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Determination of Atomic Oxygen Fluence Using Spectrophotometric Analysis of Infrared Transparent Witness Coupons for Long Duration Exposure Tests

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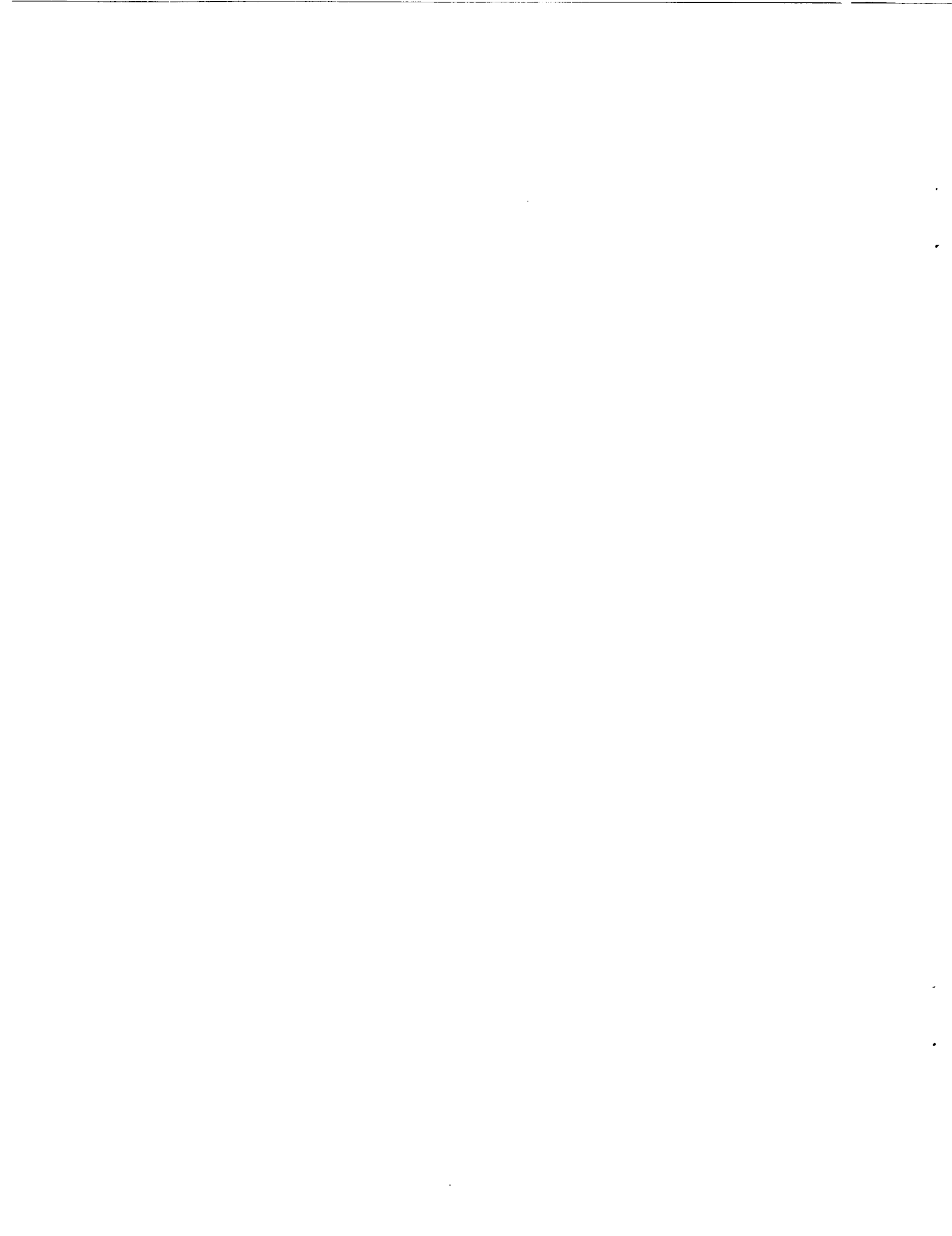


(NASA-TM-106021) DETERMINATION OF
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**DETERMINATION OF ATOMIC OXYGEN FLUENCE USING SPECTROPHOTOMETRIC
ANALYSIS OF INFRARED TRANSPARENT WITNESS COUPONS FOR
LONG DURATION EXPOSURE TESTS**

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ABSTRACT

Atomic oxygen degradation is one of several major threats to the durability of spaceborne systems in low Earth orbit. Ground-based simulations are conducted to learn how to minimize the adverse effects of atomic oxygen exposure. Assessing the fluence of atomic oxygen in test chambers such as a plasma asher over long periods of time is necessary for accurate determination of atomic oxygen exposure. Currently, an atomic oxygen susceptible organic material such as Kapton is placed next to samples as a witness coupon and its mass loss is monitored and used to determine the effective atomic oxygen fluence. However, degradation of the Kapton witness coupons occurs so rapidly in plasma ashers that for any long term test many witness coupons must be used sequentially in order to keep track of the fluence. This necessitates opening vacuum to substitute fresh coupons. A passive dosimetry technique was sought to monitor atomic oxygen exposure over longer periods without the need to open the plasma asher to the atmosphere. This paper investigates the use of spectrophotometric analysis of durable IR transparent witness coupons to measure atomic oxygen exposure for longer duration testing. The method considered would be conducive to making in situ measurements of atomic oxygen fluence.

INTRODUCTION

The harsh low Earth orbital environment poses many hazards to spaceborne systems. Among these are thermal cycling, VUV radiation, micrometeoroid and debris impact, and plasma and atomic oxygen interaction. Atomic oxygen is the predominant atmospheric component at low Earth orbit (LEO) altitude of 400 km. Reaction with atomic oxygen causes many spaceborne materials to degrade. In order to study LEO atomic oxygen interactions, materials are subjected to tests in ground-based atomic oxygen plasma chambers.

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Atomic oxygen exposure testing requires measuring the effective fluence of atoms impinging on the surface of a sample. Currently, it is common to use an organic substrate such as Kapton H or HN polyimide as a witness coupon to determine atomic oxygen fluence. Space flight data have provided an erosion yield that defines the relationship between fluence and mass loss, making this technique very effective [1]. However, there are two problems with this technique. The first problem is that the thin (5 mil) Kapton samples begin to lose their structural integrity with exposure to a fluence of approximately $1.5 \text{ E} + 21$ atoms/cm² and are nearly fully converted to gaseous species after a fluence of $4.2 \text{ E} + 21$ atoms/cm². As the sample becomes microscopically rough, the surface area of the sample changes, putting the fluence value into question. Because of this concern, it is preferable not to use the Kapton beyond the point at which noticeable structural failure begins. This only represents 72 days at LEO altitude of 400 km.¹ For long duration testing, a single Kapton witness coupon is not adequate. Therefore, multiple witness coupons must be used sequentially, necessitating the opening of the vacuum chamber of the atomic oxygen plasma asher. A second more general problem is that the test chamber must be opened in order to determine the fluence whenever mass-loss dosimetry is used. This exposes the test samples to moisture in the atmosphere.

As a result of atomic oxygen durability testing of infrared optic materials in support of the Earth Observing System (EOS), the concept of using an IR sensitive material on an IR transparent substrate for long term atomic oxygen dosimetry was conceived. Hence, an alternative dosimetry technique was found that would be effective for longer uninterrupted exposures and which would allow in situ measurements. In this paper, this secondary dosimetry technique, which uses spectrophotometric analysis of special witness coupons to assess the dosage of atomic oxygen, is described. Also, preliminary tests of dosimeter materials are discussed.

This paper is divided into several sections. The general dosimetry technique proposed is discussed first. Next, infrared spectroscopy results from screening tests performed on a variety of inorganic substrates in an effort to identify potential dosimeter materials are presented. Finally, the long term atomic oxygen exposure testing of the chosen dosimeter will be discussed. In addition, the effect of a protective coating on an IR dosimeter material was investigated.

APPARATUS AND PROCEDURES

Spectroscopically pure NaCl and KBr infrared windows were used as substrates to which dosimetry materials were applied. These were standard windows purchased from the

¹ Equivalent LEO time was determined using data from the LDEF mission. An average of the atomic oxygen flux data for a 17 day period centered around the LDEF pass through 400 km altitude was calculated. This value was determined to be $2.42 \text{ E} + 14$ atoms/cm²*s [2].

Perkin-Elmer Corporation (part nos. PE 0027-1182 and 0027-1282). The windows have dimensions 38.5 mm by 19.5 mm and are 4 mm thick. The NaCl windows were used during the screening tests while the KBr windows were chosen for the final study. These windows were chosen because of their greater than 90% IR transparency in the range 4000 cm^{-1} to 385 cm^{-1} [3], and because they were found to be durable to atomic oxygen.

Various inorganic dosimetry candidates were applied to the KBr substrates using electron beam (e-beam) deposition. This is a line-of-sight deposition technique that uses a beam of electrons to evaporate the coating material. Candidate materials included aluminum, silver, and copper. The coatings ranged in thickness from less than 100 Å up to 325 Å. A 1200 Å SiO_x coating was also applied to selected samples using e-beam deposition. The thickness of coatings was confirmed using a Sloan Dektak II profilometer.

Infrared spectra of dosimetry samples were obtained using a Perkin-Elmer Corporation Model 783 Infrared Spectrophotometer. Transmittance measurements were obtained for the range 4000 cm^{-1} to 250 cm^{-1} .

Samples were exposed to atomic oxygen in a Structure Probe, Inc. Plasma Prep II asher. The plasma is generated by 100 Watts continuous RF power applied at 13.56 MHz to air at a pressure of 50-100 mTorr. Although the plasma consists of standard atmospheric components, atomic oxygen is the only major reactive component. The other species in the plasma ($\text{N}_2, \text{N}, \text{O}_2, \text{Ar}$) are relatively unreactive. The mass loss of 5 mil Kapton witness coupons was used to determine the effective fluence of atomic oxygen. Care was taken to keep the Kapton coupons dehydrated to ensure accurate mass measurements. The fluence was then calculated according to equation 1.

$$\text{Effective Fluence} = \frac{\text{Mass Loss} / \text{Surface Area}}{\text{Density} * \text{Erosion Yield}} \quad (1)$$

The erosion yield for Kapton has been determined to be 3.0 E-24 cm^3/atom and the density equal to 1.42 g/cm^3 [1]. Because of the short life-span of Kapton coupons, a new witness coupon was used for each ashing period.

General Method of the Dosimetry Technique

Potential dosimetry materials were deposited on KBr infrared windows using e-beam deposition. The IR transmittance spectra of the unexposed samples were obtained on the spectrophotometer. Samples were then exposed to atomic oxygen in successive time periods. After each ashing period, the IR transmittance spectra of the samples were obtained. Atomic oxygen fluence was determined for each ashing period using sequential Kapton witness coupons. The IR transmittance at specific wavelengths was charted over the period of ashing to identify systematic trends in the transmittance of the candidate materials.

Once a suitable dosimetry material was identified, dosimetry samples were used to determine levels of atomic oxygen exposure. By monitoring the transmittance at certain wavelengths, charts of atomic oxygen fluence versus transmittance could be produced. In subsequent tests, the dosimetry samples could be used to determine fluence by recording the transmittance at one or more key wavelengths and comparing to the charts. Having several key wavelengths charted would act as a self-checking mechanism. By selecting materials that react slowly with atomic oxygen, a measurable fluence range much larger than that for Kapton could be established. Similarly, for smaller expected fluences, a rapidly reacting material could be selected.

Screening Tests Using An IR Sensitive Peak Method

An attempt was made to find a material that would experience either an increase or decrease in the size of one or more IR sensitive peaks with ashing. Kapton and several thin-film metals ($< 150 \text{ \AA}$) were tested for suitability. All screening tests involved using a single dosimetry sample to get a rough idea of the usefulness of the material.

Tests on Kapton were performed using the same 5 mil coupons used for standard atomic oxygen dosimetry. These were used because there was no method available to deposit Kapton on the KBr substrate. The Kapton coupons were ashed over a twenty hour period, with the IR transmittance spectrum recorded at four equally spaced intervals.

Screening Tests Using An Opaque Spectrum Conversion Method

A second method involves the use of thicker ($> 150 \text{ \AA}$) metal coatings that were IR opaque, but which formed oxides that had very characteristic non-opaque spectra. An initially opaque metal coating was exposed to atomic oxygen and over time the metal was converted to the oxide. The IR transmittance spectrum of the sample followed the conversion and transformed from an opaque spectrum to the characteristic oxide spectrum.

Long Term Atomic Oxygen Exposure Testing of Dosimeter

To determine the effectiveness of an atomic oxygen protective coating as measured by the IR dosimetry technique, 300 \AA of silver were e-beam deposited on the KBr infrared windows described earlier. One sample was then coated with 1200 \AA of SiO_x to serve as an atomic oxygen protective coating, and one sample had no atomic oxygen protective coating. The thickness of silver was chosen because it was thick enough to be initially opaque in the IR, but thin enough to allow all of the silver to oxidize in a reasonable time frame. Both samples were ashed simultaneously, and the IR transmittance spectrum of each was recorded between successive ashing intervals. The samples were ashed for a total of 54 days. The fluence for each interval was determined with Kapton witness coupons. The

total fluence was $1.9 \text{ E}+22 \text{ atoms/cm}^2$, equivalent to 2.5 years at LEO altitude of 400 km [2].

For the first two days, IR transmittance scans were taken every 12 hours. For the next four days, spectra were recorded every 24 hours, and for the remainder of the time, every 48 hours. The shorter periods at the beginning were used to accommodate for the rapid conversion of the unprotected sample. Even with this graded timing scale, the unprotected sample had very large differences between successive scans in the first few days. The 48 hour ashing period used for the duration of the study was the optimum for Kapton witness coupon durability and charting of data.

RESULTS AND DISCUSSION

Screening Test Results

Several of the IR sensitive peaks for Kapton showed systematic changes with ashing. The results from one sample are shown in figure 1. The graph for each wavenumber is linear and of roughly the same slope. This slope is representative of the erosion of the Kapton. As the sample was ashed, it became thinner and thus transmitted more light after each ashing period. Although a systematic increase in transmittance was successfully identified, the durability problem with Kapton remained. Degradation of the Kapton witness coupons occurred within 4 days of exposure in the asher.

There were many problems associated with using metals for the individual IR sensitive peak method. The coatings had to be extremely thin ($<150 \text{ \AA}$) to allow a significant IR transmittance. The e-beam deposition technique could not reliably produce uniform coatings that were so thin. For the limited number of samples that were produced, spectrophotometric data taken over periods of atomic oxygen exposure showed no conclusive trends in the transmittance spectra of the thin metal films.

It is important to note that organic and inorganic materials have very different IR transmittance spectra. Organic materials have spectra which have many characteristic peaks. This is due to the many different molecular vibrational modes of complex organic compounds, each of which produces an absorption peak. Inorganic materials generally have much simpler spectra than organic materials. That is, fewer vibrational modes and fewer absorption peaks [4,5,6]. For example, figure 5a shows the relatively smooth spectrum for 300 \AA of Ag/AgO_x on a KBr window and figure 2 shows the complex spectrum for a 5 mil Kapton coupon. The smoother shapes of inorganic transmittance spectra make them much easier to work with when trying to identify spectral trends.

Initial tests performed using a $100\text{-}125 \text{ \AA}$ aluminum coating indicated that this could be a suitable dosimetry material for long duration ashing using the opaque spectrum

conversion method. Initially, this sample transmitted only 20-25% of the IR radiation. As aluminum oxide formed, it acted as an atomic oxygen protective coating and limited the conversion of the underlying material. Because of this the change in the IR transmittance spectrum did not progress linearly. Figure 3 shows how the spectral changes in the aluminum coated sample occurred rapidly at first followed by a much slower progression as the aluminum oxide barrier became thicker. It must be noted that since the incremental change in transmittance for further atomic oxygen exposure becomes small with long ashing periods, there must be a point at which the error in the technique is of the same magnitude as the incremental change. Further work is required to determine at what fluence value the technique fails with an aluminum dosimeter.

Initial tests performed using a 325 Å silver coating showed that it too was a potential dosimetry material. However, the silver oxidized much more quickly and the silver oxide was a much poorer protective barrier. Because of this, the progression to a silver oxide spectrum occurred more quickly. The sequential IR transmittance spectra also showed that complete conversion was achieved with an ashing fluence on the order of $2.0 \text{ E}+21$ atoms/cm² (4 days in the asher used). This was an appropriate choice for the long-term test because the SiO_x-coated test sample would allow only a small amount of atomic oxygen to reach the dosimeter. Thus, to witness the complete conversion to oxide within a reasonable time frame, the dosimeter must be readily oxidized.

Long-Term Test Results For 300 Å Silver Dosimeter

Several selected wavelengths in the IR transmittance spectra of the control and test samples were charted over the 54 day period. Most of the higher wavelengths showed complete conversion from silver to silver oxide for both the control and SiO_x-coated sample within the time frame of ashing (see figures 4a,b,c). The figures all clearly show that the time required for complete conversion was increased in the sample with the atomic oxygen protective coating. This directly shows the reduction in the amount of atomic oxygen reaching the silver on the SiO_x coated sample.

However, from the graph of the transmittance values for the 2500 cm⁻¹ wavenumber, it is not apparent when complete conversion of the silver to an oxide is achieved (see figure 4d). After a fluence of approximately $9.0 \text{ E}+21$ atoms/cm², both the control and test sample transmittance values rise at a parallel slow rate. This is unusual because the control sample had already achieved and maintained what appeared to be a steady state transmittance of approximately 50%. Care must be taken when selecting wavelengths to monitor for dosimetry purposes.

Figures 4a-4c show a levelling-off at a fluence of approximately $1.0 \text{ E}+22$ atoms/cm². As a first estimate of the error associated with this technique, it is believed that the fluence versus transmittance data are usable up to approximately $8.0 \text{ E}+21$ atoms/cm². Again, the reason for this is that the incremental change in transmittance becomes small after this point

and it is likely that the error is larger than the incremental change. This fluence is equivalent to 1 year of exposure at LEO altitude of 400 km [2]. This is 5 times longer than can be measured with a 5 mil Kapton coupon. Further work is required to determine how reproducible the fluence versus transmittance data are.

Figures 5a and 5b show the final IR transmittance spectra of the control and test dosimetry samples. Several differences are apparent. The smooth region of the spectrum from 4000 cm^{-1} to 1500 cm^{-1} is fairly level on the SiO_x coated sample. On the control sample, this area slopes upward, more characteristic of a standard oxide spectrum. The location and magnitude of absorption peaks are also slightly different. This can be accounted for by the presence of the SiO_x protective coating, which absorbs some IR radiation.

With further work, this method might be modified to allow in situ fluence measurements. One method considered involves integrating an atomic oxygen plasma asher and an infrared spectrophotometer. The plasma chamber could be fitted with IR windows and the beams of the spectrophotometer could be made to pass through the chamber. While maintaining vacuum, the plasma could be momentarily shut off during the acquisition of the IR transmittance spectrum. The sample beam would pass through the IR windows as well as the dosimeter while the reference beam would only pass through the IR windows. This would completely eliminate the need to open the plasma chamber to determine atomic oxygen fluence during long term testing.

Further Considerations

When selecting a dosimeter for atomic oxygen exposure tests, it is important to be aware of the modes by which atomic oxygen attacks materials. Of particular interest are the effects of pinhole defects in coatings which provide a pathway for atomic oxygen. Much work has been done studying these effects [7,8,9].

For the Ag sample with no protective coating, the effect of pinhole defects was minimal. Although pinhole defects were inevitable in all the dosimetry coatings, the cumulative pinhole area was expected to be very small compared to the area of the sample. The entire surface was exposed and the AgO_x that formed was porous. Because of this, atomic oxygen could readily attack over the entire surface area until all of the Ag was converted to an oxide. The added effect of atomic oxygen penetrating deeper into the Ag through pinhole defects was negligible. Therefore, the rate at which unprotected Ag is converted to AgO_x is not highly dependent upon pinhole defect population.

When samples with atomic oxygen protective coatings are studied, there are three modes by which atomic oxygen can attack the underlying layer of reactive material. These are: attack along exposed edges, entering through pinhole defect pathways, and diffusion through the protective coating. Note that while SiO_x allows atomic oxygen to diffuse only

very slowly, the AgO_x is porous and allows atomic oxygen to move through to unconverted Ag much more readily. Because of this, atomic oxygen can continue to attack farther inward from the original entry point once the outlying Ag has been oxidized.

In this study, the SiO_x -coated Ag dosimeter used in the long-term test had a completely exposed edge area which was expected to be much greater than the combined area of pinhole defects on the front surface. This was the main pathway for atomic oxygen attack, and thus the controlling factor in determining the life-span of the dosimeter. This was evidenced by a ring-like discoloration that moved toward the center of the sample as ashing progressed. Since the area of defects is much smaller than that of the edges, it is expected that protecting the edges would significantly increase the usable life of the dosimeter. If the edges were protected, the pinhole size and population density would become the determining factor in dosimeter life span because that would be the main pathway for atomic oxygen attack. However, variations in the integrity of SiO_x coatings between different samples would affect the rate at which the underlying Ag was converted to AgO_x . These factors must be taken into consideration so that errors are kept to a minimum.

CONCLUSION

A secondary atomic oxygen dosimetry technique that uses infrared spectrophotometric analysis of durable IR transparent witness coupons was shown to be potentially useful for long duration exposure tests. With appropriate choice of materials, the dosimeter reaction rate could be customized to allow measurement of different magnitudes of fluence for specific applications. This method could be adapted to make in situ fluence measurements during long duration exposure tests. By applying a protective coating to a dosimeter and simultaneously exposing the test and control samples to atomic oxygen, the reduction in atomic oxygen reaching the dosimeter was observed spectrophotometrically. A dosimeter composed of 300 Å Ag coated with 1000 Å SiO_x can be used to measure fluence in the range 0 to $8.0 \text{ E}+21 \text{ atoms/cm}^2$, or 0 to 1.0 years at LEO altitude of 400 km [2]. This allows for a useful measurement span approximately 5 times greater than that for 5 mil Kapton witness coupons. However, accuracy of measurements can be affected by variations in dosimeter coating thickness and pinhole defect populations. Further work is required to determine the magnitude of error associated with this technique.

ACKNOWLEDGEMENTS

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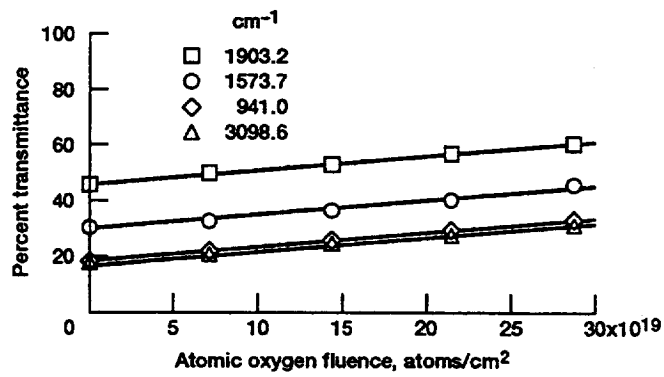


Figure 1.—Infrared transmittance measurements at selected wave-lengths for 5 mil Kapton over 20 hours of atomic oxygen exposure.

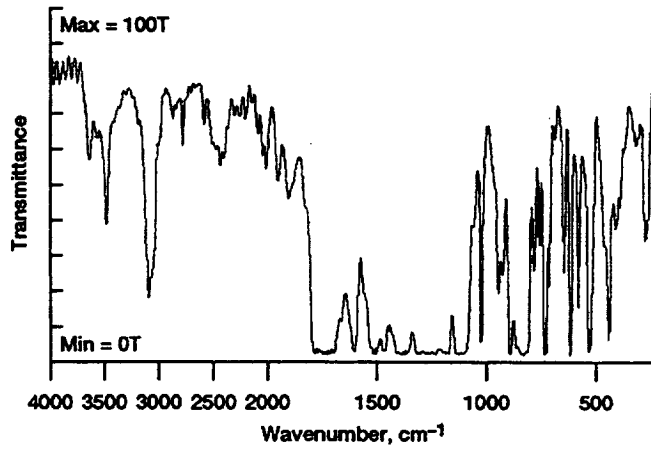


Figure 2.—Infrared transmittance spectrum for 5 mil Kapton coupon.

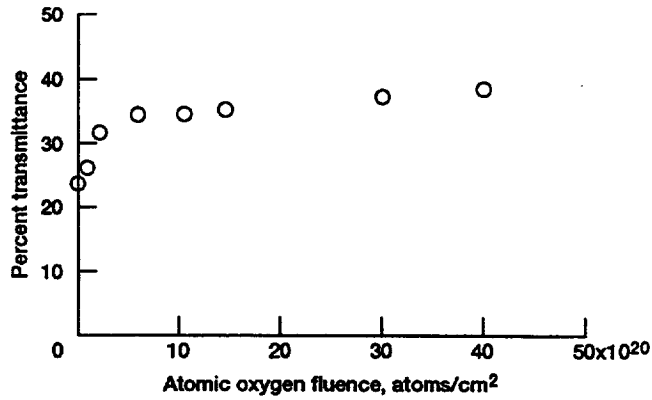


Figure 3.—Screening test results for KBr window coated with 100-125 Å Al. The transmittance measurements are for 3500 cm⁻¹.

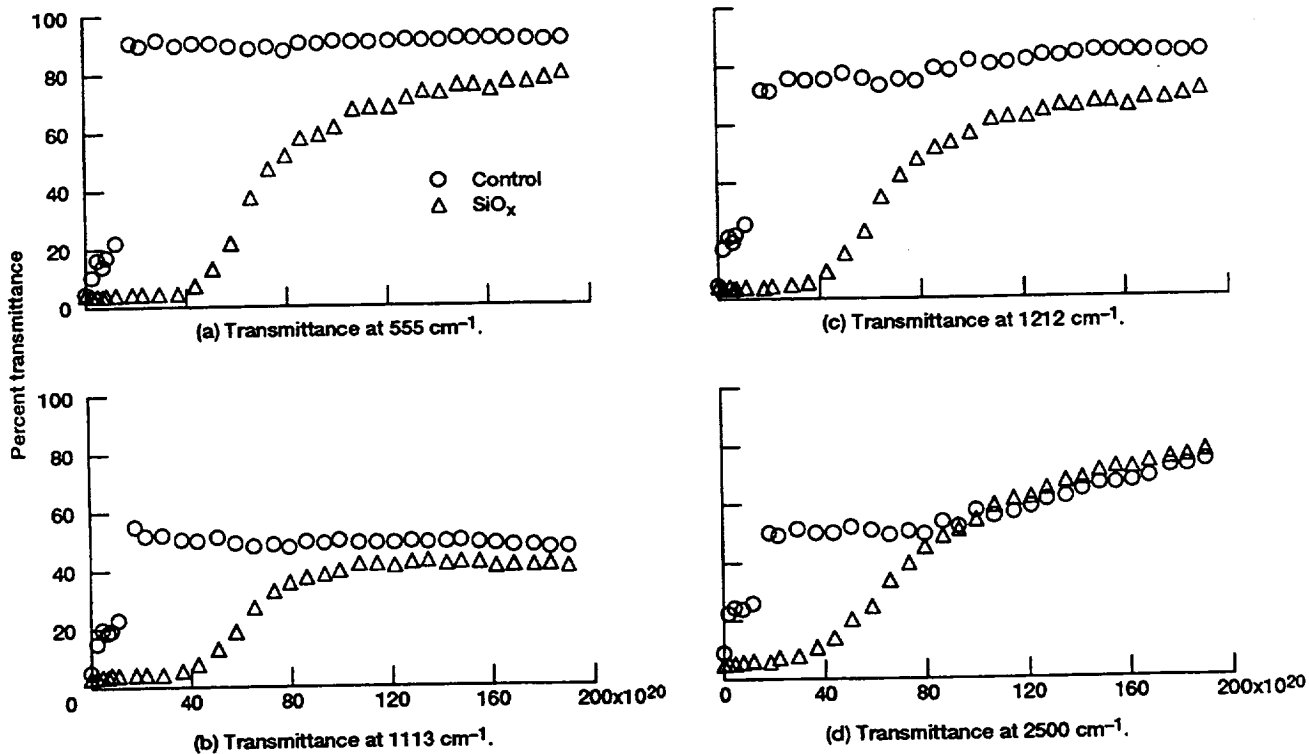


Figure 4.—Long term dosimetry results for 300 Å Ag on KBr substrate. Control curve represents normal dosimetry sample and SiO_x curve represents dosimetry sample with 1000 Å SiO_x atomic oxygen protective coating applied.

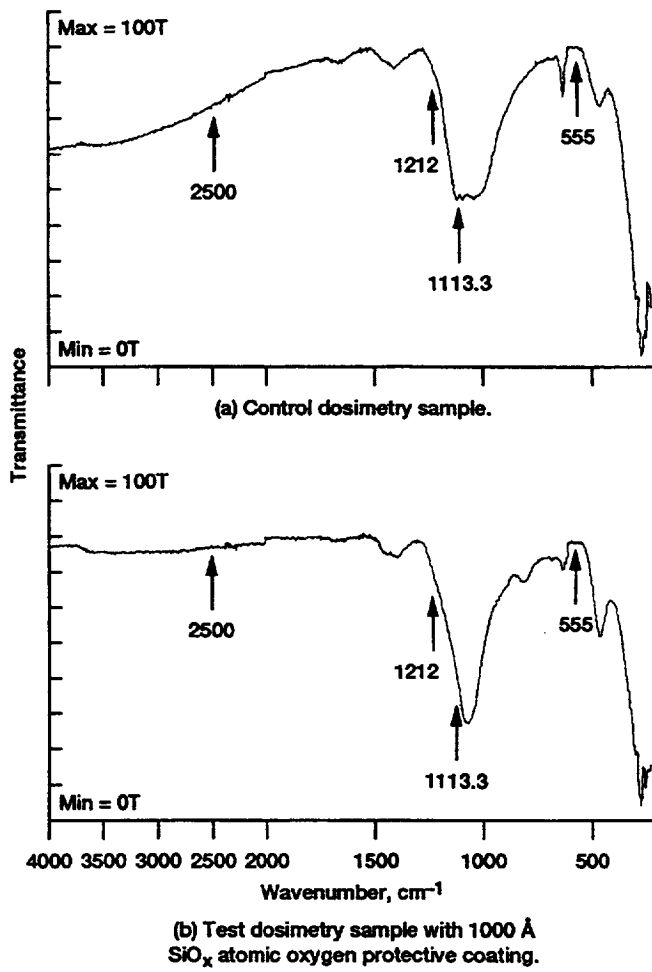


Figure 5.—Final infrared transmittance spectra and locations of selected wavenumbers for control and atomic oxygen test dosimetry samples.

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