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Indium Tin Oxide-Magnesium Fluoride Co-Deposited Films for Spacecraft Applications

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INDIUM TIN OXIDE-MAGNESIUM FLUORIDE CO-DEPOSITED FILMS FOR SPACECRAFT APPLICATIONS

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ABSTRACT

Highly transparent coatings with a maximum sheet resistivity between 10^8 and 10^9 ohms/square are desired to prevent charging of solar arrays for low Earth polar orbit and geosynchronous orbit missions. Indium tin oxide (ITO) and magnesium fluoride (MgF_2) were ion beam sputter co-deposited onto fused silica substrates and were evaluated for transmittance, sheet resistivity and the effects of simulated space environments including atomic oxygen (AO) and vacuum ultraviolet (VUV) radiation. Optical properties and sheet resistivity as a function of MgF_2 content in the films will be presented. Films containing 8.4 wt.% MgF_2 were found to be highly transparent and provided sheet resistivity in the required range. These films maintained a high transmittance upon exposure to AO and to VUV radiation, although exposure to AO in the presence of charged species and intense electromagnetic radiation caused significant degradation in film transmittance. Sheet resistivity of the as-fabricated films increased with time in ambient conditions. Vacuum heat treatment following film deposition caused a reduction in sheet resistivity. However, following vacuum heat treatment, sheet resistivity values remained stable during storage in ambient conditions.

1. INTRODUCTION

When a spacecraft in geosynchronous orbit (GEO) encounters a solar substorm environment, the spacecraft is exposed to a plasma with particle energies between 1 and 50 keV [1]. Because of the variety of materials comprising spacecraft surfaces, differential charging of different materials on spacecraft surfaces in GEO due to plasma exposure can result in the formation of locally high electrical fields leading to arcing. The Marecs-A spacecraft, operating in geosynchronous orbit, experienced a power loss on part of its solar arrays due to charging and arcing [2]. High voltage spacecraft in low Earth orbit (LEO) are also subject to plasma interactions leading to charging [2, 3]. For example, the Upper Atmospheric Research Satellite (UARS) has experienced difficulty interpreting instrument data due to charging of its solar arrays [2]. Other problems that can occur are ion sputtering damage [2, 3] and enhanced contamination whereby contaminants are attracted to charged surfaces and deposit onto them. [1, 2, 3] In order to prevent these problems, a coating that is slightly conductive has been proposed for use on spacecraft solar arrays that are vulnerable to charging problems such as those flown in geosynchronous orbit and low Earth polar orbit [4]. It is necessary that such a coating have a maximum sheet resistivity of 10^9 ohms/square for GEO applications [1] or a maximum sheet resistivity of 10^8 ohms/square for LEO polar applications [5]. The coating must have a high transmittance to minimize impact on solar cell performance. It must also be environmentally durable to solar radiation, thermal effects, and, for LEO applications, atomic oxygen. Indium tin oxide films have been recommended as conductive coatings for solar cell applications. [6, 7] However, ITO alone has been shown to undergo optical properties degradation in LEO [8] and in the simulated space environment of an RF plasma asher where samples were exposed to a plasma containing AO, charged species and electromagnetic radiation [5]. Protection or modification of ITO is required to prevent its degradation in the LEO environment.

Ion beam sputter deposition from targets composed of at least two materials has been investigated for purposes of tailoring film properties [9]. Because conventional spacecraft solar cell assemblies have used magnesium fluoride as an antireflective, external surface coating [10], it is an appropriate candidate for co-deposition with ITO to obtain a transparent, slightly conductive, LEO-durable coating. This paper investigates ion beam sputter co-deposited films of ITO and MgF_2. Films were ion beam sputter co-deposited onto fused silica substrates. Transmittance and sheet resistivity were measured as a function of calculated MgF_2 content in the films. Film sheet resistivity was monitored as a function of time after deposition, and after vacuum heat treatment. Screening tests were conducted to determine the effects of AO and VUV radiation on film optical properties.

2. EXPERIMENTAL PROCEDURES

2.1 Film Deposition

Mixed films of ITO and MgF_2 were ion beam sputter co-deposited onto fused silica substrates. Figure 1 shows the configuration of the dual ion beam sputter deposition system inside its high vacuum facility including an example of the configuration of the mixed target for co-deposition of MgF_2 (Material A) and ITO (Material B). The ITO target was comprised of 91 mole percent In_2O_3 and 9 mole percent SnO_2. The MgF_2 content in the deposited films was varied by using between 30 and 130 degrees total included angle of pie-shaped wedges of MgF_2 placed on the ITO target. Wedges were each 20 to 30 degrees included angle and were evenly spaced over the ITO target to provide the most uniform deposition over the substrate area. The hypothetical volume percent of MgF_2 in the deposited film was calculated as shown in Equation 1 where Θ represents the total included angle of the pie shaped wedges and R represents the deposition rate for each target material. Deposition rates for MgF_2 and ITO are 2.5 and 4.0 nm/min, respectively. Weight and mole percent composition were also calculated using density and molar weight of ITO and MgF_2. Table 1 shows the calculated compositions of MgF_2 for the various target configurations used.

Film deposition procedures were as follows: first, the 2.5 cm diameter ion source was used for a

$$\%\, MgF_2 = \frac{\Theta_{MgF_2} R_{MgF_2}}{\Theta_{MgF_2} R_{MgF_2} + \Theta_{ITO} R_{ITO}} \qquad [1]$$

5 to 20 minute sputter cleaning of the target. Then the 15 cm diameter ion source was used for a 2 minute sputter cleaning of the substrates. Finally, the 2.5 cm source was used to deposit the film onto the substrates. The ion sources were operated using argon. Additionally, either pure dry air or an RF discharge of pure dry air was flowed into the chamber during deposition to promote deposition of an oxide-rich film. Various flow rates of air were used to determine the effect on film properties. As air flow rate was increased, argon flow rate was decreased to maintain the desired chamber pressure during deposition. Air flow was measured in standard cubic centimeters per minute (sccm). Use of the RF air discharge provided more reactive oxygen species, atoms and ions, than molecular oxygen flowed directly into the chamber. The effect of these different types of oxygen environments on film properties will be discussed. System pressure was approximately 6.67 mPa ($5x10^{-5}$ torr) with no gas flowing into the chamber and approximately 40 mPa ($3x10^{-4}$ torr) during deposition.

2.2 Film Thickness and Optical Properties Characterization

To fabricate samples for film thickness measurement and optical properties measurement, fused silica optical flat substrates, 2 cm by 2 cm, were partially covered with polyimide tape prior to deposition. After deposition, the tape was removed and the surfaces were scanned with a surface profiler to measure the step change between the tape-covered and coated surfaces. Film thicknesses ranged between 20 and 110 nm.

Transmittance spectra in the 250-2500 nm wavelength range were obtained using an ultraviolet-visible-near infrared spectrophotometer equipped with a 15 cm diameter integrating sphere. Total solar transmittance values over this wavelength range were calculated using standard procedures [11].

2.3 Sheet Resistivity Characterization

To fabricate samples for sheet resistivity measurements, 1 cm by 2 cm fused silica substrates were covered with an aluminum mask prior to deposition to produce a bar-shaped deposited film measuring 0.3 cm by 1.9 cm with three contact arms along the edges to permit four-lead resistance measurements to be made. Electrical contact to the film was made with spring-loaded pressure contacts. Electrical measurements were made with direct currents of 5 pA to 10 mA using guarded, shielded cabling and high-impedance electrometers to measure resistive voltages. Most electrical measurements were made in the ambient laboratory atmosphere. Some samples were measured in a vacuum of approximately 5.3 Pa (40 mtorr). Three samples underwent vacuum heat treatment by heating to 400°C for 1 hour in a vacuum of 0.13 mPa (10^{-6} torr) to determine effects on sheet resistivity. Sheet resistivity is defined in Equation 2 as

$$\rho/t = Rw/\ell \qquad [2]$$

where ρ is the resistivity of the specific material, t is thickness, R is measured resistance, w is width, and ℓ is length of a resistor or conductor in sheet form. The term ρ/t is referred to as the sheet resistivity given in units of ohms/square ($\Omega/\square$), and ℓ/w is referred to as the number of "squares."

2.4 Screening Tests For Space Environmental Durability

Samples of deposited films on fused silica substrates were exposed to AO in screening tests to determine the effect of AO on the transmittance of ITO films compared to that of mixed ITO-MgF_2 films. An RF plasma asher which generates a 13.56 MHz RF-induced discharge of air in a partial vacuum of 20 Pa (150 mtorr) was used to simulate the atomic oxygen environment of low Earth orbit. A Kapton H polyimide witness coupon was exposed along with the samples, and the effective AO fluence was measured based on the mass loss of Kapton H which has a known in-space erosion yield of 3×10^{-24} cm^3/atom [12]. Samples were exposed to the RF plasma environment using the following procedure: first, samples were located inside of a Faraday cage comprised of an aluminum foil box with louvers so that samples received scattered atomic oxygen exposure, but were shielded from line-of-sight exposure to charged species and intense electromagnetic radiation. Samples were then characterized for total solar transmittance. Following transmittance characterization, the same samples were then exposed directly in the plasma where they received exposure to electromagnetic radiation, including intense VUV radiation [13], and charged species [13] in addition to AO. Following exposure, total solar transmittance of samples was measured.

Samples of mixed ITO-MgF_2 films deposited onto fused silica substrates were also screened for durability to VUV radiation. Samples were exposed to a maximum of 3 VUV suns from a deuterium lamp in the 115 to 200 nm wavelength range. This VUV deuterium lamp was

installed inside of a water-cooled copper chamber in a cryopumped high vacuum system. Pressure during exposure was approximately 1.2 mPa ($9x10^{-6}$ torr) and samples reached a maximum temperature of 120°C. The lamp was calibrated with a cesium-iodide phototube with a MgF_2 window sensitive in the 115-200 nm wavelength range. This phototube had been calibrated to a deuterium lamp of the same type which had been calibrated by the National Institute of Standards and Technology.

3. RESULTS AND DISCUSSION

3.1 Transmittance and Sheet Resistivity of As-Fabricated ITO-MgF_2 Films

Figure 2 shows total solar transmittance of samples of films deposited onto fused silica substrates as a function of calculated percent of MgF_2 in the film. Samples were fabricated with an average deposited film thickness of approximately 65 nm. The total solar transmittance of the fused silica optical flat substrates is approximately 0.93. As shown in the figure, as the MgF_2 content of the film is increased, the transmittance of the samples increased. Error in total solar transmittance values is ± 0.005. Figure 3 shows sheet resistivity of the deposited film as a function of MgF_2 content. These data show that a maximum of approximately 9.2-10 wt.% MgF_2 is necessary for mixed ITO-MgF_2 films with a maximum sheet resistivity between 10^8 and 10^9 ohms/square required for the arc-proof coating. Error in sheet resistivity measurements is ± 20%.

3.2 Effect of Air Background Gas During Deposition on Film Properties

Table 2 shows properties of ITO-8.4 wt.% MgF_2 films on fused silica substrates as a function of air flow during deposition. For films of 27-35 nm thickness prepared using various flow rates of pure dry air, sheet resistivity varied between 10^7 and 10^{10} ohms/square, and films were highly transparent. Varying the flow rate of pure dry air in the chamber did not appear to produce a significant effect on optical or electrical properties as shown in Table 2. However, use of an RF discharge of air produced samples with higher resistivity than those produced using a background of pure dry air. A sample with a 29 nm deposited film prepared using an RF discharge of air showed a higher sheet resistivity, 10^{10} to 10^{11} ohms/square, than samples prepared using air alone. Oxygen partial pressure during deposition has been shown to have an effect on the resistivity of ITO and indium oxide films [7, 14]. Reference 7 reports that at levels of oxygen partial pressure greater than approximately 13.3 mPa (10^{-4} torr), increasing oxygen partial pressure decreases oxygen vacancies in the depositing film resulting in increased resistivity. Increasing the reactivity of the oxygen at the same partial pressure, as occurs when using an RF discharge of air instead of pure dry air, should also produce the effect of decreasing oxygen vacancies and thus increasing resistivity as is observed in Table 2. Based on this observation, one would expect the increase in flow rate of pure dry air to also produce an increase in sheet resistivity. However, because the chamber background pressure remained essentially unchanged regardless of the pure dry air flow rates used, and because air contains only 21% oxygen, it is likely that increasing the flow rate of pure dry air did not produce a significant enough increase in the oxygen partial pressure to cause an increase in the sheet resistivity of the deposited films. This lack of a relationship between pure dry air flow rate and sheet resistivity of deposited films is evident in Table 2.

3.3 Sheet Resistivity Stability of ITO-MgF_2 Films in Ambient Conditions

Figure 4 shows sheet resistivity as a function of time after deposition for three identically fabricated samples of ITO-8.4 wt.% MgF_2. In general, sheet resistivity appears to increase over time after deposition and is somewhat unstable, sometimes changing an order of magnitude over a short period of time. Storage of samples under vacuum of 5.3 Pa (40 mtorr) did not stabilize sheet resistivity.

Irreversible resistivity changes have been observed in lead oxide films by heating in vacuum or hydrogen to 400°C or in oxygen or air to 450°C [15]. To determine whether this effect would also occur in and stabilize ITO-MgF_2 films, vacuum heat treatment was performed on these films. Figures 5a and 5b show the results of vacuum heat treatment of deposited films of ITO-2.4wt.% MgF_2 (21 nm film thickness) and ITO-8.4 wt.% MgF_2 (27 nm film thickness) on fused silica samples as compared with samples prepared in the same deposition batches that were not heat treated. Heating samples for 1 hour to 400°C in a vacuum of 0.133 mPa (10^{-6} torr) caused a reduction in sheet resistivity between one and four orders of magnitude. Following this treatment, sheet resistivity values remained significantly more stable as shown in Figure 5. Although these data imply that heat treatment decreases and stabilizes sheet resistivity, the resistivity stabilization of the heat treated samples may be simply due to the lower value of sheet resistivity.

Deposited films of greater thickness than those described in this paper may have more stable sheet resistivities. Electrical properties of transparent conducting oxides are known to be influenced by deposited film thickness due to the effects of increasing grain size in thicker films [16].

Samples were not prepared for analysis of the effects of heat treatment on film optical properties. However, it is expected that the lowered resistivity induced by heat treatment would also decrease the transmittance of the deposited film due to the increasing metallic nature of the film.

3.4 Space Environmental Durability Screening Tests

A sample of ITO-8.4 wt.% MgF_2 of 30 nm thickness on fused silica was exposed to atomic oxygen in an RF plasma chamber. During the first part of the exposure, the sample was located inside a Faraday cage aluminum box with louvers. This allowed scattered atomic oxygen exposure, but prevented line-of-sight radiation exposure. The transmittance spectrum was essentially unchanged after exposure to a Kapton effective atomic oxygen fluence of $5x10^{21}$ atoms/cm^2 in the Faraday cage as shown in Figure 6. This atomic oxygen fluence is representative of 1.5 years on the International Space Station for solar array surfaces. Subsequent exposure of this sample directly in the plasma to a Kapton effective atomic oxygen fluence nearly an order of magnitude less, $8x10^{20}$ atoms/cm^2, resulted in significant degradation in transmittance. This atomic oxygen fluence is representative of three months on the International Space Station for solar array surfaces. Compare this degradation to that of ITO as shown in Figure 7. The ITO film also showed durability to atomic oxygen when exposed in a Faraday cage to a Kapton effective fluence of $5x10^{21}$ atoms/cm^2 . However, also as shown in Figure 7, the ITO film was much more severely degraded than the mixed film shown in Figure 6 upon

exposure in the plasma to an effective atomic oxygen fluence of $8x10^{20}$ atoms/cm^2 indicating that the ITO-MgF_2 mixed film is more durable to the RF plasma environment than ITO alone. The RF plasma environment produces an intensity of vacuum ultraviolet radiation which is one to three orders of magnitude higher than the LEO environment. [13] It also produces an electron density which is three to seven orders of magnitude greater than LEO with electrons of higher energy. [13] Therefore, it is likely that the degradation due to plasma exposure for ITO and ITO-MgF_2 films shown in Figures 6 and 7 is due to an unrealistically severe environment. However, the comparison between the ITO and ITO-MgF_2 films does give a valid indication of the increased durability of the mixed film in the RF plasma environment.

Figure 8 shows the transmittance spectra before and after exposure of a sample of ITO-8.4 wt.% MgF_2 before and after exposure to 250 equivalent sun hours of vacuum ultraviolet radiation at an intensity of three vacuum ultraviolet suns in the wavelength range between 115 and 200 nm. This exposure caused essentially no change in the transmittance of the film as shown by the total solar transmittance values before and after exposure of 0.925 and 0.921, respectively.

In order to most accurately predict the long-term in-space durability of the ITO-MgF_2 films, longer duration AO and VUV testing is required. Also, it will be necessary to determine the effects of AO and VUV on sheet resistivity. Because of the instability of sheet resistivity in ambient conditions, the effects of AO and VUV on sheet resistivity were not evaluated as part of this effort. Further modifications to the film formulation will be made in an effort to stabilize sheet resistivity.

4. CONCLUSIONS

Co-deposition of indium tin oxide and magnesium fluoride using ion beam sputter deposition processes resulted in highly transparent films with sheet resistivity suitable for use on an arc-proof solar array. Sheet resistivity could be tailored by varying the composition of MgF_2 in the film. Mixed films of ITO-8.4 wt.% MgF_2 deposited onto fused silica substrates were fabricated with initially measured sheet resistivities between 10^7 and 10^{10} ohms/square. Total solar transmittance remained essentially unchanged upon exposure of these samples to atomic oxygen and to vacuum ultraviolet radiation in screening tests, although exposure to the combined environment of atomic oxygen, charged species and electromagnetic radiation present in an RF plasma environment caused degradation in the transmittance of this film. Transmittance degradation of the mixed film was not as severe as the degradation of an ITO film upon exposure in the same environment. Sheet resistivity of as-fabricated samples increased over time during sample storage in room air indicating instability in electrical properties. Vacuum heat treatment following film deposition caused sheet resistivity to decrease, in some cases by several orders of magnitude. However, following vacuum heat treatment, sheet resistivity values were considerably more stable during storage in room air. In general, mixed films of ITO-MgF_2 show promise for use on an arc-proof solar array; however, adjustments to thickness and composition may be needed to provide greater stability in electrical properties.

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TABLE 1 - SPUTTER TARGET CONFIGURATION AND COMPOSITION OF MIXED ITO-MgF_2 FILMS

Total MgF_2 wedge angle (degrees)	Calculated Composition		
	vol. % MgF_2	wt. % MgF_2	mole % MgF_2
30	5.4	2.45	9.68
60	11.1	5.20	19.0
90	17.2	8.37	28.1
110	21.6	10.8	34.1
120	23.8	12.1	37.0
130	26.1	13.4	39.9

TABLE 2 - PROPERTIES OF ITO-8.4 wt.% MgF_2 FILMS ON SiO_2 SUBSTRATES

Type of Air and Flow Rate During Deposition		Film Thickness (nm)	Sample Total Solar Transmittance	Sheet Resistivity ($\Omega/\square$)
Pure, Dry Air	0.6 sccm	~35	0.915	$\sim 10^9 - 10^{10}$
	1.0 sccm	~27	0.924	$\sim 10^7 - 10^{10}$
		~32	0.911	$\sim 10^8 - 10^9$
	1.25 sccm	~30	0.923	$\sim 10^8 - 10^9$
RF discharge of air, 1.25 sccm flowed into RF system		~29	0.913	$\sim 10^{10} - 10^{11}$

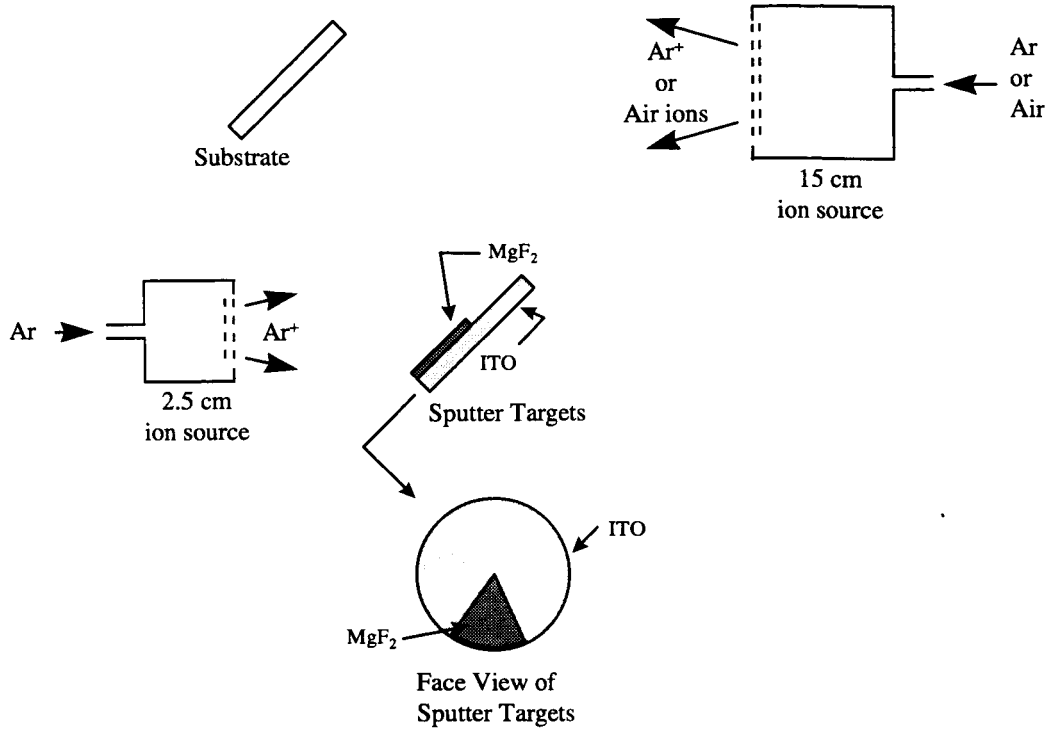


Figure 1: Configuration of ion sources, sputter targets and deposition substrates in dual ion beam sputter deposition system.

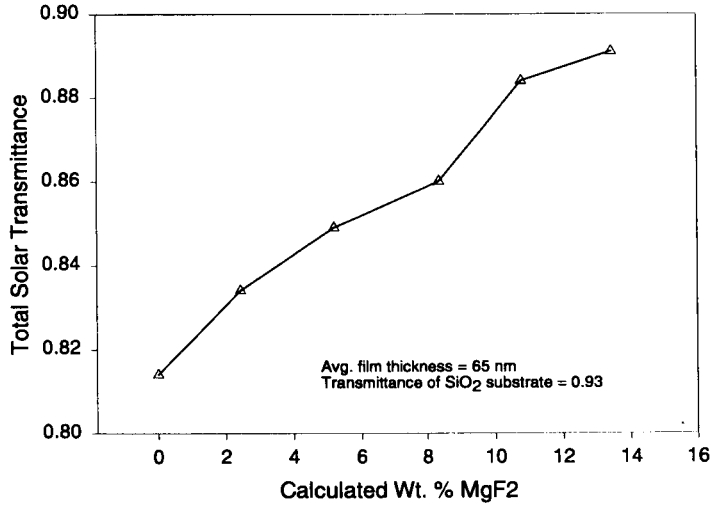


Figure 2: Total solar transmittance of samples of mixed ITO-MgF_2 films deposited on fused silica as a function of calculated percent of MgF_2.

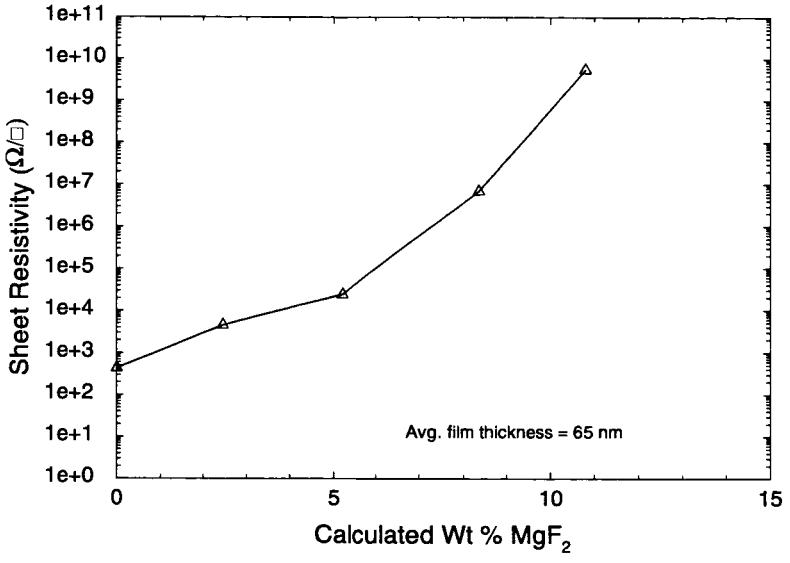


Figure 3: Sheet resistivity as a function of calculated percent of MgF_2 in mixed ITO-MgF_2 films.

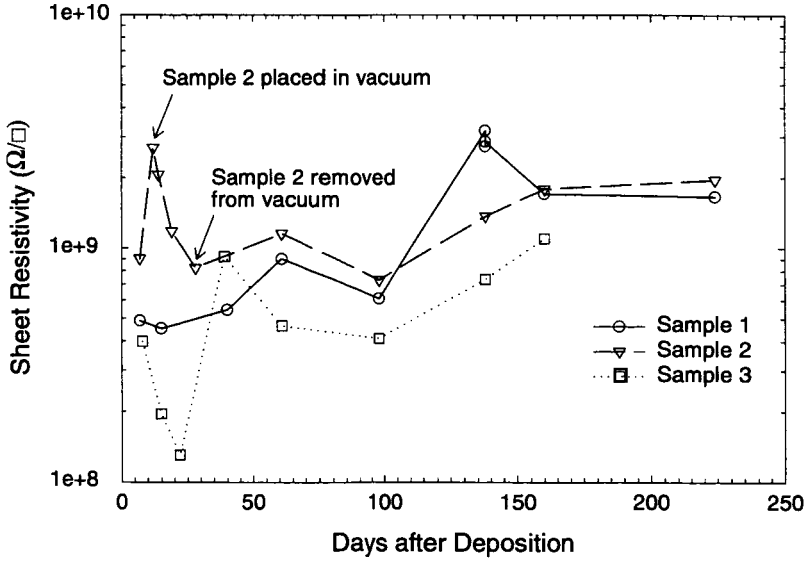
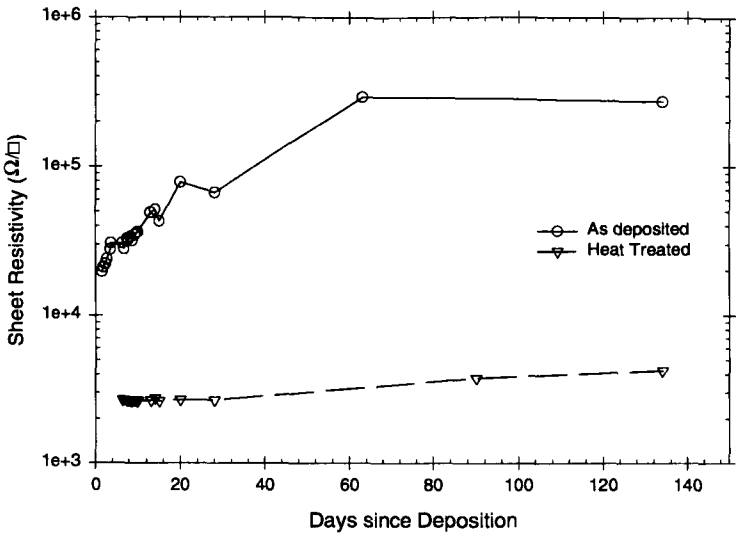
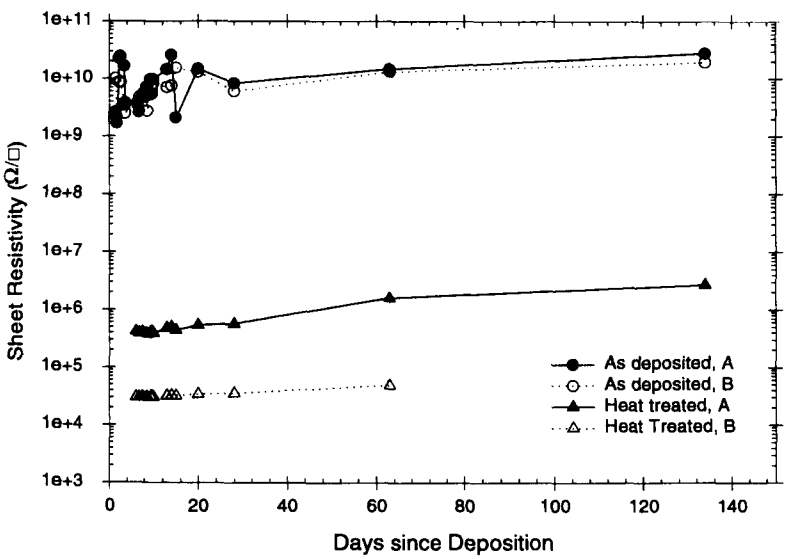


Figure 4: Sheet resistivity as a function of time after deposition for ITO-8.4 wt.% MgF_2 films.



(a)



(b)

Figure 5: Effect of vacuum heat treatment of 1 hr at 400°C on sheet resistivity of a) ITO-2.4 wt.% MgF_2 deposited films and b) ITO-8.4 wt.% MgF_2 deposited films.

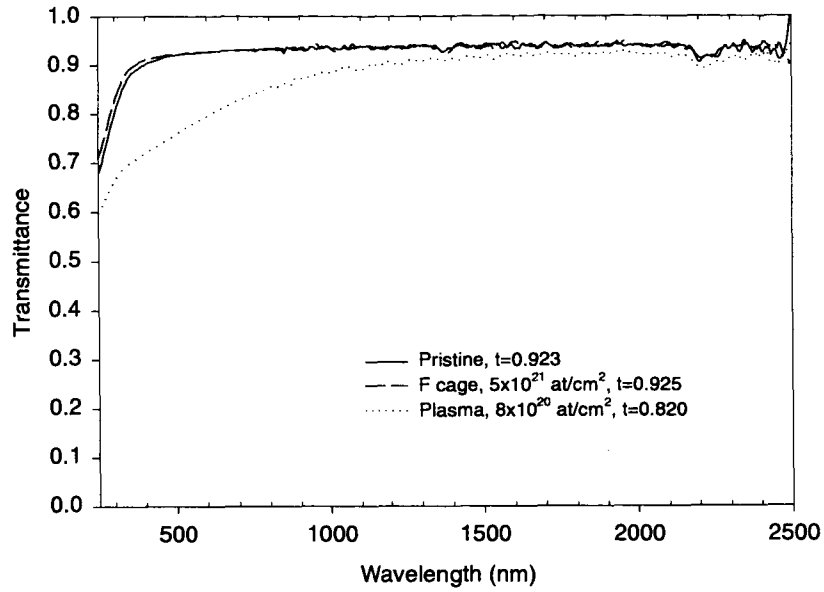


Figure 6: Effects of RF plasma exposure, in a Faraday cage and directly in the plasma, on transmittance spectra of ITO-8.4 wt.% MgF_2 film on fused silica.

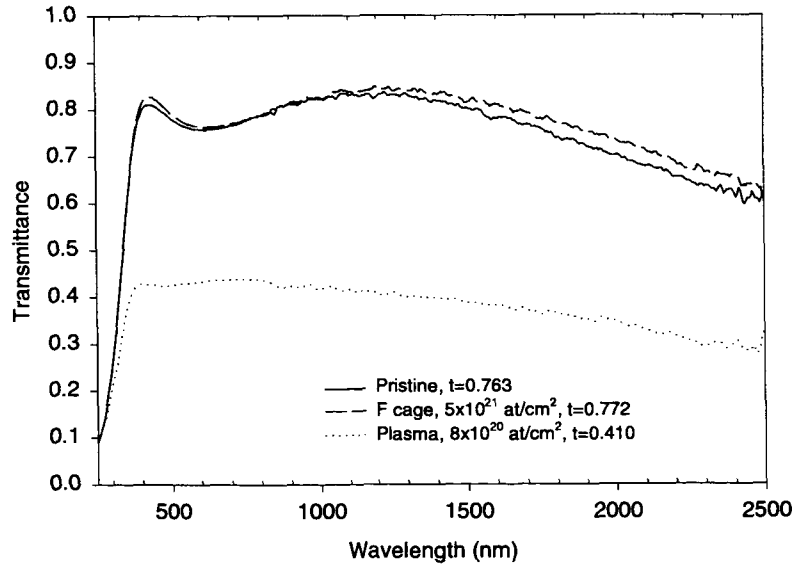


Figure 7: Effects of RF plasma exposure, in a Faraday cage and directly in the plasma, on transmittance spectra of a sample of an ITO film deposited on fused silica.

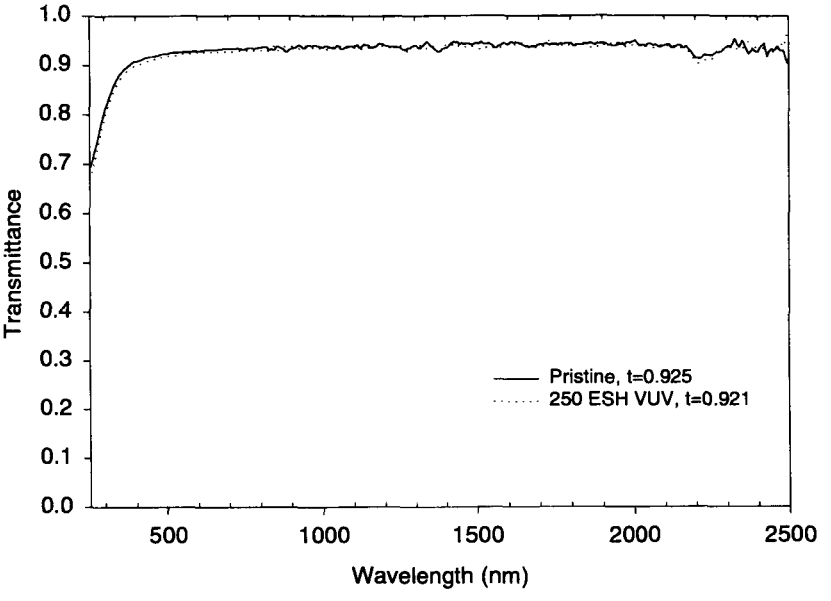


Figure 8: Effect of exposure to 250 equivalent sun hours of VUV radiation at 3 VUV suns on transmittance of a sample of ITO-8.4wt.% MgF_2 film deposited on fused silica.

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13. ABSTRACT ***(Maximum 200 words)***

Highly transparent coatings with a maximum sheet resistivity between 10^8 and 10^9 ohms/square are desired to prevent charging of solar arrays for low Earth polar orbit and geosynchronous orbit missions. Indium tin oxide (ITO) and magnesium fluoride (MgF_2) were ion beam sputter co-deposited onto fused silica substrates and were evaluated for transmittance, sheet resistivity and the effects of simulated space environments including atomic oxygen (AO) and vacuum ultraviolet (VUV) radiation. Optical properties and sheet resistivity as a function of MgF_2 content in the films will be presented. Films containing 8.4 wt.% MgF_2 were found to be highly transparent and provided sheet resistivity in the required range. These films maintained a high transmittance upon exposure to AO and to VUV radiation, although exposure to AO in the presence of charged species and intense electromagnetic radiation caused significant degradation in film transmittance. Sheet resistivity of the as-fabricated films increased with time in ambient conditions. Vacuum heat treatment following film deposition caused a reduction in sheet resistivity. However, following vacuum heat treatment, sheet resistivity values remained stable during storage in ambient conditions.

14. SUBJECT TERMS

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