LONG-TERM STABILITY OF ION-BEAM TREATED SPACE POLYMERS IN GEO-SIMULATED ENVIRONMENT

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ABSTRACT

It is a real challenge to reproduce various space environment conditions in ground-based facilities for testing of external space system materials and elements. This is due to the variety and complexity of the space environment conditions and their effects on materials and structures. The reliability of test results depends on accurate simulation of the critical factors and effects of the space environment for a particular mission. The main objectives of the simulation testing are to get the test results that are adequate to the material durability and functional behavior in a space environment.

As part of a large, multi-year program, a number of ion beam surface treatments and proprietary coatings formed on advanced space polymers by ion beam deposition were developed at ITL Inc, Canada. This technology allowed to impair controlled surface resistivity in a wide range of charge dissipation values, with negligibly low RF losses and other important functional properties, allowing using these materials and products in modern space antennae, solar arrays and other applications in GEO environment.

This paper will summarize a unique set of irradiation experiments of space polymers that were ion beam treated and coated with special coatings by ion beam deposition (IBD) for charge dissipation. The irradiation experiments were conducted in three world-recognized GEO simulation facilities, covering a wide range of irradiation conditions. New results will be presented on the radiation stability of those space polymers surfaces after the electron accelerator testing in the MeV energy range.

Key-words: *GEO radiation environment, deep charging, surface charging, Carbosurf+,*

1.0 INTRODUCTION

The work described in this paper was conducted during a number of projects on the development of special important structures for space vehicles operating in GEO long-term space missions. Not only thin polymer films, common in space vehicles' design and applications, but also comparatively thick surface treated/coated dielectrics have been considered and treated.

It is well known that spacecraft charging includes both surface charging and internal dielectric charging [1]. The consequences of charging in GEO orbits have ranged from various anomalies in space missions up to satellite failures, Most of the undesired effects of both charging types are due to the discharge arcing, and subsequently, the danger of physical materials damage. Some of the important functional characteristics of external thermal control space materials can also be changed under space radiation. The potential danger of all these space radiation effects in materials drastically increases for the modern long-duration missions and, therefore, requires the development of strong preventive and protective mitigation techniques.

In GEO environment the spacecraft experience irradiation by electrons in a wide energy spectrum that includes high-energy, but low-density electrons. Energy levels affect mostly the voltage potential, while the electron densities determine charging current density. A number of studies have been conducted recently regarding the phenomena of radiation-induced conductivity (RIC) at high-dose electron exposure of space polymers, and RIC influence on charging effects.

2.0 LOW AND MID ENERGY GEO SIMULATION TESTING OF SURFACE TREATED POLYMERS

2.1 Space polymer materials

A very important and productive approach for preventing the external dielectric space materials from charging in GEO have been developed at ITL Inc. and used in a number of applications, for example, in modern high-frequency, high pulse space antennae and for components of solar arrays. A special ion beam surface treatment technology for surface carbonization of polymers, named Carbosurf, allowed to make the surfaces of space polymers and polymer-based space products charge dissipative in a wide range of surface resistivity (SR) values. This patented treatment and the results for few space polymers, Kapton, PEEK, Mylar, Upilex and both clear CP-1 and white CP-W have been described in [2-4]). An example of SR values, achieved on three space polymers, surface treated with noble gases in two runs, (1) and (2), and by Ar+ at two doses, (I) and (II), is shown in Table 1.

Table 1. Surface resistivity of thin (1 mil) polymer films after moderate energy ion beam treatments at room temperature

Materials / Surface treatment	Surface resistivity at room temperature, ρ (Ohm/sq.)		
	Xe^{+}	Ar+ (I)	Ar^{+} (II)
CP 1 White (1)	$0.75{\cdot}10^{7}$	$2.5{\cdot}10^{8}$	$1.3{\cdot}10^{7}$
CP 1 White (2)	$0.8{\cdot}10^{7}$	$3{\cdot}10^{8}$	$3{\cdot}10^{7}$
CP 1 (1)	$0.6{\cdot}10^{7}$	$5{\cdot}10^{8}$	$1.3{\cdot}10^{7}$
CP 1 (2)	$0.75{\cdot}10^{7}$	$5.2{\cdot}10^{8}$	$6{\cdot}10^{7}$
Kapton HN (1)	$1.5{\cdot}10^{7}$	$5{\cdot}10^{10}$	$3{\cdot}10^{9}$
Kapton HN (2)	$1.3{\cdot}10^{7}$	$3.5{\cdot}10^{10}$	$1.9{\cdot}10^{9}$

While various noble gases were used successfully in ion beams treatments of a variety of space polymers, for practical applications, the surface treatment was mostly performed on 1 and 5 mil thick KaptonHN, mostly with Ar + powerful technological ion beams with an energy E=3 KeV.

The thermal optical properties may be more or less affected by this treatment, depending on the selected ion beam source and treatment conditions – see examples in Table 2 and Fig. 1.

Table 2. Thermal optical properties and SR of KaptonHN polymer films after Carbosurf surface treatment by Ar + ion beam, E=3 KeV [2].

Sample # / Thickness	Apparent Solar absorptance α_S (on Al-backing)		Apparent Thermal emittance ε (over high emissivity standard)		Surface Resistivity (MOhm/sq.)
	Pristine	$\Delta\alpha_S$ after treatment	Pristine	Δε after treatment	
#11, 1 mil	0.339	0.122	0.883	0.009	~(10-12)
#14, 3 mil	0.497	0.013	0.880	0.003	~(5-6)
#15, 3 mil	0.497	-0.031	0.880	0.004	~(20-30)
#17, 1 mil	0.339	0.138	0.883	-0.002	~(130-150)
#18a, 3 mil	0.497	-0.003	0.880	0.004	~(2-3)
#18b, 3 mil	0.497	0.016	0.880	0.008	~(0.5-0.7)
#19, 3 mil	0.497	-0.031	0.880	0.007	~(80-100)
#21, 3 mil	0.497	0.019	0.880	0.008	~(10-20)

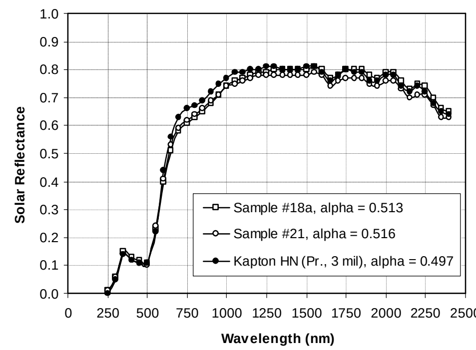

Fig. 1. Total solar reflectance spectra of Carbosurf treated and pristine Kapton HN after low-energy (3 kV) Ar^{+} beam treatment. The insert shows the calculated solar absorptance, α_S [2].

For further enhancement of material's « practical » properties like handling convenience, chemical and mechanical stability, and long lasting shelf life, a modernized Carbusurf+ technological process was developed that presents a combination of the Carbosurf ion beam treatment with a very thin ion beam deposited hydrogenated diamond-like carbon (DLC) coating that demonstrated very high radiation resistance properties.

Ground-based GEO simulation testing of CarboSurf+ treated samples was conducted at the JSC Kompozit facility [5]. The facility includes beam sources of electrons and protons in the low-to-mid energy range that can simulate the influence of GEO environment mostly on the surface properties. Table 3 shows an example of SR data results obtained for pristine and ion beams treated space polymers before and after GEO testing [3]. The samples treated by CarboSurf+ technology underwent a successful extended, 7-9 years long shelf life evaluation in laboratory conditions, practically without SR changes.

Very positive results were also received for Carbosurf+ treated space polymers in an extended GEO simulation testing program imitating up to 15 equivalent years of a GEO mission. The impaired charge dissipative SR values remained practically unchanged. The lower values of SR even decreased slightly, but non-significantly, to the end of the tests, and the rest of SRs were left completely unchanged. All those results demonstrated outstanding radiation resistance of all surface treated space polymers during the testing with comparatively low- and mid-energy irradiation sources of GEO environment [2-6].

Table 3. Surface resistivity of thin (1 mil) pristine and Carbosurf+ ion beam treated space polymer films before and after GEO-simulated testing at the Komposit facility with the irradiation conditions of ($p^+ + e^- +$ UV) equivalent to ~5-7 years in GEO orbit [2].

Material	Treatment	SR (Ohm/sq.)	SR (Ohm/sq.), Rad. Tested
Kapton HN	Pristine	$> 10^{12}$	10^9
Kapton HN	Ion beam treated	$(13\text{-}25)\cdot10^6$	$18\cdot10^6$
CP1 (White)	Pristine	$> 10^{12}$	$8\cdot10^8$
CP1 (White)	Ion beam treated	$(13\text{-}60)\cdot10^6$	$0.8\cdot10^6$
CP1 (Clear)	Pristine	$> 10^{12}$	$0.8\cdot10^9$
CP1 (Clear)	Ion beam treated	$(60\text{-}80)\cdot10^6$	$0.7\cdot10^6$
Upilex S + Ge coating	Ge (1000 Å)	$2\cdot10^7$	$1.3\cdot10^8$

2.2 Flat Cable Conductors

Carbosurf+ technology was also used successfully in another project to prevent surface charging of specific external space components – Flat Cable Conductors used on solar arrays of satellites in GEO orbits [8].

Flat Cable Conductors (FCCs) are manufactured, using Kapton100HN on both surfaces. Due to the specifics of the manufacturing process, the surfaces have a "grooved" surface relief, and one of them, that we called "back", have been contaminated with tiny embedded pumis particles. To make these surfaces charge dissipative, a modified version of Carbosurf+ treatment was used, that allowed for a simultaneous surface renewal, and that was applied to both sides. The patented modified treatment can be used, in general, for any hydrocarbon polymers with inorganic inclusions [9].

The FCCs, treated on both sides, passed successfully a full space qualification program, and the GEO simulation testing (on Komposit facility), without compromising the BOL/EOL surface properties, as is demonstrated in Table 4.

Additional GEO simulation testing under electron exposure was also conducted at the SIRENE facility in France [7]. The SIRENE facility is known for its unique capability to reproduce the GEO electron environment, using both a 20 KeV electron source and an electron accelerator with the energy up to 400 KeV, and conducting the tests in a wide space-related temperature range.

The results of the GEO simulation testing program that also included the electrostatic discharge (EDC) testing (Fig. 2) in the most dangerous, low-temperature GEO environment conditions demonstrated that the original SR values and other functional surface properties remained practically unchanged [8,9].

Table 4 Thermal Optical Characteristics of Pristine, and Carbosurf+ (Ar+ Ion Beam treated), and GEO tested FCC samples from the first set, tested at Komposit

Sample ID	Side	Solar Absorptance α	Thermal Emittance ε	α/ε
Pristine	Front	0.565	0.81	0.70
	Back	0.592	0.80-0.81	0.74
Ion-Beam Treated	Front	0.691 - 0.695	0.79-0.80	≤0.88
	Back	0.712- 0.714	0.80	≤0.89
GEO-Tested	Front	0.787	0.78-0.82	1.01
	Back	0.817-0.821	0.79-0.81	≤1.04

A conclusion was made, that the success of the developed and patented ion beam surface treatment with simultaneous surface renewal was demonstrated by the full functional durability of the charge dissipative surfaces and non-changed performance of treated FCC's in the conditions, imitating expected long-term GEO missions [8,9].

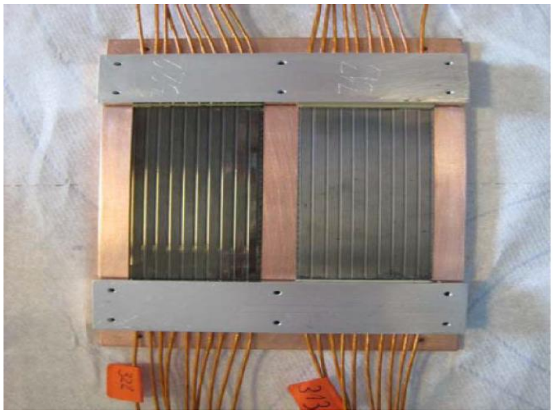

Fig. 2. FCC samples mounted on a copper plate for ESD testing in SIRENE facility: left – front side of an FCC; right - back side (8)

Figure 3 shows an example of the evolution of surface potential on FCC samples during SIRENE x4 irradiation and relaxation at – 145 °C. The complete abcense or presence of a very small non-critical, external surface potential shows that the modified Carbosurf+ treatment, when applied to both sides of the FCCs, prevents both the surface and the deep charging of the comparatively thick structures (with thickness in the mm range).

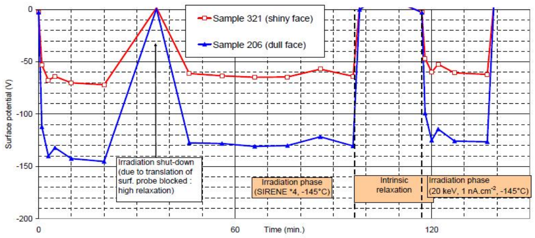

Fig. 3. Evolution of surface potential on FSS samples during SIRENE x4 irradiation and relaxation at – 145 °C (8)

3.0 HIGH ENERGY GEO SIMULATION TESTING OF SURFACE TREATED POLYMERS

As was mentioned above, for thicker dielectrics, with the thickness in the millimeter range, not only surface, but also internal, or deep charging, may be a real danger in GEO environment. We, therefore, investigated also the SR behavior of thicker Kapton and PEEK polymer samples. PEEK is used in a growing number of applications in space and aerospace industry as a rigid high performance structural material.

The pristine and Carbosurf$^+$-treated samples of Kapton and PEEK have been tested in the GEO simulation facility - an electron accelerator at the Idaho Accelerator Center (IAC) by the Utah State University (USU) group. In a collaborative project with USU and ViaSat, the SR values were monitored on 15 PEEK coupons and eight KaptonHN samples of two different thicknesses, i.e. 1 mil and 5mil that were treated at ITL with the Carbosurf+ technology and exposed to MeV electrons irradiation in three consecutive irradiation experiments.

3.1 Samples Used in the Accelerator Exposures

Irradiation tests were performed on samples on three types of substrates. Sample pre-test evaluation included size and thickness measurements and visual inspection. Eight polyimide (PI) (Kapton HN™) thin film substrates (~4 cm x 7 cm) were 29±4 µm (1 mil) and 129±5 µm (5 mil) thick, with mass density (1.42±0.02) g-cm^{-3}. Fifteen thicker poly-ether-ether-ketone PEEK) flat coupons (3 cm x 8 cm) were 123±5 µm (4 mil) thick, with mass density 1.28±0.04) g.cm^{-3}.

Each substrate was Ar+ ion beam surface treated, as for Carbosurf, and then a thin ion beam deposited diamond-like carbon (DLC) coating was formed on the polymer surface, as described for Carbosurf+ above. On PEEK and KaptonHN substrates coatings have been formed to cover the SR range between 10^6 - 10^9 Ω/sq. Fig. 4 shows visual images of Carbosurf+ treated PEEK and KaptonHN substrates. Darker regions have been treated with Carbosurf+; lighter regions were masked during the surface processing. In general, the surfaces of all exposed samples got darker after the radiation exposure experiments, similar to the effects at GEO simulation testing of Carbosurf+ treated/coated samples in other facilities, mentioned above.

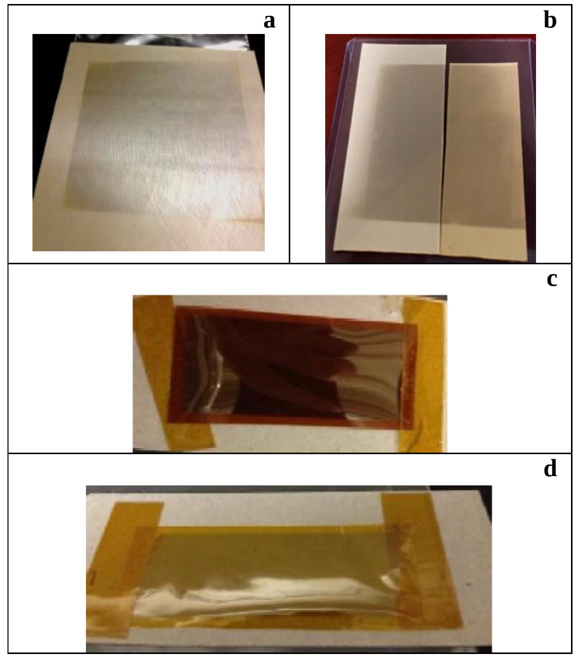

Fig.4. Optical images of samples that were irradiated at Idaho Facility.

a) Flat PEEK coupon in as deposited condition (no irradiation)
b) The sample on the left was irradiated to a dose of 30 Mrad, while the sample on the right was irradiated to the full dose of 470 MRad. Notice distinctive yellowing and darkening of the sample after the full dose.
c) Kapton sample (5 mil thick) irradiated to a dose of 470 MRad
d) Kapton sample (1 mil thick) irradiated to a dose of 470 MRad

The inspection of the sample's surface morphology, using optical and scanning electron microscopy, as well as surface roughness evaluation, have been also made. Significantly higher roughness is obvious for PEEK in comparison with KaptonHN surfaces (Table 5).

The surface morphologies of the PEEK and Kapton samples were examined before and after CarboSurf+ treatment and then again after the samples were exposed to the range of BOL/EOL irradiation conditions (Fig. 5).

Table 5. Summary of surface roughness data of the flat PEEK coupons and the Kapton samples used in the irradiation experiments.

Test	Roughness, *Ra [µm]*		
		Kapton	
	PEEK	500HN,5 mil	100HN,1 mil
1	0.69	0.025	0.052
2	0.83	0.024	0.039
3	0.85	0.031	0.035
Average	***0.79***	***0.027***	***0.042***
Std. Dev.	***0.09***	***0.004***	***0.009***

As expected, the morphology of the surfaces did not practically change, with the morphological differences between individual samples remaining the same after the deposition runs and after irradiation.

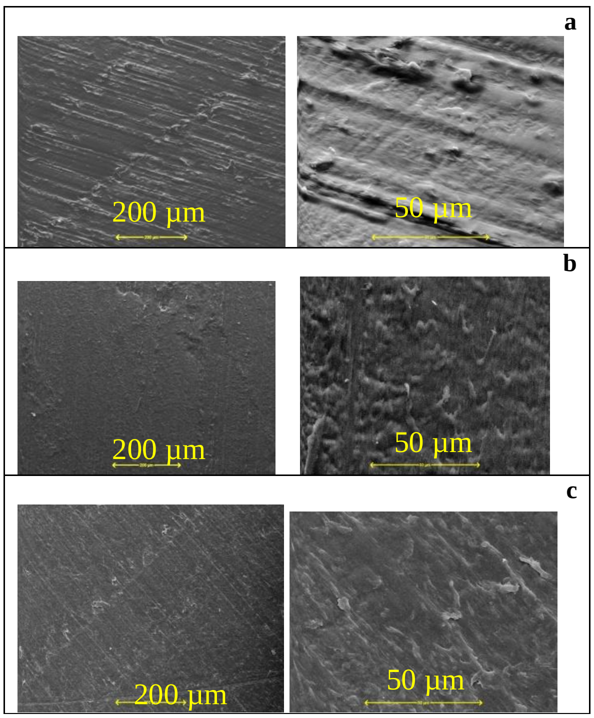

Fig.5. Scanning electron microscopy analysis (secondary electrons) of surfaces of pristine PEEK coupon (a), of PEEK coupon after deposition of the Carbosurf+ (b) and after 470 MRad irradiation dose (c).

3.2 Preparation of Sample for Testing

Samples were subjected to standard cleaning with methanol and a vacuum bake out at 383±1 K (110 °C) and $5x10^{-4}$ Pa for ~72 h prior to mounting in the sample trays to minimize absorbed water and volatile contaminates in the samples, i.e. for outgassing. They were brought in contact with a grounded conducting surface during outgassing, to dissipate potential internal charging. Once bake out was complete, samples were transferred under dry nitrogen and mounted with thin double-sided adhesive Cu tape. All of them were mounted in the base of 22 cm diameter 4 cm high trays made of 250 µm thick Al which underwent similar vacuum bake out prior to sample transfer (Fig. 6).

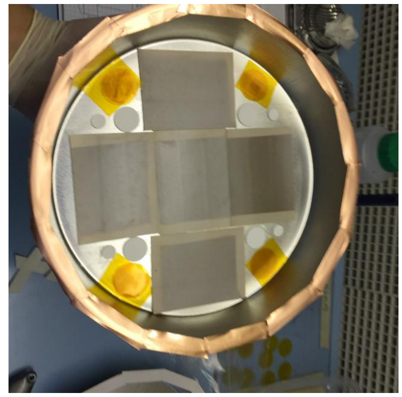

Fig.6. Optical image of some of the samples that were irradiated at IAC Facility. Samples were laid out to provide best uniformity of exposure across them.

Great care was taken to minimize changes in resistivity due to water absorption and possible chemistry modifications of the films due to exposure to oxygen and OH- radicals, especially during irradiation. Preliminary post-irradiation SR measurements were done less then in 3 hr after exposure to atmospheric conditions, with ~40% RH. More accurate SR results, shown in Figs. 7-9, were taken after longer exposure to atmospheric conditions, after more then ~ 50 hr.

3.3 Samples Irradiation

To test the effects of high energy ionizing radiation, especially for thicker samples, such as they may experience in GEO, the Carbosurf+ treated sample coupons were exposed to high energy electron irradiation in three sequential irradiation experiments. The samples in the dry nitrogen environment of the sample trays at room temperature were exposed to three sequential total ionizing doses (TID) of ~17 Mrad (Si) for 0.67 hr, then 30-170 Mrad (Si), and finally 65-230 Mrad. In total, the samples were exposed cumulatively to between ~100 Mrad and ~400 Mrad TID. The doses were selected to span the beginning of life (BOL) and end of life (EOL) conditions for typical geosynchronous orbit (GEO) missions. The electron source used was a 25 MeV LINAC accelerator operating at ~10 MeV at ~110 mA beam current in a pulsed mode with 4 µs pulse widths at a 250 Hz rep rate [9]. A 250 µm thick Al scatter foil was used to expand the beam, producing a gaussian beam profile with 16±4 cm FWHM at the sample distance as measured with a translatable ionization chamber (RadCal Corp., Model 2025). Samples, mounted on a sample tray, rotating at ~10 rpm to minimize local dose rate variations, had less than ±10% variation in dose rate across individual samples when corrected for radial distance in the sample trays. The Al scattering foil produced an electron energy distribution centered at ~7.8 MeV with a nominal width of ~1 MeV. Average dose rates for irradiation of samples at IAC were ~15 Mrad (Si) per hour

Using the data of electrons range in polymers and metals [10], it is easy to estimate, that in the Komposit facility the electron flux did not penetrate through the whole thickness of the tested samples, exposing mostly the subsurface area, in the SIRENE facility it propagated through the samples of selected thicknesses, with the USU electron beam going even through the Al holder.

The annual dose for typical mission in GEO (a 15 year mission at a longitude of 160° W in GEO orbit and a 90 transfer orbit) has a ~200 Mrad/yr total ionizing dose (TID) for unshielded aluminum including all incident energies and ~17 Mrad/yr TID through ~ 1.6 mm PI or PEEK (~0.9 µm equivalent aluminum shielding) for energies above ~500 keV. Thus, the first dose (~17 MRad) and total cumulative range of dose (~100 Mrad and ~400 Mrad) were equivalent, respectively, to ~1 month and ~½ year to 2 year TID in an unshielded GEO environment or ~1 year and ~6 to 24 year TID for electrons above ~500 eV in a typical GEO environment.

3.4 Results and discussion

Figure 7 presents the SR measurement results after irradiation exposure of a number of PEEK samples before (beginning of life (BOL)) and after three consecutive irradiation experiments (end of life (EOL)). Figures 8 and 9 present similar results for the Kapton samples. As can be seen from Figs. 7-9, all SR values remained in the pre-testing SR ranges after the irradiation experiments, with a general trend of a slight increase in the SR values for all tests, but leveling off before or close to the highest dose.

The SR values for all samples were re-measured after a prolong storage in laboratory conditions. Tables 6 and 7 present the results of all SR measurements done shortly after the irradiation exposures and repeated after almost 2 years of storage in laboratory conditions.

As can be seen from Tables 6 and 7, the SR values show a trend for a very small increase, remaining, however, in the same charge dissipative range after both, the high energy electron irradiation and storage in laboratory conditions. The small increase is more pronounced for PEEK, that might be due to it's higher surface roughness.

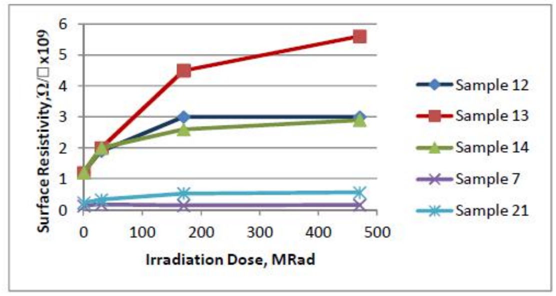

Fig. 7. Summary of the changes in SR values after three consecutive irradiation tests for PEEK coupons with initial SR values in the range 10^7- 10^9 **(Ω/□)**.

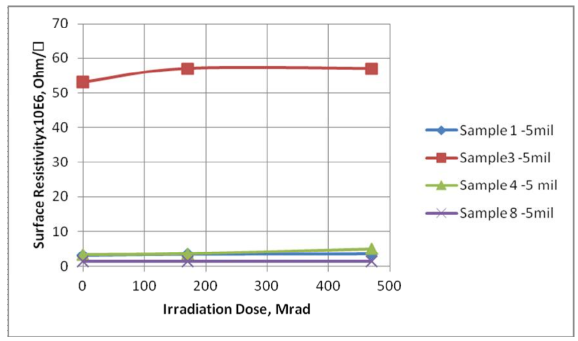

Fig.8. Summary of the changes in SR values after consecutive irradiation tests for 5 mil thick Kapton500HN.

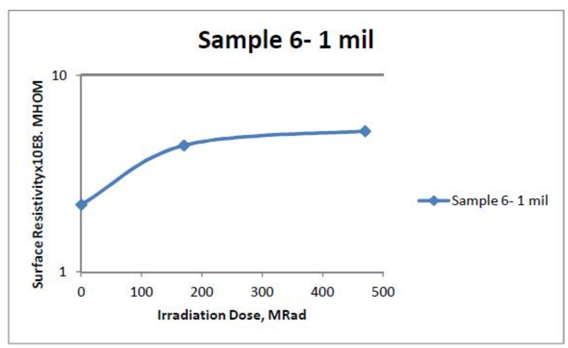

Fig. 9. Summary of the changes in SR values after consecutive irradiation tests for 1 mil thick Kapton100HN

Table 6: Summary of SR values for flat PEEK coupons before and after three consecutive BOL/EOL irradiation experiments and extended storage in the lab.

Run/Test#	Coupon #	SR of PEEK coupons, (S/□) x 10^{9}				
		Pristine	After EOL		After 2 additional BOL/EOL	
		0 MRad	~30 MRad SR Tested		470 MRad SR Tested	
		06.15. 2016	08.15 2016	06.09 2018	11.24. 2016	06.09. 2018
Run #6, 10^{8} - 10^{9}	#12	1.2	1.9	3.0	3.0	4.5
	#13	1.2	2.0	3.5	5.6	1.4
	#14	1.2	2.0	2.0	2.9	4.0
	#15	1.34	2.6	4.5	5.4	7.0
	#16	1.9	3.0	5.0	5.0	6.0

Table 7: Summary of SR values for Kapton samples before (BOL) and after three consecutive irradiation experiments (EOL) and extended storage in the lab.

ID#	Surface Resistivity of Kapton samples with CarboSurf (S/□)			
	BOL	Irradiation Dose: MRad		
		170	470	
		EOL Tested 09.30.16	EOL Tested 11.24.16	EOL Tested 09.10.2018
5 mil	$3.2\cdot10^{6}$	$3.5\cdot10^{6}$	$3.6\cdot10^{6}$	$4.5\cdot10^{6}$
1 mil	$3.9\cdot10^{6}$	$4.3\cdot10^{6}$	$6.1\cdot10^{6}$	$6.4\cdot10^{6}$
5 mil	$5.3\cdot10^{7}$	$5.7\cdot10^{7}$	$5.7\cdot10^{7}$	$8.3\cdot10^{7}$
5 mil	$3.3\cdot10^{6}$	$3.6\cdot10^{6}$	$4.9\cdot10^{6}$	$4.2\cdot10^{6}$
1 mil	$1.8\cdot10^{7}$	$2.4\cdot10^{7}$	$3.5\cdot10^{7}$	$4.0\cdot10^{7}$
1 mil	$2.2\cdot10^{8}$	$4.0\cdot10^{8}$	$5.2\cdot10^{8}$	$8.0\cdot10^{8}$
1 mil	$5.4\cdot10^{10}$	$9.0\cdot10^{10}$	$15\cdot10^{10}$	$4.0\cdot10^{10}$
5 mil	$1.3\cdot10^{6}$	$1.3\cdot10^{6}$	$1.3\cdot10^{6}$	$1.3\cdot10^{6}$

4.0 CONCLUSIONS

A unique set of collaborative Projects was conducted where ion beam surface treatment and thin DLC film ion beam deposition were applied to thin and thick space polymers and external FCC components of solar arrays, with following GEO long-term simulation testing that covered the full spectrum of electron energies and a partial spectral range of protons. It was shown that all Carbosurf+ treated and tested samples remained charge dissipative after irradiation in simulated GEO environment covering ranges of up to 15 years in GEO. A major conclusion reached in these studies is that the developed ion beam surface treatments can be used successfully to prevent or mitigate both surface charging and deep charging in dielectric polymers flown in long-term GEO missions. The obtained results provide a basis to expect that Carbosurf+ treatment when applied to various dielectric polymers and based on them external radiation sensitive space components will allow preventing the damage arising in space polymer dielectrics in GEO from surface and deep charging.

5.0 ACKNOWLEDGEMENTS

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