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Transparent Conducting Thin Films for Spacecraft Applications

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TRANSPARENT CONDUCTING THIN FILMS FOR SPACECRAFT APPLICATIONS

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Abstract

Transparent conductive thin films are required for a variety of optoelectronic applications, automotive and aircraft windows as well as in solar cells for space applications. Transparent conductive coatings of indium-tin-oxide (ITO)—magnesium fluoride (MgF_2) and aluminum doped zinc oxide (AZO) at several dopant levels are investigated for electrical resistivity (sheet resistance), carrier concentration, optical properties, and atomic oxygen durability. The sheet resistance values of ITO-MgF_2 range from 10^2 to 10^{11} ohms/□, with transmittance of 75 to 86 percent. The AZO films sheet resistances range from 10^7 to 10^{11} ohms/□ with transmittances from 84 to 91 percent. It was found that in general, with respect to the optical properties, the zinc oxide (ZnO), AZO, and the high MgF^2 content ITO-MgF^2 samples, were all durable to atomic oxygen plasma, while the low MgF^2 content of ITO-MgF^2 samples were not durable to atomic oxygen plasma exposure.

Introduction

Transparent conducting thin film materials would have wide application for heat mirrors, opto-electronic devices,[1] gas sensors, automotive and aircraft windows. The use of solar cells for space applications also requires slightly conductive coatings which are resistant to degradation caused by atomic oxygen and ultraviolet (UV) radiation.

The development of a conductive protective coating is essential for the operation and efficiency of photovoltaic systems for use in space environments. Atomic oxygen is the most predominant environmental species from an altitude of 180 km (97 nmi) to 650 km (351 nmi)[2], and degradation by atomic oxygen and or UV radiation may cause optical property changes, thus affecting the performance of the system.

Another mechanism of degradation is surface charging, which might lead to pinhole formation in the protective coatings, as well as electronic interferences with the on-board spacecraft systems. A possible solution to this problem is to

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apply a surface coating material that is conductive, transparent, flexible, and resistant to atomic oxygen and UV radiation. To discharge surfaces that are being charged by space plasmas, a high resistivity to ground can be tolerated because the plasma charging currents are small.[3] Materials applied over a dielectric area must be grounded at the edges and must have a resistivity less than 10^9 "ohms per square" (Ω/□) for geosynchronous orbit. The draining of surface charge for low Earth orbit (LEO) polar spacecraft applications, requires a surface resistivity of 108 Ω/□.[4] Several candidate materials which might provide protection are transparent inorganic oxide coatings, and doped transparent inorganic oxide coatings.

The purpose of this research is to develop a conductive transparent protective coating whose properties are durable to the space environment. Indium-tin-oxide (ITO) has been recommended as a conductive surface coating on spacecraft.[3] However, ITO atomic oxygen durability studies indicate that the solar transmittance of ITO is reduced as a result of the exposure to atomic oxygen plasma,[4] (which also produces vacuum UV radiation). Other studies have also shown an increase of the ITO surface resistivity with atomic oxygen exposure.[4-6] Zinc oxide has been used in many applications due to its piezoelectric and pyroelectric properties.[1,7,8] Its properties, as well as low cost, makes ZnO very attractive as a transparent and conductive coating material.[9] Elements such as aluminum,[1,9-12] indium,[1] and fluorine,[13] have been used to dope zinc oxide to improve its optical and/or electrical properties.

In this paper we examine the surface resistivity (sheet resistance), carrier concentration, mobility, and optical properties of sputter deposited ITO-MgF_2, ZnO, and AZO. In addition we examine the atomic oxygen durability of these thin film materials.

Deposition of Thin Protective Coatings

An Ion Tech dual ion beam sputtering system was used to sputter deposit thin film protective coatings of ITO-MgF_2 from a water cooled, pressed powder target of In_2O_3 (91 percent) SnO_2 (9 percent) with a wedge of MgF_2 on the surface, a ZnO (98 percent) Al_2O_3 (2 percent) (AZO) target, ZnO target, and a ZnO with a wedge of Al were also used. The configuration of the ion source and target is illustrated in Fig. 1. The target was located approximately 20.3 cm downstream of the 2.5 cm argon ion source. Various size wedges of magnesium fluoride (MgF_2) were placed on the ITO target for simultaneous sputter deposition with the ITO. The same procedure was used for simultaneous sputter deposition of ZnO and Al. The 2.5 cm diameter ion source was operated at an ion beam energy of 1000 eV and an ion beam current of 35 mA. The water cooled substrate holder was located approximately 15.3 cm from the target and approximately 30.5 cm downstream of the 15 cm diameter source. To improve coating adherence, a 15 cm diameter ion source was operated with argon (Ar) at an ion energy of 250 eV and beam current of 35 mA to ion beam clean the substrates for 2 min prior to deposition. Air was also introduced during deposition of ITO-MgF_2. Thin film coatings were sputter deposited on fused silica substrates at an average ITO-MgF_2 deposition rate of 31 Å/min. Film thickness was measured on the fused silica substrates with a Sloan:Dektak IIA surface profiler. The film thickness ranged from approximately 900 to 1000 Å. The average deposition rate for AZO on fused silica substrates was 11 Å/min, the film thickness ranged from 200 to 650 Å.

A compromise between electrical conductivity and optical transparency is required. An increase in thickness of the conductive coating increases the electrical conductivity but it also decreases the optical transmittance.

Optical Properties

Optical measurements of coatings deposited on fused silica substrates were made with a Perkin Elmer Lambda 9 UV/VIS/NIR spectrophotometer operated with a 60 mm integrating sphere. Integrated solar transmittance and solar reflectance values were obtained by convoluting the spectral data into the air-mass-zero (AM0) solar spectrum. The accuracy of measurements are within ±2percent. Figure 2 illustrates the spectral transmittance for ITO-MgF_2, AZO and ZnO thin films on fused silica quartz. The transmittance of the ITO-MgF_2 coating increases with an increase in MgF_2 concentration. Spectral variations at approximately 820 nm and 1920 nm are due to the instrument filter changes.

The total transmittance and solar absorptance (α) for the thin films are shown in Table 1. The absorptance was calcu-

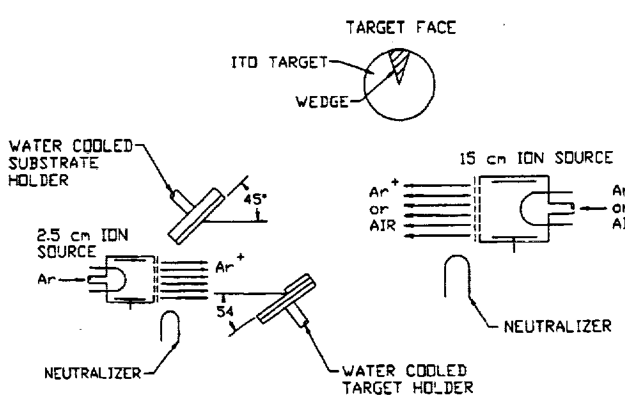


Figure 1.-Dual ion source sputter deposition system.

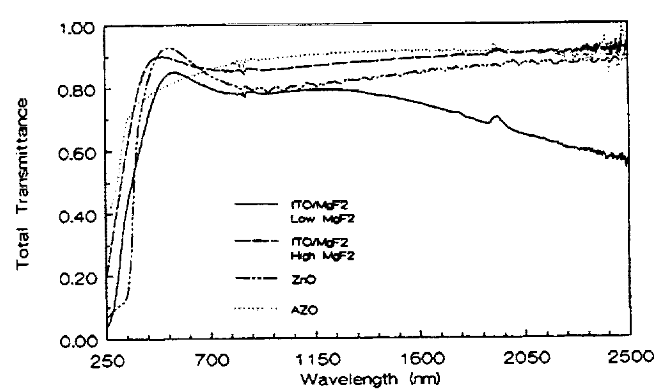


Figure 2.-Total spectral transmittance of thin films.

Table 1. Optical and Electrical properties of thin films

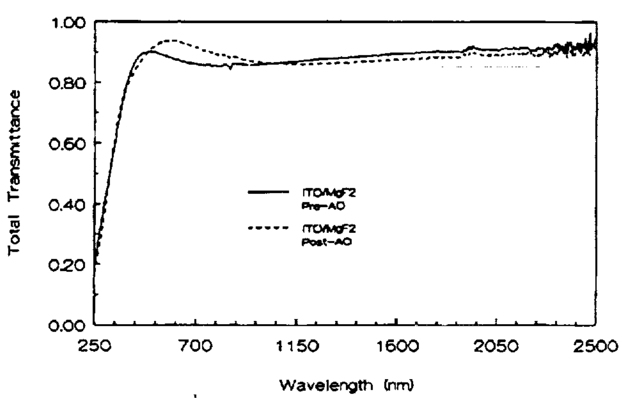
Thin Film	Total Transmittance	Solar Absorptance	Total Reflectance	Thickness (Å)	Sheet Resistance (Ω /□)
ITO	0.720	0.140	0.140	1000	10^{1}
ITO/MgF_2 (I - low)	0.750	0.130	0.120	1000	10^{2}
ITO/MgF_2 (II - high)	0.855	0.022	0.123	959	10^{9}
ITO/MgF_2 (III)	0.790	0.064	0.146	1115	10^{7}
ITO/MgF_2 (VI)	0.763	0.093	0.145	1042	10^{3}
ITO/MgF_2 (VII)	0.774	0.068	0.159	1158	10^{5}
AZO (II)	0.833	0.013	0.153	624	10^{7}
AZO (III)	0.906	0.012	0.082	200	10^{11}
ZnO	0.792	0.038	0.171	1068	10^{4}

lated from the reflectance and transmittance data. Values are typically an average of two to five samples. A decrease in is obtained with an increase of MgF_2 in the ITO films.

Atomic Oxygen Durability

Atomic oxygen exposure was conducted in a ground based-plasma facility (SPI Plasma Prep II). Atomic oxygen was generated in a small quartz vacuum chamber by RF (13.56 MHz) dissociation of air at a pressure of approximately 90 mtorr. The typical effective atomic oxygen flux during exposure was approximately 8×10^{15} atoms/cm^2-sec based on the erosion of Kapton in the plasma compared with known erosion in low Earth orbit (LEO).[14] Samples were exposed to atomic oxygen effective fluences between 5.4 and 6.4×10^{21} atoms/cm^2. Although the atomic oxygen flux is accelerated compared to what would be experienced in LEO, and the energy is lower (0.04 eV compared to 4.5 eV in LEO), plasma exposure can provide a good qualitative feel for material durability in LEO.

The changes in the spectral total transmittance of ITO-MgF_2 upon exposure to atomic oxygen can be seen by comparison on the plots shown in Fig. 3. Figure 3(a) shows the spectral total transmittance of ITO-MgF_2 (high MgF_2 concentration) before and after exposure to an atomic oxygen fluence of 5.39×10^{21} atoms/cm^2. Figure 3(b) shows the total spectral transmittance for a sample with a low dopant level of MgF_2 before and after atomic oxygen exposure to an effective fluence of 5.39×10^{21} atoms/cm^2 or to same effective fluence as high MgF^2. Figure 4 shows the total spectral transmittance for AZO (2 percent Al) before and after atomic oxygen exposure.



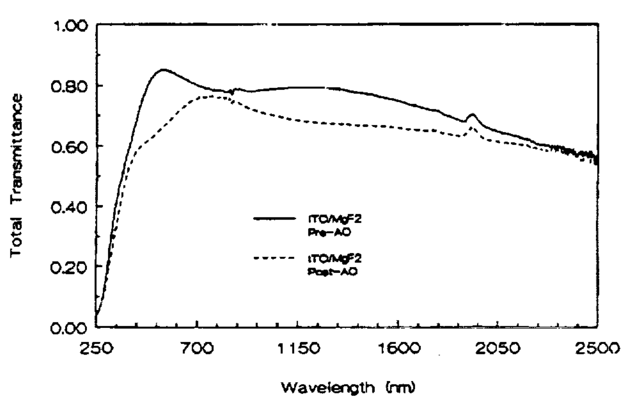


Figure 3.–Total spectral transmittance. (a) ITO/MgF (high MgF) Before and after atomic oxygen exposure. (b) ITO/MgF (low MgF) before and after atomic oxygen exposure.

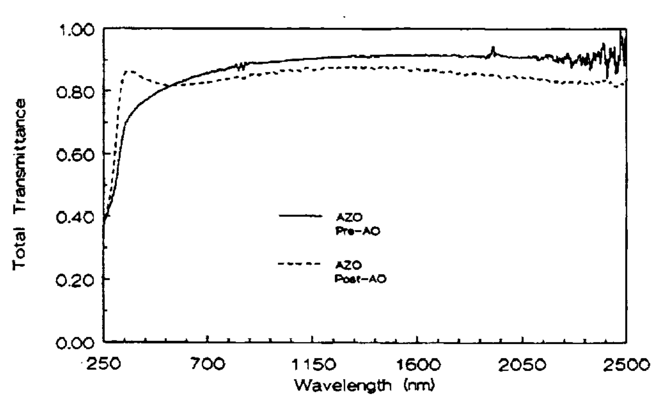


Figure 4.–Total spectral transmittance of AZO befroe and after atomic oxygen exposure.

Table 2. The integrated solar optical properties of samples before and after atomic oxygen exposure

Thin Film	Before AO			After AO		
	Transmittance	Reflectance	Absorptance	Transmittance	Reflectance	Absorptance
ITO-MgF_2 (I) (low % MgF_2)	τ_t: 0.748 τ_s: 0.741	ρ_t: 0.147 ρ_s: ——	α: 0.105	τ_t: 0.655 τ_s: -	ρ_t: 0.089 ρ_s: 0.083	α: 0.256
ITO-MgF_2(II) (high % MgF_2)	τ_t: 0.855 τ_s: 0.841	ρ_t: 0.123 ρ_s: 0.115	α: 0.022	τ_t: 0.870 τ_s: 0.860	ρ_t: 0.086 ρ_s: 0.076	α: 0.049
ITO-MgF_2 (III)	τ_t: 0.790 τ_s: 0.783	ρ_t: 0.146 ρ_s: 0.138	α: 0.064	τ_t: 0.846 τ_s: 0.835	ρ_t: 0.108 ρ_s: 0.096	α: 0.047
ITO-MgF_2 (IV)	τ_t: 0.842 τ_s: 0.834	ρ_t: 0.119 ρ_s: 0.112	α: 0.039	τ_t: 0.895 τ_s: 0.868	ρ_t: 0.085 ρ_s: 0.075	α: 0.020
ITO-MgF_2 (VII)	τ_t: 0.774 τ_s: 0.767	ρ_t: 0.159 ρ_s: 0.154	α: 0.068	τ_t: 0.657 τ_s: 0.652	ρ_t: 0.167 ρ_s: 0.162	α: 0.176
ITO-MgF_2 (VIII)	τ_t: 0.824 τ_s: 0.818	ρ_t: 0.145 ρ_s: 0.136	α: 0.032	τ_t: 0.816 τ_s: 0.809	ρ_t: 0.094 ρ_s: 0.089	α: 0.090
AZO (II)	τ_t: 0.833 τ_s: 0.821	ρ_t: 0.159 ρ_s: 0.149	α: 0.008	τ_t: 0.845 τ_s: 0.838	ρ_t: 0.136 ρ_s: 0.131	α: 0.019
ZnO (I)	τ_t: 0.792 τ_s: 0.777	ρ_t: 0.171 ρ_s: 0.157	α: 0.038	τ_t: 0.838 τ_s: 0.831	ρ_t: 0.116 ρ_s: 0.111	α: 0.046

The integrated solar optical properties (total transmittance (τ_t), specular transmittance (τ_s), total reflectance (ρ_t), specular reflectance (ρ_s) and absorptance) of all samples exposed to atomic oxygen, prior to and after exposure, are listed in Table 2. The ITO-MgF_2 samples with apparently lower MgF_2 content (I and VII) have initially lower total transmittance (0.748 and 0.774, respectively), similar to the ITO without any MgF_2 (0.720), and these samples are found to decrease in transmittance and increase in absorptance with atomic oxygen exposure. This behavior is very similar to the reaction of ITO to atomic oxygen plasma exposures.[4] Decreases in transmittance of ITO samples are generally attributed to the UV radiation which is present during plasma ashing.[4,5] The addition of greater amounts of MgF_2 to ITO (II and IV) not only results in greater initial transmittance (0.855 and 0.842, respectively), but these samples are found to increases in transmittance with atomic oxygen exposure (to 0.870 and 0.895, respectively). The solar absorptance of the lower MgF_2 content films (I and VII) increased from 0.105 and 0.068 to 0.256 and 0.176, respectively. While the absorptance of the higher MgF_2 content films (II and IV) increased and decreased, respectively. These changes in the optical properties of the high MgF_2 content ITO samples may be attributed to index of refraction changes associated with oxidation of the MgF_2. ITO-MgF_2 (II) increased from 0.020 to 0.049, while ITO-MgF_2 (IV) decreased from 0.039 to 0.020. Both the AZO and the ZnO films reacted similarly when exposed to atomic oxygen. Both the AZO (II) and the ZnO (II) films increased in total transmittance from 0.833 and 0.792 to 0.845 and 0.838, respectively, and increased in solar absorptance from 0.008 and 0.038 to 0.019 to 0.046, respectively. All samples except one (ITO-MgF_2 (VII)) experienced a decrease in both total and specular reflectance with atomic oxygen exposure. The sample which did not decrease in reflectance, increased only slightly, from a total reflectance of 0.159 to 0.167. In general, with respect to the optical properties, the AZO, ZnO, and the high MgF_2 content ITO-MgF_2 samples, were all found to be durable to atomic oxygen, while the low MgF_2 content ITO-MgF_2 samples were not durable to UV containing atomic oxygen environments.

Electrical Properties

Sheet resistance measurements were made on film samples using pressure contacts on wire leads attached with conductive silver paint. In general, alternating current (19.5 Hz) was used for samples with sheet resistance below 104 Ω/□, direct current was used for samples with higher resistance. Four-lead techniques were used whenever possible; however, some high-resistance samples were measured by two-lead methods. Power dissipation in samples was kept below 15 μW to avoid self-heating effects. Hall measurements were made by similar techniques in a magnetic field of 1.2 Tesla to obtain information on carrier concentration and mobility.

Our results on the electrical properties of ITO-MgF_2, ZnO, and AZO films are summarized in Table 3. The Hall data suggests that adding MgF_2 to ITO reduces carrier concentration and mobility, both increasing the resistance. This raises the question of stability, since the resistance of pure ITO films, prepared to have low carrier concentration and mobility, changes drastically when heated.

The sheet resistance of several samples were measured after atomic oxygen exposure, are shown in Table 4. These preliminary results suggest that the electrical properties are affected by atomic oxygen exposure. More tests are planned to investigate the effects of atomic oxygen on the electrical properties of these coatings.

Table 3. Electrical properties of thin films at room temperature

Thin Film	Thickness (Å)	Sheet Resistance (Ω /□)	Carrier Concentration (electrons/cm^3)	Mobility (cm^2/(V s)
ITO	1000	10^1	10^{20}	17 - 20
ITO/MgF_2 (I) (low % MgF_2)	1000	10^2		
ITO/MgF_2 (II) (high % MgF_2)	959	10^9		
ITO/MgF_2 (III)	1115	10^7		
ITO/MgF_2 (VI)	1042	10^3	10^{19}	2.8
ITO/MgF_2 (VII)	1158	10^5	$\geq 10^{18}$	≤ 1
AZO (I)	559	10^8		
AZO (II)	624	10^7		
AZO (III)	150	10^{11}		
ZnO	1068	10^4	10^{19}	5.8

Table 4. Sheet resistance of thin films before and after atomic oxygen exposure

Thin Film	Sheet Resistance (Ω /□) (Before AO Exposure)	Sheet Resistance (Ω /□) (After AO Exposure)
ITO/MgF_2 (III)	1×10^7	8×10^6
ITO/MgF_2 (VII)	5×10^5	1×10^6
AZO (II)	1×10^7	2×10^6

Conclusions

Transparent conductive coatings of indium-tin-oxide (ITO)-magnesium fluoride (MgF_2) and aluminum doped zinc oxide (AZO) were prepared by ion beam sputter deposition. Simultaneous ion beam sputter deposited indium-tin-oxide (ITO) and magnesium fluoride (MgF_2) at several dopant levels were investigated for electrical resistivity, optical properties and atomic oxygen durability. Results show sheet resistance values of ITO-MgF_2 range from 102 to 10 11 Ω/•, with transmittance of 75 to 86 percent. The AZO films investigated show a sheet resistance value of 107 to 1011 Ω/□ and transmittance of 84 to 91 percent. In general, with respect to the optical properties, the AZO, ZnO, and the high MgF_2 content ITO-MgF_2 samples, were all found to be durable to UV containing atomic oxygen, while the low MgF_2 content ITO-MgF_2 samples were not durable to UV containing atomic oxygen environments.

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

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