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Durability of ITO-MgF_2 Films for Space-Inflatable Polymer Structures

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July 2003

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DURABILITY OF ITO-MgF_2 FILMS FOR SPACE-INFLATABLE POLYMER STRUCTURES

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Abstract

This paper presents results from ITO-MgF_2 film durability evaluations that included tape peel, fold, thermal cycle and AO exposure testing. Polymer coupon preparation is described as well as ITO-MgF_2 film deposition equipment, procedures and film characterization. Durability testing methods are also described. The pre- and post-test condition of the films is assessed visually, microscopically and electrically. Results show that at ~500Å ITO - 9 vol% MgF_2 film is suitable to protect polymer surfaces, such as those used in space-inflatable structures of the PowerSphere microsatellite concept, during a 1-year Earth orbiting mission. Future plans for ground-based and orbital testing of this film are also discussed.

Introduction

An increasing number of NASA missions will benefit from constellations of microsatellites (or microsats) to obtain broad-area, contemporaneous measurements. These multi-kilogram class microsats tend to be power poor due to limited spacecraft surface area to body-mount photovoltaic cells. At the same time, tracking planar solar array systems are mass prohibitive for this class of microsat. An elegant solution to this power pinch challenge is the PowerSphere (Lin, et al., 2003 and Simburger, et al., 2002) concept shown in Figure 1. The PowerSphere space-inflatable, geodetic solar array provides attitude-independent microsat power with very low mass and efficient launch packaging to enable microsat constellation deployment from a single carrier spacecraft. Once inflated, an ultraviolet (UV) activated resin impregnated in PowerSphere central columns and cylindrical solar cell connecting hinges is rigidized by exposure to sunlight and Earth albedo. These inflatable/rigidizable columns and hinges as well as thin-film solar cell substrates and encapsulants are all constructed of polymer materials. During operation in Earth orbit, virgin polymer surfaces could charge to high voltage leading to damaging electrostatic discharge (ESD). In addition, the presence of atomic oxygen (AO) in low Earth orbits leads to aggressive attack of unprotected polymers.

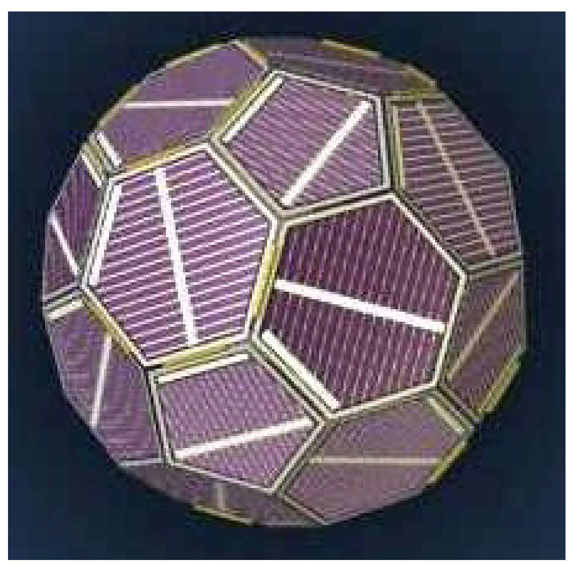


Figure 1. PowerSphere Concept

To address these design challenges, PowerSphere polymer surfaces will be coated with a thin protective film. This film must have suitable electrical sheet resistivity for ESD control, be transparent to UV and solar radiation, for photovoltaic cell operation and UV resin curing, in addition to being AO resistant. The film material selected is a co-sputtered, 91% indium oxide (In_2O_3) – 9% tin oxide (SnO_2) [a.k.a., ITO] and magnesium fluoride (MgF_2) (Dever, et al., 1996). The volume percentage of MgF_2 (typically 0-30%) can be chosen to achieve the desired sheet resistivity ($10^8 \Omega/\square$) and maximize film transmittance. The properties of these films have been the subject of research over the last decade, although the durability of this film for space-inflatable applications has not been addressed.

This paper presents results from film durability evaluations that included tape peel, fold, thermal cycle and AO exposure testing. In the following sections, polymer coupon preparation is described as well as ITO-MgF_2 film deposition equipment, procedures and film characterization. Durability testing methods and results are also discussed.

Coupon Preparation

Coupon Substrates

Two materials were coated with the ITO-MgF_2 films, both Mylar® and a Tefzel®-encapsulated amorphous silicon (a-Si) solar cell on Upilex® as shown in Figure 2. The 0.001 in. Mylar® samples were cut into 2 by 2 in. squares. Prior to deposition, two copper areas were deposited onto the surface for resistivity measurements. The 2 by 2 in. solar cell was cut in half in order to expose both sides for coating.

The target film thickness for the two depositions was 250 Angstroms and 500 Angstroms. For each deposition, two Mylar® samples were coated for testing. Tables 1 and 2 illustrate the sample nomenclature and applicable test plan, respectively.

Coupon ID	Material	Side Exposed	ITO-MgF_2 Thickness (Å)
1m250p 1m500p	Mylar®	Front	250 500
2m250 2m500	Mylar®	Front	250 500
3au250 3au500	Upilex®/a-Si /Tefzel®	Front	250 500
3bu250 3bu500	Upilex®/a-Si/ Tefzel®	Back	250 500

Table 1. Coupon Summary

Coupon ID	Peel Test	Fold Test	AO Test	Thermal Cycling
1m250p 1m500p	Yes	Yes	Yes	No
2m250 2m500	No	Yes	No	Yes
3au250 3au500	Yes	No	No	Yes
3bu250 3bu500	Yes	No	No	Yes

Table 2. Coupon Tests

Film Deposition

An Ion Tech Dual Beam Facility was used to deposit the coatings. This facility uses two ion sources to clean the target, clean the substrate for enhanced adhesion and to carry out the deposition without breaking vacuum. The facility was run on argon as the feed gas in a background of air for more complete oxidation. The pressure during the depositions was less than 4.0E-4 Torr.

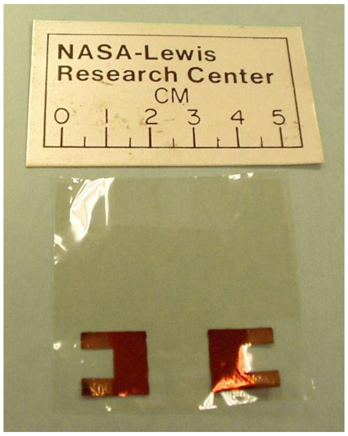
In order to obtain a mixed target, the individual deposition rates had to be determined for the ITO target and the MgF_2 target independently. Once the individual rates were determined, an aluminized piece of Kapton® was used to determine the beam center. Knowing the individual rates and the beam center, the 9 vol% MgF_2 required composition could be translated into angled wedges sitting on top of the ITO target. The mixed target was verified to give the proper deposition rate prior to coating the samples. The sample coupons were then coated in two subsequent depositions.

Film Composition & Characterization

The coating composition was based on calculations yielding 91 vol% ITO - 9 vol% MgF_2. A quartz slide masked with Kapton® tape was used in all depositions as a witness for thickness determination. The coating thickness was determined using a "Dektak IIA Profilometer" by scanning from the tape protected region of the slide to the coated portion in four places. The target 250Å deposition resulted in 275Å ± 24Å. The target 500Å deposition resulted in 535Å ± 37Å.

Durability Test Procedures

During all coupon testing, samples were handled carefully with gloved hands and plastic tweezers to minimize contamination.



(a)

(b) Front (Top) and Back (Bottom)

Figure 2. Coupons With Pristine 535Å Films: (a) Mylar® and (b) Tefzel®-Encapsulated Amorphous Silicon Solar Cell on Upilex®

Tape Peel Testing

A paper was placed over half of the coupon area and then a 1-inch length of 1/2-inch wide Kapton® tape with 3M-Y9460 "Isotac" adhesive was applied to the coupon (see Figure 3). The tape was hand smoothed with moderate pressure. After waiting approximately 2 minutes, the tape was peeled back at a 180º angle and removed from the coupon in about 2 seconds. This test was to demonstrate good film adherence to the substrate.

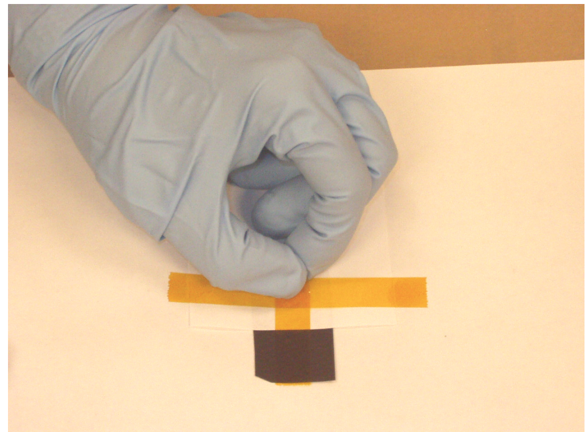


Figure 3. Film Tape Peel Test

Fold Testing

With the film-coated side outward, the Mylar® coupons were quarter-folded using tweezers. A block, approximately 2.5 cm by 2.5 cm, was gently placed on the folded coupon and then 6.8 kg of weights were gently placed upon the block to approximate an atmospheric pressure loading of 10^5 N/m^2 (see Figure 4). After about 2 minutes, the weights and block were removed and the coupon was gently unfolded for inspection. This test simulates pump-down on the PowerSphere center column and subsequent z-fold packaging. Similarly, solar cell inflatable hinges will be flattened for packaging creating single fold lines.

Figure 4. Film Fold Test

Thermal Cycling

After tape peel and fold testing, the film coated coupons were attached to the thermal cycling frame using Kapton® tape and mounted in the NASA Glenn Research Center thermal cycling facility (Scheiman, et al., 1990 and Scheiman and Smith, 1992) shown in Figure 5. While cycling, coupons are exposed to an ambient pressure, nitrogen atmosphere (chamber cold side liquid nitrogen boil-off). A single thermocouple was placed on the back of one coupon. The coupons were then cycled 5000 times between hot and cold chambers based on a measured hot temperature of 100ºC and cold temperature of -128ºC. About 250 thermal cycles were achieved per test day. 5000 thermal cycles is representative of 1-year of operation in low Earth orbit (LEO).

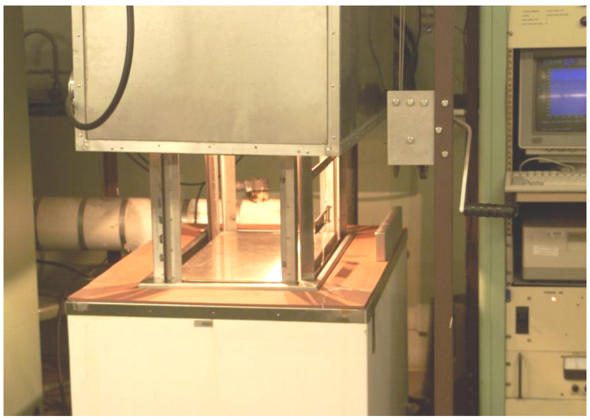


Figure 5. Thermal Cycle Chamber With Solar Cell Test Frame (Right), Hot Chamber (Top) and Cold Chamber (Bottom)

Atomic Oxygen Exposure

After fold testing, 2 Mylar® coupons, with film thicknesses of 275Å and 535Å, were placed in an SPI Plasma Prep™ II Etcher, a.k.a. "asher", which produces a plasma and atomic oxygen (AO) neutrals by radio-frequency excitation. The film coated coupons and a circular, bare Kapton-H® witness coupon were placed film side up on a glass plate and held flat by an open-wire frame placed on top of the coupons (see Figure 6). The planned AO fluence was 1.66E21 atoms/cm^2 which simulates 1-year of exposure in LEO for a ram surface. To gage accumulated AO fluence, the Kapton-H® witness coupon, with a known AO erosion rate, was periodically removed from the asher and weighed.

Figure 6. Atomic Oxygen Coupon Film Testing

Coupon Inspection Methods

The ITO-MgF_2 films were visually inspected, photographed and microscopically examined before and after durability testing. For the Mylar® coupons, film sheet resistivity was measured before and after fold testing (Cashman, et al., 2002). Mylar® coupon mass was measured before film AO durability testing.

Results and Discussion

Tape Peel Testing

Based on visual and microscopic examination of the Mylar® and a-Si solar cell coupons, the appearance of the both 275Å and 535Å ITO-MgF_2 films was unaffected by the tape peel test. The films appeared totally normal and undamaged. A typical photomicrograph of the 535Å ITO-MgF_2 film on Tefzel® is shown in Figure 7. Based on this finding, film sheet resistivity measurements were not obtained. The results indicate these sputtered films are generally robust and tenacious for the Mylar®, Tefzel® and Upilex® polymer substrates.

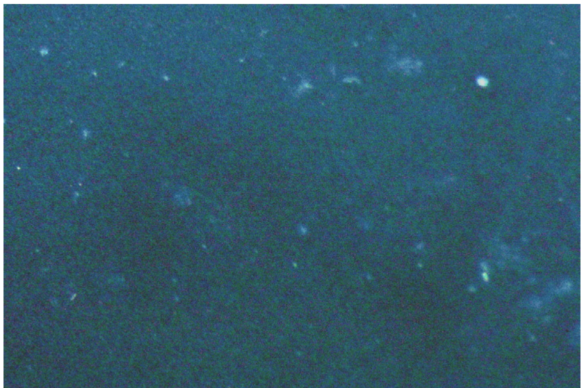


Figure 7. 535Å ITO-MgF_2 film on Tefzel® at 40X Magnification

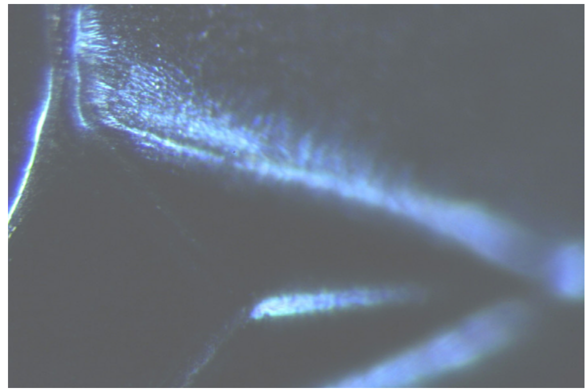


Figure 9. Double Fold Line Region at 80X Magnification; 535Å ITO-MgF_2 Film on Mylar®

Fold Testing

Based on visual inspection of the 275Å and 535Å thickness films along the single and double fold lines, the films appeared unaffected as a result of folding. Figure 8 shows a folded Mylar® coupon with 535Å ITO-MgF_2 film thickness.

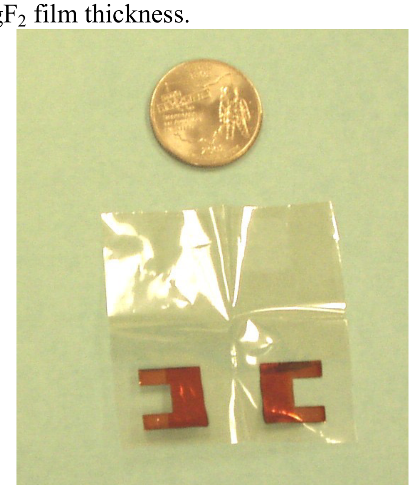


Figure 8. Folded Mylar® Coupon

Under magnifications of 15X, 40X, 80X, and 128X, no generalized film damage along fold lines was evident. Out of 4 coupons examined, only 2 isolated sites of film cracking were detected. Figure 9 shows a typical photomicrograph of the double fold region of a 535Å thick ITO-MgF_2 film on Mylar®.

Thus, based on visual and microscopic inspections, the film appeared to be undamaged after fold testing. Film sheet resistivity measurements were then made. Prior to fold testing, the dark-stabilized, ambient, film sheet resistivity was measured at 10^{10} Ω/□ for 275Å films and 10^{06} Ω/□ for 535Å films. After fold testing, film sheet resistances for all films were insulating (a resistivity >10^{13} Ω/□). This indicated that the films were substantially damaged (cracked) along the fold lines. Prior work (Banks et al., 1985) with a 500Å SiO2 film on Kapton® showed that about 2% strain in the film could be tolerated prior to brittle fracture. This strain level corresponded to a 3 mm bend radius. Although not measured, the bend radius of folded Mylar® coupons is likely much smaller resulting in excessively large film strains. Further testing is planned with a representative center column lay-up of materials, including ITO-MgF_2 coated Kynar-740 and typical z-folding. Under these conditions, a much larger bend radius is expected which should introduce much lower strain levels in the film and less film cracking. To ensure the proper ESD protection function of the ITO-MgF_2 film, a thin layer of copper (with high strain tolerance) will be deposited across fold lines to ensure electrical continuity of any isolated film segments. Since the film stays resident along fold lines, partial AO protection is afforded from a cracked film along a fold line. A more detailed discussion is given in the Atomic Oxygen Exposure section below.

Thermal Cycling

Based on visual and microscopic examination of the Mylar® and a-Si solar cell coupons, the appearance of both 275Å and 535Å ITO-MgF_2 films was unaffected by exposure to the 5000 thermal cycles. This finding held true for film virgin areas, film peel tested areas and along film fold lines. The films appeared totally normal and undamaged. Based on this finding, film sheet resistivity measurements were not obtained. These results indicate that these films, on Mylar®, Tefzel® and

Upilex® polymer substrates, are robust and tenacious for at least 1-year in a LEO thermal cycling environment.

Atomic Oxygen Exposure

The folded Mylar® coupons were removed from the plasma asher after an accumulated AO fluence of only 0.66E21 atoms/cm^2 (0.4 years ram exposure in LEO). At this point, the coupons were obviously damaged. To the unaided eye, the coupon films appeared frosted (see Figure 10 below compared to pre-AO exposure in Figure 8) and have reduced mass and stiffness (more pronounced in coupon with 275Å ITO-MgF_2 film). The outward facing fold line substrate material was substantially severed with the 275Å film thickness and only partially severed on the coupon with the 535Å ITO-MgF_2 film. Uncoated Mylar® (area masked to keep the copper pad fingers pristine for electrical test purposes) was completely eroded away in both coupons. As a result of this unprotected material loss, meaningful coupon mass change measurements could not be made. Photomicrographs revealed the 275Å ITO-MgF_2 film on the copper pads was unaffected as a result of AO exposure. This film on Mylar®, however, was found to exhibit generalized within film cracking and occasional pin hole defects and large, through film cracks near fold lines (see Figure 11).

Figure 10. Mylar® Coupon With 535Å ITO-MgF_2 Film Following AO Exposure

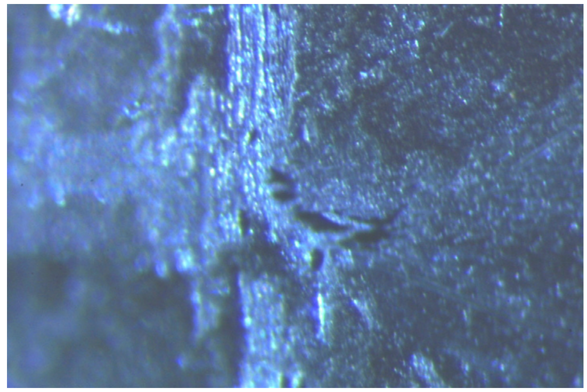


Figure 11. : 128X Magnification Photomicrograph of Mylar® Coupon Double Fold Line With 275Å ITO-MgF_2 Film Following AO Exposure

A similar frosty film appearance was obtained with the 535Å ITO-MgF_2 film coupon as shown in Figure 12 below (compare to pre-AO exposure condition shown in Figures 7 and 9). The cause for this frosty appearance is likely from the AO penetrating small film cracks, previously not visible, and attacking the Mylar® substrate. The resulting Mylar® cavity formed under the crack then scatters the light leading to the frosted appearance. Larger cracks allow the AO to completely erode away the local underlying Mylar® and thus create the appearance of black regions such as the dark pin hole defect in Figure 11. In this case, the Mylar® can no longer support the ITO-MgF_2 film and it collapses,

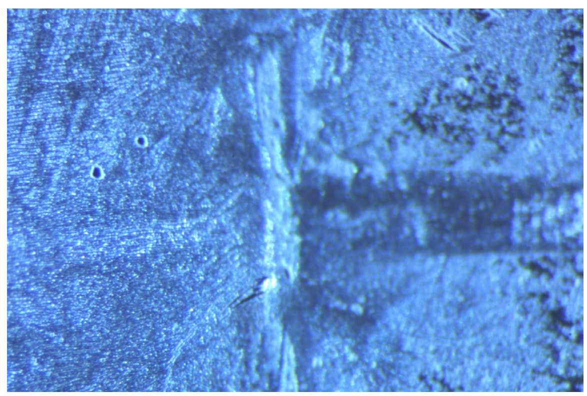


Figure 12. : 40X Magnification Photomicrograph of Mylar® Coupon Double Fold Line With 535Å ITO-MgF_2 Film Following AO Exposure

opening up the defect site for further undercutting AO attack of the Mylar® substrate. The source of the initial film cracks is likely the combination of fold test block compression forces and fold test handling with tweezers.

These AO exposure tests indicate the 275Å film thickness is insufficient to protect the Mylar® along fold lines. The 535Å film remained largely intact and held up well on the copper pads (important for electrical continuity). Since the polymer films used on the inflatable center columns and solar cell hinges are only required for the first few hours of the mission, during inflatable structure rigidization, AO-induced frosting, and the concomitant changes in optical properties, is acceptable. Given the more benign handling requirements for solar cell films (i.e., no folding or large compressive loads), film cracking and subsequent frosting behavior is less likely than with the inflatable polymer surfaces. Even if some frosting occurs, scattered light will still reach the solar cell so performance should not be greatly reduced. Further AO exposure testing is planned for coupons reflecting the final design, materials and handling procedures for PowerSphere inflatable polymer structures. If required, the film thickness could be doubled to the 1000Å range for enhanced robustness. With twice the film thickness, the resulting loss in film solar transmittance, and hence solar cell current output, was estimated as 12%.

Film Sheet Resistivity Sensitivities

The sheet resistivity of ITO-MgF_2 coatings on Mylar® substrates was found to be sensitive to light, age, and film thickness. Following long exposure to ambient room light, the sheet resistivity increased by a factor of ~2 after 20 hours of dark soak and by a factor of ~3 after 70 hours of dark soak. When measured after ~20 hours of dark soak, sheet resistivity increased by a factor of 10-30 in the period 20 to 90 days after deposition. The sheet resistivity also showed substantial thickness dependence; samples ~250 Å thick had dark sheet resistivities ~$2x10^4$ times larger than those ~500 Å thick. Qualitatively similar effects have been observed in ITO-MgF_2 films deposited by RF magnetron sputtering (Cashman, et al., 2002).

Concluding Remarks

275Å- and 535Å-thick films of ITO - 9 vol% MgF_2 were co-sputtered on space-inflatable structure polymer materials including Mylar®, Tefzel® and Upilex®. The durability of these films was assessed by conducting a regiment of tests including tape peel testing, fold testing, thermal cycle exposure and AO exposure testing. The films were characterized by visual inspection, optical microscopy and electrical sheet resistivity. Tape peel tests and thermal cycle exposure tests demonstrated that the films were tenacious and robust. Fold testing cracked the film along fold lines creating electrically isolated film segments although the film remained intact and apparently undamaged based microscopic inspections. Subsequent AO exposure produced a frosty appearance in the films presumably due to substrate attack below previously unseen film cracks. Fold lines on coupons with a 275Å thick film were mostly severed and thus it was deemed that this film thickness is insufficient. Although frosted, the 535Å thick film remained largely intact after AO exposure and should be acceptable for the PowerSphere inflatable structure polymer application. Further film durability testing with articles reflecting the final inflatable structure design, materials and handling will be conducted to ensure the performance and robustness of the ITO-MgF2 ESD/AO protective film. If necessary, a more robust 1000Å film thickness could be employed with acceptable solar cell performance impacts.

Future Work

Other ITO-MgF_2 film research is planned or in progress. This work includes film electron, proton and neutron irradiation tests on-going at The Aerospace Corporation. Film electrical and optical properties will be measured before and after the radiation exposures. Also, ITO-MgF_2 films will be deposited on prototypical PowerSphere solar cells with flex harness and included as part of the Materials International Space Station Experiment (MISSE-5) flight experiment to be launched to the International Space Station. After exposure to the LEO environment for a year or more, the MISSE-5 samples will be returned to Earth to allow ITO-MgF_2 film characterization testing. And lastly, research is underway at the Cleveland State University Department of Physics exploring the use of magnetron sputtering emission line spectroscopy for in-situ control of film resistivity.

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13. ABSTRACT *(Maximum 200 words)*

This paper presents results from ITO-MgF_2 film durability evaluations that included tape peel, fold, thermal cycle, and AO exposure testing. Polymer coupon preparation is described as well as ITO-MgF_2 film deposition equipment, procedures and film characterization. Durability testing methods are also described. The pre- and post-test condition of the films is assessed visually, microscopically, and electrically. Results show that at ~500Å ITO - 9 vol% MgF_2 film is suitable to protect polymer surfaces, such as those used in space-inflatable structures of the PowerSphere microsatellite concept, during a 1-year Earth orbiting mission. Future plans for ground-based and orbital testing of this film are also discussed.

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