

Corrosion-resistant front surface aluminum mirror coatings

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Abstract. Front surface metal mirrors need protection of the inherently fragile metal film deposited on a glass substrate. Conventional evaporated dielectric thin-film overcoats provide limited protection because of their less than dense packing. These films usually have a columnar structure with voids between the columns. The voids give access to the underlying metal film for humidity and corrosive gases or liquids. Some progress in developing better coatings was made in the early 1980s with ion-assisted deposition. Front surface aluminum mirrors with dielectric thin films deposited by reactive low-voltage ion plating have a still higher survival time, by a factor of up to 3 under comparable test conditions. The transmission of our best samples increased to only 10% when immersed in 0.2 M NaOH for 20 h. By comparison, an unprotected aluminum film dissolves in less than 5 min. Electron beam evaporated dielectric coatings provide protection for about 1.5 to 2 h in the same test solution. The reason for the significant improvement brought about by reactive low-voltage ion plating deposition, and its advantage for large-scale production is discussed.

Subject terms: coatings; corrosion testing; harsh environment; ion platings; mirrors; thin films.

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1 Introduction

Evaporated aluminum mirror coatings have been known for the last 80 years or so,¹ used in particular for astronomical mirrors.² They are probably the most widely used coatings in optics and astronomy, because of the relative ease of manufacturing and their high reflectance over a wide spectral range.³ Achieving high-reflecting aluminum films with conventional (thermal) evaporation requires a low background pressure in the vacuum system and high evaporation rates.⁴ The composition of the residual gas can also influence the reflectance of aluminum layers. The correlation of deposition conditions and reflectance has actually been used for controlling the consistency of aluminization in semiconductor manufacturing.⁵

While bulk aluminum is relatively stable in normal environment, evaporated aluminum films are more fragile because of their columnar microstructure.⁶ This microstructure consists of voids between the columns, giving easy access of environmental contaminants, including humidity, to the inside of the coating and its interface to the substrate. This access is particularly pronounced at growth defects known as nodules,⁷ which typically have gaps around them.⁸ The columnar microstructure makes the aluminum films soft and susceptible to mechanical damage by abrasion or scratching, and can lead to noticeable light scattering.^{9,10} Nodules tend to break out either all by themselves because of intrinsic or

thermally induced stress, or upon external mechanical interaction with the surface of the thin film (cleaning, abrasion), leaving pinholes behind.

A pair of dielectric layers on top of the aluminum film serves the dual purpose of reflection enhancement and environmental protection,¹¹ particularly against uptake of humidity into the aluminum film and oxidation, which may degrade its longevity and reflectance, respectively. The reflection enhancement is based on constructive interference within the two dielectric layers. Both layers have quarter-wave optical thickness (QWOT) at the center of the spectral region for which the reflectance enhancement is wanted, for example, the visible or the ultraviolet.¹² Dielectric overcoats also enhance the hardness and mechanical resistance of aluminum front surface mirrors to an extent that allows them to pass customary abrasion tests.¹³

2 Experiments

2.1 Thin-Film Deposition

High vacuum box coating equipment of the type Balzers BAK 760 has been used at Opto Mechanik, Inc. (OMI) for conventional electron beam and thermal evaporation. Two electron-beam evaporation sources of the type Balzers ESQ 110 and one thermal evaporation source (directly heated by high current) are mounted in the base plate of the coating chamber. The substrates to be coated are located on three rotating planets. The vacuum system and the evaporation processes are controlled by a microprocessor-based process control unit Balzers BPU 100. The rate of evaporation and the thickness of the aluminum and dielectric films is monitored by a quartz-crystal microbalance device. At CREOL, high vacuum box coating equipment of the type Balzers

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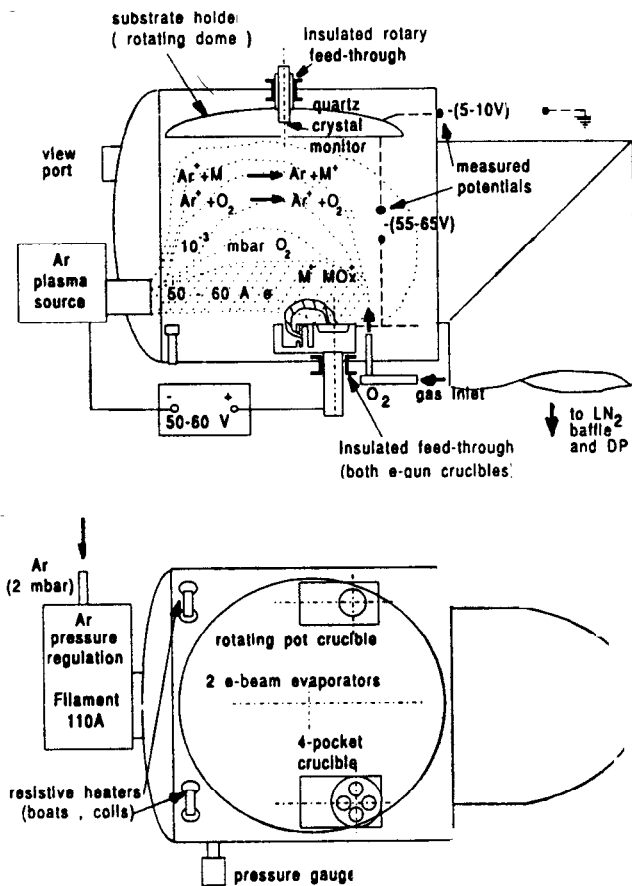


Fig. 1 Schematic of coating equipment used at CREOL for reactive low-voltage ion plating (Balzers BAP 800).

BAP 800 was used, which employs a low-voltage, high-current plasma source (ca. 55 V, 55 A) for the ion-plating deposition process,^{14,15} which is described elsewhere in more detail.^{16,17} The equipment, shown schematically in Fig. 1, has two electron beam evaporators (one ESQ 110 and one ESQ 113, the latter used with a four-pocket crucible) mounted in the base plate, insulated from ground potential. The single rotation rotary dome is also insulated from ground potential. The film thickness monitor is of the quartz-crystal type.

Protected aluminum mirrors produced in the BAK 760 coating equipment at OMI served as a reference, representing conventional state-of-the-art deposition. For these coatings, glow discharge is being used routinely before starting the actual aluminum evaporation, to enhance the adhesion of the aluminum film to the substrate. At CREOL, we did not use the glow discharge cleaning for the first set of experiments, but later on before the thermal evaporation as well as ion plating of aluminum films.

2.1.1 Thermal evaporation of aluminum

For the evaporation of aluminum at the necessary high rates, we have been using the standard method of resistive heating of helical tungsten coils mounted between high-current feedthroughs. The aluminum is evaporated quantitatively, i.e., until no more aluminum is left on the tungsten coils. With the given geometry the amount of evaporated aluminum is sufficient to deposit an opaque layer of aluminum, about 100 nm thick. After completion of the aluminum layer,

dielectric layers (for example, SiO₂, TiO₂) were deposited for reflectance enhancement and protection, using reactive evaporation (at OMI) or reactive ion-plating deposition (at CREOL).

These processes are described in more detail below. For aluminum films, the substrates to be coated are not heated as is usual for thermal or electron beam evaporation of dielectrics for better durability. Otherwise, the aluminum film would grow with pronounced crystallites,^{9,10} giving rise to enhanced light scattering (hazy films).

2.1.2 Electron beam evaporation of dielectrics

Electron beam evaporation has become the standard technique for vacuum deposition of almost any kind of materials, metals, semiconductors, and dielectrics alike. Some of the advantages of electron beam evaporation over boat evaporation are higher local temperatures of the source material in a smaller area, giving higher evaporation rates at reduced thermal radiation load. By melting the starting material in a limited area, contamination problems with foreign crucible material do not occur, since the molten puddle is surrounded by solid material of its own. Some oxides such as the widely used TiO₂ may lose oxygen upon heating in the crucible because of thermal and electron beam induced dissociation. Sometimes suboxides (Ti₂O₃, SiO, TaO_x, with $x < 2.5$) are used as the starting material because of lower melting point and higher vapor pressure at a given evaporation power and temperature. To balance the oxygen deficit, molecular oxygen is supplied to the coating chamber at a partial pressure in the high 10^{-5} to the low 10^{-4} mbar range.^{18,19} Oxygen molecules dissociate partially upon adsorption on the surface of the substrate and growing thin film. The resulting oxygen atoms react with the suboxide molecules or metal atoms upon their arrival at the surface. We used this classical reactive evaporation process in the BAK 760 at OMI for the dielectric layers on top of front surface aluminum mirrors. The substrates were only moderately heated ($\leq 150^\circ\text{C}$) during the deposition of the dielectric films in order to avoid recrystallization of the freshly deposited aluminum. Some heating was necessary to assure proper environmental stability.

2.1.3 Low-voltage ion plating

The process of low-voltage ion plating as implemented in the Balzers BAP 800 equipment used at CREOL begins by first melting the starting material for the thin film (aluminum, silicon, zirconium, titanium oxide, tantalum pentoxide) in one of the two electron beam evaporators. After achieving a uniform melt, an argon plasma discharge burns between the hot filament plasma source (located in the coating chamber door) and the crucible (Fig. 1). The crucible of that particular electron beam evaporator, insulated from ground, serves as the anode of the discharge. A low-voltage, high-current power supply delivers 50 to 60 A of electron current at an arc voltage of 50 to 60 V between the hot filament and the crucible. The argon in the plasma source chamber (at 2 to 4 mbar pressure) is ionized by the temperature of the filament. The argon plasma created in the hot filament source effuses into the coating chamber, via a 1- to 2-mm orifice which acts as a differential pressure stage. The partial pressure of the argon in the coating chamber is about 10^{-4} mbar during the ion-plating process. The dis-

Table 1 Thin films, starting materials, and process parameters of the (reactive) low-voltage ion-plating process.

Starting Material	Deposited Film	Index of Refraction	Plasma Current	Arc Voltage	Deposition Rate	Oxygen Pressure
		@ 550 nm	[A]	[V]	[nm/s]	[10 ⁻³ mbar]
Al	Al	-	n.a.	n.a.	7.0	0
Al	Al	-	50	55	4.0	0
Si	SiO ₂	1.49	55	55	0.5	1.1
TiO	TiO ₂	2.45	55	70	0.25	1.5
Ta ₂ O ₅	Ta ₂ O ₅	2.31	55	65	0.3	0.9
Zr	ZrO ₂	2.27	55	80	0.4	1.3

charge current between the plasma source and the crucible ionizes the evaporant by electron impact.

Because of the high current flowing through the melt, the starting materials (Table 1) need to have some electrical conductivity. This requirement excludes most of the full oxides as starting material for the deposition of oxide coatings. The apparent exception in Table 1, tantalum pentoxide (Ta₂O₅), dissociates easily during the premelting procedure before the ignition of the plasma discharge, and gains sufficient conductivity by becoming partially metallic.

A rotating dome, insulated from ground potential, above the electron beam evaporators holds the substrates to be coated. The insulation of the dome allows dielectric substrates in particular, but also metallic surfaces like the aluminum films and the dome itself, to attain a negative self-bias of -5 to -20 V relative to ground potential. These surfaces receive more electrons out of the plasma sheath than the less volatile ions. Positive ions (M⁺, MO_x⁺) created in the vapor above the crucible (by electron impact), and ions generated in the plasma (for example, by an electron transfer process Ar⁺ + M ←→ Ar + M⁺) arrive at the substrate surface with an energy (per charge) in the order of 10 to 20 eV.²⁰ The negatively biased substrates attract the positively charged ions (argon, oxygen, and evaporant-metal and/or suboxides) which follow electrostatic field lines that terminate normal to the surface. Hence, the angle of ion impact on the surface of the substrate and the growing film is normal, irrespective of the inclination of the local surface element with respect to the line-of-sight to the vapor source.

The kinetic energy of the ions transforms partly into thermal energy and into enhanced surface mobility on the substrate and the growing film. The momentum transfer of the accelerated ions impinging onto the growing film by collision cascades and the conversion of kinetic into thermal energy yield a very dense microstructure for most ion-plated coatings. The film formation process might be seen as a transient liquid film deposition (because of the equivalency of their energy with high local temperature), with immediate quenching from the liquid into the solid state because of the actually relatively cool substrate.²¹ Accordingly, the resulting films are very dense and glass-like (vitreous) and have smooth surfaces, which were found to have even lower rms surface roughness in some cases than the respective substrate.²²

For ion plating of aluminum films we used a pure argon plasma. The protective oxide coatings need a reactive oxygen plasma (in addition to the argon plasma). Oxygen was bled into the coating chamber by means of a regulated valve,

at partial pressures in the low 10⁻³-mbar range (see Table 1). The reactivity of the ionized and activated oxygen allows for the deposition of fully stoichiometric oxides from metals or suboxides as the starting materials in the electron beam crucible. The ionization and accelerated attraction of the starting material and the oxygen resulting in more densely growing films allows for the coating of unheated substrates. However, we found it necessary to apply glow discharge cleaning of the substrate surfaces immediately prior to coating them. The ions formed in a glow discharge bombard the substrate surface and destroy hydrocarbon film contamination that may have stayed on after the solvent cleaning process. They also remove the physisorbed monomolecular water layer which otherwise decreases film adhesion. Initially we used room air admitted to the coating chamber through a regulated valve for sustaining the discharge. Later, we changed to dry nitrogen because of cleanliness concerns. The film adhesion was not noticeably different for air or nitrogen sustaining the glow discharge.

2.2 Environmental Testing

All of the environmental tests were performed at OMI, using a Thermotron SM 32 S "Minimax" humidity chamber and a Harshaw No. 22 salt fog test chamber. The environmental durability tests, including the cheesecloth abrasion test and the Scotch-tape adhesion test, were in accordance with the widely used military standard MIL-M-13508 C.¹²

2.3 Corrosion Testing of Protected Aluminum Front Surface Mirrors

To evaluate the anticipated improvement of environmental protection brought about by the higher density and literal imperviousness of ion-plated thin films, we adopted an accelerated corrosion test used earlier by Sainty, Netterfield, and Martin.²³ The aluminum coatings protected with various single- or double-layer dielectric thin films were immersed into a 0.2 M solution of NaOH. A photoresistor monitored the light transmitted through the aluminum mirror (Fig. 2) as it became more transparent upon the dissolving of the aluminum film in the case of imperfect protection. The voltage drop across the photoresistor was measured as a function of the immersion time.

3 Results and Discussion

3.1 Summary of Coating Runs and Environmental Test Results

Table 2 summarizes the coating experiments, both for aluminum mirrors and all-dielectric high reflectors, and the various environmental test results achieved with them. The first coatings made with the BAP 800 at CREOL were inferior in their environmental durability compared to the state-of-the-art reference samples produced at OMI by standard thermal and electron beam evaporation of the aluminum and the dielectric thin films, respectively, for two reasons. First, the high thermal evaporation rate of the aluminum from a tungsten helix placed at a fairly eccentric location in the coating chamber (see Fig. 1) caused a large and random variation of the angle of aluminum vapor incidence on the substrates. Because of the rapid evaporation of the aluminum for achieving high reflectance, the position of the substrates on the rotary dome during the few seconds of evap-

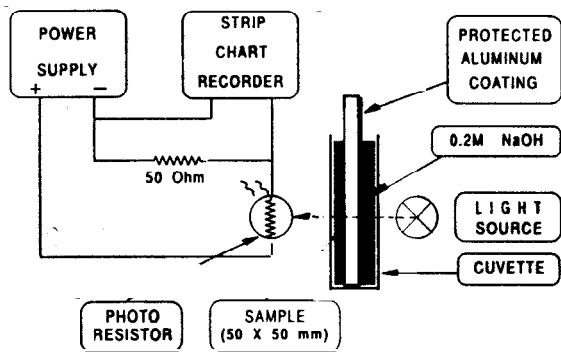


Fig. 2 Accelerated corrosion testing of protected front surface mirrors, by immersion into a 0.2 M NaOH solution. Mirror transmittance is monitored as a function of time.

Table 2 Summary of coating runs and environmental test results.

Run Nr.	Layer Sequence (1)	Al-Dep. (2)	Glow Disch. (3)	Cheese-Cloth Abrasion Test (4)	Scotch-tape Adhesion Test (4)	24 hours Humidity Test (4)	48 hours Salt Fog Test (4)
508	Si Al Ti	N	passed	passed	passed	passed	
515	Al Si Ti	N	sleeks easily	failed			
516	Al Si Ti	N	light sleeks	passed	failed		
518	Al Si	N	passed	light removal			
519	Al Si Ta	N	light sleeks	failed			
611	Si Al Zr	N	light sleeks	failed			
617	Al Si Zr	N	light sleeks	failed			
704	Si Al Si Ta	Y	passed	passed	passed	passed	
707	Al Si Ta	Y	passed	passed	passed	passed	
714	Si Al Si Ta	Y	passed	passed	passed	passed	
804	Al Si	Y	passed	passed	small pinholes		
805	Si Al Si	Y	passed	bad	blisters		
806	Al Ta	Y	small sleeks	passed	passed	passed	
810	Al Zr	Y	small sleeks	passed	passed	passed	
811	Al Si	Y	passed	passed	passed	passed	
812	Al Si Al	Y	sleeks	passed	milky		
813	Al Ti	Y	sleeks	passed	passed	passed	

(1) Counted from the substrate. Al is the metallic aluminum film; Al, Si, Ti, Ta, and Zr indicate thin films of the respective oxides, deposited by RLVIP.
 (2) Deposition of the aluminum film: T = thermal evaporation, I = low voltage ion plating.
 (3) Before deposition of the first layer; N = no, Y = yes.
 (4) Tests performed according to MIL-M-13508 C.¹³

oration was rather erratic, giving rise to a significant variation of the angle of vapor incidence. Second, we did not use glow discharge at CREOL for the first set of coating experiments. The substrate surfaces may have retained some hydrocarbon or water film contamination that reduced the adhesion and hardness of the aluminum film. Although the protective dielectric coatings were deposited at CREOL in all cases by reactive ion plating, their usual hardness and density may have made up incompletely for the weakness of the aluminum films. Glow discharge prior to the aluminum evaporation in later coating runs improved coating adhesion. We kept using the glow discharge precleaning when we changed from thermal evaporation of aluminum to electron beam evaporation with argon plasma assistance (low-voltage ion plating of the aluminum films), because

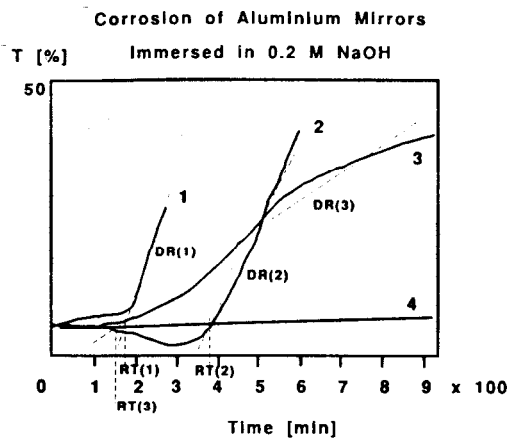


Fig. 3 Increase of the transmission of some protected aluminum mirrors (see Table 3) upon immersion into 0.2 M NaOH solution, as a function of time.

Table 3 Select samples used for corrosion testing (see Fig. 3).

Graph Nr.	Sample Nr.	Sub-Layer*	Aluminum Deposition*	Protective Coatings: Material / Deposition*
1	OMI	none	TE	SiO ₂ + TiO ₂ / EB
2	806	none	IP	Ta ₂ O ₅ / IP
3	810	none	IP	ZrO ₂ / IP
4	714	SiO ₂ / IP	IP	SiO ₂ + Ta ₂ O ₅ / IP

*Deposition processes: TE = Thermal evaporation, EB = Electron beam evaporation, IP = low voltage ion plating

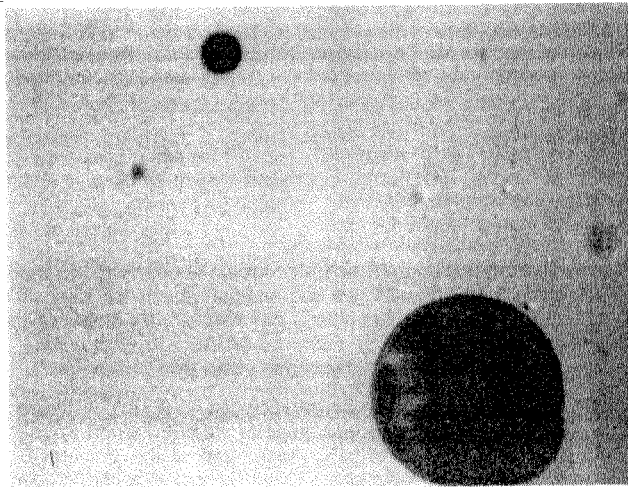
the relatively low energy of the argon plasma employed in the particular ion-plating process is probably insufficient to remove (sputter off) such contamination layers.

3.2 Accelerated Corrosion Testing

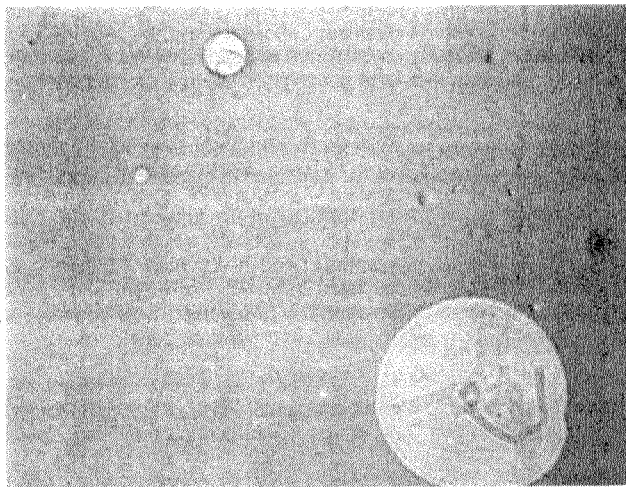
The caustic solution of 0.2 M NaOH dissolves an unprotected aluminum film of the thickness used for these experiments (≈ 100 nm) in just a few minutes. Conventional protective layers, e.g., a pair of SiO₂ and TiO₂ thin films of QWOT in the visible, reactively evaporated with an electron beam source, provide for a retention time of about 1.5 to 2 h. Sainty, Netterfield, and Martin²³ reported retention times as long as 400 min (approximately 3.5 h) for their ion beam-assisted deposited protective layers. Figure 3 shows that we obtained similar retention times with reactive ion-plating deposition of the protective layer(s) on aluminum directly deposited onto the glass substrates (Table 3), with varying degradation rate as shown in Table 4. However, with an ion-plated SiO₂ underlayer between the substrate and the aluminum thin film, the retention time increased dramatically to more than 20 h. After this time, the mirror still had 90% of its initial reflection. The transmission increase of about 10% of these "best" mirrors was clearly due to individual pinholes etched into the aluminum film by the corrosive solution at defect sites in the coating. Optical micrographs obtained with a Nomarski differential interference contrast microscope at $\approx 1000\times$ magnification (Fig. 4) show indeed defects in the center of the etched pinholes. Transmission illumination also shows that the protective coating is still present as a transparent pellicle above

Table 4 Evaluation of corrosion tests shown in Fig. 3 for retention time (RT) and degradation rate (DR).

Graph Nr.	Sample Nr.	Retention Time [min]	Degradation Rate [%T/100min]
1	OMI	180	22.3
2	806	360	16.9
3	810	150	7.0
4	714	n.a.	< 1.0



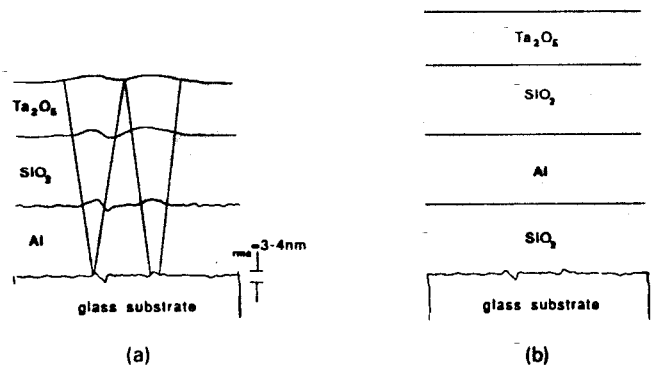
(a)



(b)

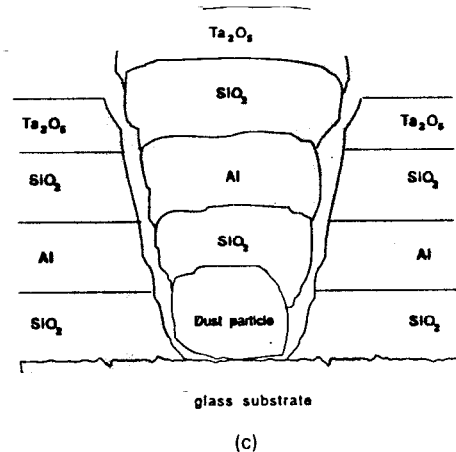
Fig. 4 Optical micrographs (Nomarski differential interference contrast at $\approx 1000\times$ magnification) of aluminum mirror protected by a pair of dielectric thin films, and SiO_2 between the aluminum film and glass substrate: (a) in reflected light, (b) in reflected and transmitted light.

these pinholes. Figure 5 gives a schematic representation of a possible reason for the dramatic increase in survival time, and indicates that there is still room for improvement. While the ordinary glass substrates that we used have a surface roughness of about 3 to 5 nm rms, as measured with a WYKO TOPO 2-D (courtesy of F. Boero and C. Masser, Newport Corp.), the SiO_2 layer deposited onto such a glass



(a)

(b)



(c)

Fig. 5 Schematic of the film structure of protected aluminum films deposited on (a) ordinary sheet glass and (b) on a SiO_2 underlayer, which provides a smooth surface. (c) Gaps around an overcoated (dust) particle provide access for the caustic solution to the aluminum layer.

surface may be as smooth as 0.2 to 0.6 nm rms.²² The difference in the roughness of the surface to be coated with the aluminum and subsequent dielectric layers is very likely to make a difference for the durability of these films. A rougher surface may cause intrinsic growth defects [voids, cracks, Fig. 5(a)] which allow the corrosive solution to access the aluminum film more or less uniformly, with some delay time (survival time). The smooth surface provided by the SiO_2 underlayer [Fig. 5(b)] may be replicated by uniformly dense coatings, aluminum as well as the dielectrics, preventing the solution from attacking the aluminum layer. The solution can then get to the aluminum only through gaps formed at point defects such as dust particles or possibly spatters or flakes originating during the deposition process, if these particles are overcoated incompletely [Fig. 5(c)].

4 Conclusions

We have shown significant improvement of the corrosion resistance of aluminum thin films with dielectric protective overcoats when deposited by (reactive) low-voltage ion plating, as compared to state-of-the-art thermal and electron beam gun evaporation. In particular, an ion-plated SiO_2 layer on the surface of ordinary glass substrates extended significantly the retention time and decreased the degradation rate of a subsequently ion-plated aluminum film with

protective overcoats. Our results give directions for the development of environmentally stable mirrors that are needed, for example, in astronomical, solar energy, and military applications.

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Karl H. Guenther Biography and photograph appear with the paper "Metal-oxide thin films with reduced residual absorption deposited in a reactive low-voltage ion-plating process with N₂ activation" in this issue.



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He moved to the Instrument corporation of Florida in 1963. He formed Willey Corporation in 1964, which functioned in consulting, optical system development, and limited production. In 1981, he joined Martin Marietta and was director of the Optical Component Center, which performs development and production of optics, coatings, and assemblies. He became vice president of technology at Opto Mechanik in 1984, where he is responsible for the development of all new instruments, test equipment, and coatings. He is a fellow of the Optical Society of America.