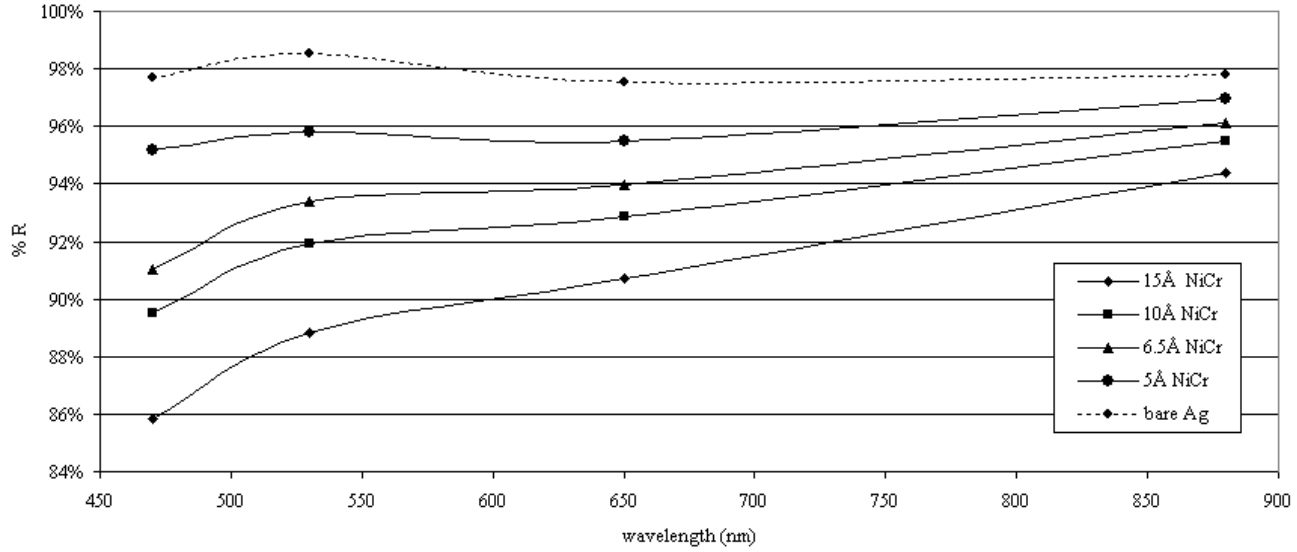

Figure 3: absorption of the NiCr layer on top of the Ag layer

3.3 Emissivity results

For reference samples, we use mirrors coated at NOAO in 1992 and measured between 1992 and 1995 with the 'blue toad' emissometer. By re-measuring them with our EMU, we are able to compare our samples directly to the ones obtained in the demonstration phase and to known standards. The following table compares the results and indicates the difference between instruments (also likely due to age of sample), giving an idea of the error bar of the current absolute measurements. Overall, we estimate that the error bar of our emissivity measurements is ±20%.

	Dec93 and Jun94		**Aug03 and Oct03**		
	average ε	st.dev.	average ε	st.dev.	difference
Ag+Al2O3	3.14	0.06	3.55	0.01	-12%
Ag+ThF4	0.79	0.04	0.54	0.06	37%
Ag+Y2O3	0.77	0.01	0.80	0.18	-4%
Al	2.00	0.07	2.02	0.07	-1%
Au	1.06	0.03	0.96	0.05	10%

We have measured Al, bare Ag and protected-Ag samples, either just after coating or after some exposure. We have also been able to measure the emissivity of M1 and M2 in the telescope.

Sample	$\varepsilon_{3.8\mu m}$
Al 16-month old (lab)	2.60
Al 24-month old (M2)	2.60
Al 22-month old (M1, washed once)	7.80
Ag fresh	0.63

Ag 10-month old (lab)	0.88
Ag 12-month old (down-looking)	0.90
Ag 10-month old (up-looking, very tarnished)	3.19
Ag 12-month old (up-looking and washed regularly)	1.25
3-layer fresh	0.88
3-layer exposed 7 months (up-looking)	2.68
4-layer fresh (sample and M1)	1.0 and 1.3
4-layer exposed 2 months (up-looking)	1.52
4-layer ACT	1.00

From the PE983G reflectivity data, we had $R_{3.8\mu m} = 98.5\%$ for the bare and protected-Ag (3 and 4 layers), so there is a slight mismatch between reflectivity and emissivity measurement. Our samples have nevertheless achieved the same emissivity as the demonstration phase samples. We also deduce an emissivity increase of up to 0.25%/month for the up-looking samples (no cleaning). Overall, with the current 4-layer Ag coatings on both M1 and M2, we are close to achieve the goal of $\varepsilon_{telescope} = 2\%$ at 3.8μm (and based on reflectivity measurements and $\varepsilon = 1-R$, we will achieve $\varepsilon_{telescope} = 4\%$ at 2.2μm).

Emissivity measurements are also taken directly through observation with NIR and MIR instruments on the telescope at night. The ε_0 and ε_1 references are usually measured by imaging the center of the cold Lyot stop and the black instrument cover at ambient temperature, respectively. With the telescope looking at the sky, one can measure the pupil emissivity (sum of sky, telescope and instrument) and also estimate the sky emissivity (either by subtracting measurements at two different airmasses or by measuring directly through the M2 central hole if there is a pupil viewing mode in the instrument), therefore allowing to deduce the telescope (mirror coatings mainly) contribution to the total emissivity (7% obtained in the best cases including sky, instrument and telescope). So far, we have measured the following telescope emissivity with several instruments:

Old Al on M1, clean Al on M2 and clean protected Ag on M3	8.0%	11 February 03	GN, NIRI, 4μm
Old Al on M1 and clean Al on M2	6.5%	23 February 03	GN, Michelle, 12 μm
Clean Al coatings on both M1 and M2	3.5%	13 July 03	GS, T-ReCS, 10μm
Clean Al on M1 and 3-layer on M2	3.0%	12 October 03	GS, T-ReCS, 10μm
Clean Al on M1 and 4-layer on M2	2.5%	27 April 04	GS, T-ReCS, 10μm

The small gain seen with protected Ag coatings on M2 is expected. After all mirrors are silver-coated, the limitation will be the instrument (about 1-2% entrance window emissivity) and the sky (typically 1-2% for a clear dry night at Cerro Pachon).

4. DURABILITY TESTING AND MAINTENANCE

4.1 'Real-life' test setup

In parallel with the coating development, we have been conducting an intensive durability campaign with tens of samples exposed in different places at both sites. Most of the samples are 30x30cm and coated in pairs: one is immediately exposed in the dome and the other is kept in a closed wooden box (not special sealing) inside the building. The exposed samples are located (see figure 4) on the elevation platforms of the telescope mount, at the same level as M1, under a small roof (some 15cm above the sample) that prevents particulates from falling straight down onto the sample, but allows air to flow across the sample from the 4 sides (especially when the lateral vent gates of the dome are opened at night). Unless otherwise noted, all the durability tests described were conducted at Cerro Pachon in Chile.

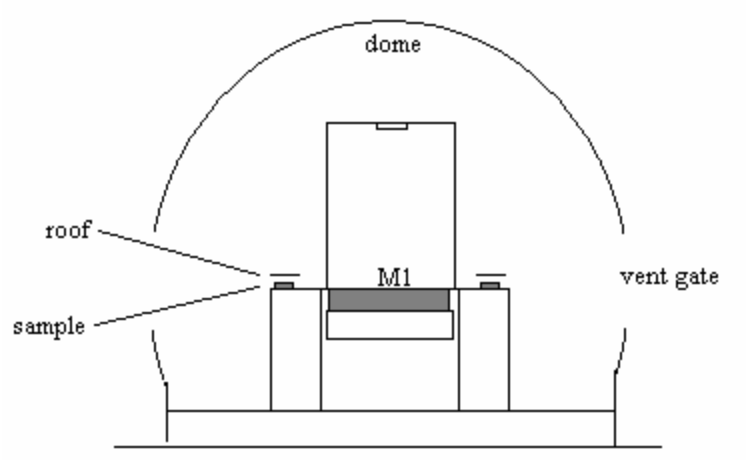

Figure 4: exposure setup for coated samples

This partial exposure setup attempts to simulate the real exposure of M1: fully covered during the day and fully exposed during the night. Except in one case further described, the samples were not cleaned during the aging exposure so they obviously collected dust. We used an Al witness sample, exposed the same way as the family of Ag-based samples (under the small roof), to determine the reflectivity loss due to the dust only. Aluminum is protected by the Al_2O_3 layer that naturally forms on it and doesn't degrade because of corrosive effects. It degrades when the dust sticks so much to it that it becomes embedded into the film and cannot be cleaned. In order to confirm this fact, we washed the Al dust reference plate 7 months after starting the exposure and recovered the fresh coating values to within 0.2% (figure 5).

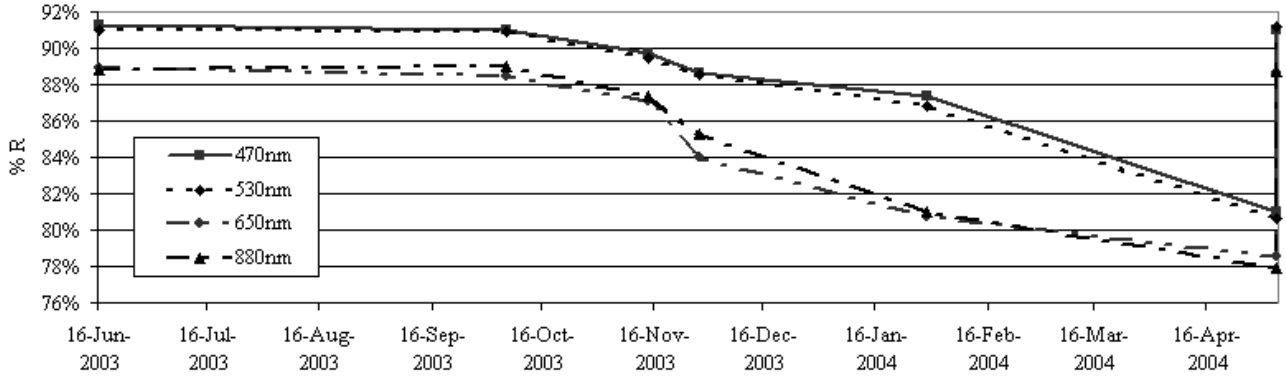

Figure 5: reflectivity loss on Aluminum dust reference sample (exposure started in October)

This experiment indicates a 10% visible reflectivity loss in 7 months (1.4%/month), which is significantly worse than the 0.35%/month that we see on our GS M1 (data available on internal Gemini web site, average of several periods between coatings and washings over 4 years; at GN the average on M1 is 0.17%/month). Therefore our exposure setup provides a harsher environment than that seen by telescope mirrors in routine use. In the next section, if one wants to assess with the graphs the reflectivity loss of the Ag-based samples due to corrosive effects only, the effect of the dust previously calculated should be corrected (subtracted) unless noted. We do not present systematic reflectivity loss data in the NIR since our Cary 500 was not available to make regular reflectivity measurements until late in the aging program.

4.2 Reflectivity loss and tarnishing

4.2.1 Reference samples

It is well known[9] that a tarnish film forms on freshly deposited silver when exposed to atmosphere. The tarnish film is silver sulfide (Ag_2S), a semiconductor that is black in the visible region but has an absorption edge at about 1.4μm and thus is transparent in the IR. Sulfides are by far the most damaging environmental constituent for Ag coatings. This

motivated us to survey the ambient telescope environment and remove several sources of sulfides (batteries outgassing hydrogen sulfide, generator exhaust, etc…) whereas other products like lubricants are still being investigated.

Our first important observation is that the Ag coating samples, protected or not, kept in a box, in an office, for up to 20 months, do not undergo any cosmetic deterioration! The bare Ag samples show a visible R loss of 1.1% on average after the first 20 days, but thereafter their reflectivity is very stable (within the 0.6% accuracy of the reflectometer). The protected Ag samples exhibit no loss at all.

Another bare-Ag sample was kept in the same office but out of the box, and facing down to avoid dust accumulation. Curiously, this sample took a yellowish tint. There are several yellow Ag compounds like Ag_2CO_3, $AgNO_2$ but we have yet not identified which one is formed (it might also be an early form of Ag_2S tarnishing).

Clearly, the only environmental difference in this case is the exposure to fluorescent lights of the office. We conclude that the reaction is induced by light, a fact consistent with the photosensitivity of many Ag compounds used in photographic processes. Previous studies[10] have reported on this deterioration mechanism. This observation is confirmed on the bare-Ag samples exposed facing downward in the dome (also illuminated all day with fluorescent tubes) that have also taken a yellow tint at the 8th month of the exposure. We have no evidence of yellow color on 3- and 4-layer samples under the roof.

4.2.2 Bare Ag durability: dependence on orientation and gain by washing

The second observation we present concerns the durability of three sets of bare-Ag samples (all exposed in the same area under the small roof):

- Figure 6 shows the reflectivity of two samples facing up (like M1), one with a regular wash and one with no wash. We see the chromatic effect of tarnishing (faster at bluer wavelength) and the lower loss rate due to washing: 4x less at 880nm and 2x less at 470nm. Although the regular washing helps to retard tarnishing, it is not sufficient to maintain the reflectivity of an Ag-coated mirror above 90% for more than about 6 months. As for cosmetic degradation, the washed sample only presented a yellow coloration with few brown tarnish spots, whereas the non-washed sample was nearly fully covered with large tarnished spots (very faint brown spots are already seen 2 months after the beginning of the exposure). Even under weak magnification (5-15x), one can easily appreciate how the tarnish grows along the exposure lifetime: round brown spots form first around the largest pinholes and eventually around all imperfections in the coating covering nearly the whole surface. Meanwhile, the spots around the largest pinholes become thinner and end up being transparent, i.e. with no coating left (the worst case we have seen is a Ø4mm hole grown after 550 days). At 470nm, the bare Ag loss rate is 4.73%/month (dust excluded).

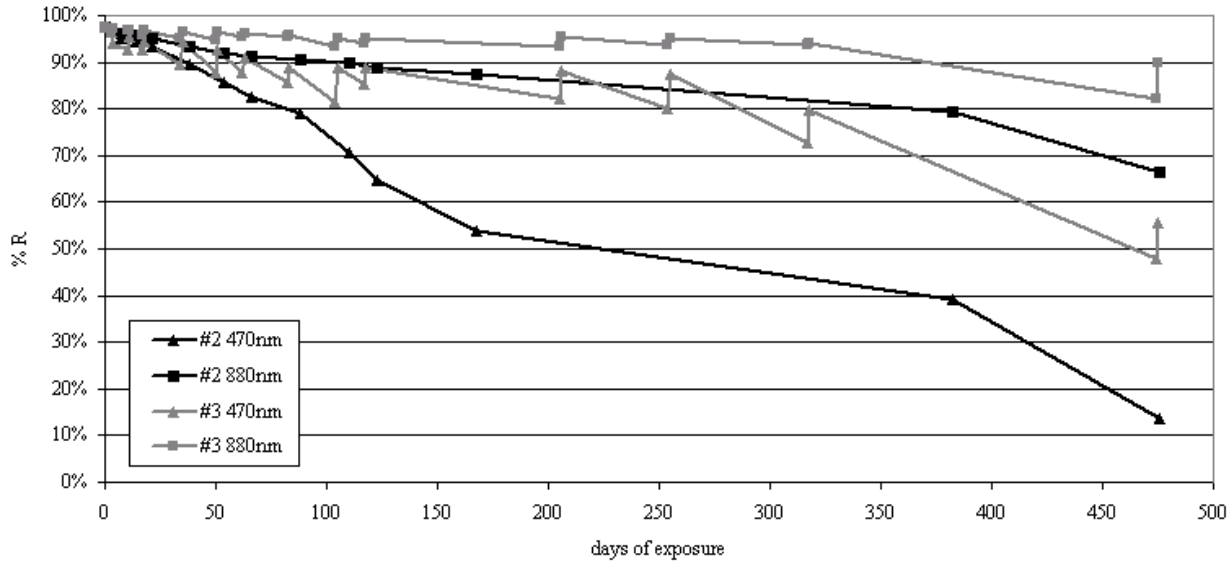

Figure 6: samples facing up, effect of washing a bare Ag sample (#3) vs. non-washing (#2) at 2 wavelengths

- Figure 7 shows a downward-facing sample (like M2). The sample was originally facing up. On exposure day 67, it was washed and from then exposed facing down. In this orientation there is virtually no deposition of dust on the mirror. Looking at the slopes of the curves, we notice the significant change in the reflectivity loss rate between both orientations. After a year, a bare Ag-coated M2 would still have reasonable reflectivity in the visible (4% loss at 470nm and 0.5% loss at 880nm). Cosmetically, the sample is barely yellowish (mostly near the edges where it was supported) and almost no tarnish spots. Another sample that was exposed facing down from the beginning also slowly ended up degrading (yellowish color and R loss).

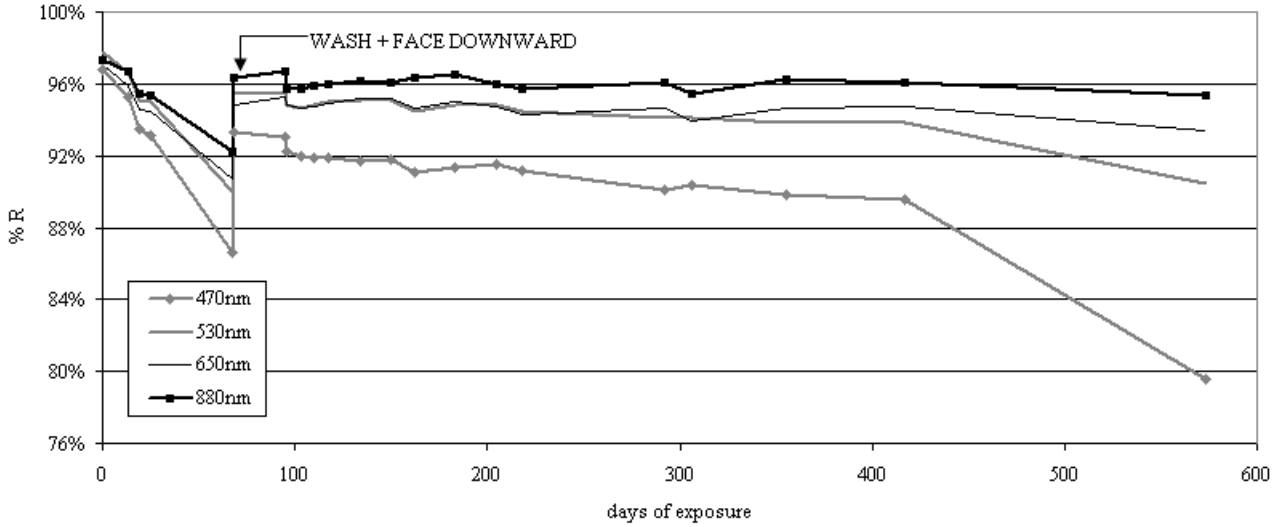

Figure 7: reflectivity loss of a sample facing down

From the observation of these bare-Ag coatings, we learned of another important mechanism influencing the tarnishing process. Since exposed but dust-free samples do not present brown spots, the main source for tarnishing is clearly the dust that collects on the mirror. The deposited particles are what bring sulfide contaminants onto the film. The upward looking samples collecting dust are also likely less protected against condensation than downward looking samples. So it is the combination of sulfur compound and water that starts the tarnishing (exposure to sulfides in dry atmosphere doesn't create tarnishing). We did not conduct chemical analysis of the soil surrounding the observatory to understand the composition of the dust as this was far too ambitious, and we cannot change our site and environment.

4.2.3 Protected-Ag durability

The third observation concerns upward-facing protected Ag samples. We experimented with a bare Ag sample that we put back into vacuum, 3 months after depositing the Ag film, and coated directly with SiN_x, and later compared with the unprotected Ag sample. The cosmetic difference between the samples is striking. The SiN_x-coated sample shows slight yellowing and with very few tarnish spots. In contrast, the unprotected-Ag sample shows significant yellowing and advanced tarnishing. The SiN_x coating has decreased the reflectivity loss rate significantly (down by a factor of 1.6x at 470nm, 1.3x at 530nm, 1.2x at 650nm and no effect at 880nm). At 470nm, the SiN_x protection yields a loss rate of 2.28%/month (dust excluded).

Figure 8 shows the relative durability of 3-layer coatings due to variations in the NiCr top layer thickness (6.5Å, 10Å and 15Å). The exposure lasted 411 days and the samples were washed before final measurement. Cosmetically, they all have very few small tarnish centers. But clearly, thicker top layer maintained better the initial reflectivity. Curiously, the R loss is less at bluer wavelengths. For the thickest NiCr layer sample, the data actually show a slight blue reflectivity gain which might not be real but is still presented because the values are outside our typical error bar and the data set is quite consistent. At 470nm, the NiCr protection (standard thickness of 6Å) loss rate is 0.28%/month (dust excluded).

These results would tend to indicate that a 3-layer Ag coating has sufficient durability, as outlined in the feasibility study. However, as our negative experience with M2 describes below illustrates, our on-telescope experience has proven otherwise.

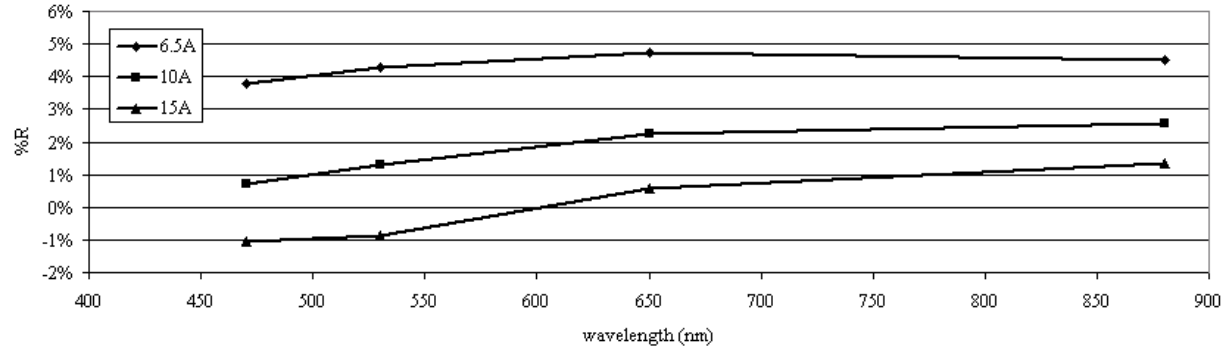

Figure 8: total R loss after 411 days of exposure of 3-layer samples

Figure 9 shows that after 260 days, after washing the sample, our first ever 4-layer sample exhibits absolutely no loss in visible reflectivity. Cosmetics are truly excellent. The reflectivity loss rate (at 470nm) during the test is exactly 1.4% just like our Al dust reference plate. The data shows weak chromaticity, the redder being more affected, maybe due to particle size.

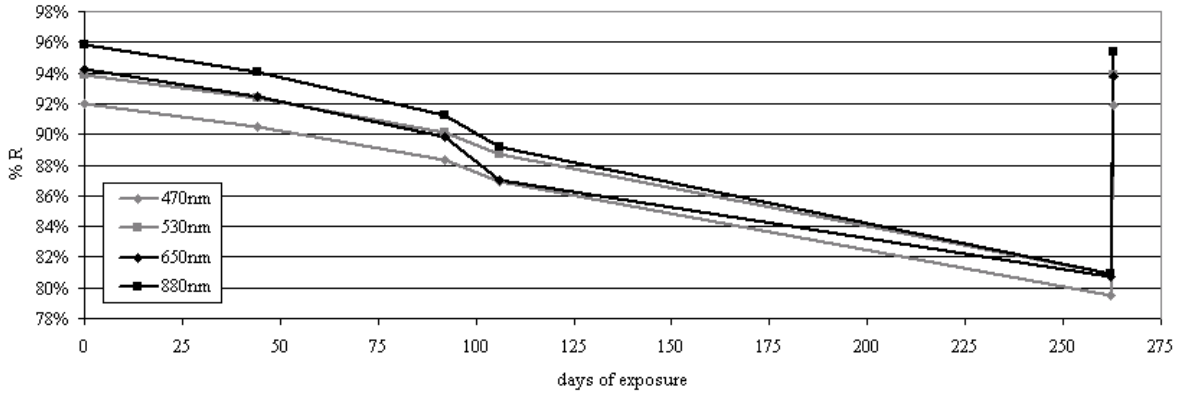

Figure 9: reflectivity loss of the 4-layer recipe

Finally, figure 10 shows a 4-layer sample exposed outdoor (facing upwards) on the prevailing wind side of the dome, about 2m above the ground. This test attempts to understanding exposure resistance under the worst possible conditions: full exposure to all atmospheric events including dust, condensation during high humidity, rain and even a snow storm (a few suspicious scratches also make us think a bird visited the sample!). Cosmetically, the coating presents no yellowish tint nor tarnish centers, which gives some important clues on the durability we have achieved. At the beginning of the sample's outdoor exposure, which started 34 days after being coated, we covered it with a thin layer of dirt collected on the ground; it was later washed after each of the two measurements taken so far (day 82 and 254). The average visible reflectivity loss is 0.75%/month and is due only to dust embedded in the film.

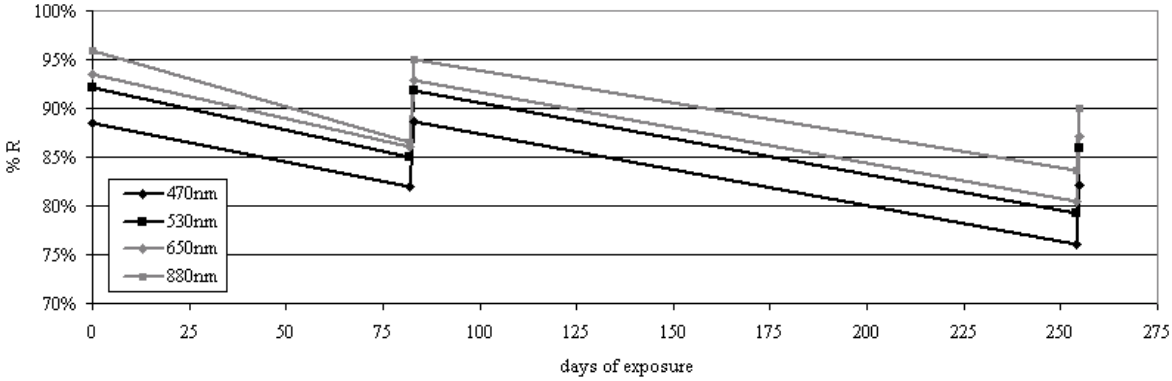

Figure 10: reflectivity loss of a 4-layer Ag sample exposed outdoor (day 0 is coating)

The following table summarizes the degradation rates we observed and confirms the importance of the NiCr layer between Ag and SiN_x[11,12]. We also include the durability data obtained on samples exposed in the GN dome in Hawaii. At that site, there are several environmental conditions different from GS in Chile: the mountain top soil is made of volcanic cinders, the elevation is much higher (4300m versus 2700m), there is an active volcano nearby, and there are more 100% humidity events. Curiously the tarnishing of bare Ag is much slower at Mauna Kea, but the 3-layer protected Ag has deteriorated faster. The 4-layer coating is resisting equally-well at both sites.

Coating	**Monthly loss rate at 470nm at GS** (dust loss excluded)	**Sample age** days	**Monthly loss rate at 470nm at GN** (dust loss excluded)	**Sample age** days
Bare Ag	3.98 %	566	1.47%	566
SiN_x-protected Ag	2.28 %	315	-	-
3-layer Ag	0.28%	411	0.47%	263
4-layer Ag	0 %	263	0 %	263

4.2.4 First real-life experience with M2

In October 2003, we coated the GS M2 with the 3-layer recipe since it appeared to provide sufficient protection for a downward looking mirror. Figure 11 shows the reflectivity loss over time. The coating behaved well for the first 45 days until some 'event' seems to have triggered a rapid degradation during which the R loss averaged 0.23%/day! None of the similar 3-layer samples exposed under the roof degraded in the same way, so the particular exposure of M2 at the top of the dome is highly suspicious. The cosmetics are very peculiar and not the typical tarnishing: the coating is free of dust but looks very hazy and took a pronounced yellowish to greenish tint with darker spots and extended areas, especially near the inner and outer edges. Despite an exhaustive investigation, we do not have a definitive explanation for the problem but we suspect a photoreaction of the Ag layer (yellowish tint already reported) and an oxidation of the top NiCr layer (various nickel oxides and chromium oxides are greenish). The effect is likely caused by the combination of a reaction to the ambient lights in the dome (compared to the 3-layer samples exposed under the roof, M2 has no protection and is directly illuminated by ambient light) and some other 'triggering' contamination. Among all the possibilities, we examined contaminants outgassing from the substrate, contamination during the coating process, outgassing from batteries or exhaust from fork lift on observing floor, and CO2 cleaning (done once only). We think that the exhaust fumes from our auxiliary generator, used once at night in a period of unfavorable winds, might be the problem.

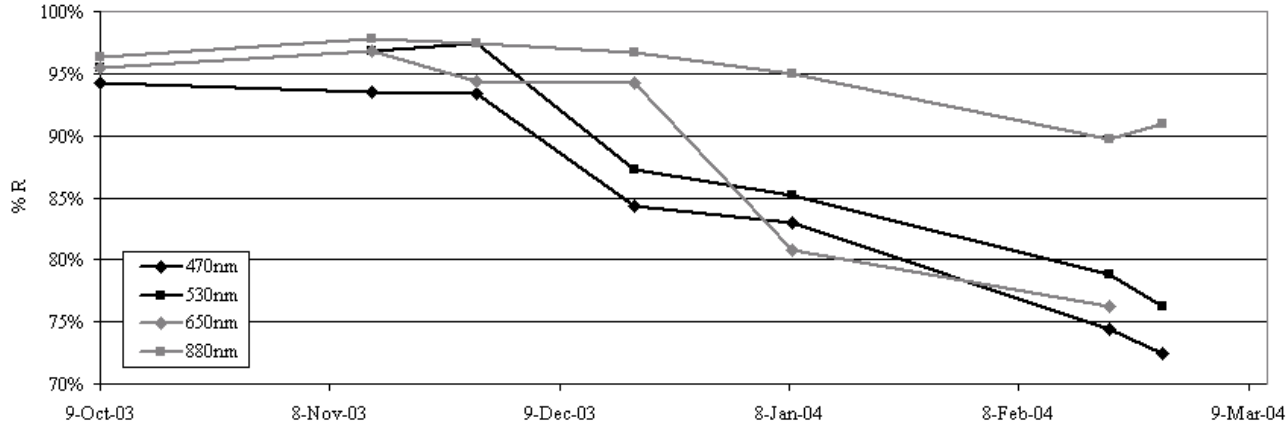

Figure 11: reflectivity loss of M2 coated with 3-layers

4.3 Adhesion and abrasion tests

Adhesion is checked by a pull-test with Scotch tape. We have experienced both adhesion problems of the film onto the substrate and between the layers. These issues have been solved experimentally with specific combinations of power level, throw distance and pulsed power depending on the materials being sputtered. The first NiCr adhesion layer between the substrate and Ag is clearly essential (bare Ag samples have started to peel off after 400 days under soft sponge washing). Under proper sputtering conditions, both 3- and 4-layer samples pass the adhesion test.

We also conducted standard abrasion tests:

- Severe abrasion (2.5 lb eraser) following MIL-C-675C 4.5.10: both 3- and 4-layer samples were scratched.
- Moderate abrasion (1 lb eraser with cheesecloth) following MIL-M-13508C 4.4.5: the 3-layer sample was always scratched and sometimes the coating peeled off, whereas the 4-layer sample was barely scratched.

4.4 Accelerated durability testing

Accelerated durability tests were subcontracted to ODA because of their prior experience with similar tests in our feasibility campaign. Durability of 3- and 4-layer samples was measured in three tests done in environmental chambers, some of which are standard in the industry:

- Temperature/humidity done at ENKI Technologies : 24h at 55°C and 98% RH without condensation, followed by 6 four-hour cycles (5-15°C T and 30-95% RH linear increase in the first hour, stay at 15°C for 2h, 15-5°C T and 95-30% RH linear decrease in the fourth hour). All the samples passed the test with no reflectivity loss.
- Salt fog exposure done at Dynamic Labs (5% NaCl salt solution at 35°C) for 48h following MIL-STD-810F (method 509A) specification: after washing a white deposition left on the samples, there was no change at all for the 4 layers and a uniform 1.5% reflectivity loss (0.3 to 3μm) for the 3 layers.
- H_2S fog exposure done at ODA following the ISO Standard 9022-20 (temperature of 35 ± 2°C): the 3-layer sample failed almost immediately whereas the 4-layer sample resisted well until 500ppm-hour (the later compares favorably with the old ACT sample that started to fail around 650ppm-hour; the difference might be due to the faster dosage put on our sample: 3 days compared to 10 days for the ACT sample)

	3-layer			4-layer		
	$R_{0.5\mu m}$	$R_{1\text{-}3\mu m}$	$R_{10\mu m}$	$R_{0.5\mu m}$	$R_{1\text{-}3\mu m}$	$R_{10\mu m}$
10 ppm-hour	15%	5-45%	83%	89.7%	94-96%	98.7%
500 ppm-hour				88.3%	94-96%	98.7%
520 ppm-hour				78.3%	84-87%	96.6%

The last accelerated and extreme test worth mentioning was conducted at Gemini by exposing for 5 min a 3- and 4-layer samples in the generator exhaust plume (facing down into the fume, about 10cm above the duct end). The samples got covered with black soot, but after washing, the 4-layer sample recovered its previous shiny appearance (0.7% visible R loss at 500nm) whereas the 3-layer sample looked very hazy (44% loss).

4.5 Coating preparation and maintenance

It is well known[13] that particles on the substrate prior to coating form pinholes in the coating that are the main entries for the contaminants into the film. We have recently started a refurbishment plan of our coating area to transform it into a cleaner room since the plant is located in the basement of our building with no special consideration for cleanliness. The environment is characterized with a dust monitor (model GT-480 from Met One Instruments) located outside and inside the coating vessel. This device processes air at a rate of 2.8 lpm (0.1cfm). We have measured the average number of 0.3μm and 5μm particles per liter of air to be: 50,400/6,000 and 36,000/3,000 outside and inside the vessel respectively. Therefore, in our 150m^3 vessel, we have the order of 450 million of 5μm particles (!) that a HEPA system should be able to eliminate. We don't know how many particles are pumped away during roughing and how many deposit on the substrate because of turbulent flow. Our coating quality inspection includes a rough pinhole evaluation done by naked-eye counting in transparency with back illumination (only large pinholes detected). This is really more a qualitative test than quantitative although we attempt to determine some numbers: for reference, at a recent coating of the GN M1 we had about 10 pinholes between 0.1 and 0.5mm per square inch and about 5 pinholes >0.5mm per square foot, which is an indicator that our substrate cleanliness should still be improved dramatically. We estimate that all these pinholes can represent up to 0.4% of the M1 surface (and account for 25% of the total emissivity). We observed that as soon as M1 was dried after the stripping, it was already covered with dust. In the 1h-period needed to transport the mirror from the wash area to the vacuum vessel, we also collect more dust. Final blowing with CO_2 snow inside the vessel is not efficient so we implemented both a HEPA-filtered air system from the top port of our vessel, maintaining positive pressure inside, and a CO_2 'shower' across the mirror as it enters the vessel. Initial analysis of dust count data after installing the filtered-air fan shows that we lowered by a factor 50 and 100 the amount of 0.3 and 5μm particle size respectively in the vessel. Our last coating inspection (M3) showed no pinholes larger than about 10μm, and an average of 5 pinholes about 10μm-size and 5 pinholes < 5μm-size per square inch.

In order to fulfill the demanding emissivity requirement in operation, we have implemented an in-situ wash process of both M1 and M2 in the telescope. The technique is standard contact-wash with natural sponges and soap followed by de-ionized (DI) water rinsing and drying. We have retrofitted the telescope with all the hardware needed to make a quick and safe in-situ process. For M1, we use the mirror cover as access to the glass while the telescope is zenith-pointing. Water, soap and DI water are spread with 80 nozzles at the OD and collected in a drain pan installed in center hole in lieu of the light baffle. Special sealing with air bladders, backed up with a water detection system, prevents leakage into the mirror cell or down to the telescope focus and instrumentation. Drying is done with small portable air knifes. The whole process takes about four hours and will ultimately be performed within a single day without impact for science observations at night (the process is currently being practiced and tuned while delicate instrumentation is off the telescope). With this process, we hope to maintain low-emissivity surfaces permanently and extend coating lifetime significantly. Extrapolating the numbers indicated previously, we anticipate washing M1 approximately every 4 to 6 months, or in the case of a sudden major contamination.

As for etching the 4-layer film, we have experimented with our standard recipe and found with relative surprise that the 'green river' solution (HCl) is capable of stripping all the layers, although the base NiCr layer required more wetting time (20min). Other chemicals like the Schweig solution (based on potassium ferricyanide and sodium thiosulfate) can be used to accelerate the process. The stripping effluent was analyzed and met the 5.0ppm EPA regulation for Ag and chromium.

5. CONCLUSIONS

We have now fully coated our southern telescope with protected silver coatings and reached $\varepsilon_{3.8\mu m} = 2.6\%$ at the RC focus. By the end of 2004, we should have the northern telescope in a similar state thus providing improved mid-IR capability at the Gemini Observatory.

Because of the encouraging result from our 4-layer samples (the longest exposure is 10 months) and the fact that they are subjected to a more extreme environmental exposure than M1, we have strong indications that the Ag coating recipe recently applied to M1 should have sufficient protection against tarnishing and maintain its reflectivity/emissivity performance for more than a year with appropriate in-situ washing maintenance. Of course, due to large-size scaling effects, only real-life experience will tell us exactly what the durability is like for a large 8m mirror.

We plan to continue to monitor the optical quality of our witness samples as well as our primary optical surfaces. Further, we will investigation techniques to increase coating lifetime (lighting, flushing with dry air). We plan to continue to improve the performance of the coating process (increase the 2.2μm reflectivity by another 1%) and to fully automate it. Testing will continue on recipes including both Al and Ag that would boost visibility in the blue, providing an improvement to both visible and infrared telescopes. We also have plans to implement more monitoring devices for sulfide compounds, and possibly implement the surface heating technique engineered in the Gemini design phase.

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