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HIGH-REFLECTIVE COATINGS FOR GROUND AND SPACE BASED APPLICATIONS

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I. INTRODUCTION

High-reflective coatings are indispensable in order to manufacture mirrors with highest possible reflectivity. The maximum reflectivity can be achieved by all-dielectric coatings; however, the spectral bandwidth of these mirrors is limited. For astronomical applications metal based coatings (Al, Au, Ag) are commonly applied, as they allow high reflectivity and at the same time a broad spectral bandwidth.

Silver based coatings are the only possible choice for numerous applications, as from all metals silver provides the highest reflectivity from VIS to IR. The optical properties of sputtered and of evaporated silver films are related to their morphology. A model has been developed which relates the reflectivity of as deposited samples and samples after aging to the surface morphology of the Ag films and allows the determination of effective optical constants for the respective silver films.

During aging, corrosion products like Ag_2S and AgCl are formed and reduce the reflectivity of silver mirrors. Thus, a protective layer is required to prevent the corrosion and preserve high reflectivity of a silver reflector. Even space-based telescopes require a protection because assembling, transport and storage takes place at ground under various environmental conditions. The protective layer on the mirror front-side influences the optical properties of the film, therefore transparent dielectric materials like SiO_2, Al_2O_3, etc. are commonly applied as protective layers. However, even the deposition of a pinhole-free protective layer seems to be only a necessary but not a sufficient condition for the effective protection of silver films: hydrophobic particles containing Cl and/or S in combination with a humid ambient can potentially harm even silver coatings with defect free protective layer.

For some applications the optical performance of protected silver coatings is not sufficient. In these cases enhanced Ag-coatings might be a suitable option. For example, a considerably higher reflectivity in the spectral range 350 – 450 nm can be achieved with an UV-enhanced Ag-coating while the coating still maintains a high reflectivity in the IR range. This UV-enhancement can be achieved by a multilayer approach for the protective coating which at the same time considerably increases the stability of protected silver coatings.

In a separate part of this work the properties of dielectric coatings deposited by plasma enhanced atomic layer deposition (PEALD) were investigated. ALD is a very promising technology for the optical coating of components with complex shapes, however, there are not many data available on the optical properties of ALD deposited films. It will be shown, that ALD coatings have a high potential for a successful application in optical coatings, however, some process optimization will still have to be made.

II. EXPERIMENTAL

A. Coating deposition

Most of the silver coatings and protected silver coatings presented in this article were prepared by magnetron sputtering in an inline-system equipped with 8 sputtering cathodes for the deposition of metal layers by DC sputtering and of dielectric layers by reactive MF sputtering in two separate vacuum chambers. The evaporated silver coatings were prepared by thermal evaporation in a Leybold Syrus Pro coating system.

ALD coatings were deposited by Plasma enhanced ALD in an Oxford Instruments OpAL open load reactor. For the SiO_2 coatings Tris[DiMethylAmino]Silane was used as Si precursor and O_2 plasma as the oxidant. The process parameters were optimized at various substrate temperatures from 100°C to 300°C. The pulse and purge times were reduced to the shortest values that still were sufficient to ensure a saturated ALD regime. With a total cycle time of 14.4 s and a growth rate of about 1Å/cycle the deposition rate amounts to approx. 25nm/h. The Al_2O_3 films have been deposited using alternating sequences of trimethylaluminum (TMA) and oxygen plasma at 120 C°. A 30 ms TMA dose and 5 s Plasma (O_2) with 3.5 s purge times between each precursor have been used. Working pressure was about 250 mTorr.

B. Coating characterization and testing

Reflectance and transmittance measurements were conducted using a spectrophotometer Perkin Elmer Lambda 950 equipped with a VN-accessory for R- and T-measurements at 6° angle of incidence. For the analysis of defects a Zeiss scanning electron micrograph (SEM) Sigma, an Oxford Instruments Inca x-act energy dispersive X-ray detector and a Leica light microscope CTR 6000 were applied. For infrared measurements a Varian 3100 FTIR (Fourier transform infrared spectrometer) was used.

III. INVESTIGATION OF HIGH-REFLECTIVE SILVER COATINGS

A. Optical properties of silver films

The properties of silver films depend strongly on the deposition technology and the deposition parameters. Film properties like grain sizes, orientation of grains, density and impurities have a large impact upon the optical constants of the respective silver film. Silver surfaces are rough due to the crystalline structure of the silver film and tarnishing processes after exposition of a freshly deposited unprotected silver film to atmosphere. Figure 1 (left) shows measured reflectivity data of differently prepared silver films (S1, S2, S3, and E1) together with simulated reflectivities based on two different sets of optical constants found in the literature (Wilson [1] and Johnson and Christy [2]). The experimental data was measured at an evaporated silver film (E1) and three silver films deposited by magnetron sputtering (S1, S2, S3) using different sets of deposition parameters. It can be seen that the evaporated film shows a higher reflectivity than the sputtered films, and that there are also considerable differences between the three sputtered films due to the different deposition parameters.

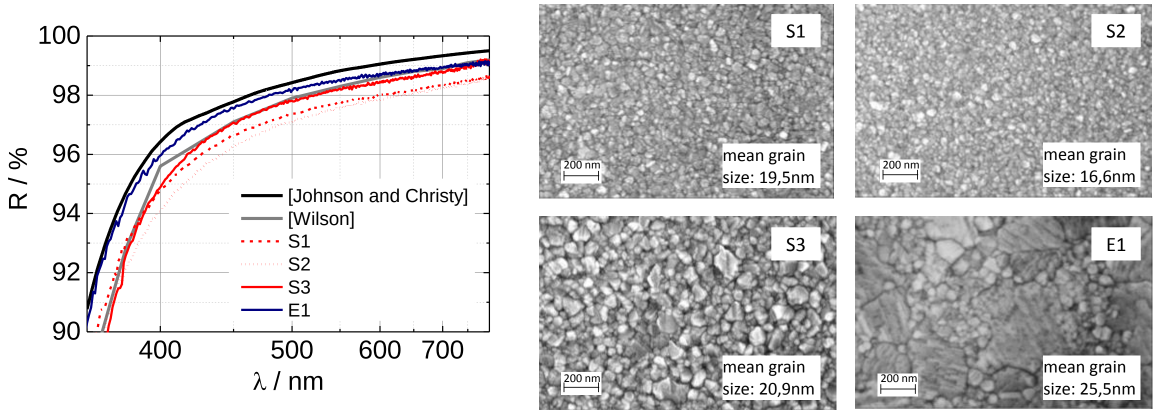

Figure 1: Left side: simulated reflectivity of silver films based on optical constants from Wilson and from Johnson and Christy and measured reflectivity of evaporated (E1) and sputtered (S1, S2, S3) silver films. Right side: Scanning electron microscopy images of the deposited films.

The scanning electron microscope images at the right side of Fig. 1 show that the reflectivity data of the differently deposited silver films can be correlated to the microstructure of the films. A larger grain size leads to higher conductivity and therewith higher reflectivity of the films. The reflectivity is influenced by the surface roughness, which leads to the formation of a more or less pronounced absorption feature in the UV reflectance spectrum, which is located around 340 nm. Additionally, the reflectivity of silver films changes during storage at ambient atmosphere due to the formation of corrosion products like Ag_2S at the surface. The combined effect of the interface roughness and the covering absorbing debris layer leads to a spectral shift of the plasmonic absorption feature. In combination with the appearance of a spectrally broad absorption tail mainly originating from the intrinsic absorption of silver sulfide [3], this mechanism can explain the observed differences between the films and also the loss in reflectance over the entire UV-VIS spectral region as a result of ageing. These influences upon the reflectance of silver films can be modelled [3] applying a model including an absorbing overlayer and a rough silver surface. With this model effective optical constants can be calculated even for aged silver films. In addition the model can be applied to predict the optical properties of protected silver surfaces.

B. Investigation of a defect mechanism in protected silver coatings

As silver films are prone to corrosion, silver mirrors have to be protected by an additional dielectric protective layer. The protective layer prevents corrosion and the formation of defects. There are numerous approaches to developing protected silver coatings which combine an effective protection of the silver film with good optical properties. Several investigations of the durability of protected silver have already been published [4, 5, 7-10]. The most investigated coatings are based on the patented coating design from Wolf et al. [6]. Advanced telescopes, e.g. the Gemini twins or the Kepler Space Telescope [4, 5], are using mirrors with this kind of coating. However, even though protected silver coatings were extensively studied and optimized in the recent years, the appearance of defects after extensive exposure of protected silver mirrors to humid atmosphere remains still an important topic [7].

In order to prevent defects on protected silver or other metals, it is important to avoid defects in the protective layer caused for example by particles or substrate imperfections. One of the most promising recent approaches is to use protective layers deposited by conformal coating methods in order to avoid pinholes and seal defects. In many of these experiments ALD is used to deposit the protective layer [9, 11, 12]. The pinhole-free protective layer can prevent a permeation of corrosion products and thus a damage of the silver film. However, new investigations of defects at protected silver reveal, that the deposition of a defect-free protective layers is a necessary but not a sufficient condition for stability at ambient conditions. At ambient conditions hygroscopic air borne particles can be adsorbed on protected silver surfaces. In combination with humidity these particles can damage the protection and subsequently damage the silver [13]. This damage mechanism could even be observed in case of protective layers which are known to be nearly pinhole-free directly after the deposition of the coating.

For example defects were observed on silver coatings with Al_2O_3 protective layer after environmental testing or after storage at ambient conditions. An investigation of these defects revealed that in the centre of each defect, a particle or a contamination containing Cl and/or S appears as the origin [13]. Cl and/or S are commonly contained in hygroscopic airborne particles [14, 15]. The presence of such particles on the surface of mirrors and samples is very likely. These hygroscopic particles can attract water molecules from the ambient and form a droplet at the surface. Components of these particles can be solved in the droplet. Thus, a solution of Cl and/or S can be formed on the surface of mirrors and samples already at a humidity of 35-80 % RH [16].

These solutions affect the protective layer. At the investigated defects, the Al_2O_3 layer was damaged in the surrounding of the particles which appears as the origin of a defect. FTIR-measurements confirm that sputtered Al_2O_3 protective layers and also Al_2O_3 protective layer deposited by ALD become converted into hydrolysed products $(AlO_x(OH)_y)$ due to exposition to water vapour. At 45 °C ± 5 °C, the conversion of a complete Al_2O_3 layer of 110 nm thickness takes 12h [13]. A similar transformation or dissolution is expected with other protective layer materials. The transformation or dissolution of the protective layer leads to a weakening of the protection and enables permeation of the solution. The permeated solution contains dissolved elements from the particles like Cl or S. These corrosive agents cause agglomeration of the silver grains and therewith a damage to the silver layer. The same kind of agglomeration can be observed at unprotected silver after exposure to NaCl-solution. This is a further indication of damage due to permeated Cl-solution. The described damage mechanism is schematically shown in Fig. 2.

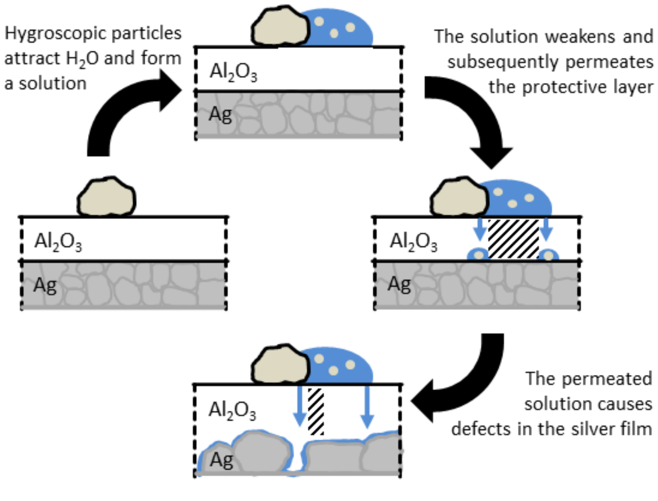

Figure 2: Schematic illustration (cross section) of the discovered damage mechanism at protected silver caused by hygroscopic airborne particles.

In order to further examine the described damage mechanism, protected silver samples have been artificially contaminated with small NaCl particles. Subsequent to the deposition of these particles on top of silver protected by an Al_2O_3 layer, the samples were stored at > 75 %RH at room temperature or exposed to 85 %RH at 45°C. After these environmental tests defects could be observed. The appearance of these defects was very similar to single defects which had been observed after environmental testing or after storage for long times at ambient conditions without artificial contamination. The characteristic agglomeration of the silver layer appears in the surrounding of the (NaCl) particle. This is another indication for damage due to permeated Cl-solution. Very similar defects occur also at silver films protected with Al_2O_3 deposited by ALD after the same period of time. As ALD-layers are known to be nearly pinhole free it can be concluded, that these defects can occur due to hygroscopic particles which damage the protective layer, subsequently permeate the protective layer and finally damage the silver. This finding shows the importance of clean and dry storage conditions. Furthermore, the test method of artificial contamination enables a direct comparison between different protective layers whereby a systematic improvement and optimization is possible [13].

C. Enhanced Ag-coatings with improved stability

It is known that different coating materials have different resistances against different kinds of contamination. Hence, by the use of a multilayer as a protective system a high resistance against different contaminations can be achieved. Tests with combined contamination of NaCl and Na_2S and subsequently exposure to moisture have been performed. As a first result a protective coating with a multilayer consisting of Al_2O_3 and SiO_2 showed a considerably better stability than single protective layers consisting either of Al_2O_3 or SiO_2 alone. This approach of improving the durability of protected silver coatings by application of multilayer protective coatings consisting of different materials can be combined with an improvement of the optical performance.

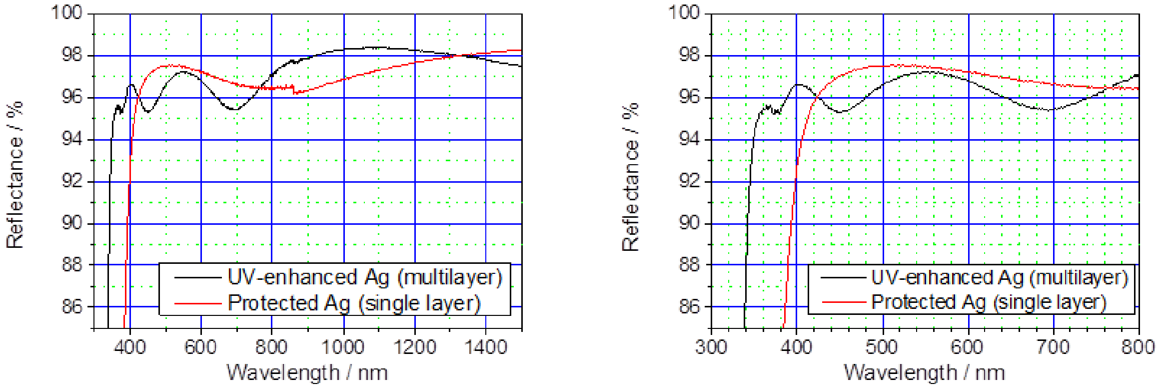

Figure 3: Reflectance of UV-enhanced Ag-coating protected by multilayer coating (black curve) in comparison to conventional protected Ag with single protective layer (red curve) at 6° angle of incidence.

Fig. 3 shows the optical performance of an UV-enhanced protected Ag-coating based on this multilayer approach in comparison to a conventional protected Ag-coating with single-layer protection. For the UV-enhanced coating a reflectance larger than 95% can be achieved for $\lambda > 350$ nm while for the conventional protected Ag the reflectance is larger than 95% for $\lambda > 420$ nm. Furthermore this multilayer UV-enhanced coating consisting of three different layer materials (SiO_2, Al_2O_3, Nb_2O_5) shows a considerably better stability than silver coatings with single layer protection of either of the three layer materials and identical overall thickness.

Table 1 shows the result of a comparative study of the stability of silver coatings with different protective layers. In order to allow a comparison of the different samples the protective layers have an identical thickness for all samples. The samples were artificially contaminated with NaCl or Na_2S and subsequently exposed to high humidity for several hours. After the testing all samples were carefully inspected. As can be seen in Tab. 1 only the silver film with the multilayer protective layer does not show any defects even after contamination with NaCl and Na_2S and subsequent exposure to high humidity. All protected silver coatings with single-layer protection show defects in the silver film after the testing with both contaminants.

Table 1: Results of comparative testing of silver layers with different protective layers by artificial contamination + exposure to humidity. All protective layers have the same thickness.

	Contamination with NaCl + humidity	Contamination with Na_2S + humidity	Contamination with NaCl + Na_2S + humidity
Ag + single layer SiO_2	✗	✓	✗
Ag + single layer Al_2O_3	✗	✗	✗
Ag + single layer Nb_2O_5	✗	✗	✗
Ag + protective multilayer	✓	✓	✓
✓	No defect in silver layer after contamination + exposure to humidity	✗	Silver layer has defects after contamination + exposure to humidity

IV. DEVELOPMENT OF ALD-COATINGS FOR OPTICAL APPLICATIONS

Atomic layer deposition (ALD) is a powerful deposition technique that enables a very precise thickness control on atomic level as well as conformal coating on surfaces with complex shapes or structures with high aspect ratio. These advantages make this technology also interesting for the application in precision optics, e.g. manufacturing interference systems on curved surfaces. Since ALD technology was preferably developed for semiconductor industry, there is still the need to investigate also fundamental questions about the achievable layer properties for optical applications. The following section presents an investigation of the properties of SiO_2 and Al_2O_3 layers deposited by ALD. These materials are very important layer materials for optical coatings and the goal of the study is to evaluate the suitability of ALD-deposited SiO_2 and Al_2O_3 layers for application in precision optics.

Fig. 4 shows the optical transmittance and reflectance spectra of a 300 nm SiO_2 layer (left) as well as a 300 nm Al_2O_3 layer (right) deposited on quartz substrates. The films are homogeneous, i.e. they do not show a refractive index gradient in z-direction since the identified halfwave points of the layer spectra are in agreement with the spectra of the uncoated substrate.

The optical losses of the silica and alumina films calculated by 100%-R(%)-T(%) are displayed in Fig. 5. Within the measurement accuracy of the spectrophotometer the films do not show any significant losses within the measured spectral range of 250 nm to 1100 nm, i.e. they are in the range of the uncoated substrate.

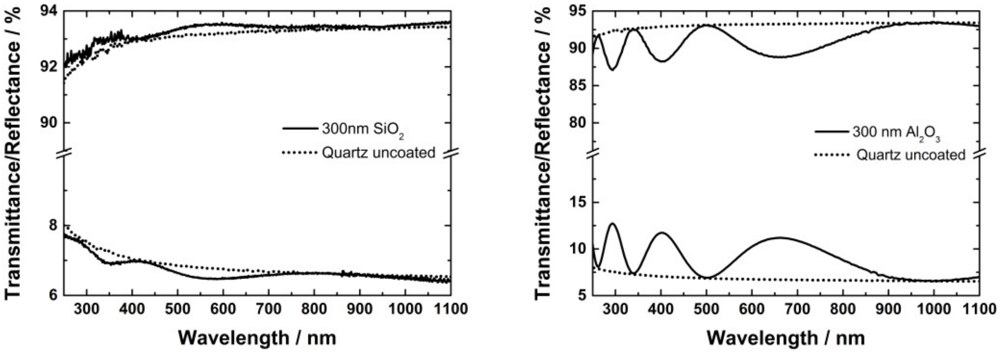

Figure 4: Transmittance and reflectance spectra of a 300 nm SiO_2 layer (left) and a 300 nm Al_2O_3 layer (right) deposited on quartz substrates.

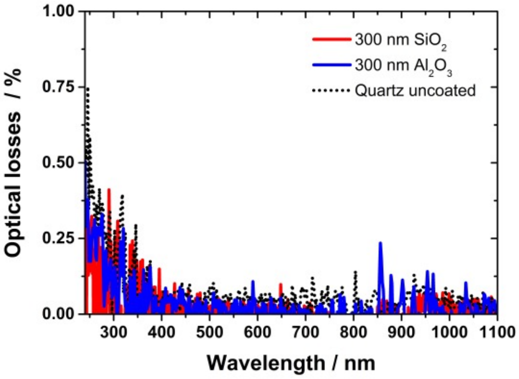

Figure 5: Optical losses of the SiO_2 and Al_2O_3 films in comparison to the uncoated quartz substrate.

The determination of the resulting film thickness and the optical constants of the coatings was implemented using an absorption-free Cauchy model. The resulting dispersion curves can be seen in Fig. 6. The SiO_2 film exhibits a refractive index of 1.452 at 550 nm whereas the index of the higher refractive alumina film amounts to 1.63.

A variation of the substrate temperature during the SiO_2 deposition only resulted in a change of the growth rate whereas the refractive index of the SiO_2 layers did not vary significantly. The growth rate decreased from 1.17 Å/cycle to 0.95 Å/cycle when increasing the substrate temperature from 100°C to 300°C. This behavior was observed before and can be attributed to a decreased precursor adsorption at higher temperatures [17].

However, the refractive index of the Al_2O_3 film could be increased from 1.63 to 1.66 as the substrate temperature was increased from 120°C to 300°C. Again, the growth rate showed the same behavior as it was observed for SiO_2 coatings.

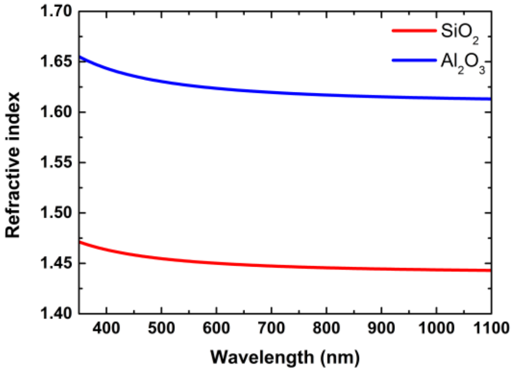

Figure 6: Refractive index of silica and alumina layers prepared by PEALD on quartz substrates.

The analysis of the structure and morphology via X-Ray diffraction (XRD), X-Ray reflectivity (XRR) and scanning electron microcopy (SEM) has shown that the SiO_2 films are completely amorphous without any crystalline structure. The density of the films was in the range of 2.2±0.2 g/cm³ (XRR analysis).

The film stress was determined measuring the curvature of a silicon wafer prior to and after the deposition with a Tencor system and the application of Stoney's equation [18]. The silica layer deposited at 200°C showed slight compressive stress in the range of -30 MPa that relaxed towards tensile stress with time when it was stored under ambient laboratory conditions. This behavior was reported before for SiO_2 layers prepared by physical vapor deposition techniques, e.g. e-beam evaporation, without or with only very weak ion assistance [19]. It indicates that the films are not highly densified and exhibit open pores that can be filled with water molecules when the film is stored under ambient conditions. In order to verify this conclusion an FTIR spectrum of a SiO_2 film coated on silicon was recorded (not shown here). Typical Si-O-Si and Si-OH bonds could be identified, but no organic residuals - indicating a complete oxidation reaction of the precursor material [20, 21]. In the FTIR spectrum a broad peak around 3387 cm^{-1} corresponding to a water absorption band [22] revealed that there is physically absorbed water within the films or pores of the films, respectively. This was confirmed by a thermal vacuum to air shift measurement. The shift has been determined by first measuring the transmittance spectrum on atmospheric conditions at room temperature. Afterwards, it was repeated in an evacuated and heated (>100°C) measurement chamber. The film exhibited a negative thermal shift of -2.1%

which is caused by a decrease in film refractive index under vacuum conditions [23]. The resulting effective refractive index of a porous film can be calculated using a mixture model that considers the volume fractions of the film material and pores. Following the assumption of an open porous film, the refractive index is higher under ambient conditions (pores filled with water) than in vacuum (water can desorb) as it was observed for the above shown SiO_2 film prepared via PEALD.

The Al_2O_3 films exhibits relatively high tensile stress of -500 MPa. Although the high tensile stress of the layer would be indicative that the films are not highly densified, the films did not show a vacuum to air shift. This could be attributed to a closed porous structure which does not cause any shift [23].

The presented results indicate, that dielectric films deposited by ALD are very promising candidates not only for the optical coatings of components with complex shape, but also for the protection of silver films. ALD coatings have very good optical properties, are known to be nearly pinhole free and are compatible with the very promising multilayer approach for the protection of silver films.

V. SUMMARY

The optical properties of differently deposited silver films can be simulated applying a model including grain size, roughness of the film and an absorbing debris layer consisting of Ag_2S. This model helps to describe real deposited and aged silver films and is also useful for future optimization of the deposition parameters for silver coatings.

As unprotected silver films are prone to corrosion a protective layer on top of the silver is necessary in order to preserve the high reflectivity of the mirrors. However, a defect mechanism has been studied that can potentially harm even protected silver coatings with pinhole-free protective layer. Hygroscopic particles containing Cl and/or S at the surface can harm the protective layer if the mirror is stored in a humid atmosphere. In a later stage a Cl or S-containing solution can permeate the protective layer and damage the silver film locally. The understanding of this defect mechanism is important in order to improve storage conditions for silver mirrors and provides valuable information about possibilities to further improve the environmental stability of protected Ag coatings.

Protective coatings consisting of at least two different layer materials and designed according to a multilayer approach can considerably improve the environmental stability of protected silver coatings. This was demonstrated in an experiment based on artificial contamination of different protected silver coatings in order to qualitatively compare their stability. At the same time this multilayer can potentially be used to increase the reflectivity of protected Ag coatings in the wavelength range 350 nm to 450 nm.

SiO_2 and Al_2O_3 coatings were prepared using the PEALD technique. The films were homogeneous in z-direction and showed no detectable losses within a spectral range from 250-1100 nm. The refractive indices of the films were close to the indices known from established PVD techniques. The investigated Al_2O_3 coatings were dense without open pores while for SiO_2 films further developments are necessary in order to achieve shift-free SiO_2-coatings. Due to their good optical properties and the fact that ALD coatings are known to be nearly pinhole-free, ALD coatings are not only suitable for the deposition of optical coatings on substrates with complex shape, but also very promising for the effective protection of high-reflecting silver coatings.

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