Antireflection Coating Production Process without an E-Beam Source and without Scattering

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ABSTRACT

The most common antireflection coatings in the industry today use electron beam sources for their deposition which require a large capital investment and have some other undesirable characteristics. These characteristics might include: lack of uniformity and reproducibility due to melt-in variations, sweep variations, and rate instabilities. The predominant low index material used throughout the world is silicon dioxide, which also can have uniformity and reproducibility problems. A production process is described which uses only resistance sources, which are much less complex, are more reproducible, and are easier to control. Magnesium fluoride is a most desirable material to use because of its low index of refraction and reproducible deposition results, but it tends to have scattering and stress problems. It is possible to reduce the scattering and stress by interjecting thin high index layers into the thicker MgF_2 layers. These thin layers can be accommodated in the "normal" designs without significant effect on the spectral performance. Experiments to optimize these "lamination" processes and designs are described.

INTRODUCTION

The goal of this work has been to minimize the capital investment needed to produce good antireflection (AR) coatings for the visible spectrum on glass substrates in a chamber without an electron beam evaporation source or an ion source. The most common practice in the world at this time for ARs in the visible spectrum appears to be the deposition of SiO_2 for the low index layers and high index layers of TiO_2, ZrO_2, or Ta_2O_5 from an E-gun. The high index materials can be evaporated from resistance sources (boats), but a satisfactory process for evaporating SiO_2 from a boat has yet to be reported. If a proper ion source is available, SiO can be evaporated from a resistance source and converted to SiO_2 by oxygen ion assisted deposition (IAD) [1]. However, an IAD source requires a capital expenditure of the same order as that for an E-gun. Magnesium fluoride (MgF_2) has been used here for the low index material because it evaporates easily from a resistance source and it has the lowest index of practical materials in the visible region. The high index material used here is Dralo [2].

PROBLEMS

The evaporation of SiO_2 from an E-gun has uniformity and reproducibility problems. MgF_2 can have stress and scattering problems.

SiO_2 (silica) is commonly evaporated from its granular form in an E-gun. We had found that the deposition of critical optical thin film stacks with silica from an E-gun is severely limited by the stability of the evaporation pattern or angular distribution of the material [1]. As illustrated in Figure 1, the amount of material deposited on a central monitor chip or control crystal in a coating chamber did not have a reproducible ratio to that received at other positions in the chamber. The evaporated "cloud" may be broad or narrow and may not necessarily be normal to the general surface of the evaporating material if there is any "tunneling." This is explained by the erratic melting/evaporation of silica surfaces in both granular and solid disc forms of fused silica (FS).

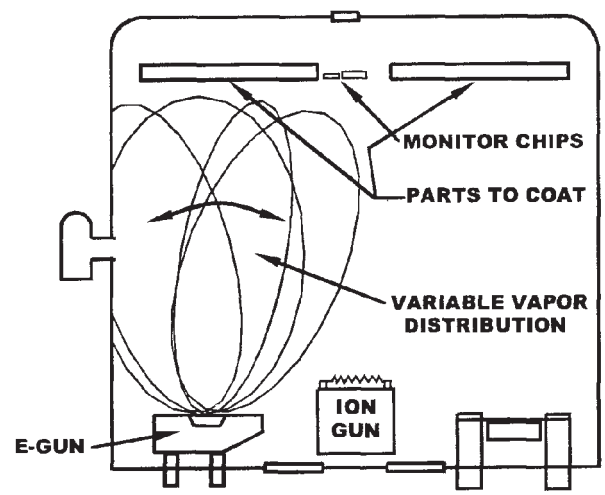

Figure 1: The variable distribution of silicon dioxide evaporant from an E-gun as is commonly experienced in physical vapor deposition.

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The hypothesis for this unstable behavior is illustrated in Figure 2. The surface of the silica is locally heated with an E-gun by the electrons impinging from above in Figure 2, and the surface becomes white hot. A great deal of the impinging energy is dissipated in the form of heat radiated by the white hot surface. If this surface is primarily horizontal and upward facing, as in the left side of Figure 2, the energy will radiate into the relatively colder environment of the chamber and be lost from the silica. However, if a groove has formed in a flat surface or between granules of silica, as in the right side of Figure 2, much of the radiating energy will fall on the adjacent radiating face. This results in less radiant energy loss in the groove so that it gets still hotter and more material will be evaporated from these surfaces. Two effects of this can be seen. One is that the groove will tend to get deeper or "tunnel," and the other is that the direction in which the evaporating silica travels will be quite different from that of the flat horizontal surface on the left side of Figure 2.

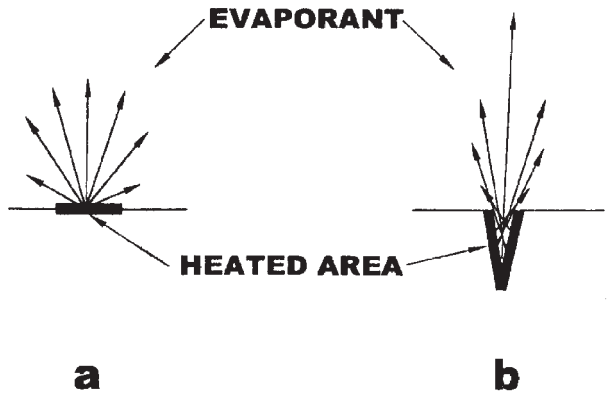

Figure 2: The vapor distribution of silicon dioxide evaporant from a flat surface of SiO_2 versus a groove or tunnel where the radiant heat is confined and causes extra heat and a higher evaporation rate.

Our preferred solution to this SiO_2 problem is the deposition of SiO from a resistance source as in Figure 3 and with IAD using oxygen and argon [1] to produce SiO_2. However, this approach requires an ion source and therefore does not support the goal here of minimizing the capital investment.

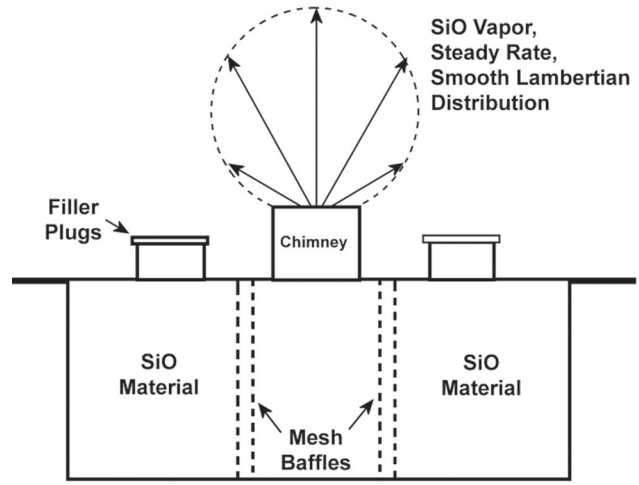

Figure 3: The vapor distribution of silicon monoxide from a resistance source is Lambertian, steady, and reproducible.

Much of the problem with stress in MgF_2 coatings appears to come from its high coefficient of thermal expansion (CTE) which is at an average of ~11.5×10^{-6}/°K as compared to ~$6\text{-}8\times10^{-6}$/°K for most glass substrates. There is also the possible influence of the strong difference in the CTE on its crystal axes (8.9 versus 14.0×10^{-6}/°K). MgF_2 is commonly evaporated onto substrates heated to 250-300°C in order to obtain dense enough films to withstand abrasion. When these coatings and substrates cool to room temperature, the MgF_2 film contracts more than the substrate, and the film is then under tensile stress. MgF_2 on a FS substrate with a CTE of 0.55×10^{-6}/°K should be an extreme case of such a CTE mismatch. This tensile stress can result in cracking of the coating and causing scattering. The stress can also cause warping or bending of the substrates. The effects of CTE mismatch could be reduced if the process were at room temperature as demonstrated by Selhofer, et. al. [4]. Such a film would have poor abrasion resistance. A low-temperature MgF_2 process can be achieved with an ion source in order to have reasonable durability as in the process reported earlier [5]. The high temperature process used in the current work also excludes the use of plastic substrates.

SOLUTION

With no E-gun and no Ion-Source, the solution used here for high- and low-index materials was Dralo [2] and MgF_2 evaporated from tungsten resistance sources (boats). Their indices are about 2.2 and 1.37 at 550 nm. Dralo is TiO_2 mixed with some alumina, which lowers stress and provides smoother evaporation. Pulker et. al. [3] did much of their work with TiO_2 from tungsten boats, but they indicated that tantalum boats might be preferred.

The chamber used here was a Balzers BAK760. The process temperature was 290°C and the start pressure was $1.5\text{-}2.0 \times 10^{-5}$ mbar. The deposition rate for the Dralo was 0.15 nm/second with an oxygen flow of 70 SCCM which gave a background pressure of $3.2\text{-}3.3 \times 10^{-4}$ mbar. The rate for the MgF_2 was 1.0 nm/second.

Figure 4 shows the actual spectral result of an AR coating with the process reported here. The coatings showed no significant scattering or stress on substrates like N-BK7 and FS.

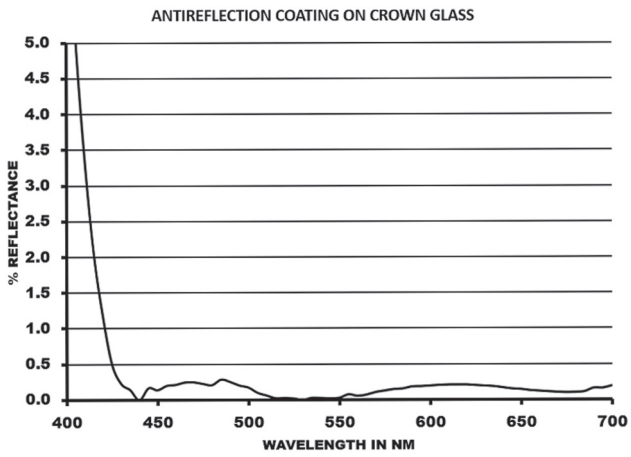

Figure 4: Spectral reflectance of AR coating using process described.

STRESS AND SCATTERING

Test runs were made to characterize stress and scattering. All runs were done at 290°C with FS, H-K9L (N-BK7), and H-ZF2 substrates of 25.4 mm diameter and 1.1 mm thickness. The CTE of FS is 0.55×10^{-6}/°K , N-BK7 is 8.3×10^{-6}/°K, and H-ZF2 is 8.8×10^{-6}/°K. The FS substrates are expected to show the most stress, crazing, and scattering because of their greater CTE difference between the MgF_2 versus the N-BK7 and H-ZF2. Tests were done with single layers of MgF_2 of thicknesses 50, 100, 200, and 400 nm. The thickest layer in the AR coating of Figure 4 is less than 100 nm. No significant scattering was seen in the AR coating.

To test the effects of the insertion of thin high index layers into the thicker MgF_2 layers to reduce stress, samples were made with structures of 200L 10H 200L, 200L 5H 200L (3-layer), 133L 5H 133L 5H 133L (5-layer), and 100L 5H 100L 5H 100L 5H 100L (7-layer). These were each a total thickness of MgF_2 of approximately 400 nm, where L represents MgF_2 and H is for Dralo. Figure 5 illustrates the structures of these layer stacks.

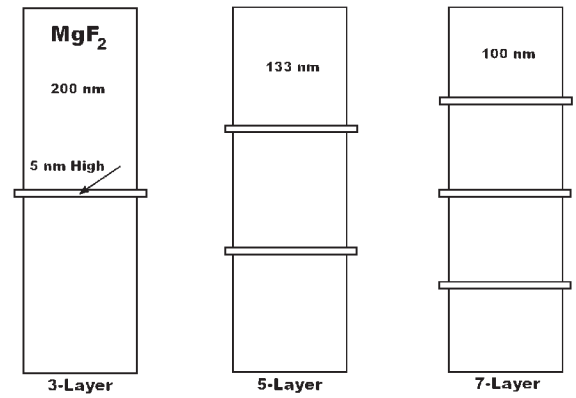

Figure 5: Layer structures of 400 nm stacks of MgF_2 with 5 nm layers of Dralo inserted.

The equipment to quantify the stress and scattering of these samples was not available to the authors at this time; further testing is anticipated. Qualitative observations of these test samples were made under 150 watt fiber optic light source illumination and 3X eye loupe inspection.

1. The 100 nm MgF_2 film on all substrates was smooth without any visible granular microstructure.
2. The 5-layer test (133/5…) showed: FS-Faint regular (fine) granular microstructure; H-BK7-Very faint (barely visible) granular microstructure; H-ZF2-Smooth film (like 100 nm MgF_2), no microstructure visible.
3. The 7-layer test (110/5…) showed: FS-Pronounced granular microstructure, H-BK7-Faint (fine) granular; H-ZF2-Faint (regular) granular microstructure.

These observations are consistent with the hypotheses that: 1) MgF_2 films at 100 nm thickness or less are smooth with stress low enough to avoid scattering; 2) substrates with high CTE have less structure and scattering problems than low CTE substrates; and 3) insertion of thin high index layers into thick MgF_2 layers effects the microstructure (further work needed here).

If a coating design requires thick MgF_2 layers and the production of the design has stress and scattering problems, the design of the thick layers can be adjusted [6] to have nearly the same spectral performance while reducing stress and scattering.

CONCLUSIONS

It has been demonstrated that good AR coatings for the visible spectrum on glass can be produced without E-guns or Ion-sources. Stress and scattering in such coatings does not

appear to be a problem. The mismatch of CTE between the MgF_2 and the substrates, and the deposition temperature used for the combination, are major factors in the residual stress of the coating-substrate system. The insertion of thin high index layers in thick MgF_2 coating layers can effect stress and scattering.

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