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PHOTOCONDUCTIVITY OF HIGH-VOLTAGE SPACE INSULATING MATERIALS-- MEASUREMENTS WITH METAL ELECTRODES

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SUMMARY

The electrical conductivities of high voltage insulating materials were measured in the dark and under various intensities of illumination. The materials investigated included FEP Teflon, Kapton-H, fused quartz, and parylene. Conductivities were determined as functions of temperature between 22 and 100°C and light intensity between 0 and 2.5 kW/m². The thickness dependence of the conductivity was determined for Teflon and Kapton, and the influence of spectral wavelengths on the conductivity was determined in several cases. All measurements were made in a vacuum to simulate a space environment, and all samples had metallic electrodes. The conductivity of Kapton was permanently increased by exposure to light; changes as great as five orders of magnitude were observed after six hours of illumination. Since this material is being used as the substrate for satellite solar cell arrays, prolonged exposure to sunlight might lead to degraded solar panel performance.

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I INTRODUCTION

This program was undertaken to generate experimental data on the photoconductivity of materials used for high-voltage insulation on spacecraft, and to correlate these data with available theoretical analyses. It is anticipated that the results of this work will assist designers in selecting materials and in predicting changes in the electrical conductivity of these insulators in the presence of solar illumination.

The materials used in these studies include FEP teflon, kapton-H, fused quartz, and parylene. Teflon is a well known insulating material used in a wide variety of applications and, because of its extensive use, was regarded as a particularly important material to evaluate. Kapton has good dark insulating properties, is mechanically stable at high temperatures, is reported to retain its good mechanical characteristics after UV radiation, and is being used with increasing frequency in thermal-control blankets and other space applications. These desirable characteristics and its frequent usage suggested that this material should also be evaluated. Fused quartz is used as cover material for solar cells and for back-surface mirrors for thermal control on some spacecraft. Unusual dielectric breakdown phenomena, possible leading to RF pulses that interfere with digital equipment, have been observed in silvered fused quartz samples in our laboratories. This usage, and the possibility of quartz breakdown interference, led to the selection of quartz as a material for study.

Parylene-coated samples are relatively new, but hold promise for extensive use in future spacecraft applications. The unique feature of parylene is that it is deposited at subatmospheric pressures and

polymerizes when it contacts the substrate. This has led to unusually pin-hole-free coverage on samples having complex geometries--for example, on circuit boards. Its photoconductive properties were also measured.

Bulk-conductivity measurements were made on samples of each of these materials both in the dark and with various intensities of illumination from a xenon lamp. Surface conductivities were measured on samples of each material in the dark, and in some cases with visible and infrared light. Each type of material was studied at three temperatures--22, 66, and 100°C. All measurements were made in a vacuum of 10^{-5} torr or less, the typical vacuum being 2×10^{-6} torr.

In order to separate conduction effects caused by the electrode from those intrinsic to the material, two types of measurement are being made. One type utilizes metallic electrodes deposited on both sides of the sample, and the second type utilizes either ions or electrons as one of the electrodes. This interim report discusses only the results obtained on samples with metallic electrodes on both sides.

Analysis of the results will be postponed until the results of the experiments with nonmetallic electrodes are available. The complexity of the mechanisms involved in the conduction process will preclude any but a cursory analysis. The experimental results and some preliminary interpretations are presented in later sections of this report.

II CONDUCTION PROCESSES

Although the processes involved in the conduction of electricity are well understood in metals, semiconductors, and ionic fluids, the analogous processes in insulators are not well established. Least understood of all are the organic insulators, or organic semiconductors as they are sometimes called.

Several factors contribute to the present uncertainties with respect to conduction in dielectrics. First, is the difficulty associated with having several possible conduction mechanisms operating simultaneously. The second is the alteration of the conduction current by the type of electrode used, and the third is the alteration of the characteristics of the sample in the course of measuring its properties. The results obtained on a particular material can depend significantly on the history of the sample at the time of measurement.

When the electric field applied to a sample is small, electrical conduction is due to the motion of ions or electrons. Since the energy gap between the valence and conduction bands is large, little electronic conduction is experienced at low temperatures. Trapped electrons, however, can have energies close to the conduction band and can contribute to the conduction process. Ionic conduction can occur either by migration of the ions of which the sample is composed, or by migration of impurity ions in the sample.

The conduction process in intense electric fields can be dominated by the electrodes. In this case, charge carriers are emitted from the electrodes into the conduction band. Electrons can be freed by electron tunneling or by thermionic emission from the cathode into the sample.

High field conduction can also be dominated by ions, leading to a linear dependence of the current density on the hyperbolic sine of the field.

Photoconduction generally occurs when electrons are excited from the valence band to the conduction band by absorption of an incident photon. Similar effects can occur when the photon's energy is absorbed by an electron or ion trapped at an imperfection in the lattice. The latter mechanism leads to an enhanced conductivity after the illumination is extinguished, since some time is required before the charge carriers again become trapped at an imperfection.

Many of the above mechanisms involve the removal of a charge carrier from a trapping center and therefore inherently result in a recombination time for the carrier and the trap. Recombination times can be very short or very long--in some cases, weeks. It was impractical, in the experiments discussed herein, to observe these long recombination times although some short-time measurements were recorded.

Some of the above phenomena will be identified in the data presented in later sections of this report.

III APPARATUS, SAMPLES, AND METHODS OF MEASUREMENT

Samples of kapton were cleaned with isopropyl alcohol, lightly brushed, and dried in clean air prior to the sputtering of electrodes onto the surfaces of the samples. Teflon was treated in the same manner, but was not brushed due to the resultant surface abrasion. Parylene samples were deposited on polished copper plates by the Hughes Corporation and were tested as received. Quartz was cleaned as described above.

All samples were 0.1 m (4-in) square or round. The front-surface gold electrodes were nominally 20 nm (200 \AA) thick; the back-surface gold electrodes were opaque. Electrodes were deposited on the front surface as follows. A central circular electrode 0.0302 m in diameter was deposited and served as a guarded electrode for all bulk-conductivity measurements. A second annular electrode having an inside diameter of 0.0332 m and an outer diameter of 0.076 m was used as the guard electrode. The 0.0015-m gap between the two electrodes was used for surface-conductivity measurements.

The samples were mounted on a flat aluminum plate and pressed to it by a circular brass electrode placed against the guard ring. Contact to the center electrode was via a small brass rod. The sample mount was heated by an electrical heating element on the dark side of the mount, and cooled either by air or water. Temperatures were measured by a thermistor on the aluminum plate and controlled by a proportional temperature regulator.

The samples, holder, and heater were located in a vacuum chamber that was pumped by an oil diffusion pump equipped with a water baffle and a liquid nitrogen cold trap. Illumination of the sample was provided by

a xenon lamp, and entered the chamber through a quartz window. Neutral-density filters were used for light attenuation.

Voltages from a highly regulated (0.025%) power supply were applied to the sample through a 10-megohm resistor and were measured using a 100:1 voltage-reduction probe. The bulk and surface currents were measured using two HP 425 picoammeters. One of these was placed in the high-voltage line to the sample and was powered through an isolation transformer. The second was connected between the guard electrode and ground and was used to measure surface current. For consistency, all dark-current measurements were made 60 seconds after the voltage was applied. All photocurrents were measured after the voltage was applied in the dark for 30 seconds, and the sample was exposed to the light with the voltage applied for an additional 30 seconds.

Dark, bulk conductivity measurements at various temperatures and voltages were made prior to illumination of the samples. This was necessitated by permanent changes induced in some of the samples by the xenon light. Consequently, dark, bulk conductivity and photoconductivity measurements were not always made in succession.

Dielectric breakdown effects were avoided when possible, and measurements were made below the voltage at which breakdowns previously had occurred in similar samples.

IV EXPERIMENTAL RESULTS

The data obtained from the experiments performed in this program to date are summarized in this section. To facilitate comparisons between, and selections of, materials, the results of measurements on all materials are combined in discussing the various phenomena studied.

A. Dark, Bulk Resistivity

Measurements of the dark, bulk resistivity were made on all samples tested. As mentioned earlier, variations were observed in the resistivities of kapton, teflon, and parylene after they were installed in the vacuum chamber. Variations were also noted from one sample to the next. In order to achieve an acceptable degree of confidence in the results, several samples of teflon and kapton having the same thicknesses were measured and the results averaged in obtaining the results presented here. Quartz was measured in only the thickness previously used in satellite back-surface mirrors (2.54×10^{-4} m). Parylene samples were received late, and only one sample of 6.1×10^{-5} m thickness was tested.

Changes in the resistivity of kapton after installation in the vacuum chamber were substantial, and a considerable effort was expended in achieving reproducible results on this material. By heating these samples for several hours at 100°C , and by repeatedly applying high electric fields to them, it was found that the resistivity could be stabilized, and reproducible measurements could be made. Further, after this preconditioning, the agreement between different samples was as good as, or better than, that found in teflon. Similar effects were found in teflon and parylene but were much smaller in magnitude.

Figures 1, 2, and 3 present the results at 22, 66, and 100°C on all the materials tested. The bulk resistivity of fused quartz at 22°C was at the limits of the sensitivity of the apparatus for this thickness. At 39.4 MV/m (1 kV/mil), the resistivity was about 10^{16} ohm-m; the resistivity increased in lower fields. Due to the scatter in these data, they are not presented. The dielectric strength was determined to be 50 MV/m in one quartz sample.

Dielectric breakdown in thick teflon and kapton samples occurred at lower field strengths than in thinner samples of the same materials. This factor limited the field-strength range over which measurements could be made in these samples. Fused quartz and teflon exhibited the best average dielectric properties of the materials tested, but the former suffered from a very low dielectric strength. The resistivity of teflon in samples with thicknesses of 5.08×10^{-5} and 1.27×10^{-4} m exceeded the resistivity of samples of kapton with comparable thicknesses by factors of 3 to 8, depending on the electric field and temperature.

The thinnest teflon exhibited resistivities that varied significantly in the three samples tested. The very rapid decrease in resistivity with temperature in high electric fields was apparent in all three samples.

Parylene had the lowest resistivity of all the materials tested. The results presented here were obtained on one sample and could deviate from the usual behavior. No attempt was made to establish the dielectric strength of this material, and measurements were restricted to conservative electric fields.

With the single exception of the thinnest teflon samples, all the samples exhibited a common behavior at high electric fields. The resistivity of all the materials decreased with increased electric field, and the field level at which the downturn in the resistivity occurred was dependent on the temperature. At temperatures above 66°C, the slope of the

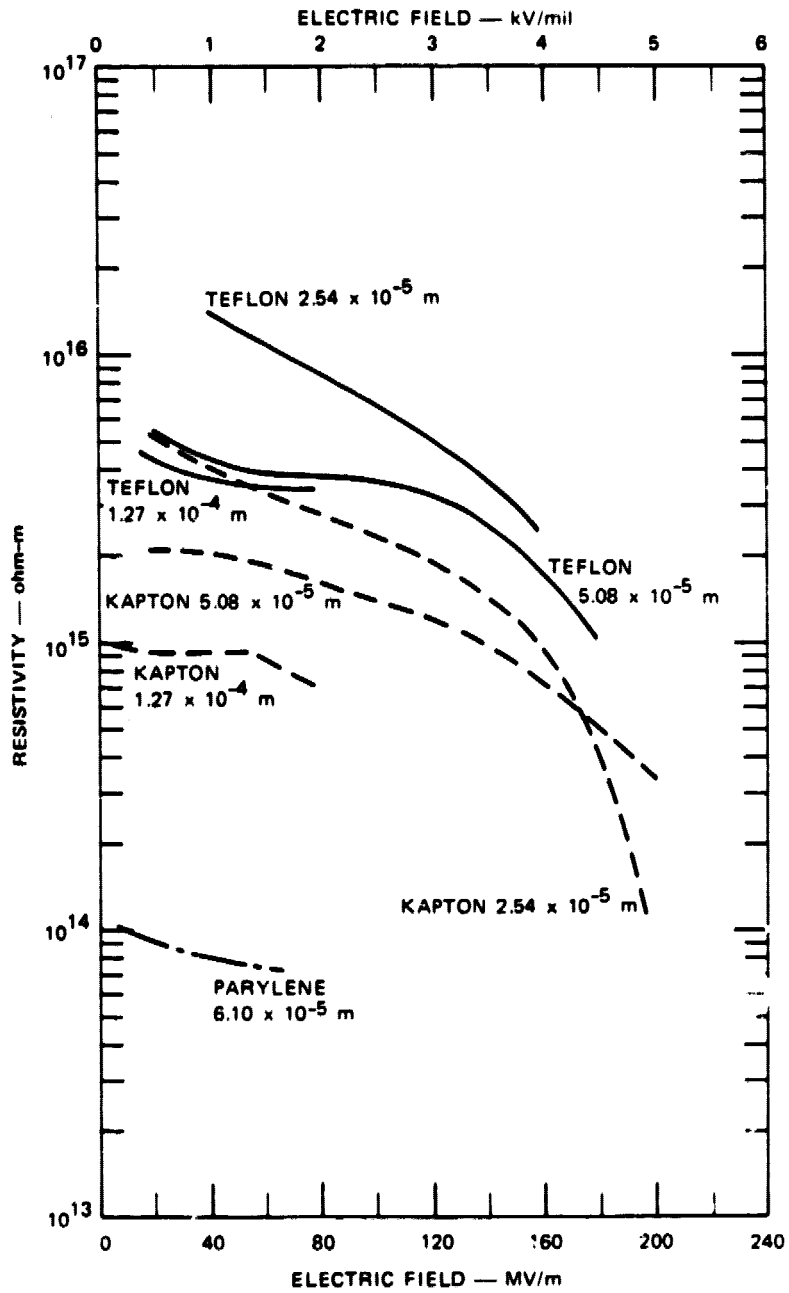


FIGURE 1 DARK, BULK RESISTIVITIES OF INSULATORS vs ELECTRIC FIELD AT 22°C

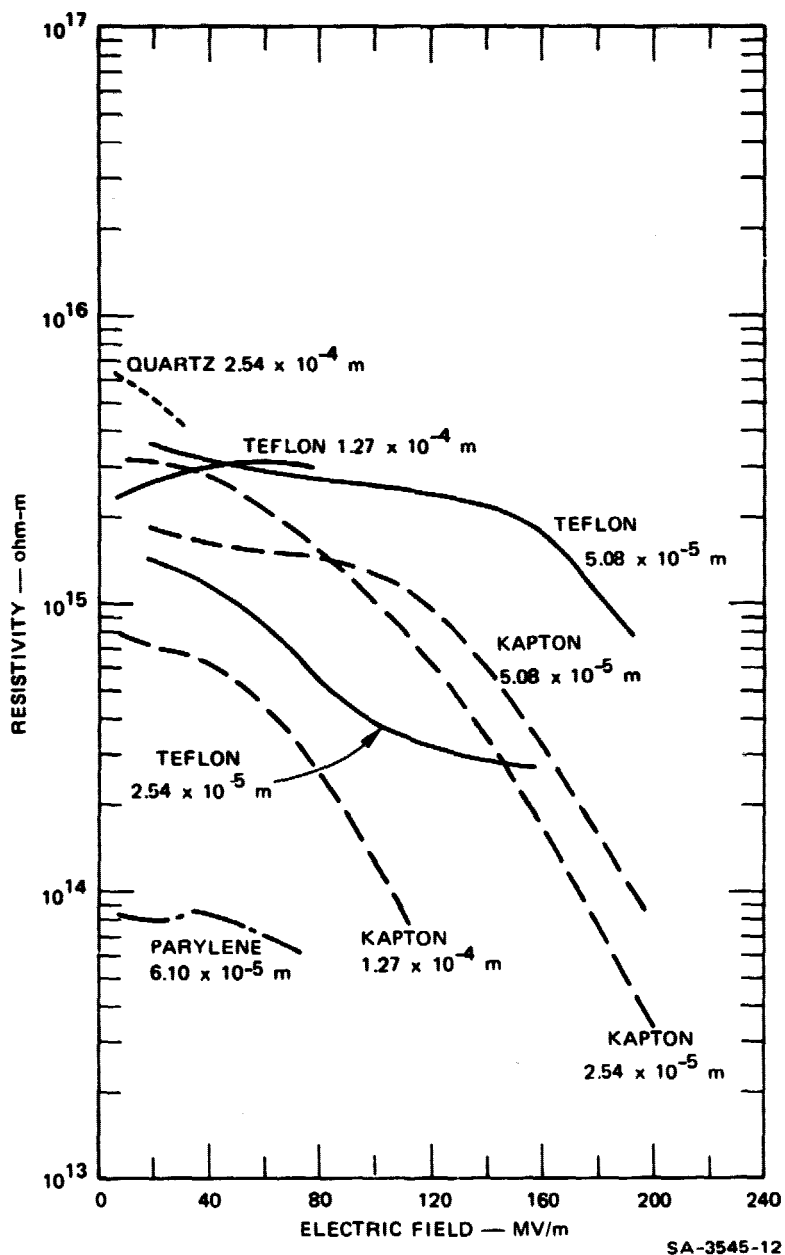


FIGURE 2 DARK, BULK RESISTIVITIES OF INSULATORS vs ELECTRIC FIELD AT 66°C

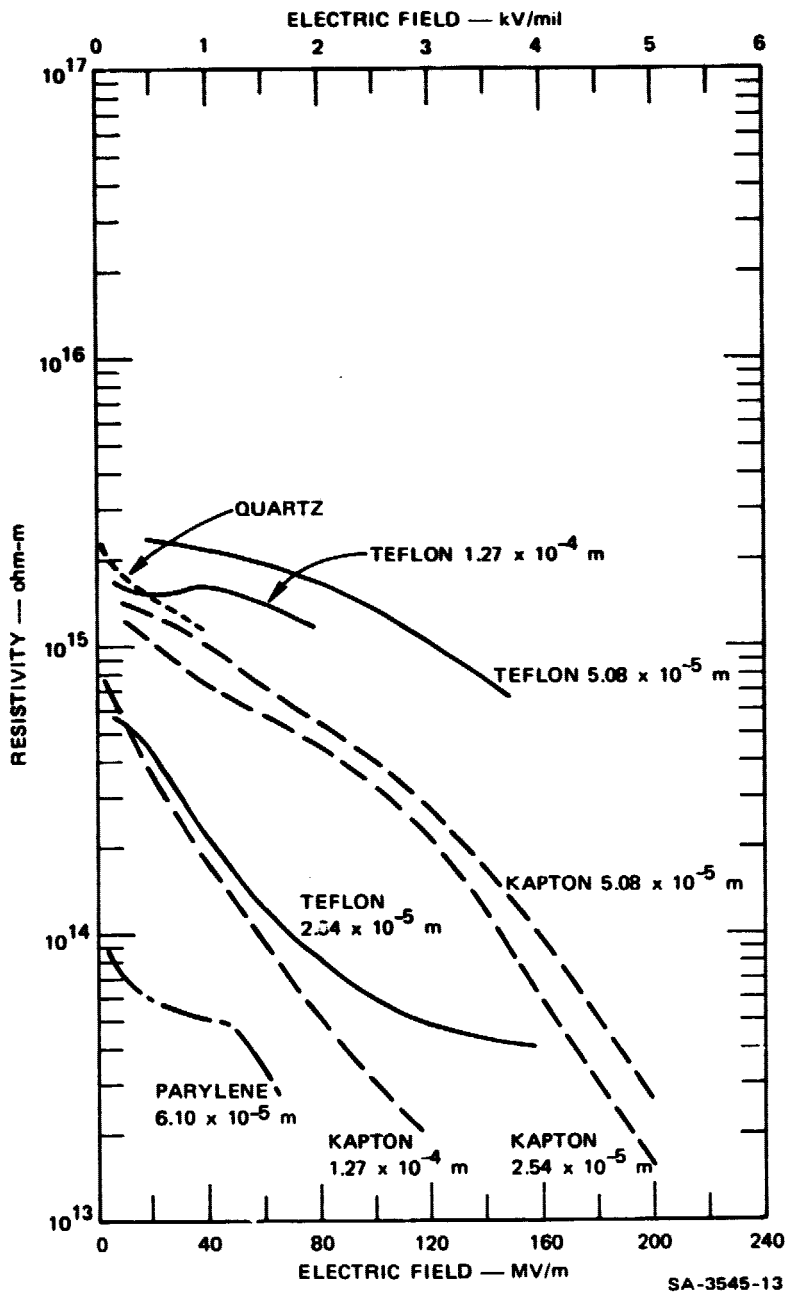


FIGURE 3 DARK, BULK RESISTIVITIES OF INSULATORS vs ELECTRIC FIELD AT 100°C

resistivity-versus-electric-field curves appears to be approaching the same limit in all materials.

The data of the 1.27×10^{-7} -m kapton sample at 100°C are replotted in Figure 4 on a Schottky plot, where it is seen that the data are well fitted by an $\exp(aE^{1/2})$ function. This function describes the electric-field-enhanced thermionic emission of electrons from the negative electrode into the dielectric. The data obtained at 66°C in the same samples indicate similar behavior at high fields, as do the data for the thinner kapton samples at 100°C . An inspection of Figures 1 through 3 shows that a similar behavior occurred in teflon and parylene. At lower fields and at lower temperatures, however, conduction is clearly due to different processes. Further, the characteristic shape of the curve for the kapton current fitted in this figure evolves only after the sample has been thoroughly outgassed.

The data shown in Figure 5 were obtained on a single kapton sample 1.27×10^{-4} m thick, and show the effects of outgassing and the application of an electric field on the conductivity of kapton samples. The curvature of the j -versus- E curves also changes upon outgassing. After outgassing, the j -versus- E curves becomes linear at high fields while the same curves in untreated samples have some curvature in this region. This suggests that field emission occurs in both cases but that an additional conduction mechanism also occurs in the virgin sample. Since it is known that kapton outgasses substantial amounts of water vapor, it is reasonable to expect that the higher conductivity in unoutgassed materials is due to ionic conductivity by the water.

No attempt has been made at this time to clearly define the conduction processes in any of these materials.

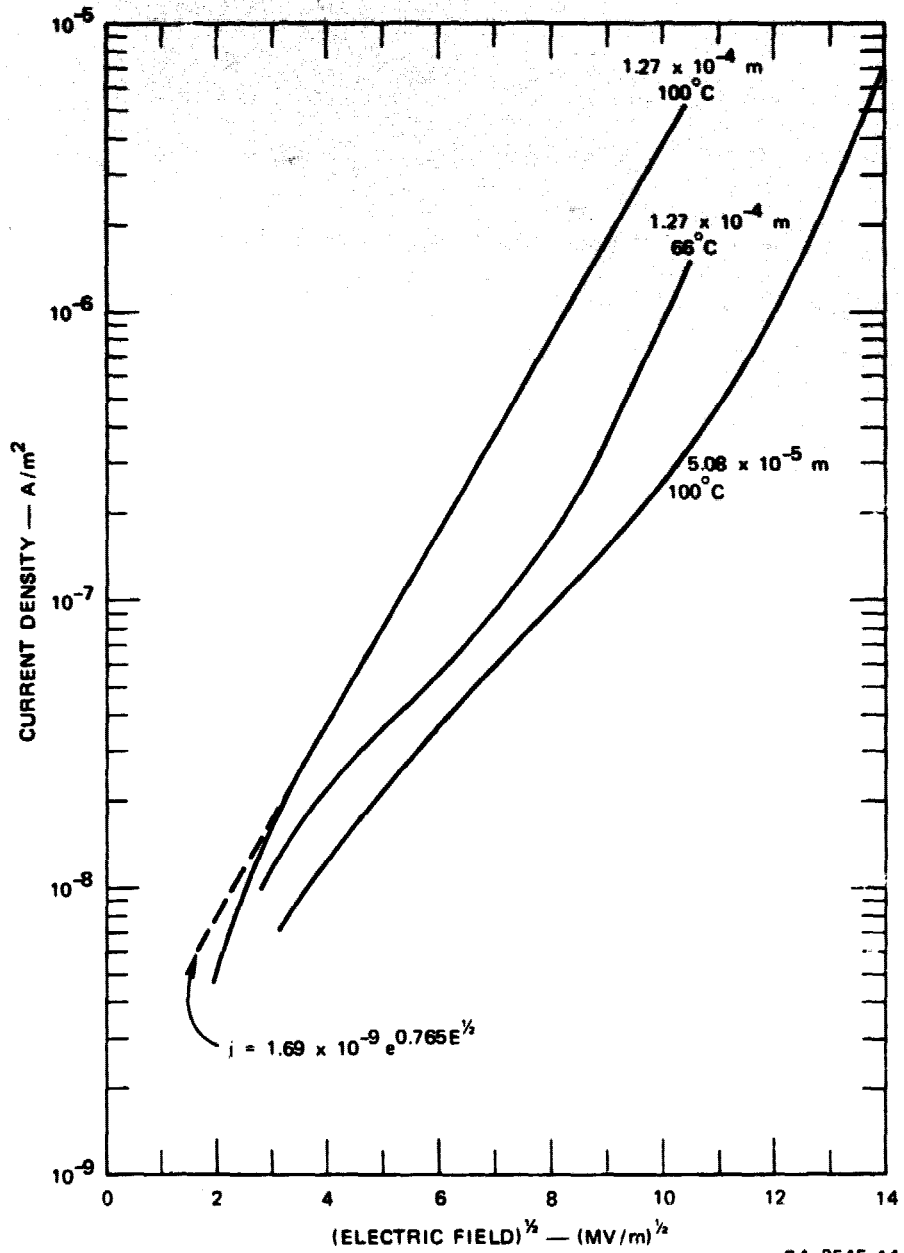


FIGURE 4 CURRENT DENSITY vs (ELECTRIC FIELD)^{1/2} — KAPTON

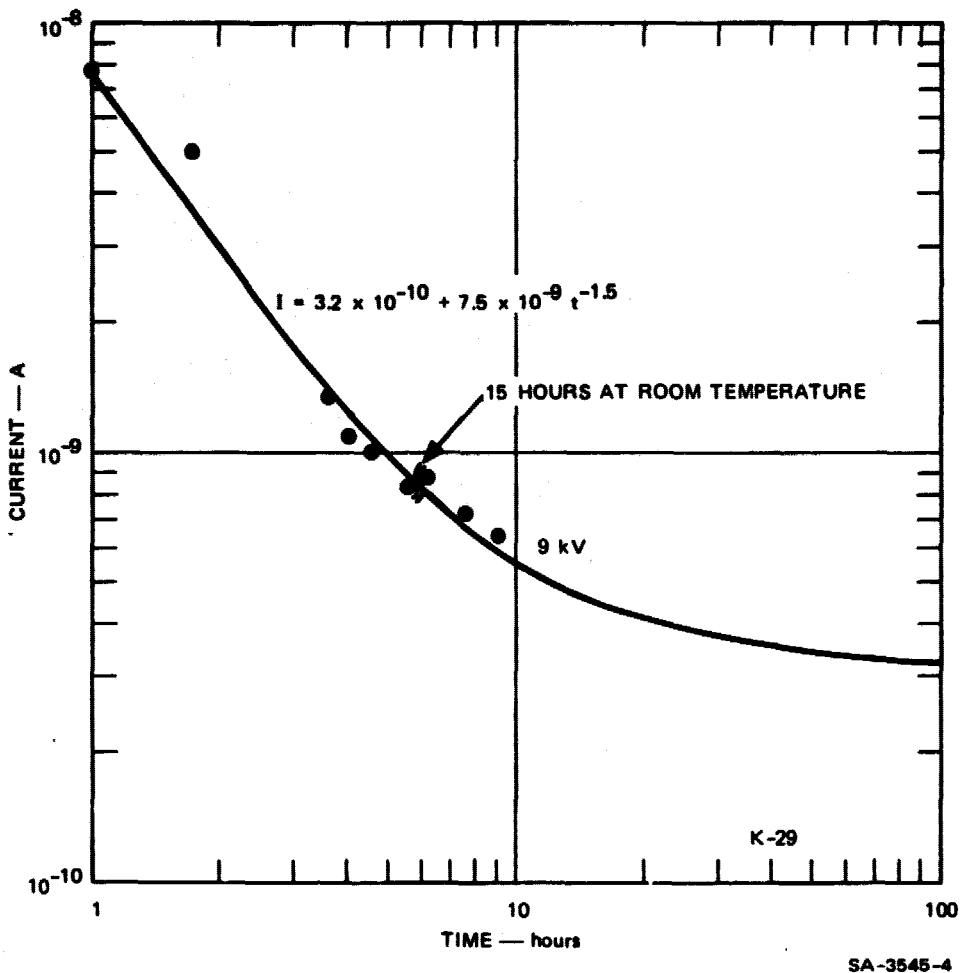


FIGURE 5 DARK, BULK CURRENT IN KAPTON 1.27×10^{-4} m THICK AT 100°C vs TIME IN VACUUM

B. Dark, Surface Resistivities

Surface resistivities are known to vary with the condition of the surface in all materials, and are as much a function of the contamination on the surface as they are of the material itself. In the samples studied, the surface resistivity was immeasurably large in almost all cases when the samples were well outgassed in the vacuum. Consequently, in most cases, only lower limits could be placed on the resistivity. The highest measurable resistivity at 10 kV was 6.3×10^{17} ohms. Measurements of the surface resistivity were somewhat erratic, perhaps because of changes in

the surface state upon application of electric fields. This behavior reduced the practical limit of the resistivity measurements to values only slightly in excess of 10^{17} ohms. These resistivities are several orders of magnitude higher than the resistivities usually measured at atmospheric pressure.

The surface resistivity in quartz at 100°C was determined to be approximately ohmic in nature and had a value of 7.9×10^{15} ohms. At 22°C , the surface resistivity exceeded 1.7×10^{17} ohms at all electric fields up to 31.5 MV/m.

Both kapton and teflon had surface resistivities that varied widely from sample to sample. It was assumed that the highest resistivities correspond to the true surface resistivities of the materials and that low resistivities correspond to surface contamination. In both cases, the highest resistivities exceeded 10^{17} ohms. Also in both cases, one or more samples were found that diverged widely from this result. One sample of teflon had a surface resistivity lower by 10^4 than other samples and one sample of kapton was lower by a factor of 10^2 . These anomalous resistivities may possibly be the result of diffusion of gold under the mask onto the surface during the deposition of the electrodes, although there was no visible evidence to confirm this possibility. In the case where the surface conductivity was anomalously high, the bulk conductivity was comparable to other samples of the same material.

All the polymeric materials became polarized after they were subjected to high electric fields, and this resulted in large currents with no voltage applied. This was particularly true in the case of the surface currents in parylene. Consequently, changes were sought in the initial current upon application of a surface voltage. The lower limit on the surface conductivity of parylene at 100°C determined in this way was 1.5×10^{16} ohms, increasing to 1.5×10^{17} ohms at 22°C .

C. Bulk Photoconductivity

Measurements of the bulk photoconductivity in insulators yielded conductivities that differed by large factors from conductivities measured in the dark. Although significant changes were observed in all the materials studied, the most significant results were obtained in kapton, which was permanently changed by illumination. Since the behavior of kapton was unlike that of the other materials it will be discussed separately.

During the course of bulk-photoconductivity measurements it was found that light having an appreciable UV content produced major changes in the conductivity of teflon and quartz, as shown in Figure 6. Through filtering of the incident light, it was determined that approximately 90% of the photocurrent in quartz and about 95% of the photocurrent in teflon was produced by radiation in the 200-to-300-nm band.

This spectral band also contains photons of sufficient energy (4.1 to 6.2 eV) to cause photoemission from gold or copper, suggesting that electrons are being emitted from the negative back electrode by UV transmitted through the sample. Parylene was relatively unaffected by the UV radiation. This discrepancy can be explained by the sharp increase in the optical density of parylene below 400 nm, as shown in Figure 7. At 300 nm, only 10% of the UV was transmitted in a sample 2.54×10^{-5} m thick. The sample used in the photoconductivity measurements was 2.4 times this thickness and would have absorbed most of the UV capable of causing photoemission from the electrode. Since the UV blocking filter necessarily transmits some UV below 300 nm, and since the cutoff is not perfectly sharp at this frequency, some of the conductivity observed with the UV "blocked" may still be caused by the UV.

This result suggests that the controlling factor in the conduction process in quartz and teflon is not the dielectric properties but the photoemissive properties of the metallic coating and the UV content of

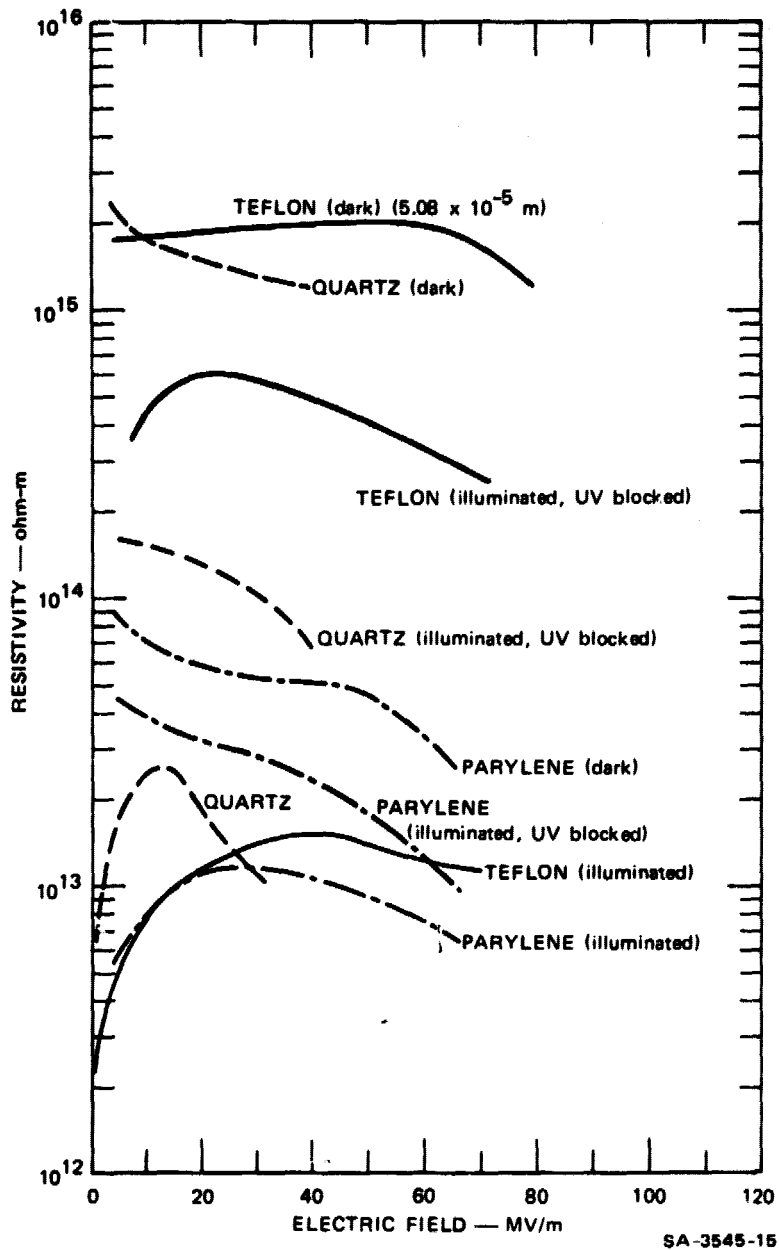


FIGURE 6 DARK, BULK RESISTIVITIES AND PHOTORESISTIVITIES OF TEFLON, QUARTZ, AND PARYLENE WITH AND WITHOUT UV RADIATION — 100°C

the incident light. When the UV is prevented from reaching the metallic electrode, the resistivity is not seriously degraded.

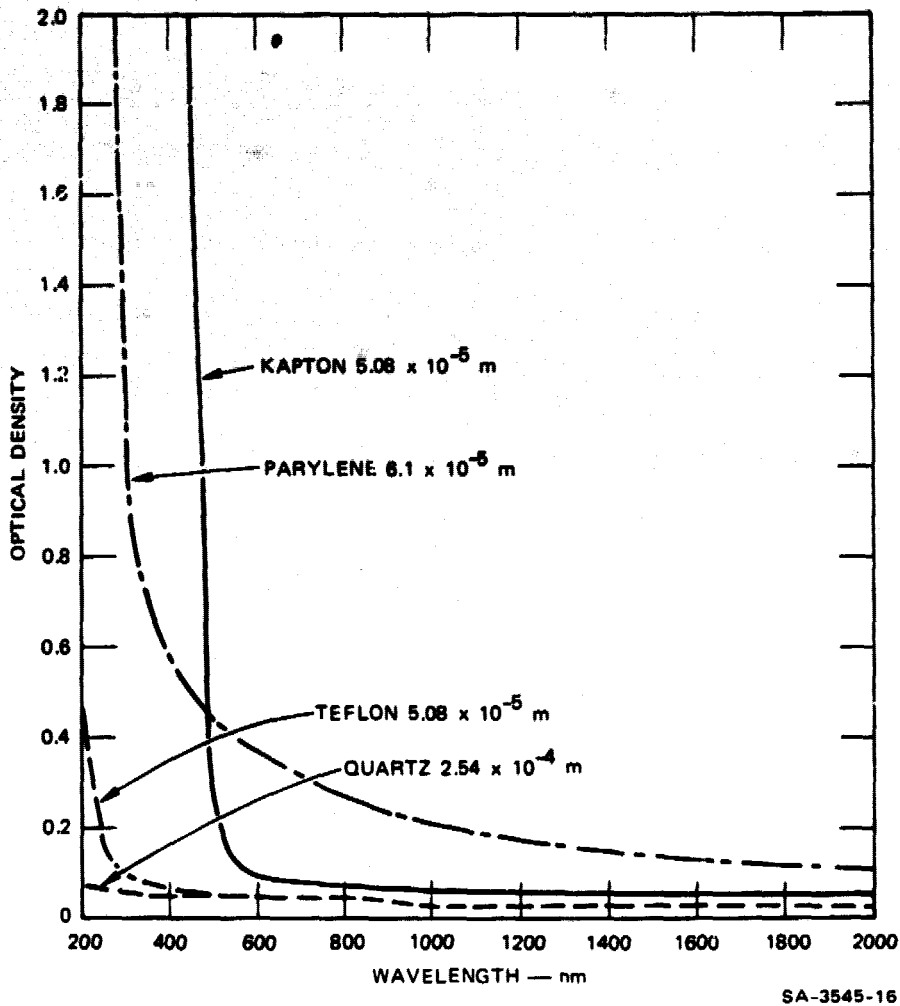
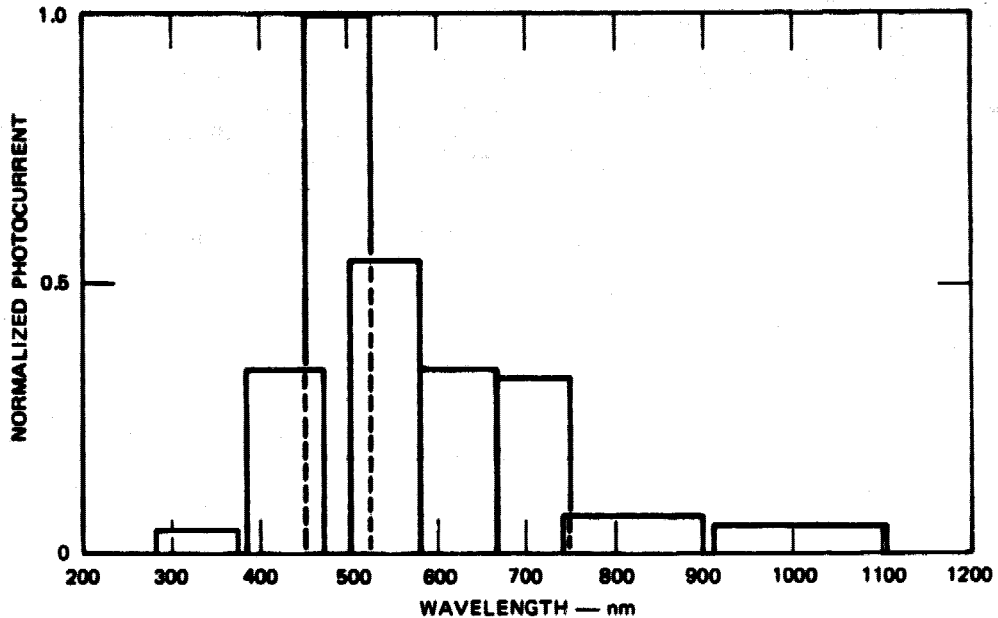


FIGURE 7 OPTICAL DENSITY OF TEFLON, PARYLENE, KAPTON, AND QUARTZ vs WAVELENGTH

The photocurrents were found to be only slightly temperature-dependent when the UV was blocked. Parylene was virtually independent of temperature, and between 22 and 100°C, quartz varied by factors of 3 and 7 at 39 MV/m and 8 MV/m, respectively, and at 2.5 kW/m² of incident radiant power. Reducing the incident power had little effect on these ratios. At low power levels and high voltages, teflon varied by a factor of 6 in the conductivity.

The spectral response of the photocurrent was determined in parylene and teflon with the normalized results shown in Figures 8 and 9. The results were normalized to remove the spectral variations in the output of the source and the bandwidth of the filters. Parylene has a broader photoresponse than does teflon and also was more responsive in the violet and near-UV.



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FIGURE 8 NORMALIZED PHOTOCURRENT vs WAVELENGTH — TEFLON, 100°C, 39.4 MV/m

Kapton differs from the three materials just discussed in that illumination by xenon light permanently altered the resistivity of the kapton. It was shown in Figure 1 that kapton samples 1.27×10^{-4} m thick had a resistivity of 9×10^{14} ohm-m in the dark with an applied field of 39.4 MV/m (5 kV). In Figure 10, it is seen that after illumination of the sample for 6.5 hours at an intensity of 1.2 kW/m^2 the dark resistivity at the same voltage and temperature decreased to 9.4×10^9 ohm-m, a reduction in resistivity of almost 10^5 in six hours. The numbers at the bottoms of the curves indicate the dark currents measured prior to illumination.

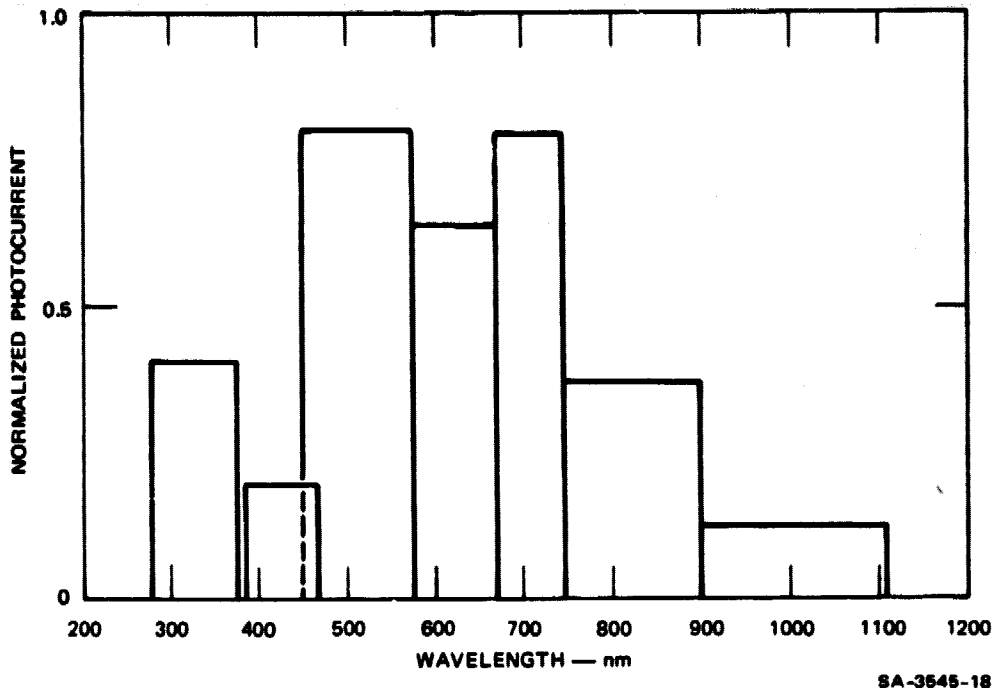


FIGURE 9 NORMALIZED PHOTOCURRENT vs WAVELENGTH — PARYLENE, 22°C, 32.8 MV/m

The extrapolated lines indicate the approximate currents during the first hour of illumination. The first hour of illumination occurred while controlled measurements of the photoconductivity as a function of light intensity were attempted. Most of this period was devoted to measurements at light intensities below 1 kW/m^2 , indicating that the initial increase in conductivity with exposure time was very rapid.

Although the measurements shown in Figure 10 were made in the dark, they were made as soon as possible after the light was extinguished. This leaves open the question of whether partial or complete recovery of the initial resistivity would occur with time. That at least a significant fraction of the decrease is permanent is shown in Figure 11, where the preillumination dark current is contrasted with the dark current 65 hours after the last illumination of the sample. While illuminated, this sample conducted a current of $6 \times 10^{-6} \text{ A}$ at only 100 V with

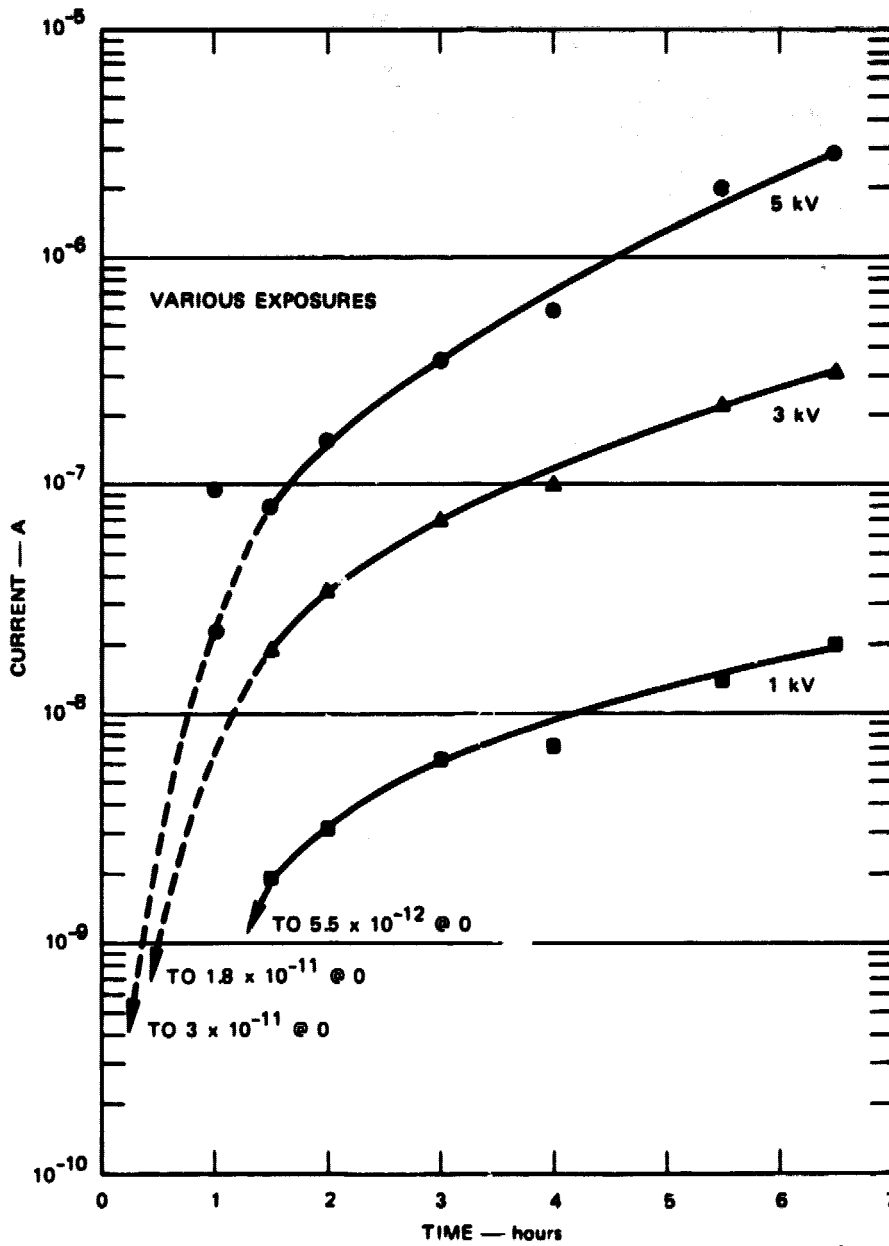


FIGURE 10 DARK, BULK CURRENT IN KAPTON 1.27×10^{-4} m THICK AT 22°C vs TIME OF EXPOSURE AT 1.2 kW/m^2

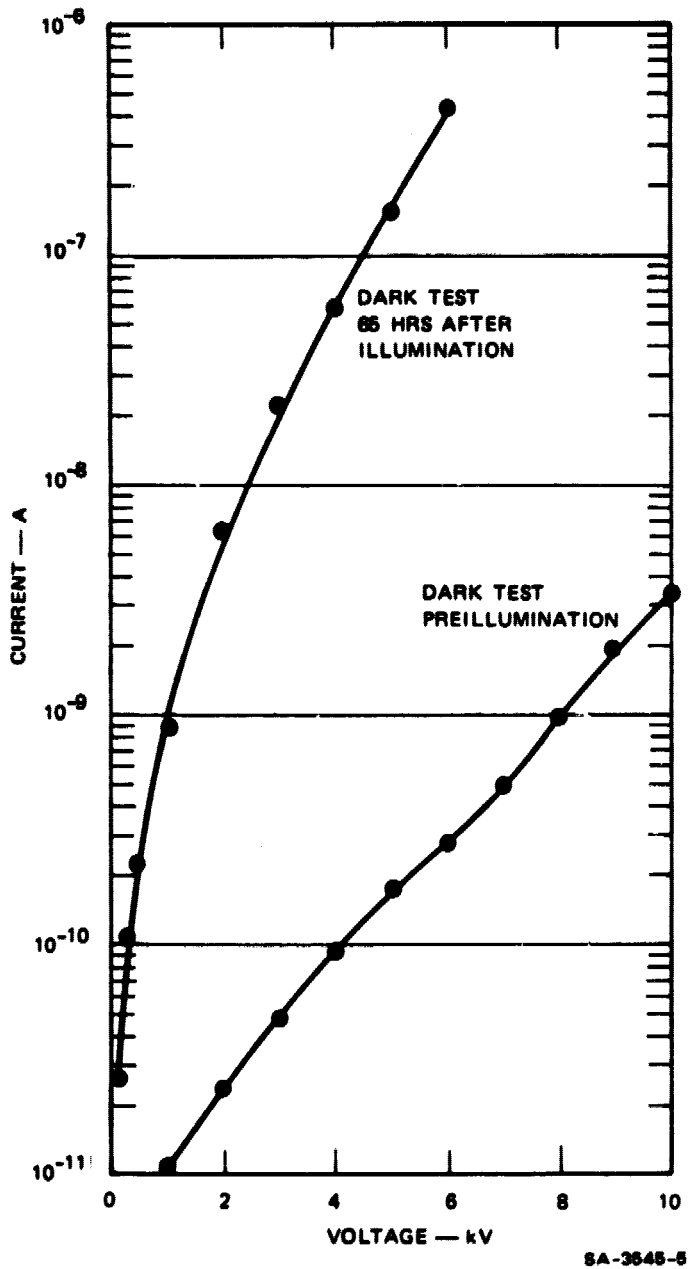


FIGURE 11 DARK CURRENT vs VOLTAGE IN KAPTON 5.08×10^{-5} m THICK AT .00°C

3.73 kW/m² of incident light, corresponding to a resistivity of 1.6×10^8 ohm-m. Referring to Figure 6, it is seen that this conductivity is four orders of magnitude below the worst case shown there.

The rate of recovery of the resistivity after illumination and the change in resistivity during exposure was investigated using the filters then available, as shown in Figure 12. These results indicate that exposure to red and infrared light (including the peak in the spectral emittance of the xenon lamp) had comparatively little effect on the photocurrent and little effect on the residual resistivity. In fact, the current decreased while the light was on, indicating recovery from preceding dark tests. Referring to Figure 7, it is seen that the optical density of kapton is low in this region, indicating little absorption of light. This places the spectral region producing the major photoconductivity below 680 nm.

The second test, encompassing the 300-to-760-nm band, produced a major increase in the photocurrent and resulted in a large change in the residual resistivity. That damage to the sample occurred is also evidenced by the increase in current during the illumination.

Subsequent exposure in the 280 to 380-nm UV band produced a current that is substantially lower than in the preceding test even when corrected for the reduced incident power. This result is surprising in view of the large optical density in this spectral band. From these results, it is evident that the photocurrent and the photodegradation occur primarily as a result of radiation in the 380-to-680-nm region of the spectrum. Broadband irradiations produced photocurrents and changes in the residual resistivity that are consistent with this conclusion. At the inception of these tests, the dark current was 9×10^{-12} A at 3 kV, and the resistivity was 4.7×10^{15} ohm-m. When the tests were completed the resistivity was 7.7×10^{13} ohm-m, 61 times lower although the total time of exposure was only 18 minutes. The 3-kV voltage remained on throughout

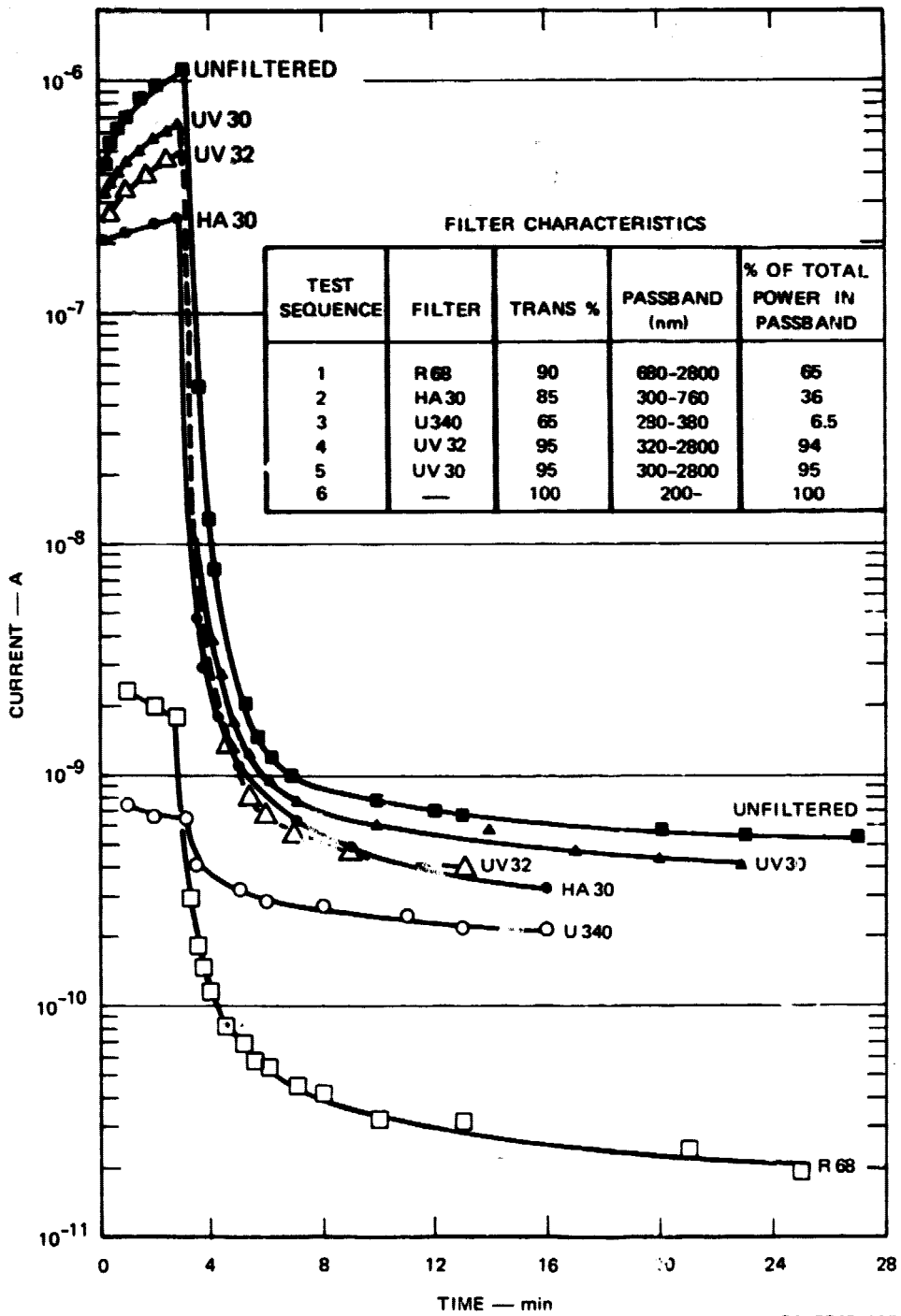


FIGURE 12 CURRENT DURING AND AFTER 3-MINUTE EXPOSURES OF KAPTON 5.08×10^{-5} m THICK AT 66° WITH VARIOUS FILTERS. Incident power on filters = 2.3 kW/m^2 .

these tests. Breakdown occurred in this sample at a current of 6×10^{-6} A after an additional 37-minute exposure.

No attempt was made to determine whether the resistivity of kapton stabilizes after prolonged exposure to radiation. The changes observed, however, suggest that caution be exercised when kapton is used for electrical insulation in a radiation environment, even at electrical fields as low as 2 MV/m (50 V/mil).

V CONCLUSIONS

Although the measurements to date are restricted to samples having metallic electrodes, some conclusions can be drawn from this work.

- The bulk conductivity of kapton was greatly enhanced and permanently altered by exposure to illumination from a xenon lamp. Applications of kapton where the conductivity is a governing factor should be carefully evaluated in the light of these results. The use of kapton as the substrate for solar cell arrays is particularly questionable and might lead to degraded performance after a sufficiently long time.
- FEP Teflon exhibited the best overall insulation characteristics of the materials investigated. In very thin samples, however, the insulation properties were poor and varied from sample to sample.
- Parylene had the highest conductivity of any of the materials tested, but it was relatively unaffected by temperature and exposure to xenon illumination.
- Fused quartz was found to have a high bulk resistivity, but its low electrical breakdown voltage restricts its utility.
- Dark surface resistivities were found to be several orders of magnitude higher in a vacuum than comparable values reported at atmospheric pressure. Under illumination, the surface resistivity is reduced by photoemission of electrons from the negative electrode.