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Ion beam-treated space polymers: long-term stabilityin GEO-simulated environments

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Abstract

As part of a large program conducted between years 2010 and 2018, a number of ion beam surface treatments and proprietary coatings formed on advanced space polymers by direct ion beam deposition were developed at Integrity Testing Laboratory (ITL) Inc, in Canada. This technology allowed producing surfaces with controlled surface resistivity in a wide range of charge dissipation values, with negligibly low additional RF losses and other important functional properties, that allowed using such treated materials and products in modern space antennae, solar arrays and other external applications on spacecrafts in GEO environment. This paper will present an overview of results achieved throughout the years on enhancement of radiation stability of space polymers that were ion beam treated and coated with special multifunctional thin coatings by direct ion beam deposition. The treated/coated subjects have been ground-based tested in a range of radiation conditions, simulating the GEO radiation environment, conducted in three world-recognized GEO simulation facilities.

Keywords GEO radiation environment · Deep charging · Surface charging · Surface resistivity · Carbosurf+

1 Introduction

The work presented in this paper was conducted during a number of projects on the development of special important surface treatment technologies for external environmentally sensitive space materials and structures for vehicles operating in GEO long-term space missions. Both, the thin polymer films, common in space vehicles' design and applications, and comparatively thick surface-treated/coated space dielectric-based structural components have been considered for these surface treatments and the following ground-based GEO space environment long-term simulation testing.

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Consequences of charging in GEO orbits have ranged from various anomalies in space missions up to satellite failures. Most of the undesired effects of charging are due to the discharge arcing, and subsequently, the physical materials damage [1–3]. 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 modern long-duration missions, and therefore, requires the development of strong preventive and protective mitigation techniques.

In the GEO environment, the spacecraft are experiencing irradiation by electrons in a wide energy spectrum with low-density electrons fluxes. 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, 3]. The influence of proton irradiation, as well as UV exposure, sometimes needs to be considered at GEO simulation testing as well.

2 Medium- and low-energy GEO simulation testing of surface-treated polymers

2.1 Space polymer materials

A very innovative and productive approach for preventing the external dielectric space materials from charging in GEO has been developed at ITL Inc using ion beam treatments and special direct ion beams deposition of multifunctional thin coatings. It was used in a number of applications, for example, in modern high-frequency, high-pulse space antennas and for components of solar arrays. Results of bombardment of organic polymers in vacuum with medium or low-energy ion beams are considered to be very specific in comparison with other types of materials. Due to polymer chain scission and destruction processes, the volatile elements from bombarded areas can diffuse to the surface and finally be released in vacuum, leaving behind more or less carbon enriched, or carbonized, subsurface layers. Diffusion of some hydrogen, oxygen and nitrogen, the gaseous atoms that are common elements in hydrocarbon polymers, was shown to be high enough in ion-bombarded polymers, to migrate from the damaged area to the surface and for their extensive release in vacuum as various volatile compounds, leaving behind the areas of strong carbonization. Depending on ion bombardment conditions, temperature and ion beam dose, or ion fluence, drastic re-arrangements have been shown to happen in the carbonized layers, up to formation of amorphous hydrogenated diamond-like new structures. Such regions commonly attain surface conductivity in a wide, few orders of magnitude charge-dissipative range.

For instance, we used the results from our earlier work on ion implantation of semi-metal or metal elements, such as Si and Al, or any of them in combination with B, at energy E = 30KeV and fluence 5×10^{16} cm^{-2} for successful erosion protection of space polymers in LEO environment from atomic oxygen [4–6]. The results from secondary ion mass spectrometry (SIMS) depth profiling of implanted ~ 100-µm-thick KaptonHN polyimide had shown the enrichment of subsurface layers with the implanted elements, strong N and O depletion and carbon enrichment in the implanted area. The treated surfaces demonstrated induced surface resistivity (SR) in a charge-dissipative range. In this case, the implantation was the primary process, and carbonization has been the secondary one. Successful protection from the atomic oxygen erosion have been shown in tests in ground-based simulated LEO environment and in LEO space exposure in MISSE experiments at International Space Station [6].

In case of surface treatment of space polymers for GEO application, where the carbonization effects are to be of primary concern, we used ion beam treatments with rare gases. Positive results have been achieved not only with medium energy ion beam treatments at dozens of KeV but also with low-energy rare gas ions, mostly using Ar + ion beam, at E ~ 3 keV.

Earlier results, confirmed recently in [7], had shown that diffusion coefficients for He, Ne and Ar in polymers are sufficiently high to prevent any accumulation of rare gas atoms in the polymers in ion beams treatments. We established that by varying rare gas ion fluencies in the range 5×10^{16} cm^{-2}–3×10^{17} cm^{-2}, the required for GEO performance charge-dissipative values of SR can be achieved that range from 10^9 Ω/sq to 10^6 Ω/sq, in addition to other attractive surface properties.

In addition, a special tendency of the same sputter rate change as a function of ion beam fluence in gaseous ion beam treatment of many polymers in the energy range E = 1–5 keV was found in [8]. The sputter rates of the polymers decreased strongly with increased ion fluence, followed by a very low constant removal rate in the steady-state region. The decrease in the sputter rate is, typically, around one order of magnitude [8], and should be limited, as we can predict, by the sputtering coefficient of carbon—the lowest sputtering coefficient of all chemical elements. Our computer simulation results using SRIM program [9] had shown that the depth of the altered layer in polymers at Ar + ion beam bombardment with E = 3 keV is expected to be around 15–20 nm. The affected layer depth may increase to some extent, especially if samples are heated during treatment, due to diffusion and re-arrangement effects in the altered area and around it.

A special high-fluence ion beam surface treatment technology, named Carbosurf, has been developed and used at ITL for surface carbonization of space polymers [10–12]. The surfaces of space polymer films and polymer-based space products were made charge dissipative in a wide range of SR values when treated by the developed and patented Carbosurf technology. Carbosurf is a surface modification process that provides surface carbonization of polymers, based on ion beam surface treatment. This treatment enables the transformation of a very thin subsurface layer of the polymer into a highly carbonized structure, to get the requested SR value in a static-dissipative range, while remaining transparent to radio-frequency (RF) wavelengths. This material proved to be superior to alternate static-dissipative coatings such as Germanium on KaptonHN and Upilex polyimides (Ge/Kapton or Ge/Upilex) in many aspects (robustness, resistance to humidity, possibility to get SR in a wide range, low dependence of SR over temperature, i.e., SR(T)). The ability to provide charge-dissipating properties is of crucial importance when very high RF power handling is required as in modern space antennas.

To improve further the durability of the materials, in particular, the stability of the surface to handling, chemical and mechanical resistance, as well as stability over time in lab conditions (shelf life) an enhanced version of Carbosurf was developed. In the improved version, named Carbosurf+(or sometimes "Carbo+ " for short), a thin multifunctional hydrogenated Diamond-like Carbon (DLC) film deposited by ion beam technique was added on top of the treated material, to provide the mentioned above additional durability properties. The developed technology was scaled-up around 2012–2013 for batch manufacturing of flight materials and was applied mostly to 1- to 5-mil-thick KaptonHN and some space grade poly-ether-ether-ketone (PEEK) films. A production vacuum facility was used that combined a gaseous linear powerful technological ion beam source with the energy up to E = 3 keV, a magnetron source, and a special designed heater with automatic temperature control. When used as a batch coater, the facility allowed to treat space polymer films up to 1.8 × 0.5 m in size.

The Carbosurf treatment and the results for a number of space polymers such as Kapton, PEEK, Mylar, Upilex, clear CP 1 and white CP-W have been described in [10]. Examples of SR values in the charge-dissipative range, achieved on three space polymers that were Carbosurf treated with Ar and Xe noble gas ion beams at high fluencies in three runs are shown in Table 1. Ion beams of Xe+at an energy E = 15 keV and total fluence 10^{15} cm^{-2}, and Ar + ion beams at an energy E = 25 keV and total fluence 3×10^{16} cm^{-2} (run I) and 5×10^{16} cm^{-2} (run II) have been used, with samples being kept at room temperature in all experiments.

A number of analytical techniques were used to evaluate and characterize different properties of the treated materials. Thus, SR and its variation with temperature, and thermo-optical characteristics, including the solar absorptance (α) and thermal emittance (ε), were measured after Carbosurf treatments and after testing in GEO-simulated environments. In addition, resistance to radiation in GEO simulation facilities was evaluated for equivalent of 5 and 15 years in GEO environment conditions. The structure and composition of pristine and treated/deposited materials were studied using surface analysis techniques that included optical imaging, SEM/EDS, survey and high resolution XPS, surface roughness measurements. The data from XPS surveys confirmed that the top surface of the treated polymers contains ~ 98.7% at. carbon.

Table 1 Surface resistivity of thin (1 mil) polymer films after moderate energy ion beams treatments at room temperature [10]

Material	Surface resistivity at room temperature, ρ (Ω/sq.)		
	Xe^+	Ar+ (run I)	Ar^+ (run II)
CP 1 White	0.75×10^7	2.5×10^8	1.3×10^7
CP 1 White	0.8×10^7	3×10^8	3×10^7
CP 1	0.6×10^7	5×10^8	1.3×10^7
CP 1	0.75×10^7	5.2×10^8	6×10^7
Kapton HN	1.5×10^7	5×10^{10}	3×10^9
Kapton HN	1.3×10^7	3.5×10^{10}	1.9×10^9

As can be seen from Table 1, the surface resistivity values depend significantly on the type of used ions and the treatment conditions, like energy and total fluence. For the same material, changing the type of gas or the fluence, a 200 times difference in SR values has been achieved. It was also found that the SR values are sensitive and dependent on substrate temperature.

Based on the encouraging results of surface modification of polymers with various noble gas ion beams at moderate energies and the strong surface carbonization at high-dose, low-energy ion beam bombardment, we conducted a number of successful studies with treatment of space polymers with the ion beams in the low-energy range [11, 12].

The temperature dependence of the SR, i.e., SR(T), is of particular interest for space applications, due to the wide temperature range experienced by satellites in space. The results of the measurements of SR(T) after the Carbosurf treatment of polyimide KaptonHN films are presented in Fig. 1. A lower T-dependence was found and confirmed in a wide SR range, when compared with the data for regular used Ge/Kapton that is highly beneficial for antenna applications in space [10]. The linear dependence SR($T^{-1/4}$), following [13, 14], means that the variable range, 3-dimensional hopping conductivity is the mechanism that provides charge dissipation of our surface-treated materials.

The thermal optical properties may be affected to a certain extent by Carbosurf treatment, depending on the

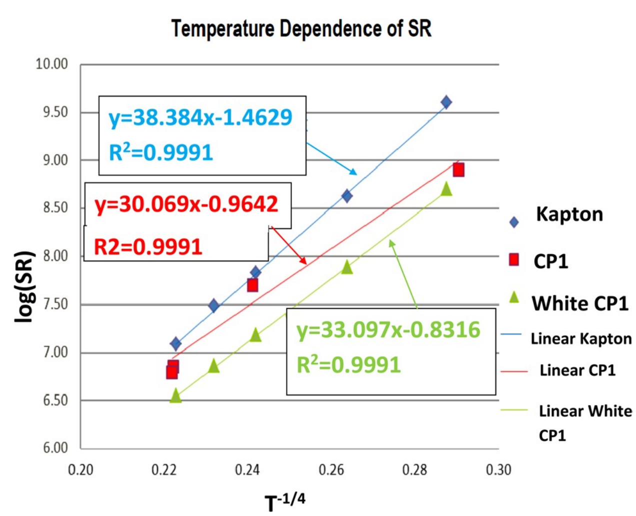

Fig. 1 Semi-log graphs of the $T^{-1/4}$ temperature dependence of the surface resistivity of various space polymer materials with Carbosurf

Table 2 Thermal optical properties and SR of KaptonHN polymer films after Carbosurf surface treatment by Ar + ion beam, E = 3 keV [12]

Sample #/thickness	Apparent solar absorptance α_S (on Al-backing)		Apparent thermal emittance ε (over high emissivity standard)		Surface resistivity (MΩ/□)
	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)

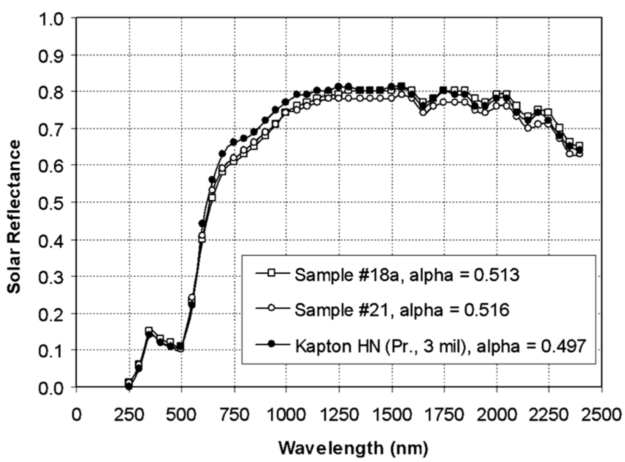

Fig. 2 Total solar reflectance spectra of Carbosurf treated and pristine Kapton HN after low-energy (3 kV) Ar^+ beam treatment. The insert shows the calculated solar absorptance, α_S [12]

selected ion beam sources and treatment conditions, as shown in Table 2 and Fig. 2.

Samples of all treated polymers were tested and successfully passed the in-house thermal cycling, humidity, flexibility test by bending over a mandrel, as well as in-vacuum outgassing test.

To improve further the quality of the treatment, a more versatile Carbosurf+ technological process was developed later at ITL, that included direct IBD. It is well known that the direct IBD technology is a low-rate vacuum deposition process, used mostly for thin film's deposition at comparatively low temperature, allowing applications to thermally sensitive substrates. However, it provides the conditions for top, superior quality thin coating's formation, with high density, uniformity, low stress level, low defects, etc. This additional stage in the process was performed without breaking vacuum, by changing the ion beam working gas to a special mixture of carbonaceous gases. Deposition

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 Kompozit facility with the irradiation conditions of ($p^+ + e^- +$ UV) equivalent to ~ 5–7 years in GEO orbit [10]

Material	Treatment	SR (Ω/□)	
		As deposited	Rad. tested
Kapton HN	Pristine	$>10^{12}$	10^9
Kapton HN	Ion beam treated	$(13–25) \times 10^6$	18×10^6
CP 1 (white)	Pristine	$>10^{12}$	8×10^8
CP 1 (white)	Ion beam treated	$(13–60) \times 10^6$	0.8×10^6
CP 1 (clear)	Pristine	$>10^{12}$	0.8×10^9
CP 1 (clear)	Ion beam treated	$(60–80) \times 10^6$	0.7×10^6
Upilex S + Ge coating	Ge (1000 Å)	2×10^7	1.3×10^8

conditions have been made adjustable to meet various customer requirements.

The most appropriate gas composition, deposition regimes and temperature range for a number of applications have been found as an outcome of significant amounts of experimental work and computer simulations.

The Carbosurf+ -treated space polymer thin films and some space products successfully passed, in addition to ITL in-house testing, mentioned above, a full space qualification program that was performed at two reputable space companies, MDA (Montreal, Canada) and Airbus (Munich, Germany).

Ground-based GEO simulation testing of Carbosurf and Carbosurf+ -treated samples was conducted at two world-class simulation facilities, the JSC Kompozit facility in Russia [11] and the SIRENE facility in France [15, 16]. The Kompozit facility includes beam sources of electrons and protons in the low-to-mid energy range that can simulate the influence of the GEO environment mostly on the surface properties of tested external space materials and structures, and a UV source. All environmental factors can be applied sequentially or simultaneously. Table 3 shows an example of SR data obtained for pristine and ion beam-treated space polymers before and after one of such GEO simulation tests. As can be seen in Table 3, even in pristine space polymer films, like KaptonHN polyimide or partially fluorinated polyimide CP 1, some RIC was observed after irradiation exposure that was simulating the equivalent of 5–7 years in GEO. The SR for pristine materials changed from ~ 10^{12} Ω/□ to ~ 10^{9} Ω/□. It is also clear from Table 3 that for the treated polymers after the GEO-simulated exposures, the SR changed insignificantly.

The samples treated by Carbosurf+technology underwent also a successful extended, 7–9 years long shelf-life evaluation in laboratory conditions (with temperature in the range 15–25 °C, and humidity in the range 35–55%, practically without SR changes.

It is interesting to mention that accidental immersion of Carbosurf+ -treated samples in water for 48 h (roof damage) with a follow-up drying in lab conditions did not cause any SR changes in the affected samples.

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 provided charge dissipative SR values also remained practically unchanged.

The lower values of SR even decreased slightly, but notsignificantly, to the end of the tests, and the rest of SR values were left completely unchanged. All these results confirmed an outstanding radiation resistance of all Carbosurf and Carbosurf+-treated space polymers during the testing with comparatively low- and mid-energy irradiation sources for simulating GEO environment [11, 12].

An additional benefit of depositing a DLC-like coating in the Carbosurf+ process is the ability to get differently doped thin DLC-like surface coatings by adding a particular type and amount of selected gas components, such as silane, nitrogen, and fluorine, to the ion beam gaseous mixture. Formation of such doped coatings allowed to extend the amount of useful space materials properties, for instance, providing enhanced atomic oxygen erosion resistance in LEO using Si doping. Such materials are needed in case of Electric Orbit-Raising (EOR) launches to bring spacecraft to GEO [17]. It was shown that while depositing the doped coatings, not only was it possible to achieve SR in the static-dissipative range, but it was confirmed that the SR(T) behavior was the same as for non-doped films. This can be seen in Fig. 3 for the case of Carbosurf+treatment of Kapton500HN and for two Si-doped Carbosurf+ samples of Kapton100HN. The SR(T) variation of these samples can also be presented in a similar way as in Fig. 1, to confirm the same variable range hopping conductivity mechanism.

The results in Fig. 3 clearly show that the conductivity mechanism in the optimally Si-doped ion beam-treated thin coating-deposited surfaces is the same, i.e., hopping conductivity. Therefore, all the benefits, mentioned above for use of these materials in antennae applications, can be easily forcasted. As it was mentioned in our patent[12], as an example, this material was used as a horn aperature cover on high power space antennas. There is no other material on the market presently that can withstand the very high RF power density seen on some horn aperture covers, while meeting the ESD requirements of being static charge-dissipative. The RF power handling of Carbosurf+ before thermal runaway proved to be at least 5 times higher than conventional semicondutive coatings like Germanium on KaptonHN. This material was used already on a total of 30 antennas/feeds installed on 11 different spacecraft.

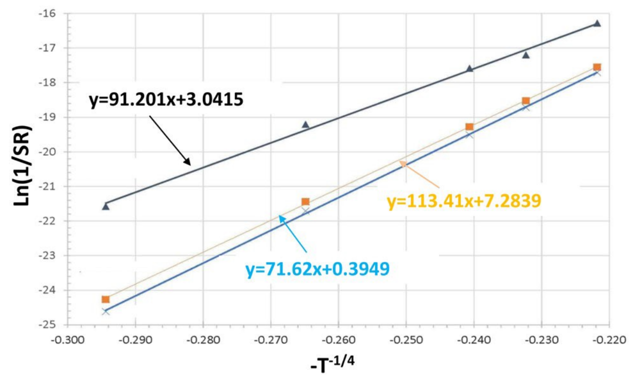

Fig. 3 Semi-log graphs of the $T^{[-1/4]}$ temperature dependence SR (RT) of the surface resistivity of Kapton500HN polyimide with Carbosurf+treatment and two Si optimally doped Carbosurf+-treated Kapton100HN with different SR at room temperature.—Carbosurf+ : Red box—on 5 mil Kapton; X—Si-doped Carbosurf+on 1 mil Kapton, High SR; ▲—Si-doped Carbosurf+on 1 mil Kapton, low SR value [17]

2.2 Flat cable conductors

The developed Carbosurf+ technology was also used successfully in another project to prevent surface charging of specific external space structural components—Flat Cable Conductors (FCCs), used on Solar Arrays of satellites in GEO orbits [18].

The FCCs are manufactured by forming special structural components, using thin Cu wires encapsulated from both sides in thin Kapton100HN films (see Fig. 4). Due to the specifics of the manufacturing process, the FCCs have on both sides a “grooved” surface relief. One of the formed surfaces (identified as “back” here), due to the manufacturing process, has been contaminated with tiny embedded pumice particles. To make the contaminated surfaces of the FCC’s charge dissipative, a modified version of Carbosurf+ treatment was used that allowed for a simultaneous surface renewal during ion beam treatment. The patented modified surface treatment version can be used, in general,

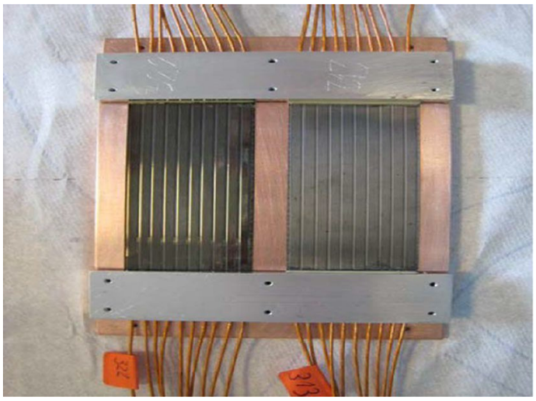

Fig. 4 FCC samples, treated by Carbosurf+ , mounted on a copper plate for ESD testing in SIRENE facility: left—front side of a treated FCC; right—back side [18]

for hydrocarbon polymeric materials and products with any inorganic inclusions [19].

The FCCs, treated on both sides, passed successfully a full space qualification program at Airbus (Munich, Germany) and the GEO simulation testing at JCS Kompozit facility, without compromising the Beginning of Life/End of Life (BOL/EOL) thermal surface properties, as shown in Table 4.

Additional GEO simulation testing under electron exposure in a wide energy range, closely resembling a real GEO environment, was conducted at the SIRENE facility in France [15, 16]. 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 (ESD) testing (Fig. 4) in the most dangerous, low-temperature GEO environment conditions demonstrated that the original SR values and other functional surface properties remained practically unchanged [18, 19].

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 FCCs in the conditions, imitating expected long-term GEO missions [18, 19].

Figure 5 shows an example of the evolution of surface potential on FCC samples during SIRENE × 4 irradiation and relaxation at − 145 °C. The complete absence 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

Table 4 Thermal optical characteristics of pristine, Carbosurf+ (Ar + ion beam treated), and GEO-tested FCC samples, tested at JCS Kompozit

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

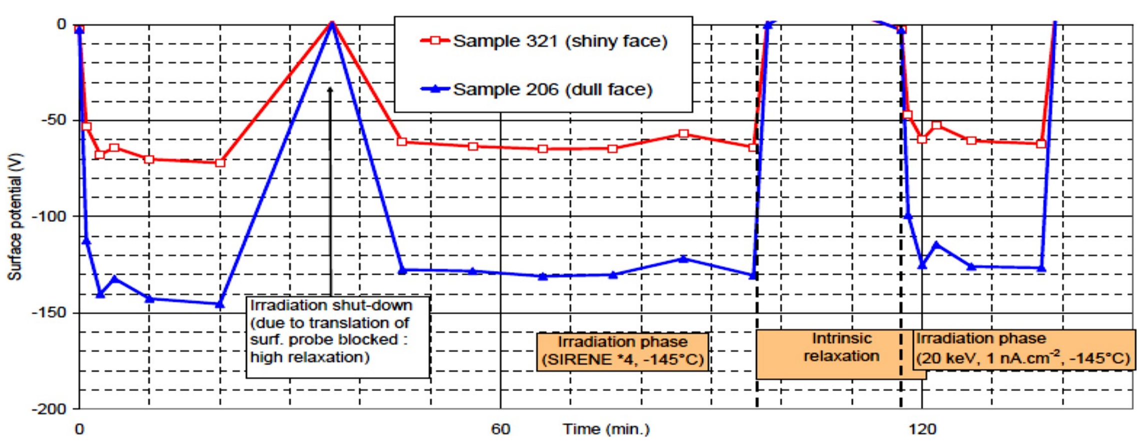

Fig. 5 Evolution of surface potential on FSS samples during GEO simulation SIRENE × 4 irradiation and relaxation at − 145 °C [18]

deep charging of the comparatively thick structures (with thickness in the mm range).

3 High-energy GEO simulation testing of surface-treated space 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, with results to be presented elsewhere. 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 KaptonHN 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 8 KaptonHN samples of two different thicknesses, i.e., 1 and 5 mil. They were treated at ITL with the Carbosurf+technology and exposed to MeV electron irradiation during testing in three consecutive irradiation experiments.

3.1 Samples used in the accelerator exposures

Irradiation tests were performed on Carbosurf+ -treated samples that included KaptonHN thin film substrates and PEEK flat coupons.

First, each substrate was surface treated with an Ar+ion beam, 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+. On both PEEK and KaptonHN materials, the coatings have been formed at different deposition conditions, to cover an SR range of 10^6–10^9 Ω/□.

Figure 6 shows the images of Carbosurf+ -treated or treated/GEO-tested PEEK and KaptonHN substrates. The light color frames around the circumference of the samples represent regions that were masked during the Carbosurf+treatment. The dark middle regions were treated with Carbosurf+ . The surfaces of all polymer samples got darker after the radiation exposure, similar to the effects in GEO simulation testing of Carbosurf+-treated/coated samples in other facilities, as was discussed above.

The surface morphologies of PEEK and KaptonHN samples were examined before and after Carbosurf+treatment and again after they were exposed to the range of irradiation conditions from BOL to EOL, using optical and scanning electron microscopy (Fig. 7), as well as surface roughness evaluation (Table 5). As can be seen from Table 5, significantly higher roughness is obvious for PEEK in comparison with KaptonHN surface.

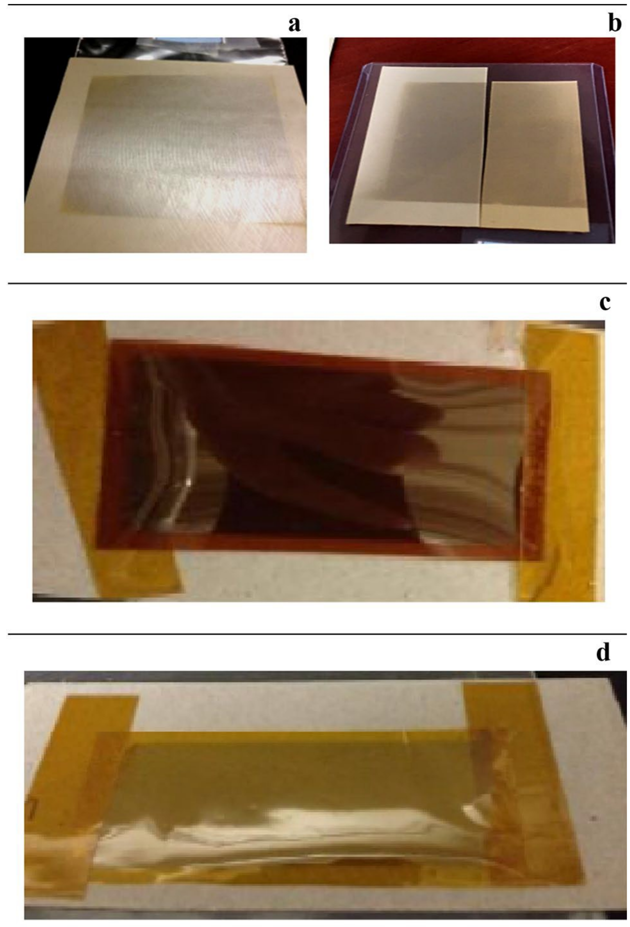

Fig. 6 Optical images of samples that were irradiated at Idaho Facility. **a** Flat PEEK coupon, "as deposited" condition (no irradiation). **b** The PEEK sample on the left irradiated to a dose of 30 Mrad, the sample on the right irradiated to the full dose of 470 Mrad. Notice distinctive yellowing and darkening of the sample on the right. **c** Kapton500HN sample (5 mil thick) irradiated to a dose of 470 Mrad. **d** Kapton100HN sample (1 mil thick) irradiated to a dose of 470 Mrad

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.

3.2 Preparation of samples for testing

Samples were subjected to standard cleaning with methanol and a vacuum bake out at 383 ± 1 K (110 °C) and 5×10^{-4} Pa for ~ 72 h prior to mounting in the sample trays to minimize absorbed water and volatile contaminants 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 the

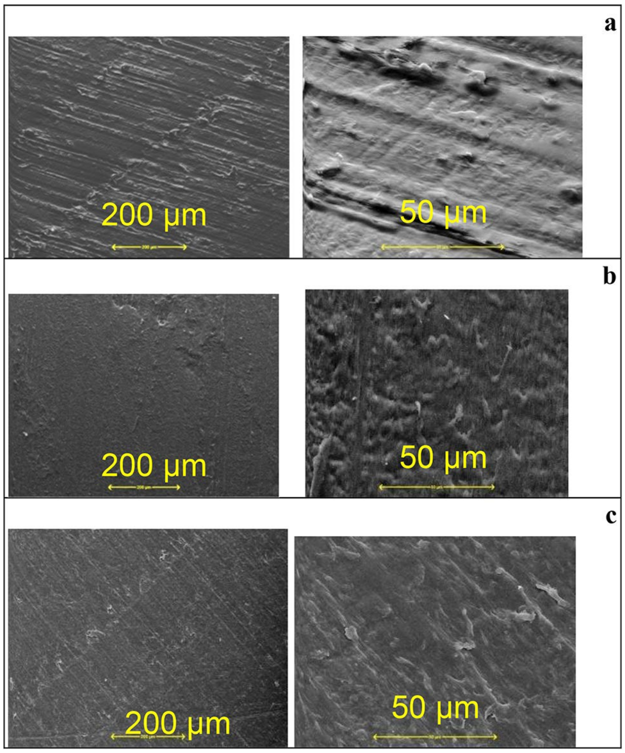

Fig. 7 Scanning electron microscopy analysis (secondary electrons) of surfaces of pristine PEEK coupon (**a**), of PEEK coupon after Carbosurf+(**b**) and after 470 Mrad irradiation dose (**c**)

Table 5 Summary of surface roughness data of the flat PEEK coupons and the KaptonHN space film samples used in irradiation experiments

Test	Roughness, Ra (µm)		
	PEEK	Kapton	
		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

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 and 4 cm high trays made of 250-µm-thick Al which underwent similar vacuum bake out prior to sample transfer (Fig. 8). 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 than in 3 h after radiation testing, when in atmospheric conditions

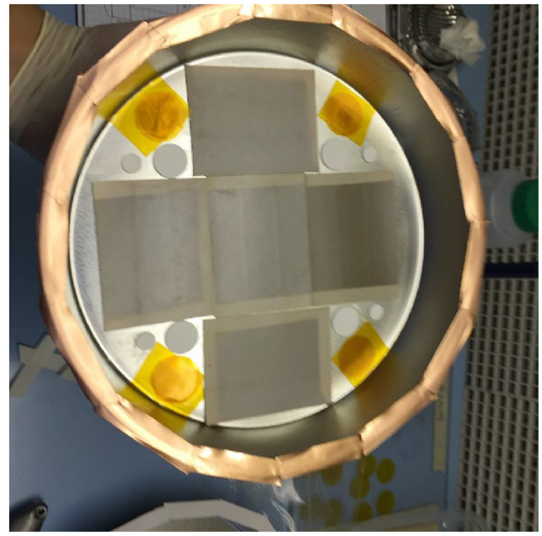

Fig. 8 Optical image of some of the samples that were irradiated at IAC Facility. Samples were laid out to provide best uniformity of exposure across them

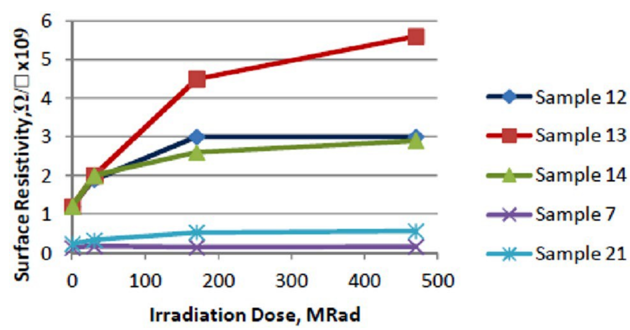

Fig. 9 Summary of the changes in SR values after three consecutive irradiation tests for PEEK coupons with initial SR values in the range 10^7–10^9 (Ω/□)

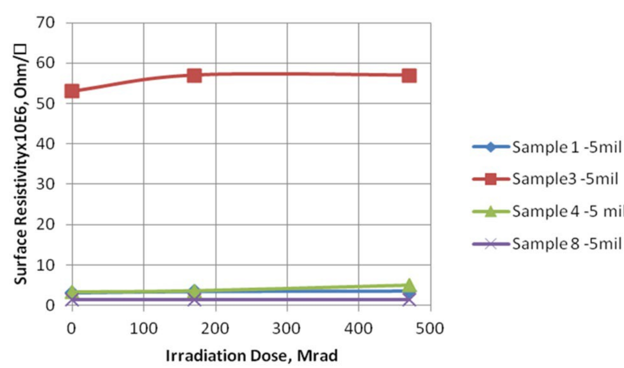

Fig. 10 Summary of the changes in SR values after three consecutive irradiation tests for Kapton500HN, 5 mil thick

with ~ 40% RH. The SR results, shown in Figs. 9 and 10, are after longer post-testing exposure to atmospheric conditions, in more than ~ 50 h.

3.3 Sample 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), 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 BOL and EOL conditions for typical 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 [20]. 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 [21], it is easy to estimate, that in the Kompozit 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 years TID in an unshielded GEO environment or ~ 1 year and ~ 6 to 24 years TID for electrons above ~ 500 eV in a typical GEO environment.

4 Results and discussion

Figure 9 presents the SR measurement results after irradiation exposure of a number of Carbosurf+-treated PEEK samples, starting with BOL values and the SR values after every of three consecutive irradiation experiments, till EOL. Figure 10 presents similar results for the 5-mil-thick KaptonHN samples. As can be seen from Figs. 9 and 10, all SR values remained in the pre-testing 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. Very similar results were also received for the 1-mil-thick KaptonHN samples.

The SR values for all samples were re-measured after a prolonged storage in laboratory conditions. Table 6 presents 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 Table 6, the SR values show a trend for a very small increase, remaining, however, in the same charge-dissipative range after both, a high-energy electron irradiation and the storage in laboratory conditions. The small increase is more pronounced for PEEK that might be due to its higher surface roughness.

It is interesting to note that in the last few years, we have been testing a number of worldwide selected advanced

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

Sample/ID#	Pristine	SR tested after 2 additional BOL/EOL	
	0 Mrad	470 Mrad	
	Year 2016	Year 2016	Year 2018
SR of PEEK samples with Carbosurf+before and after testing, (Ω/□) × 10^9			
#12	1.2	3.0	4.5
#13	1.2	5.6	1.4
#14	1.2	2.9	4.0
#15	1.34	5.4	7.0
#16	1.9	5.0	6.0
SR of KaptonHN samples with Carbosurf+before and after testing (Ω/□) × 10^6			
5 mil	3.2	3.6	4.5
1 mil	3.9	6.1	6.4·
5 mil	53	57	83
5 mil	3.3	4.9	4.2
1 mil	18	35	40
1 mil	2.2×10^8	520·	8.0×10^8
5 mil	1.3×10^6	1.3	1.3

external space materials and structures for potential Lunar applications using an ITL designed and developed Lunar Dust Simulator vacuum facility. The ion beam-treated space polymers demonstrated significant dust mitigation properties [22], that is highly promising for their use in future Lunar exploration missions.

5 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 a 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 surface charging in external 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 charging. Those treated polymers have also shown promising results in mitigation of Lunar dust adhesion on external sensitive materials and structures and may be, therefore, considered in future Lunar exploration.

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Declarations

Conflict of interest All the authors have no conflicting or competing interests.

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