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Electron, Proton, and Ultraviolet Radiation Effects on Thermophysical Properties of Polymeric Films

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The response of coated thin polymer films to simultaneous ultraviolet, electron, and proton radiation has been evaluated by selected measurements in situ and in the laboratory. Exposures simulated the radiation environment near the Earth–sun Lagrangian points 1 and 2 for five years and ~1000 ultraviolet equivalent solar hours. Materials evaluated were both commercially available and newly developed aromatic polyimide films aluminized on one side and exposed as second surface mirrors. Effects on solar absorptance, thermal emittance, and tensile properties were measured. The in situ changes in solar absorptance of Kapton® and Upilex® were less than 0.1, whereas the solar absorptance of TOR and CP films increased by more than 0.3 without saturating. Thermal emittance measurements also showed that the Kapton and Upilex materials increased only 1–2%, but the remaining materials increased 5–8%. Tensile property measurements made in air following the test showed the failure stress of every type of polymer film decreased as a result of irradiation.

Introduction

SIGNIFICANT attention has recently been given to the concept of using lightweight, compliant structures that can be folded into the compact volumes of conventional launch vehicles and subsequently deployed onorbit as a means to achieve very large structures in space, hence the term gossamer structures. Relative to onorbit construction, this approach can offer significant reductions in cost, complexity, and risk to astronauts. These structural concepts typically comprised a support structure that can be deployed via mechanical, inflatable, or other means and subsequently rigidized and a thin film that serves as an antenna, sail, reflector, concentrator, sunshade, etc. Recent demonstrations of large deployable/inflatable film-based space structures such as a 20-m solar sail (Znamya-2, New Light) and a 14-m lenticular inflatable antenna have provided glimpses into future possibilities for gossamer spacecraft. Consequently, a significant number of future missions utilizing gossamer spacecraft concepts have been proposed by NASA, the Department of Defense, and other U.S. government and foreign space agencies.

Materials are one of many technologies that must come together in an integrated fashion to successfully advance the gossamer spacecraft concept to realization. Materials are enabling for both the support structure and the film portion of gossamer spacecraft. They must have specific combinations of properties and maintain these properties over the life of the mission. The property requirements can vary significantly, depending on the mission and space environment.

Earlier programs and space flights have laid a good foundation for materials now being developed. Polyimides, polyesters, and perfluorinated ethylene–propylene copolymers have been used extensively on spacecraft designed for a wide range of environmental exposure levels. Some copolymers have been reasonably stable in reflectance when exposed to UV near 1 astronomical unit (AU). However, they lose significant amounts of reflectance and change reflective character if exposed to electrons at fluxes near the peak rates measured at synchronous altitudes. Electrons typically cause a bulk effect, with the polymer becoming a light-scattering medium.[1] Polyimide films such as Kapton® have performed well in space and have survived well in simulations of low-energy protons such as the 1–10-keV solar wind. However, they have degraded heavily at high proton fluences, or when exposed to UV at intensities representative of near-Sun missions, unless protectively overcoated.[2] The U.S.–German HELIOS spacecraft survived thermal loading up to ~10 suns for several years with metalized polymer films and other advanced technologies including control of electrical conductivity systemwide.[3] The HELIOS program studied gossamer films. Poly(paraxylylene) supported in a stainless steel mesh (a potential weight penalty) performed marginally, but bonded Al_2O_3/Al-overcoated quarter-mil Kapton was stable even at accelerated simulation intensities. The latest generation of materials includes self-supporting films having ~13 μm (half-mil) thickness. The Boeing Company's irradiation of samples of these materials has been documented for NASA[4] and is the subject of this paper.

Experimental Approach

Radiation Environment

It was the goal of the program to provide a five-year simulation of two regions of space, the environment at 0.98 AU, where the Geostorm satellite will orbit and the environment at the second Lagrangian point (L2), where the Next Generation Space Telescope will be positioned. The Geostorm location between the sun and the Earth is far beyond the influence of the Earth's magnetic field, which makes the environment of interest that of the solar wind and solar events. The L2 position, on the other hand, is located on the far side of the Earth away from the sun. At this position, a spacecraft would pass through the Earth's geotail created by the interaction of the geomagnetic field with the solar wind. The best estimate available for the radiation environment was arrived on by researching available information and by discussions with experts from NASA, academia, and industry. The levels present in these regions are continually being refined; however, it is understood that by far the major contribution to both environments was the solar wind.

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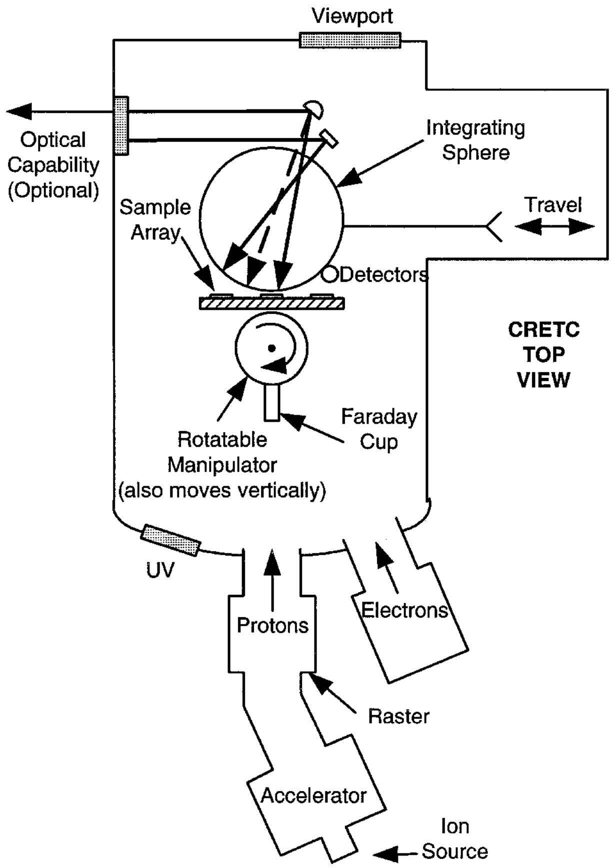

Fig. 1 CRETC layout.

The electron and proton fluence levels were determined by first generating a depth-dose profile of a representative material (Kapton in this case) for the solar wind at L1, then matching the profile using the individual depth-dose profiles of energies and particles available in the chamber. For this test 40-keV protons with a range of 0.52 μm (0.02 mils) were used to deliver the very high dose indicated near the surface, whereas electrons of 40-keV energy with a much deeper depth-dose profile were used to deliver the bulk dose. Several energies of electrons can be used to improve the match; however, the increase in test duration was prohibitive.

Exposure Facilities

The Boeing Company's Combined Radiation Effects Test Chamber (CRETC) (Fig. 1) provided the appropriate experiment environment. The chamber is equipped with sources for both a 10–80-kV rastered proton beam and a 30–60-kV electron beam. A water-cooled 6000-W xenon arc lamp provides continuum UV exposure from 200 to 400 μm at an intensity of 1–3 UV suns. The clean, cryopumped chamber is also fitted with a spectrophotometer that enabled in situ reflectance measurements to be performed periodically during exposures. It is not necessary to remove the samples from the sample holder or the vacuum to make the reflectance measurements.

The UV intensity was measured across the overall beam space that the specimen array occupied and was uniform within $\pm 10\%$ when using approximately 1.5 UV suns. Characteristics of the proton and electron beams were determined with Faraday cups that track the chamber horizontal and vertical centerlines (bisecting the array of specimens). It was determined that the 40-keV electrons were quite uniform to $\pm 5\%$. The 40-keV proton beam, which is rastered with significant overlaps to provide uniformity along with a larger beam size, was uniform to $\pm 15\%$ over the sample array.

Test Specimens and Fixture

NASA Langley Research Center provided the test specimens consisting of seven types: commercially available Kapton E and HN, Upilex® S, CP-1 and CP-2, and two experimental films, TOR-RC and TOR-LMBP. CP-1 and CP-2[§] are manufactured by SRS Technologies, Huntsville, Alabama. TOR-RC and TOR-LMBP[¶] are manufactured by Triton Systems, Inc., Chelmsford, Massachusetts. Kapton and Upilex are mature film products that have been optimized for thermal and mechanical properties through synthesis and processing. Consequently, they exhibit significantly higher strengths, moduli, and strains to failure than the batch-cast experimental films.

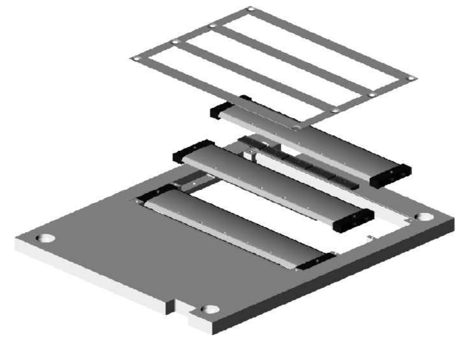

Fig. 2 Exploded view of specimen fixture.

The maximum number of samples allowed in the test was set by the available exposure area size and the minimum sample size required for the measurements. The exposure area of the CRETC allowed for specimens to be placed within an area approximately 80 by 80 mm. In addition, the tensile strength measurements required a length of unexposed material at each end. Therefore, a custom test fixture sized for specimens approximately 75 mm long and 16 mm wide, with a central exposure and measurement section 20 mm long was designed. Each row of specimens was mounted to a slightly curved shape mandrellike bar that secures each test specimen in place during exposure. Each specimen was secured from one end on the backside of the fixture, shown in Fig. 2. One feature of the fixture was a thin shield between the rows of test specimens, to provide for a well-defined central irradiation section. The result was an array of 5 samples in each of 3 horizontal rows wrapped on mandrel bars for a total of 15 specimens.

Specimen Preparation and Mounting

Thin films are difficult to handle in general, and for these samples, a cutting template tool was machined to aid in cutting the specimens from the larger sheets provided by the customer. Microscopy was used to determine machine direction (if applicable) as well as to assure that the films would be exposed as second-surface mirrors. The more fragile experimental polymer films were the most difficult to cut. Specimens that developed ragged edges or tears were not used in the irradiations, but were set aside as extra controls. The fabricated test fixture was washed with isopropyl alcohol, ultrasonically cleaned in a detergent wash, rinsed, and finally given an ethanol solvent rinse and dry.

Sample integration was performed using cleanroom gloves inside a clean laminar flow bench. The cut specimens were attached at one end under metal screwdown clips on the upper back side of the bars then wrapped down and around the front surface looping over the top of the bar to end in the back again, where small weights were attached to keep each specimen in mild tensile stress, but with freedom to shrink or elongate in response to radiation. The front cover shield was then attached, to define the overall exposure area of each specimen exactly.

Property Measurement

Reflectance

The Boeing Company CRETC has a double-beam spectrophotometer that is optically coupled to the locations of test samples in the vacuum chamber (Fig. 1). With appropriate measuring light sources [UV to near-infrared (IR)] and with light detectors

[§]Data available online at URL: http://www.stg.srs.com/advanced_polymers.htm.

[¶]Data available online at URL: http://www.tritonsys.com/special-polym.html.

in situ, the value of a test surface's spectral reflectance, as modified by radiation or perhaps other stresses, is determined during measurements and retained for computer analysis. In The Boeing Company's facility, an integrating sphere in the test chamber, between the detector and a sample being measured, produces a measurement of hemispherical reflectance. The spectral range is from 250 nm to about 2500 nm. A sample is illuminated spectrally because the spectrophotometer optical path includes the monochromator after the light source(s). The spectral illumination begins with longest wavelength light (lowest electron volt value), and the measurement proceeds to shorter wavelengths. This is a nondestructive measurement. With opaque samples, solar absorptance is derived by simple subtraction (using the appropriate solar wavelength weighting).

Emittance

A nondestructive measurement using near-IR radiation can be given to a film sample by laying it over an aperture provided in a Gier–Dunkle Emittance Inspection Device (DB100). The Boeing Company performed a series of these room temperature measurements as part of this program, in air following the in situ irradiation. A number of unirradiated samples, cut from the same polymer sheets, were used as unexposed comparison samples so that all specimens were measured in the same run. The measuring device illuminates each sample with polychromatic radiation, and the apparatus circuitry computes a weighted IR reflectance value internally. With the opaque specimens in this program, the values of thermal emittance coefficients were derived by simple subtraction from the measured reflectance values.

Tensile

After completion of the emittance measurements on all exposed samples, as well as on selected comparison or unexposed samples, mechanical property measurements were made. A MII-50 UD Satec universal test machine with a 440-kg load cell was used for the property testing. The cell is calibrated down to 880 g with a resolution down to 0.44 g. Instron hydraulic grips with rubber pads were used to clamp each test film in turn. All measurements were made at room temperature.

Experimental Data

Exposure Summary

The simultaneous exposure of protons, electrons, and UV simulating a 5-year (60-month) mission at L1/L2 was divided into five exposure segments. Table 1 lists the proton and electron fluences and the equivalent UV exposure hours for each segment. Although the total proton and electron fluences simulated the entire 60-month mission, it was not possible to provide a UV exposure that simulated the full mission within the scope of the contract. Therefore, the highest amount of UV exposure possible was accumulated as dictated by the exposure times of the protons and electrons.

The average flux over the entire exposure period was $4.8E8$ protons/cm$^2 \cdot$ s, $3.9E9$ electrons/cm$^2 \cdot$ s, and 1.5 equivalent UV suns. The chamber vacuum pressure level began at $9E-7$ at the start of the exposure and quickly leveled out at $2E-7$ torr. The CRETC exposure systems are designed to operate continuously except for brief periods each day devoted to dosimetry, to cleaning of the UV source, and during scheduled reflectance measurements.

Solar Absorptance

The solar absorptance values were calculated from the spectral reflectance data measured by the spectrophotometer. From the 240 specific wavelengths available from each data set, 100 wavelengths representing the relative spectral weighting of the sun's radiance curve were used in the calculation of solar absorptance. As the exposure continued, the TOR-LMBP sample type began to tear, and by the third measurement level, both specimens had torn and were unmeasureable. In Table 2 all individual values are listed, and Fig. 3 shows the average increase in the solar absorptance for each specimen type as a function of the exposure.

Thermal Emittance

The emittance results for both the unexposed and exposed samples were calculated from the measured reflectance values by subtraction

Table 1 Exposure summary

Exposure segment	Equivalent mission duration, months	Proton fluence, protons/cm^2	Electron fluence, electrons/cm^2	UV exposure, h
1	~3	$3.6E+13$	$5.0E+14$	90
2	12	$2.0E+14$	$1.6E+15$	330
3	24	$3.9E+14$	$3.2E+15$	480
4	42	$7.1E+14$	$5.7E+15$	685
5	60	$1.0E+15$	$8.0E+15$	1000

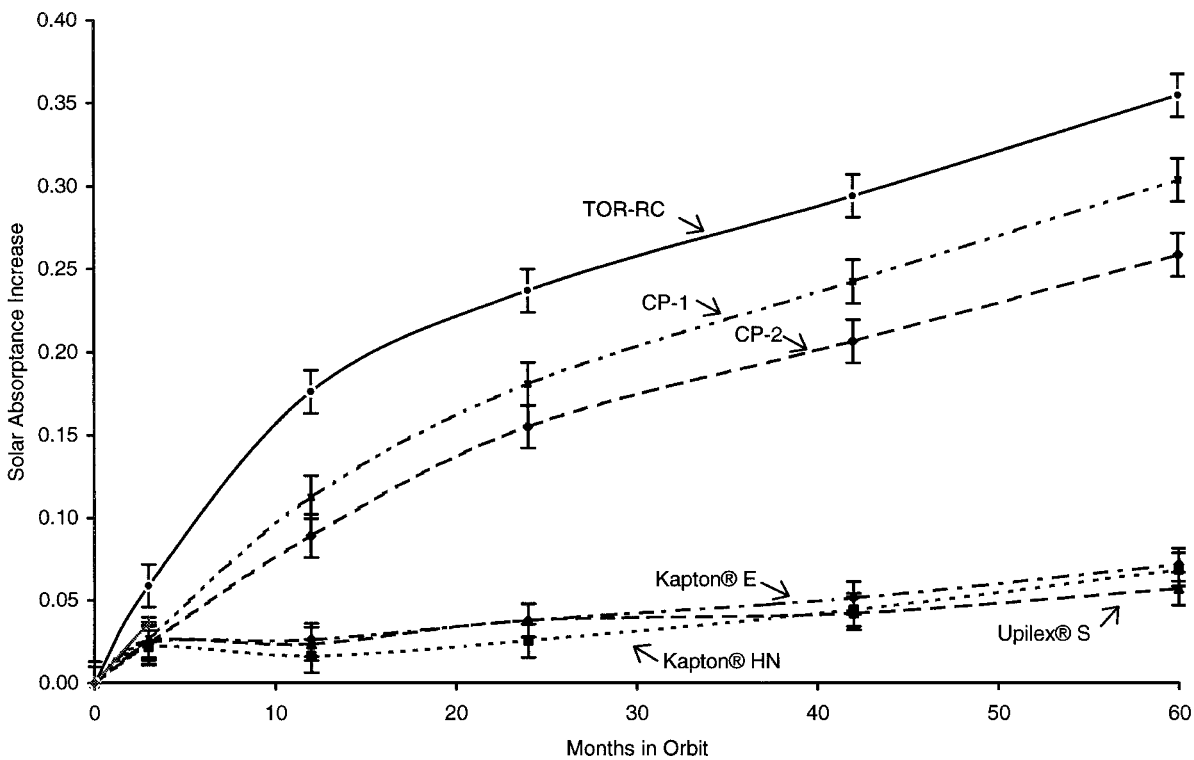

Fig. 3 Increase in solar absorptance as a function of exposure.

Table 2 Individual specimen in situ solar absorptance values

Sample		Exposure segment					
Material	Identification	0	1	2	3	4	5
Kapton E	2	0.300	0.326	0.328	0.335	0.352	0.373
Kapton E	7	0.304	0.326	0.328	0.340	0.352	0.373
Kapton E	15	0.304	0.329	0.330	0.346	0.356	0.375
Kapton HN	1	0.318	0.337	0.329	0.335	0.356	0.380
Kapton HN	8	0.314	0.339	0.337	0.349	0.365	0.389
Upilex S	4	0.351	0.376	0.370	0.383	0.392	0.407
Upilex S	11	0.355	0.381	0.383	0.398	0.399	0.413
CP-1	9	0.213	0.246	0.339	0.409	0.473	0.546
CP-1	14	0.217	0.238	0.316	0.382	0.441	0.491
CP-2	3	0.215	0.241	0.315	0.376	0.432	0.484
CP-2	6	0.211	0.233	0.289	0.360	0.406	0.458
TOR-RC	10	0.194	0.258	0.374	0.440	0.496	0.560
TOR-RC	13	0.193	0.246	0.365	0.421	0.478	0.536
TOR-LMBP	5	0.233	0.252	——	Failed during test		——
TOR-LMBP	12	0.227	0.280	——	Failed during test		——

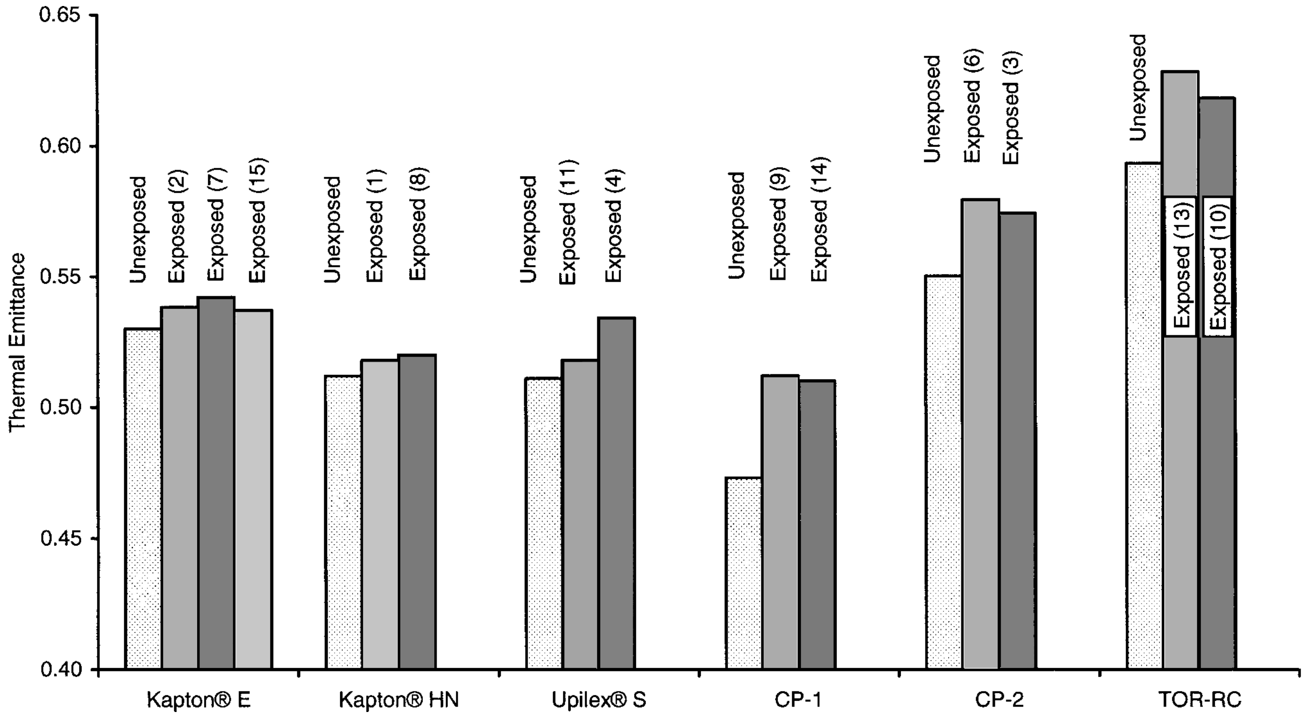

Fig. 4 Thermal emittance results. (Sample identification indicated in parentheses.)

for these opaque materials. The bar graph in Fig. 4 gives a quick visual summary of the results.

Tensile Properties

Figures 5–7 summarize the results in bar graph format. The modulus values were not affected appreciably by the radiation exposure. However, the failure stress and strain were generally affected. Several exceptions include TOR-RC's failure strain values, which were very low to begin with and which remained low, and stress at failure for Upilex S showed a decrease in only one of the two samples. These remarks illustrate that the small number of exposed samples available combined with the difficulty of making this type of measurement on very thin films reduces the usefulness of the results primarily to indicating trends.

Discussion of Findings

The computed values of each sample's solar absorptance increased, as measured in situ, as exposure to radiation continued, to the end of the test without saturation. Certain polymer films that were colorless before irradiation became considerably more absorptive and acquired a "bronze" color, during irradiation. The polymers that originally were colorless more than doubled their solar absorptance (from about 0.2 to nearly 0.5). On the other hand, Kapton specimens increased about 0.07 in solar absorptance, from base values of about 0.3. Upilex S was slightly more stable for solar absorptance, increasing about 0.06 (from base values of about 0.35). TOR-RC nearly tripled in solar absorptance by the end of the test (from base values of approximately 0.2). TOR-LMBP distorted and then disintegrated during the first quarter or so of the test period. Figure 3 summarizes the increase in solar absorptance obtained on each of the irradiated polymer films types. The experimental data divide into two principal groups, one of them having much more stable reflectance than the other. The changes in solar absorptance from Kapton and Upilex samples remain less than 0.1, whereas the solar absorptance changes in TOR and CP film samples were more than 0.3, without saturating.

The thermal emittance values measured (in air) on the Kapton specimens remained essentially unchanged within experimental uncertainty. On the other hand, the emittance of polymer CP-1 increased about 8% in air (from about 0.47 to about 0.51 decimally) as a result of the combined UV/proton/electron irradiation performed. The emittance of CP-2 and TOR-RC increased perhaps half as much.

The polymers most stable in reflectance, namely, Upilex S and Kapton, were also the strongest in tension before irradiation, and they retained the greatest percentage tensile strength. The films less stable in reflectance were also weaker in tension and lost more tensile

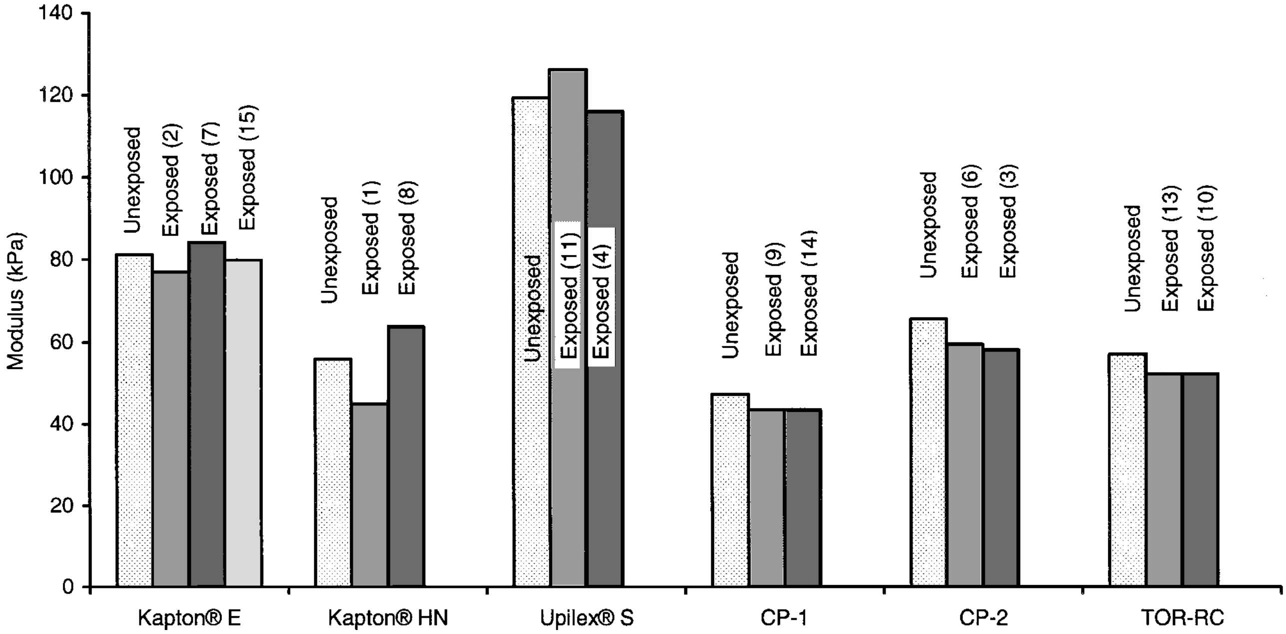

Fig. 5 Modulus summary. (Sample identification indicated in parentheses.)

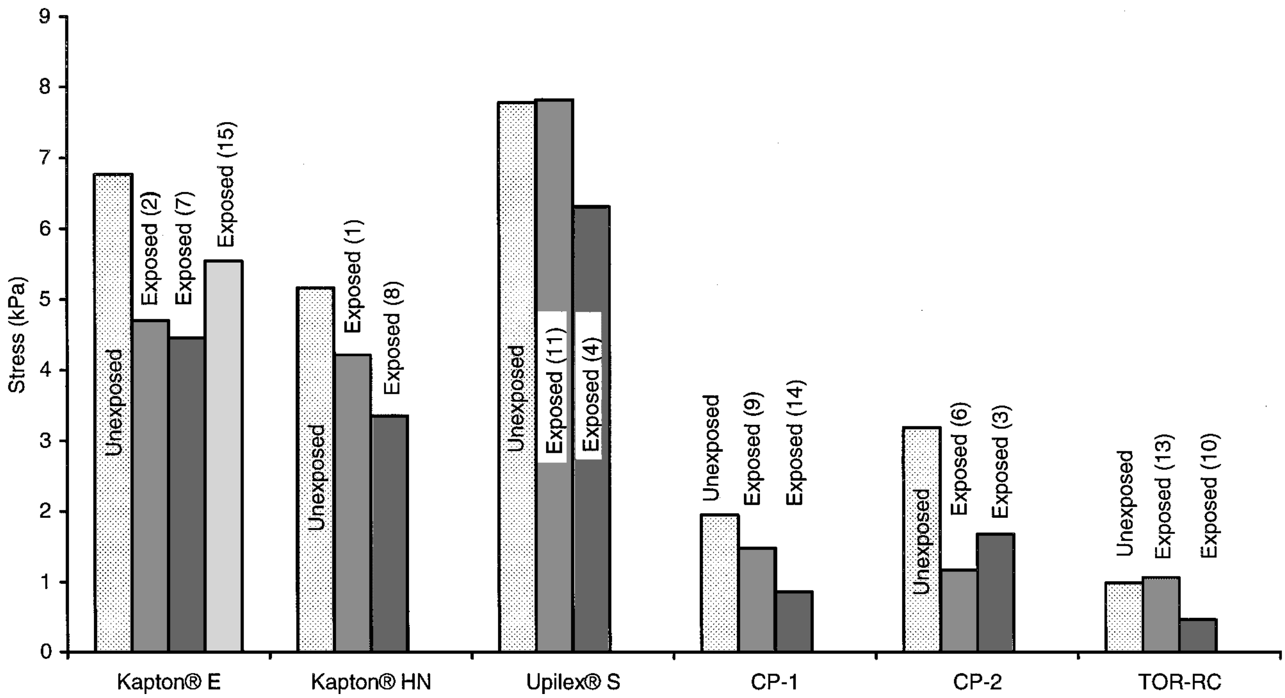

Fig. 6 Failure stress summary. (Sample identification indicated in parentheses.)

strength as a result of irradiation as shown in Fig. 6. The failure strain (as a percent of original gauge length) of every type of polymer film except TOR-RC, decreased as a result of irradiation. The decrease was "dramatic" in Kapton. Modulus generally decreased so slightly due to irradiation that the changes are not very significant. The presumed explanation for the loss of strength is the chemical reactions caused by the irradiation. The reduction in strength without a concurrent reduction in modulus suggests that the films are becoming embrittled due to crosslinking.

No direct measurement of film temperature was made during irradiation. However, when preparing the same test fixture for a later irradiation of similar film materials,[5] a cavity was machined in one sample-holding mandrel to place a thermocouple near one corner specimen. The cavity shielded the thermocouple from direct exposure to the simulated sun source. For both irradiations, all mandrels wrapped with film samples were cooled by the same technique: Adjacent water jackets cooled the perimeters of the sample-array areas, and limited heat exchange was possible through the mechanical joints between the mandrels and the perimeter (Fig. 2). The mandrel temperature was approximately 19°C when the chamber interior was dark. Under an intensity of approximately 2 UV suns, the mandrel temperature rose about 5°C (from ~19 to 24°C or less) within minutes after beginning the exposure of samples to the solar beam.

Further analysis has been done to estimate the temperature of the polymer films themselves during irradiation. The film samples were wrapped around, but not bonded to, their mandrels. Most films had the majority of their area in contact with their mandrels. However, although weighted, some specimens did not hang straight. Despite being thin, they were not fully compliant with efforts to have them

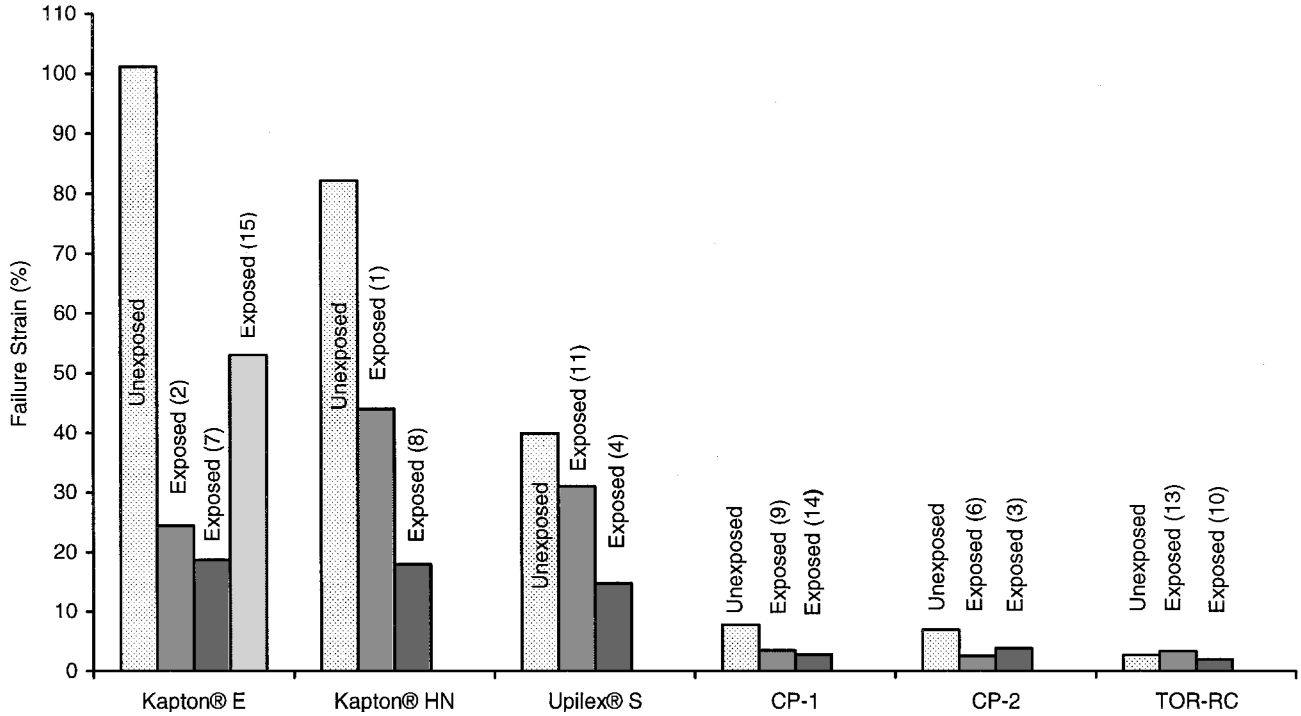

Fig. 7 Failure strain summary. (Sample identification indicated in parentheses.)

be draped uniformly around their mandrels. Therefore, portions of those samples did not have total thermal contact with their mandrel surfaces. Those portions of films with no or poor thermal contact would rise to higher equilibrium temperature values during irradiation, depending on their absorptance and emittance properties, the intensity of the simulated sun, and the temperature of adjacent radiative surfaces such as the mandrels. Calculations show that detached portions of low-absorptance films such as the CP series may equilibrate at about 80°C when illuminated by 1.5 suns, but could rise to ∼170°C in their degraded states (Table 2 and Fig. 3). In contrast, portions of Kapton and Upilex samples that are irradiated while detached from cooled substrates would equilibrate and remain under 150°C because those materials are more stable under UV, proton, and electron irradiation.

More sophisticated instrumentation, designed to reveal stress patterns during tensile-property testing, for example, might indicate effects of film temperature excursions during irradiation. The Boeing Company did not employ such instrumentation during this work. We observed no visual differences in the responses or appearances of various portions of samples, during irradiation or during post-irradiation testing; the only exception was the physical tearing and distortion noted earlier for TOR-LMBP. Therefore, the extent of sample contact (or lack of it) with its underlying mandrel/substrate probably had no substantial effect, and film temperature excursions during irradiation were probably not great enough to cause fundamental changes in polymer structure or strength.

Conclusions

1) Film reflectance measured in situ was always found to decrease after exposure to simulated space radiation.

2) Based on tensile property measurements made in air following the test, the failure stress of every type of polymer film decreased as a result of its being irradiated.

3) The space radiation simulation that we conducted was effective in determining the most stable and the least stable films for solar absorptance and tensile strength.

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