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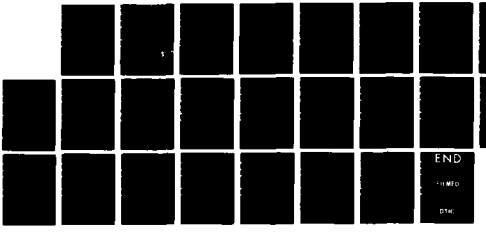

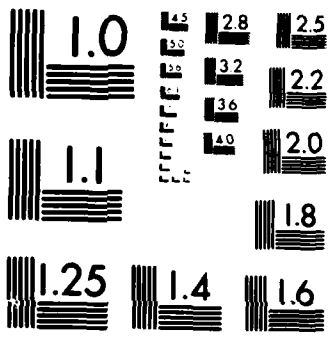

REPORT SD-TR-84-41

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Long-Term Light-Induced Changes in Dark Conductivity of Kapton

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The light-induced increase in dark conductivity of Kapton in space and simulated space environments has been investigated. It was found that a long-term increase in conductivity is caused by an accumulation of electrically active centers generated by solar radiation in the bulk of the material, if maintained in vacuum. An analysis of the increase in conductivity as a function of light exposure time indicates that the dark

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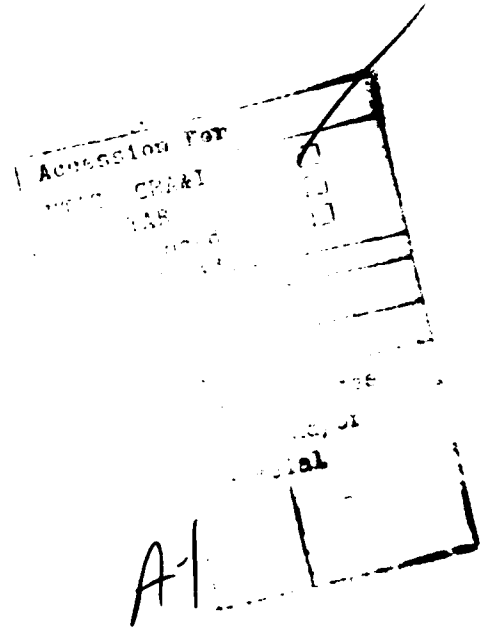
20 ABSTRACT (Continued)

conductivity of Kapton can be raised to a level where the film can become sufficiently conductive to be antistatic by simply exposing it to solar radiation in orbit.

PREFACE

The authors wish to acknowledge the many helpful technical discussions with Dr. H. K. A. Kan, the encouragement and support from Dr. H. E. F. Wang, and the effort Mr. Robert Broussard contributed in administering this program. A major part of this investigation was funded by Aerospace-Sponsored Research.



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I. INTRODUCTION

Kapton is a polypyromellitimide film whose properties have been investigated extensively because of its wide usage in both terrestrial and space applications. Because of its low conductivity and high dielectric strength, Kapton has been used as an electrical insulator in terrestrial environments. In space, Kapton is used most commonly as the outermost surface of a spacecraft thermal control blanket. When bombarded with electrons from the hot, tenuous plasma found in space during geomagnetically disturbed times, the bare Kapton surface tends to acquire a high electrostatic potential. A number of techniques have been devised in an attempt to render the surface of Kapton antistatic in order to reduce the risks arising from spacecraft charging. One such approach relies on perforating the Kapton film to allow the electrons accumulated on the front surface to drain off through the edge of the perforation to the aluminized back surface.[1] The most popular one involves overcoating the Kapton front surface with a transparent, conductive oxide.[2] In this report, we wish to present the results of our investigation in which we found that the dark conductivity of Kapton can be raised to a level where the film itself can become sufficiently conductive to be antistatic by simply exposing it to solar radiation in orbit.

II. BACKGROUND

Although photoconductivity of Kapton has been studied for some time, the effects of light on the dark conductivity of Kapton in vacuum are not well understood. There were some early ground test data indicating that the dark current measured after exposure to solar radiation in vacuum appeared to be much higher than before exposure.[3] No explanation was given at the time for the observation. The first indication that solar-exposure-induced increases in dark conductivity may be important in space came from in-situ measurements of the electrical properties of Kapton obtained by the Satellite Surface Potential Monitor (SSPM).

The SSPM was an experiment payload flown on the P78-2 Spacecraft Charging at High Altitudes (SCATHA) satellite.[4] The purpose of the experiment was to study the electrical behavior of typical spacecraft insulating materials in the near geosynchronous orbit. P78-2 was a spin-stabilized satellite with its spin axis normal to the satellite-sun line. There were three SSPM Kapton samples on board the spacecraft in different locations. Two samples were mounted in such a way that they rotate into and out of sunlight; the third was mounted in the shadow at the end of the spacecraft. On-orbit SSPM data showed that the Kapton samples that were exposed to solar radiation tended to charge less frequently and to progressively lower levels.[5] With the surface potential of an isolated gold-plated conductor as a reference, the data showed that the ratio of Kapton surface voltage to the reference voltage decreased monotonically as a function of time (Fig. 1). This decrease was observed only in the Kapton samples exposed to sunlight in orbit and not in the sample in the shadow of the spacecraft.

A more detailed analysis of the current vs voltage (I-V) behavior of Kapton in orbit is shown in Fig. 2.[6] The plot designated by triangles (Δ) represents the I-V curve for Kapton measured before launch. The squares (□) represent on-orbit I-V measurements of the SSPM Kapton samples obtained in two charging events separated by 15 mo. These measurements were chosen for comparison because charging took place in rather intense but stable

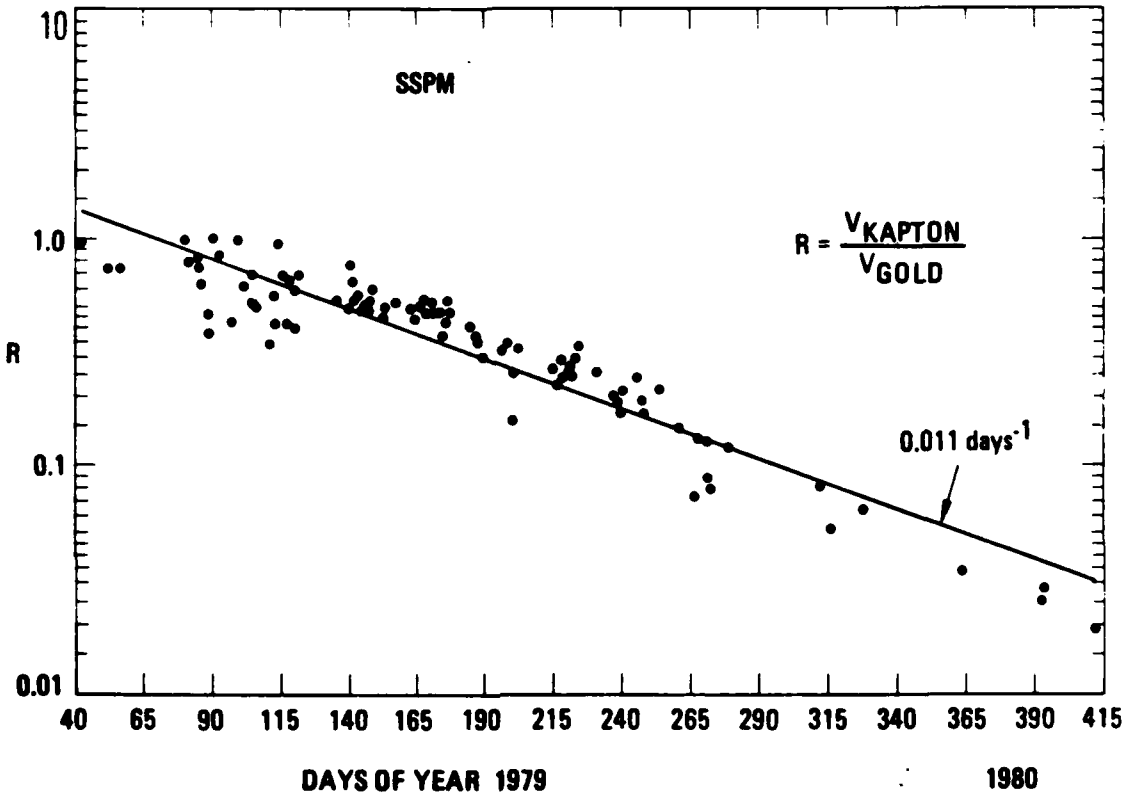

Fig. 1. Plot of the Ratio of Kapton Surface Voltage to the Reference (Isolated Gold Plate) Voltage. Data were obtained by the Spacecraft Surface Potential Monitor (SSPM) on board the P78-2 satellite..

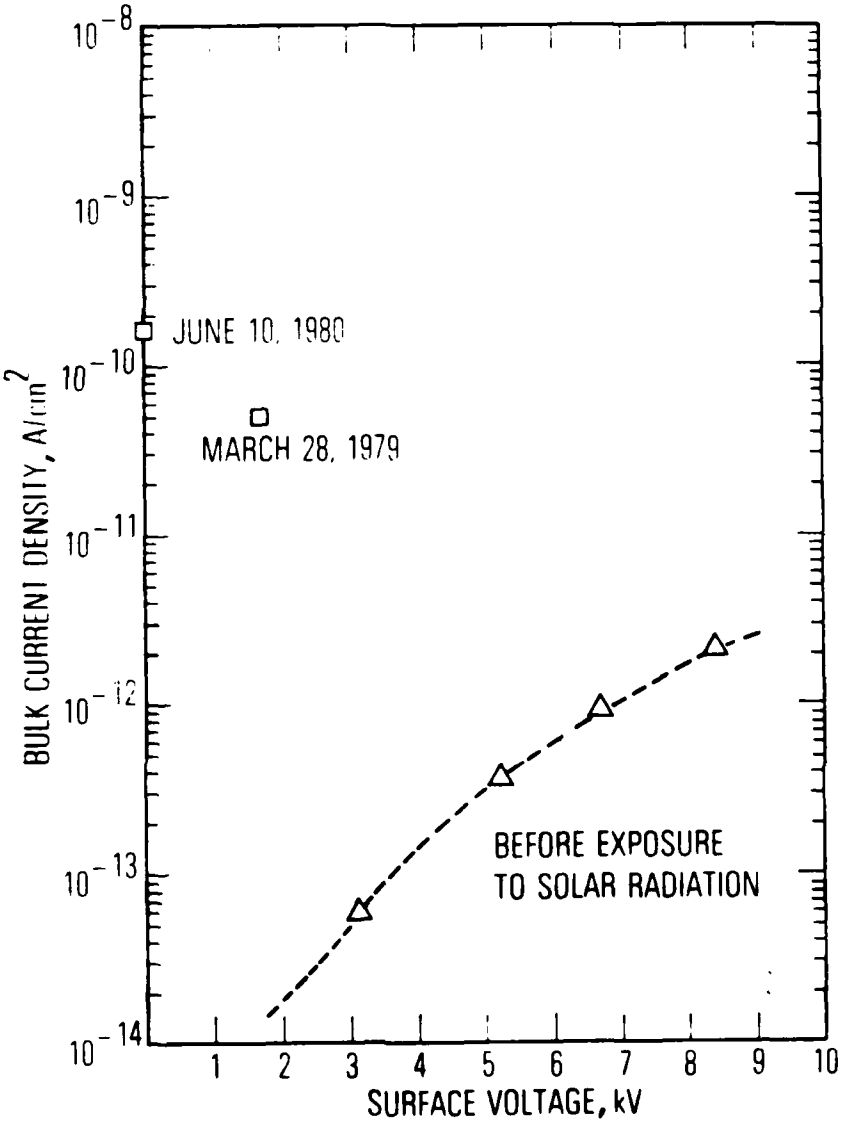

Fig. 2. I-V Characteristics of Kapton Samples in Orbit and Before Launch.

environments and under conditions that allow us to obtain a quasi-equilibrium I-V measurement.

In the 28 March 1979 charging event, the bulk current of Kapton showed a drastic increase when compared with the prelaunch measurements. During this event the surface charged to more than a kilovolt. This behavior indicates that the conductivity of the material 2 mo. after launch was considerably higher than expected, but still in the range where the surface charging is largely determined by the secondary electron emission. Approximately 15 mo. later, in the 10 June 1980 charging event, the bulk current was four times higher than that observed in the 28 March 1979 event, and at the same time the Kapton samples registered only small surface voltages. This behavior suggests that the Kapton samples exposed to the sun had become so conductive that they were no longer capable of supporting a significant surface charge buildup. A possibility exists that the conductivity of Kapton can be raised within a reasonably short time in orbit to a level where no appreciable charging can occur even in the plasmas found in periods of intense substorm activity. As a result, an experimental investigation based on ground simulation was initiated to study the change in dark conductivity of Kapton caused by exposure to solar radiation in orbit. The particular areas of interest included the spectral- and time-dependence of the change in conductivity.

III. EXPERIMENTS

Exposure to simulated solar radiation and measurement of I-V behavior of Kapton were carried out in a vacuum chamber evacuated to 10^{-7} Torr. A helium cryopump was used to maintain the pressure in the chamber. In Fig. 3 is shown the schematic of the measurement setup. The samples consisted of 5- and 2-mil Kapton films aluminized on one side.* The aluminized surface of each sample was attached by conductive epoxy to an etched copper circuit board. A diagram of the sample holder is given in Fig. 4. An electron gun was used to charge the unmetallized Kapton surface while a noncontacting electrostatic potentiometer was used to measure the surface potential developed. For a 4-kV charging beam, the surface potential of Kapton was found to be 3 kV. This result agrees with our previous findings, and the difference between the charging beam potential and the Kapton surface potential is well understood.[7]

The procedure for carrying out the electrical measurements followed the general format given below. The specimen was irradiated in the vacuum chamber with the xenon lamp for a given period of time. Interference bandpass filters were used to isolate the particular wavelengths in the different spectral regions. The filters are designated by the wavelengths (in nm) at which the transmission maxima occur. For example, filter 425 has a transmission maximum at 425 nm. The bandwidth of these filters measured at half height is approximately 50 nm. The spectral regions chosen for study in this experiment cover three separate portions of the Kapton absorption spectrum, regions where Kapton is strongly absorbing, partially absorbing, and nearly transparent. A summary of the absorption characteristics of both the filters and a 5-mil Kapton film is given in Fig. 5. The dark conductivity of the specimen was determined after each period of irradiation. In order to measure the dark conductivity, the I-V behavior of the specimen was determined in the absence of light and after the specimen temperature and current were allowed suffi-

*All of the Kapton films used in our study were obtained from Sheldahl in Northfield, Minnesota.

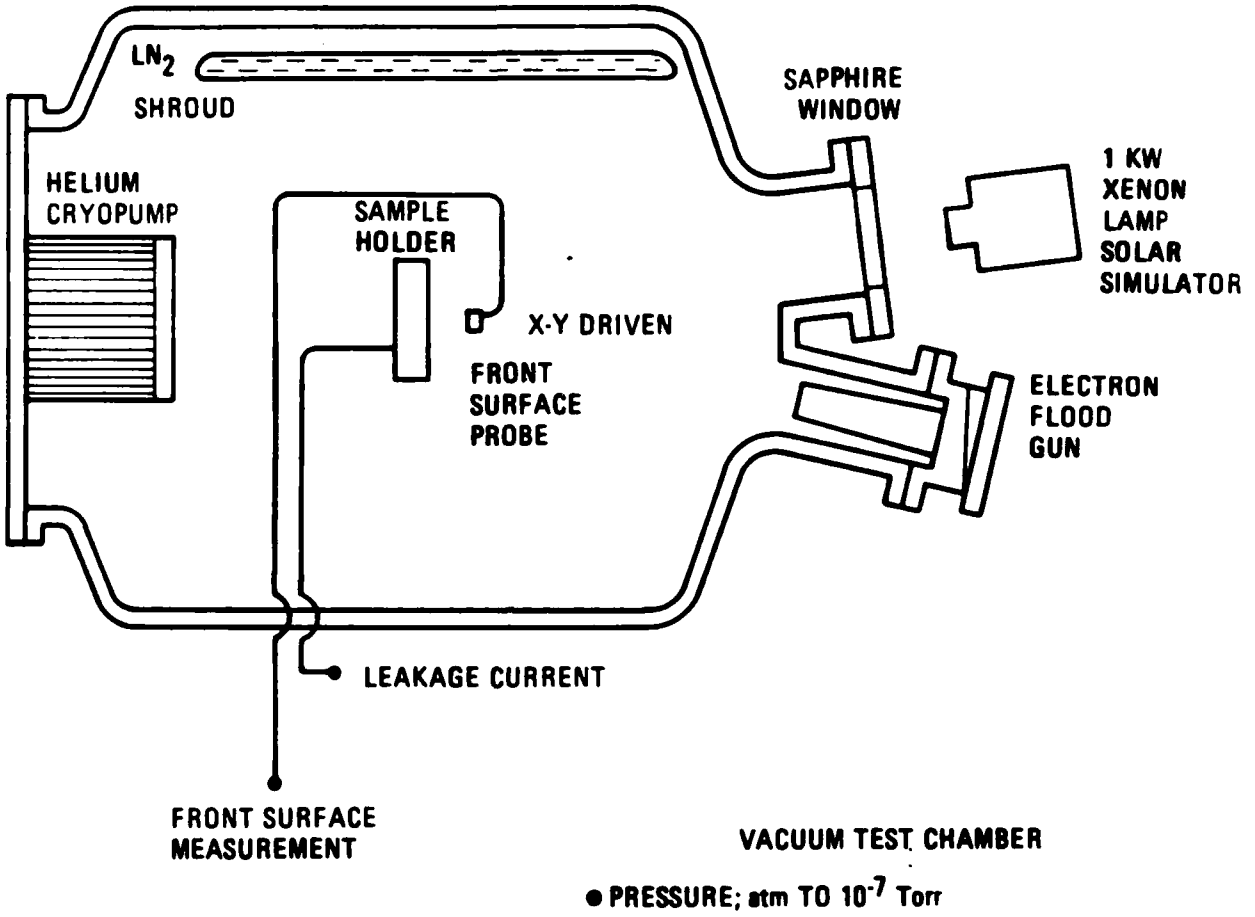

Fig. 3. Schematic of the Test Chamber.

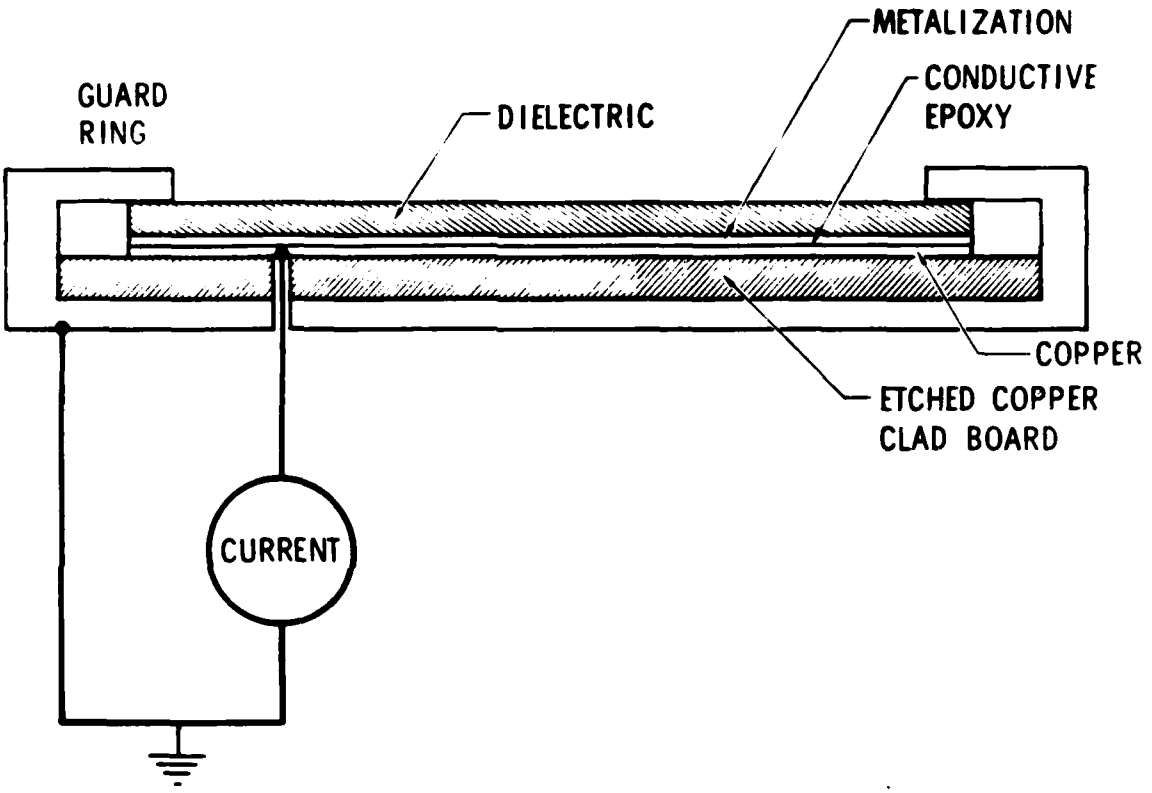

Fig. 4. Diagram of the Sample Holder.

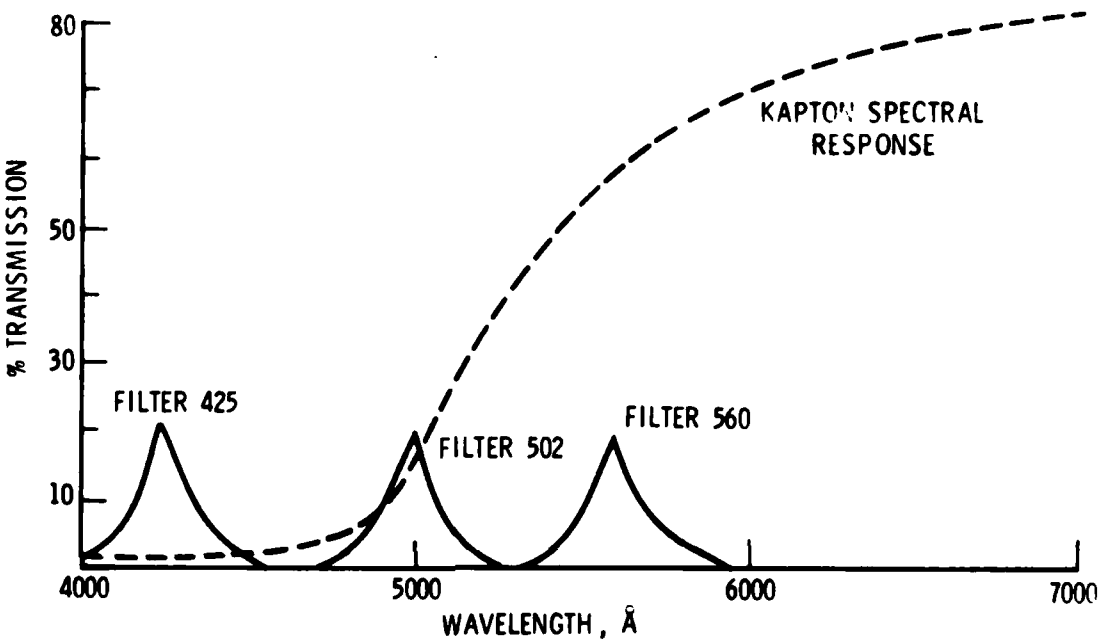

Fig. 5. Absorption Characteristics of 5 mil Kapton and the Filters Used.

cient time to come to equilibrium. Similar measurements were carried out on 2- and 5-mil Kapton specimens prepared from different production batches. For the exposure time dependence study, unfiltered output of the xenon lamp was used.

IV. RESULTS AND DISCUSSION

Three sets of experiments have been carried out: (1) investigation of the spectral dependence of the increase in dark conductivity of Kapton, (2) determination of the effects of different gaseous environment on quenching the increased conductivity, and (3) determination of the dark conductivity increase as a function of exposure time. Our results indicate that photochemical reactions are ultimately responsible for the increase in dark conductivity observed in Kapton. Furthermore, our results show that Kapton can be made conductive in a much shorter time than we expected earlier. The following sections discuss our findings in detail.

A. SPECTRAL DEPENDENCE

A summary of our ground test results on spectral dependence is given in Table 1. The difference in bulk current before and after exposure is an indication of the increase in conductivity, since there was no appreciable change in the surface potential.* Filter 502 produced the largest increase in bulk current for both the 2- and 5-mil Kapton samples, as shown in Table 1. Filter 425 produced the smallest increase. Strictly speaking, from an energetics point of view, photons transmitted by filter 425 are more energetic and more likely to induce a conductivity change in the material. However, these photons are also strongly absorbed, and as a result, do not penetrate very deeply into the material. The absorption spectrum of Kapton in Fig. 5 reveals that, at the wavelength where filter 425 is transmitting, less than 1% of the photons can penetrate the entire thickness of a 5-mil Kapton sample. Since the change in conductivity is measured through the film, it is critical that the photons responsible for the conductivity change should penetrate the entire thickness of the material. Consequently, although the photons have

*Results of our previous studies on surface charging of insulators indicate that, when the bulk current is small compared to the incident charging current, the surface potential is controlled by the secondary electron emission characteristics of the material and is not dependent on the conductivity of the material.

Table 1. Change in Dark Current in Kapton After Exposure to Selected Portions of the Solar Spectrum

	Filter	Exposure Time (h)	ΔI (pA)	ΔI per hour (pA/h)	Average	Ratio[a]	Adjusted[b] Ratio
2-mil Kapton	425	15.8	15	0.95	1.10	0.11	0.15
		16.0	20	1.25			
	502	15.6	135	8.66	9.99	1.00	1.00
		16.8	190	11.32			
	560	15.8	75	4.74	4.25	0.43	0.40
		16.0	60	3.75			
5-mil Kapton	425	16.3	0	0.00	0.00	0.00	0.00
		16.0	0	0.00			
	502	18.4	60	3.26	3.30	1.00	1.00
		15.0	50	3.33			
	560	15.8	10	0.63	0.94	0.28	0.26
		16.0	20	1.25			

[a]Normalized to the largest change in bulk current.
[b]Adjusted for filter transmission, spectral output of 1000-W xenon lamp, and relative photon flux.

sufficient energy to cause the changes that are responsible for conductivity increase, they fail to penetrate enough to produce a significant increase in bulk current for the 5-mil Kapton sample. However for the thinner 2-mil Kapton sample, more photons can penetrate the entire thickness of the film, and a slight increase in bulk current was observed.

On the other hand, according to the absorption spectrum of Kapton, a large percentage of photons from filter 560 can penetrate both the 2- and 5-mil Kapton films. These longer-wavelength photons lack the energy required to cause the chemical changes necessary to increase the conductivity of the film. As a result, only minor increases in the bulk current were detected. Filter 502 was chosen because its transmission maximum coincided with the absorption-band edge of Kapton. The photons in this spectral region have both depth penetration and the energy appropriate for photochemical reactions. Under these conditions, electrical conductivity through the thickness of the film was found to increase dramatically.

B. QUENCHING STUDY

The results we have presented so far can be explained by the formation of electrically active centers generated in the bulk of the material by photochemical reactions. In an attempt to identify which chemical species produced by the photochemical reactions are electrically active, we carried out a series of experiments in which the quenching effects of various gases were studied. In these experiments, Kapton specimens were irradiated with a 1000-W xenon lamp. After the increase in dark current was measured in a vacuum of 10^{-7} torr, a particular gas was admitted to the vacuum chamber, and the pressure of the chamber was raised to 400 torr. The specimen remained in this gaseous environment for 16 h. At the end of this period, the change in dark current was measured after the chamber had been reevacuated to a pressure of 10^{-7} torr. The gases used in this study were dry N_2 and dry air. We found dry N_2 had no effect on the dark current of the specimen, but dry air was extremely effective in quenching the increase in dark current.

These results suggest oxygen is responsible for deactivating the electrically active centers in the specimen. It is well known that organic radicals

or radical ions are byproducts of photochemical dissociation of polymeric materials such as Kapton. Acting as donors or acceptors, these radicals are responsible for the observed increase in conductivity. When exposed to dry air, these radicals can react with oxygen to form more stable peroxy radicals that appear to be electrically inactive. As a result, the increase in conductivity is quenched. This finding is consistent with our conjecture that the conductivity increase is photochemical in origin. Although we did not perform a long-term study to determine the stability of these electrically active centers, our results suggest that these centers appear to be stable at 10^{-7} torr. During the several weeks in which the experiment was performed, no appreciable degradation in dark conductivity of the sample was observed. This evidence is indirect, however, because the sample was continually being irradiated. A more direct experiment is needed to define the relationship between ambient oxygen partial pressure and the rate of decrease in dark current. Such a study would provide the information necessary to determine the stability of these electrically active centers in different environments.

C. EXPOSURE TIME DEPENDENCE

In order to make use of the light-induced increase in conductivity to control electro-static change buildup, it is essential to determine how the conductivity of Kapton varies with exposure time. A plot of the change in I-V measurements as a function of irradiation time for a 5-mil Kapton film is shown in Fig. 6. The electron beam voltage used in this particular case was 4 kV. The increase in the measured dark current shown in Fig. 6 is accompanied by a decrease in the surface potential indicating that the conductivity of the specimen is increasing as a result of irradiation.

Further investigation of the I-V behavior of the specimen revealed that the potential of the specimen measured after irradiation became less dependent on the incident charging beam voltage (Fig. 7). More importantly, the I-V curve shown in Fig. 8 obtained after 706 min of irradiation was found to be linear. Furthermore, the surface voltage was found to increase linearly with the incident beam current density and not the beam voltage. These results indicate that the specimen was behaving like a resistor, and the electron beam

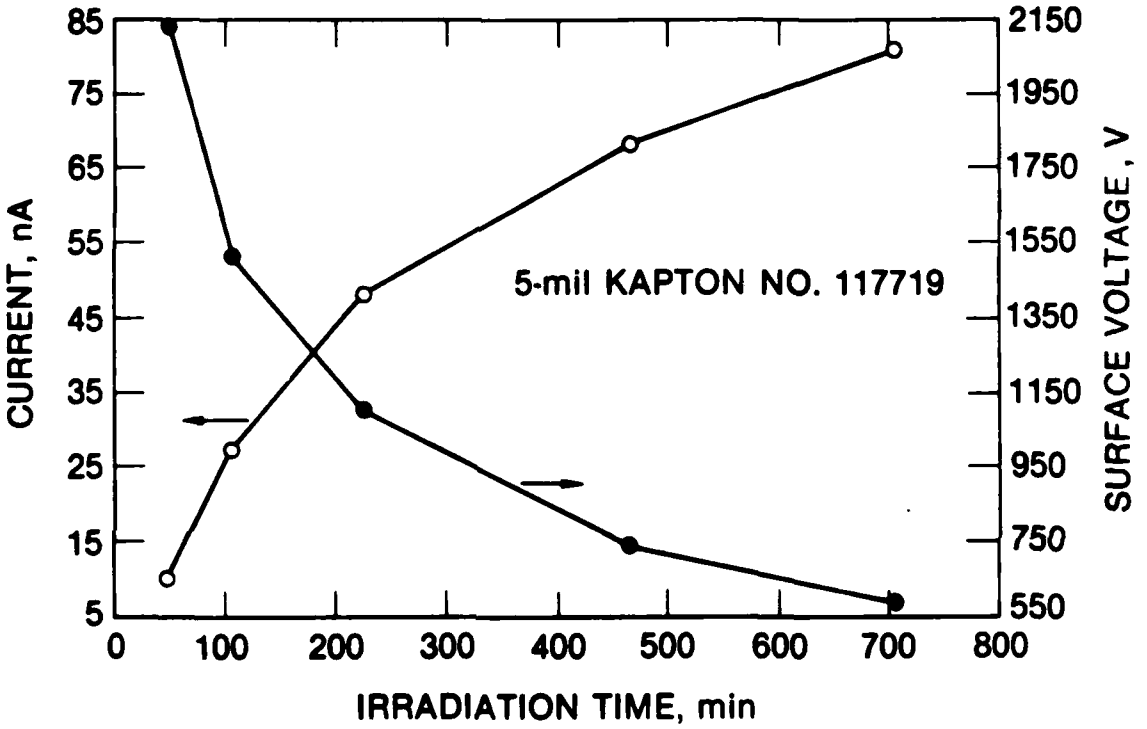

Fig. 6. Plot of I-V Measurements as Function of Irradiation Time.

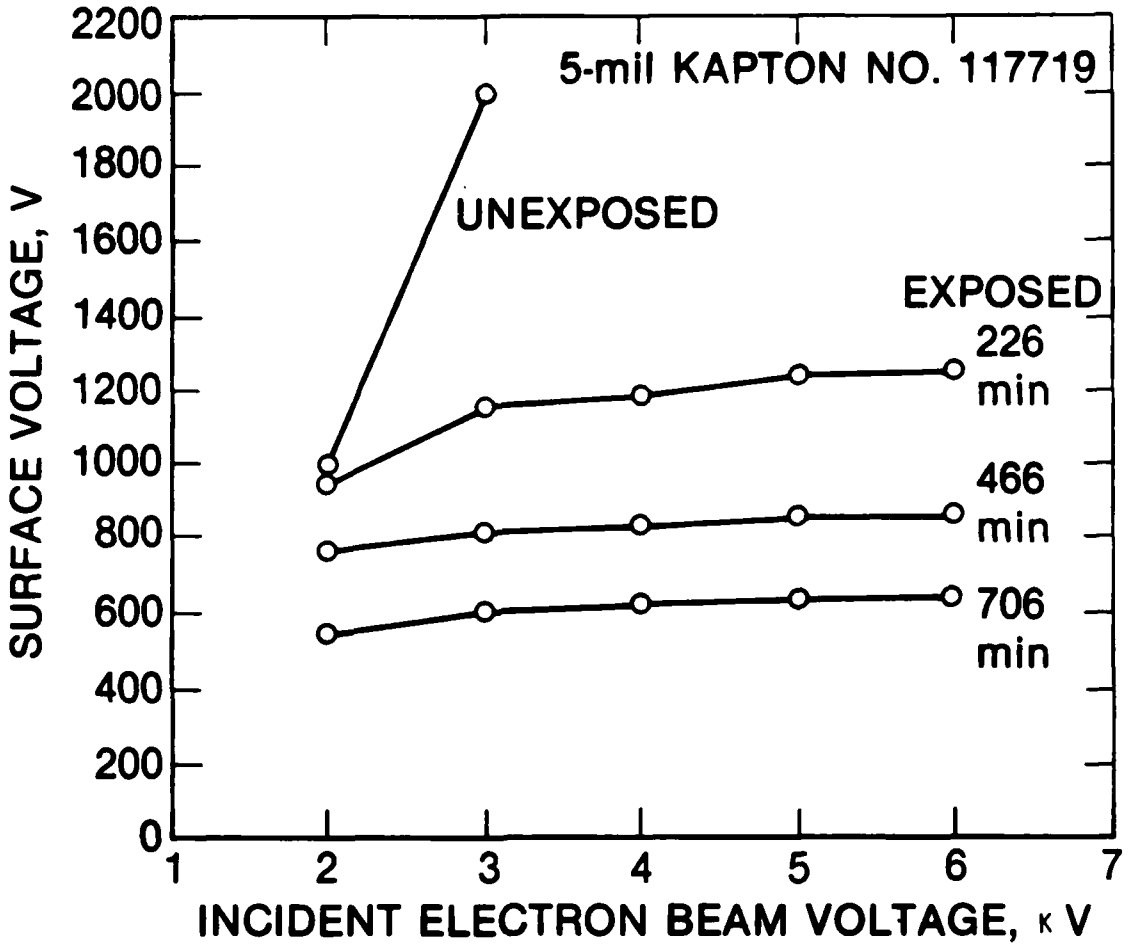

Fig. 7. Charging Behavior of Specimen Before and After Irradiation.

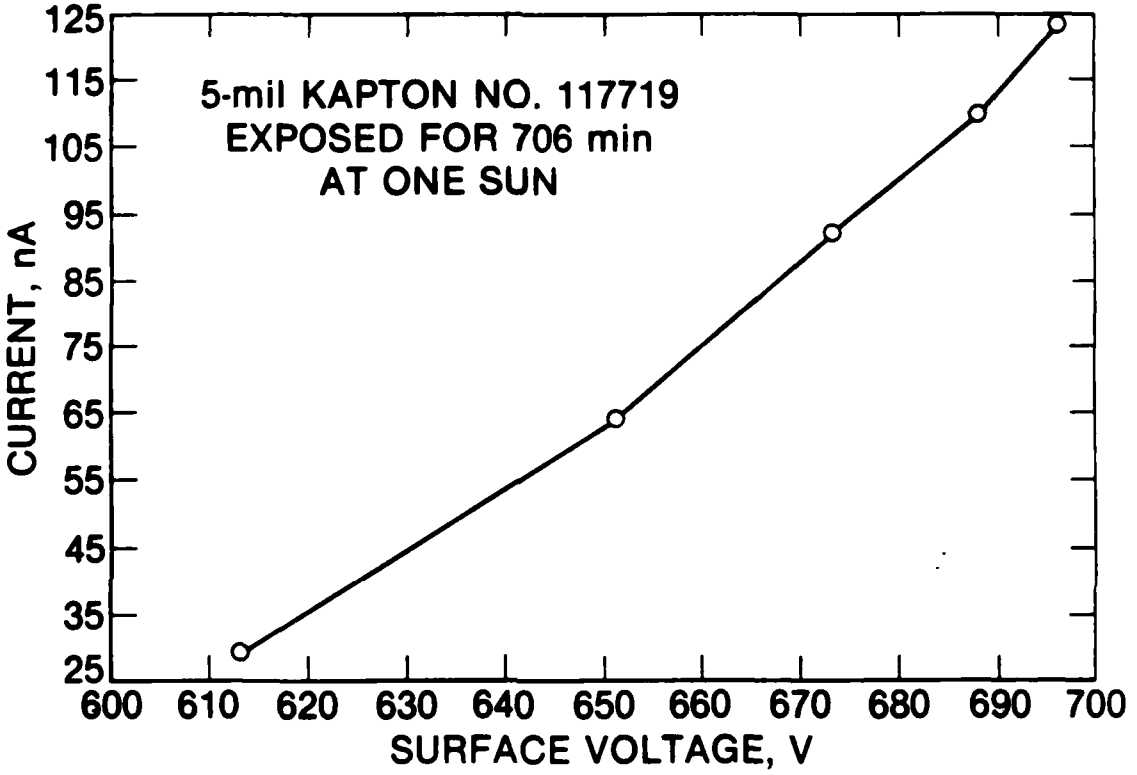

Fig. 8. I-V Characteristics of Specimen After 706 min of Irradiation.

was acting as a current source. This behavior represents a drastic departure from the I-V characteristics of the material before it was irradiated. Before irradiation, the surface potential varied linearly with the incident beam voltage and did not depend on the beam current density at all. This effect had been studied in detail by the authors earlier and found to be the result of secondary electron emission-controlled charging.[7] This phenomenon occurs only in low-conductivity materials where the major part of the incident beam current has to be balanced at the sample surface by secondary electron emission instead of conduction. For the specimen that is more conductive, the material will exhibit I-V characteristics more closely resembling those of a resistor.

In the case under study, the conductivity increases were caused by exposure to solar radiation, and we were able to follow the I-V behavior as the conductivity of the specimen was increased stepwise (Fig. 9). A detailed analysis of the I-V curves was performed to obtain an estimate of the resistance of the specimen at different exposure times. The resistivity data are summarized in Table 2. We find that the increase in conductivity is directly proportional to the increase in exposure time. This finding agrees with our earlier observation that the conductivity increase in Kapton was caused by the accumulation of electrically active centers produced by photochemical reactions. The agreement can be explained by examining the basic charge transport equations.

$$j = \sigma E \tag{1}$$

and

$$\sigma = e\, n\, \mu \tag{2}$$

where j, σ, E, e, n, and μ are current density, conductivity, electric field, electronic charge, and carrier density and mobility in Kapton, respectively. Since carrier mobility is a bulk property, it is not expected to be affected by minor chemical changes in a low-mobility polymeric material such as Kapton. Therefore, the increase in conductivity must be largely the result of

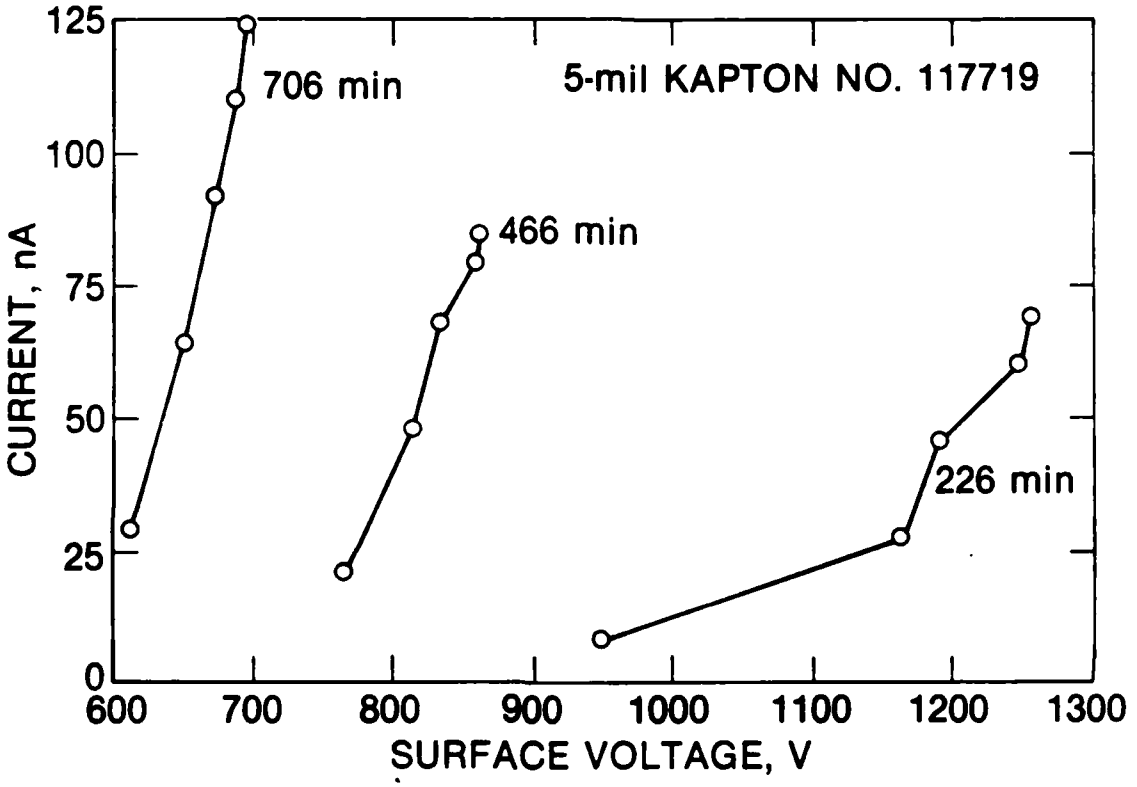

Fig. 9. I-V Characteristics of Specimen at Various Irradiation Times.

Table 2. Change in Dark Conductivity of Kapton With Exposure Time

Exposure Time (min)	Increase in Exposure Time (min)	Resistivity ρ_R Ωcm	Conductivity σ $(\Omega cm)^{-1}$	Increase in Conductivity $(\Omega \cdot m)^{-1}$
226		19.4×10^{12}	0.5×10^{-13}	
	240			1.5×10^{-13}
446		5.1×10^{12}	2.0×10^{-13}	
	240			1.4×10^{-13}
706		2.9×10^{12}	3.4×10^{13}	

an increase in the carrier density which, in turn, is controlled by the number of electrically active centers present in the material. At the same time, because the active centers are produced photochemically, the concentration of these centers should be directly proportional to the total dosage of light received by the specimen. In unimolecular photochemical reactions, the change in product concentration, $\frac{dn}{dt}$, is directly proportional to the light intensity, I, or

$$\frac{dn}{dt} = \eta I \tag{3}$$

when η is the quantum yield for the reaction. Integrating the change over time at constant light intensity,

$$\Delta n = \eta I \Delta t \tag{4}$$

and, therefore

$$\Delta\sigma = e \mu \eta I \Delta t \tag{5}$$

Hence, it is reasonable to expect the increase in conductivity of Kapton to be directly proportional to the exposure time, which is exactly what we found from our measurements.

In addition, we have uncovered a rather unexpected result from studying Kapton specimens prepared from different production batches. We found that different batches of Kapton exhibited similar increases in conductivity but differed markedly in the rate of increase under the same exposure conditions (Fig. 10). The specimen from batch numbers 117719 required approximately 10 times less light than the specimen from batch number 25976 in order to reach the same level of conductivity. This finding has important ramifications on the development of antistatic thermal control surfaces for future spacecraft. Our results show that the increase in conductivity of Kapton can be accelerated or inhibited by varying processing conditions or additives. More

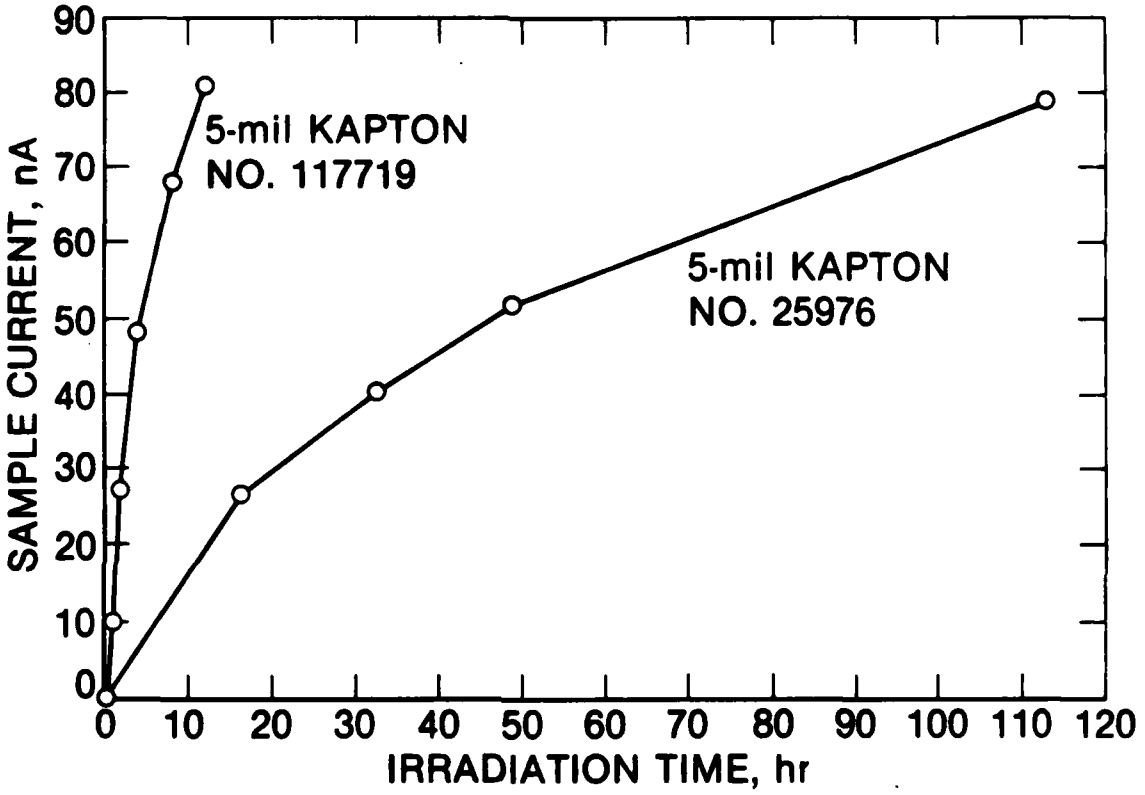

Fig. 10. Different Batches of Kapton Exhibiting Markedly Different Rate of Dark Conductivity Increase Under the Same Irradiation Conditions.

specifically, it means that the conductivity of Kapton in space can be tailored to match the requirements of specific end-use applications, provided that the basic mechanism controlling the rate of conductivity increase is known. A series of additional experiments had been conducted to investigate the chemical differences that may exist between the different batches. No extraneous differences in chemical additives were detected in the infrared spectra of the films or in the gas chromatographic analyses of extracts from samples from the two different batches. However, the specimen from batch number 25976 appears to be darker and the absorption spectrum shows a shift of the absorption band edge to a longer wavelength. Since no measurable difference in additives was found, we speculate that the shift in absorption could be the result of a higher degree of thermal crosslinking.* Kapton is a polyimide made up of monomers derived from benzene. The polymer is yellowish brown in color because of the localization of the π electrons in the benzene ring. Crosslinking the polymers increases the interaction between the π electrons and gives rise to a more intense optical absorption band as well as a shift of the band edge to a longer wavelength. The results of the spectral dependence study show that only a narrow portion of the visible spectrum, near the onset of the absorption band, is effective in producing a conductivity increase; which means that the threshold energy for photochemical reaction is only slightly lower than the absorption band edge. Consequently, a shift of the absorption band to lower energy can drastically reduce the number of photons available in the bulk of the material for reaction and thus have a significant effect in lowering the rate of conductivity increase. So far, this explanation is only a speculation, even though it seems to be consistent with all our previous findings.

*It was found that density, color intensity, and viscosity increase as the Kapton film receives greater thermal treatment.[8]

V. SUMMARY AND CONCLUSION

The spectral dependence of the dark conductivity of Kapton has been determined. It was found that only a very narrow portion of the solar spectrum is effective in causing an increase in the through-film dark current of Kapton. Experiments were also carried out to study the efficiency of various gaseous environments in quenching the light-induced dark current. The results indicated that the increase in the dark conductivity of Kapton is caused by an accumulation of electrically active centers produced photochemically in the bulk of the material and these active centers should have an extremely long lifetime in the absence of oxygen. This finding suggests that the light-induced increase in conductivity of a Kapton film can persist for a long time in orbit where there is a very low density of oxygen atoms or molecules.

The increase in conductivity of Kapton as a function of exposure time has also been determined and analyzed in detail. The analysis showed that the conductivity increase is directly proportional to the exposure time. This observation is consistent with the concept that the increase in conductivity is photochemical in origin. Markedly different rates of increase in conductivity were also observed in Kapton specimens from two different production batches. The difference in rates can be explained by the shift of absorption band edge to a longer wavelength. One important conclusion that can be drawn from our results thus far is that it is possible to raise the conductivity of Kapton to a level where it can be used as an antistatic spacecraft surface in a reasonably short time by exposing Kapton to solar radiation in space. However, before Kapton can be recommended for use as an antistatic spacecraft surface, more work would be needed to determine how long the increased conductivity will persist in space.

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