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Charging Properties of Space Used Dielectric Materials

Thierry Paulmier, Bernard Dirassen, Mohamed Belhaj, David Rodgers

***Abstract*— Charging effects in space are widely dominated by bulk conductivity (intrinsic and radiation induced [instantaneous or delayed]) of the irradiated materials. These data are needed by the community to perform charging predictions for assigned missions. This study was dedicated to the characterisation of bulk and radiation induced conductivity of polymers and non-polymers (ceramics and glasses) and their evolution in regard of sample reproducibility, composition, structure variation and environmental conditions (radiation dose rate, temperature, electric field). Three different polymers have been tested in this study: FEP, PEEK, ETFE. Three non-polymer materials have been tested as well: two borosilicate glasses and Aluminium oxide Al_2O_3. The major objective was, for polymers, to understand physical variations of conductivity for similar materials and, for non-polymers, to get better knowledge on the main physical contributions steering bulk and radiation induced conductivity at low temperature.**

Keywords— Spacecraft Charging, Bulk conductivity, Radiation Induced Conductivity, Polymers, Glass, Ceramics, Charge Transport

I. INTRODUCTION

For space scientific missions, charging levels need to be assessed very accurately which implies developing dedicated experimental methods for physical parameter extraction, including intrinsic and radiation induced surface and volume conductivities. Databases have been established which enable designers to get information on defined physical properties but the existing physical data are still sparse, and some important key properties are not included (for instance delayed radiation induced conductivity). The reason is that the different key properties are highly dependent on the environmental irradiation conditions (incident flux and energy, temperature, charging history, …). Evolution of radiation induced conductivity is especially a major concern since this effect strongly depends on various parameters (radiation dose rate, total radiation dose, temperature, electric field). The physical mechanisms underlying these effects are still not quite well understood and have not been fully integrated in databases or simulation tools for surface and internal charging prediction for planetary missions. Recent and preliminary experimental studies, performed especially at ONERA have revealed that high radiation dose received by space materials (especially polymers) must induce significant ageing, delayed effects (such as delayed radiation induced conductivities) and alter the material properties to a significant level [1-3]. These effects should therefore be taken into account in the numerical codes to achieve realistic prediction. The need of material characterisation under realistic extreme environments to account for instantaneous radiation and low temperature effects on material physical parameters becomes then compulsory for the construction of this material database and to carry out satellite design validation.

The overall objective of this study was to provide material data information and understanding of charging properties and environmental effects. The project was especially focused on the internal charging issue. This study was especially dedicated to the characterisation of bulk and radiation induced conductivity of polymers and non-polymers (ceramics and glasses) and their evolution in regard of sample reproducibility, structure variation and environmental conditions (radiation dose rate, temperature). For polymers, the objective was to understand physical variations of conductivity of similar materials. Intrinsic and Radiation Induced Conductivity (RIC) have been assessed at room temperature on three kinds of polymer materials : FEP, PEEK, ETFE. For non-polymers, the objective was to get a better knowledge on the main physical contributions steering bulk and radiation induced conductivity at low temperature. Bulk conductivities and RIC have been analysed on three different materials: two different borosilicate glasses (CMG and borofloat from Schott) and alumina Al_2O_3. We demonstrated in this study that bulk conductivity and RIC strongly depends on structure, manufacturer, composition of the tested materials. Physical mechanisms underlying these conductivities, as well as environment effects (temperature, radiation, delayed effect) vary with material nature.

II. EXPERIMENTAL SET-UP AND PROTOCOLS

A. Experimental facilities

The experiments have been performed in the unique irradiation test facilities (SIRENE), funded by CNES and installed at ONERA (Toulouse, France) which allows charge characterization under GEO-like electron irradiation spectrum. Fig. 1 shows the electron beam spectral characteristics of the SIRENE facility with an energy spectrum (Kp>5) approaching that of the geostationary charging environment.

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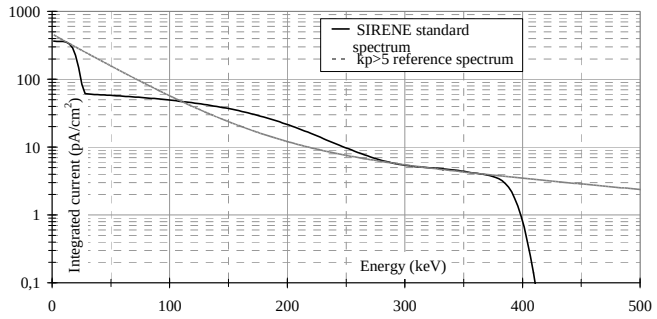

Fig. 1. SIRENE standard spectrum and reference KP>5 spectrum

SIRENE electron spectrum ([20keV, 250pA/cm2] + [0-400keV, 50pA/cm2]) experimental simulation is achieved with the use of two monoenergetic electron beams. In order to get a spacelike electron beam, the 400keV electron beam, passing through complex diffusion foils, is dispersed in energy from 0 to 400keV. The nominal fluxes used for the 20keV monoenergetic beam and the distributed 400 keV one are respectively equal 250pA/cm^2 and 50pA/cm^2 but can be raised respectively up to 1nA/cm^2 and 200pA/cm^2. A pumping system allows experiments at vacuum of around 10^{-6} mbar. The temperature of the sample holder can be controlled in the range of -180°C to 250°C allowing to reproduce the temperature variation of materials on flight. The evolution of charging potential, during and after beam cut-off are monitored using a non-contact electrostatic probe (Kelvin probe TREK 3455ET) coupled with an electrostatic voltmeter (TREK 341B).

An other facility called SPIDER (Storage of Post Irradiated Dielectrics for Electron Relaxation) was used in this study: this facility enables storing irradiated materials under vacuum (up to 10^{-6} hPa) for a long period of time (up to 1 or 2 years) to study relaxation physical processes of these materials through various in-situ, controlled and automatic measurements (surface potential with Kelvin probe, leakage and displacement currents, Non-contact Pulsed Electro-acoustic Method [PEA]). Fig. 2 presents a view of this SPIDER facility installed at ONERA (Toulouse). It was especially used in this study to performed long term surface potential measurements (up to one year storage under vacuum) of different dielectric materials previously irradiated in SIRENE at given potentials and then transferred under vacuum with a third transfer unit called STRASS.

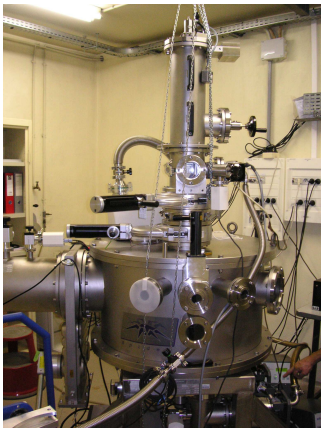

Fig. 2. View of the SPIDER facility

These different facilities allowed accurate assessment of RIC and bulk conductivities of the different dielectric materials.

B. Experimental protocols

Dedicated methods have been devised at ONERA for the evaluation of intrinsic and radiation induced conductivities. Characterization of space material conductivities is then usually performed in our studies using the surface potential decay method. For this method, the experimental procedure is summarised hereafter:

- The sample was first exposed to charging by the means of the low energy electron beam (20 keV, Beam 1 of Fig. 3). The back face of the sample was previously metal-coated to connect properly this face to the ground and avoid any stray capacitances (due to void gaps) between this face and the grounded support.

- The sample irradiation was then shut down, and its surface potential continuously monitored by use of an electrostatic (contact-less) surface voltmeter, yielding to the decrease of surface potential versus relaxation time, $V_s(t)$.

- The model of a capacitor relaxing its charge through a resistance (see Fig. 3) was then applied. The time constant of the discharge (RC) was determined and ultimately the resistivity ρ (Ω.m) or the inverse, its conductivity.

$$\sigma=\varepsilon_0.\varepsilon.dV_s/dt/V_s\ (\Omega.m)^{-1} \quad (1)$$

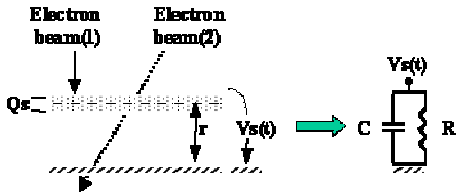

Fig. 3. The surface voltage decay method for intrinsic and radiation induced conductivity evaluation (electron beam 1: charging beam – electron beam 2: decay stimulating beam for RIC assessment)

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In the conductivity evaluation approach, when the surface potential is allowed to decay "naturally" (Beam 2 in Fig. 3 is not used), the end result is the so called dark conductivity, which is thought to depend, for most dielectrics (the conduction of which is not ohmic), on the electric field, temperature, and on total dose (effect of ageing or delayed radiation induced conductivity).

Radiation induced conductivity (RIC) depends on the dose rate dD/dt and on two parameters k and Δ characteristic of each material:

$$\sigma = k.(dD/dt)^{\Delta} \quad (2)$$

where σ is the radiation modified conductivity, D the radiation dose, dD/dt the dose rate, Δ a coefficient (without unit) with a value generally between 0.5 and 1 and k is the radiation induced conductivity coefficient

For space application, the charge decay method is usually applied for RIC measurements. However, instead of letting the charges leak "naturally" in the volume, a penetrating ionizing beam (Beam 2 in Fig. 3) is now used to stimulate the decay. To determine the k and Δ factors, these measurements must be repeated at three levels of dose rate. The k and Δ parameters can be extracted by representing $Ln(\sigma)$ as a function of Ln(dD/dt). Using equation 2, we can then write $Ln(\sigma)=Ln(k)+\Delta.Ln(dD/dt)$ and then derive k and Δ.

For RIC evaluation, the samples have been irradiated with 20 keV electron beam up to a given surface potential. The irradiation was then stopped and the potential relaxation was stimulated using a high energy electron beam (400 keV in SIRENE) at a given flux in regard of the desired radiation dose rate. The stimulated relaxation lasted around six hours. Prior to these tests, the samples were outgassed in the irradiation chamber under vacuum (10^{-6} hPa) for at least 72 hours. The materials have been tested at room temperature for radiation dose rates close to the one that we can meet in Jovian Environment. The tests complied with the particle flux (or radiation dose rate) in worst case conditions received by the materials during the overall dedicated mission. It has been decided to work with the following three radiation dose rates:

- 0.24 mGy/s (corresponding to a incident 400 keV electron current equal to around 0.1 $pA.cm^{-2}$)
- 1.2 mGy/s (corresponding to a incident 400 keV electron current equal to around 0.5 $pA.cm^{-2}$)
- 2.4 mGy/s (corresponding to a incident 400 keV electron current equal to around 1 $pA.cm^{-2}$)

Bulk conductivities have been assessed using SIRENE (for irradiation) and SPIDER (for long term relaxation) facilities installed at ONERA (Toulouse, France). For RIC extraction, the SIRENE facility, equipped with a 400 keV electron accelerator and a 7-100 keV electron gun, has been used.

C. Tested materials

This study was divided in two different tasks :

- **The first task** was devoted to study the evolution of bulk and radiation induced conductivity of polymers in regard of sample reproducibility and modified sample (in regard of additives, composition and material manufacturer). Three kinds of polymers have been tested: FEP, PEEK, ETFE.
- **The second task** was devoted to characterise bulk and radiation induced conductivities of borosilicate glasses and ceramic space materials (alumina) at low temperature and derive related conductivity parameters. The major objective of this task was to understand the physical mechanisms steering these different conductivities through representative Jovian temperature tests. Delayed effect on radiation induced conductivities of ceramics and glasses has been characterised as well in this study.

In the first task, the different tested samples are the following:

1. FEP materials (influence of manufacturer)

- Teflon® FEP (from Dupont) - 100 µm thick
- Neoflon® FEP (from Daikin) - 100 µm thick
- Norton® FEP (from Saint Gobain) - 100 µm thick

2. PEEK materials (influence of structure and manufacturer)

- Semi-cristalline PEEK Aptiv 1000 (from Victrex) - 100 µm thick
- Amorphous PEEK Aptiv 2000 (from Victrex) - 100 µm thick
- PEEK VESTAKEEP (from Evonik) - 100 µm thick

3. ETFE materials (influence of structure and composition)

- Non-crosslinked ETFE sheet (provider: Goodfellow) - 100 µm thick
- Crosslinked red ETFE cables (ESCC reference: ESCC3901 012 10) - 200 µm thick
- Crosslinked white ETFE cables (ESCC reference: ESCC3901 012 10) - 200 µm thick

The experimental tests for the first task have been performed at room temperature (temperature of internal equipment on many spacecrafts). Bulk conductivities and RIC have been extracted for the different materials.

For the second task, bulk conductivities and RIC (instantaneous and delayed) have been analysed on three different materials: two different borosilicate glasses (CMG and Borofloat® from Schott) and alumina AL_2O_3.

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III. EXPERIMENTAL RESULTS

A. *Conductivities of space used polymers*

The experimental results are presented hereafter by material type in each material family. Bulk conductivity and radiation induced conductivity are presented at the same time to get a clear view on the different amplitudes and notice the effect of RIC on the overall relaxation process.

1) FEP materials

Three Teflon® FEP samples have been stored for 10 months in SPIDER for bulk conductivity evaluation. Fig. 4 presents the evolution of potential during the intrinsic relaxation after surface charging with 20 keV electrons. Relaxation kinetics is very low and decreases over time. We can then reach relaxation kinetics as low as 0.4 V per day. For sample 1, the surface potential decrease is equal to 200 V for 10 months relaxation. Fig. 5 presents the evolution of bulk resistivity as a function of relaxation time: we can notice a constant rise of this parameter since other physical mechanisms (surface radiation induced conductivity, polarisation, electron emission, electric field effect on conductivity) fades away during relaxation. It is worth mentioning that evolution of resistivity with electric field does not fit with any conventional Poole-Frenkel and Adamec-Calderwood formula. Significant fluctuations can be observed on this parameter since relaxation kinetics between two points can be lower than the measurement resolution. 45 days relaxation duration is therefore necessary at the end of the relaxation between two measurement points to exceed the resolution of the measurement chain. Considering this amplitude, we can assess bulk resistivity for Teflon® FEP equal to 4.10^{19} Ω.m (relative permittivity is equal to 2.2, has been assessed from the charging kinetics at the irradiation start). We can notice that reproducibility is very good with an average bulk conductivity equal to 2.8 10^{-20} $\Omega^{-1}.m^{-1}$ and a standard deviation equal to 9 %.

The same procedure has been applied for the other two FEP materials. We have been able to observe that the three FEP materials (see Table I) present similar bulk conductivities, close to 10^{-20} $\Omega^{-1}.m^{-1}$.

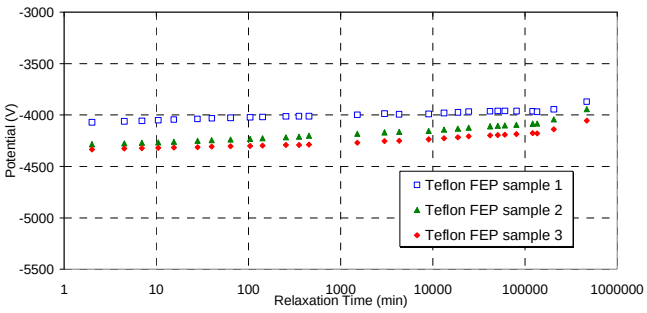

Fig. 4. Evolution of surface potential measured on Teflon® FEP samples for Bulk conductivity evaluation

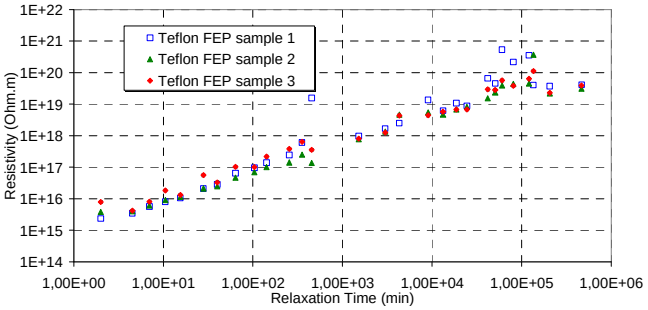

Fig. 5. Evolution of bulk resistivity during the overall relaxation phase for Teflon® FEP samples

TABLE I. BULK CONDUCTIVITY FOR THE DIFFERENT POLYMER MATERIALS

	Bulk conductivity ($\Omega^{-1}.m^{-1}$)	**Relaxation time**
Teflon®	$2.7\ 10^{-20}$	143 days
Neoflon®	$2.1\ 10^{-20}$	192 days
Norton®	$2.1\ 10^{-20}$	192 days
Victrex PEEK 1000	$4\ 10^{-20}$	166 days
Victrex PEEK 2000	$1.1\ 10^{-19}$	147 days
Evonik VESTAKEEP	$1.8\ 10^{-19}$	138 days
Non-crosslinked ETFE	$5.6\ 10^{-20}$	186 days
AXON red ETFE cable	$1.6\ 10^{-19}$	176 days
AXON white ETFE cable	$4.5\ 10^{-19}$	176 days

Radiation induced conductivity has been assessed following the stimulated potential relaxation method described in section II.B. Fig. 6 presents the evolution of surface potential during irradiation and relaxation phases. We can clearly observe a noticeable increase of the relaxation kinetics once the 400 keV irradiation is switched on (initiation of RIC). Fig. 7 presents evolution of induced resistivity for the radiation dose rate equal to 0.24 mGy/s. We can notice that RIC first increases and then decreases over time. This can be explained by the fact that RIC depends as well on radiation dose (and not only on radiation dose rate) and electric field: the first step (decrease of resistivity) is due to the rise of RIC with radiation dose (density of free holes in the Valence band increases) [4] . The second phase (increase of resistivity) is ascribed to the decrease of electric field which acts then on RIC. Adamec and Calderwood formula can not be fitted on these experimental results. The reason is that effect of electric field is certainly counterbalanced by radiation dose effect which gradually tends to increase either trapped or free charges density. For extraction of k and Δ parameters, it has been decided to take the maximum value of RIC for each radiation dose rate, which corresponds, in average, to the time required for RIC to reach its nominal value. We noticed very good

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reproducibility from one sample to the other. Table II presents average values of RIC for the three applied radiation dose rates. From this value, we can draw the evolution of RIC with radiation dose rate (Fig. 8) and extract k and Δ (Table III).

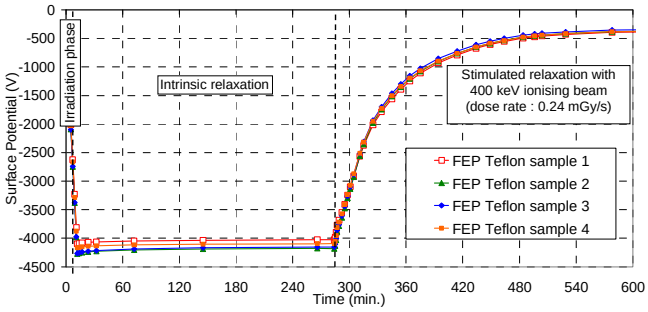

Fig. 6. Evolution of surface potential during the different steps for evaluation of RIC on Teflon FEP samples (dose rate : 0,24 mGy/s)

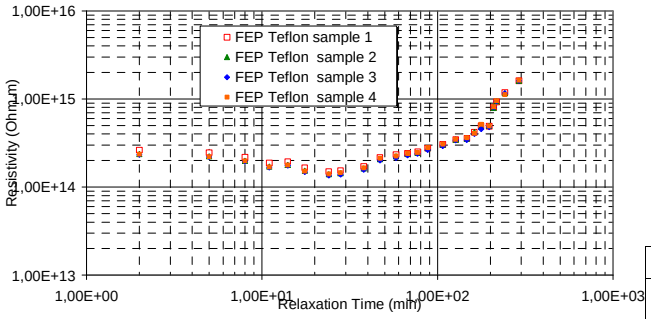

Fig. 7. Evolution of induced resistivity during relaxation stimulated phase for Teflon FEP samples (dose rate : 0.24 mGy/s)

TABLE II. RIC ASSESSED ON TEFLON® FEP FOR THE THREE RADIATION DOSE RATES

	RIC ($\Omega^{-1}.m^{-1}$)	Standard deviation
0.3 mGy.s^{-1}	7 10^{-15}	9 %
1.4 mGy.s^{-1}	1.8 10^{-14}	3.9 %
2.6 mGy.s^{-1}	2,2 10^{-14}	3.6 %

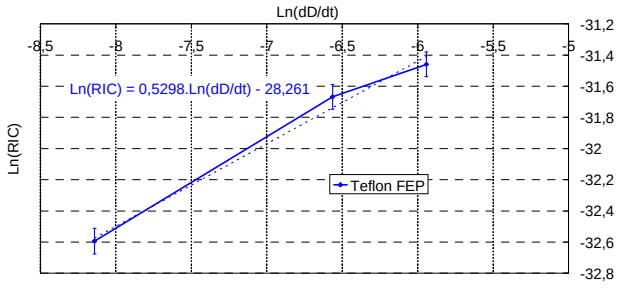

Fig. 8. Evolution of radiation induced conductivity [Gy] as a function of radiation dose rate [Gy.s^{-1}] for Teflon FEP samples

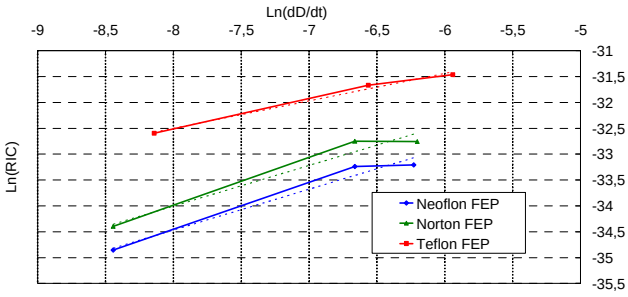

Fig. 9. Comparison of RIC [Gy] between the three tested FEP materials (radiation dose rate in Gy.s^{-1}): Dupont Teflon® FEP, Saint-Gobain Norton® FEP and Daikin Neoflon® FEP

TABLE III. RIC PARAMETERS FOR THE DIFFERENT POLYMER MATERIALS

	k ($\Omega^{-1}.m^{-1}$)	Δ
Teflon®	5.3 10^{-13}	0.53
Neoflon®	6 10^{-13}	0.79
Norton®	9.3 10^{-13}	0.78
Victrex PEEK 1000	7.5 10^{-13}	0.98
Victrex PEEK 2000	1.8 10^{-13}	0.77
Evonik VESTAKEEP	2.2 10^{-12}	1
Non-crosslinked ETFE	1.5 10^{-13}	0.87
Red ETFE cable	NA	NA
White ETFE cable	NA	NA

We noticed (Fig. 9) that RIC is 1 decade higher for Teflon® than the other two FEP materials in this range of radiation dose rate. However Teflon® presents a lower Δ parameter which could lead to RIC for Teflon equal or even lower than Neoflon® and Norton® RIC if the materials are submitted to higher radiation dose rates. Neoflon® presents a slightly lower RIC than Norton®.

2) PEEK materials

Fig. 10 compares RIC profiles for the three tested PEEK materials (PEEK 1000, PEEK 2000, VESTAKEEP). We can

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notice that PEEK 1000 and PEEK 2000 present the same RIC profile with equivalent amplitudes. There is only a slight higher RIC for higher radiation dose rate on PEEK 1000. However, Evonik PEEK presents a much higher RIC than Victrex PEEK which could be of interest for the reduction of charging hazards. Discrepancies between the different materials might come from different cristallinities which can act on charge carrier trapping processes and mobility. It is interesting as well to notice that RIC of PEEK (Fig. 11) keeps up the same value along the stimulated radiation, contrary to what was observed on other materials such as FEP or Kapton® [5]. Delayed RIC on PEEK quickly fades away after the high energy electron radiation shut-down (Fig. 11). We observed as well that bulk conductivity of the three PEEK materials is very low (Table I).

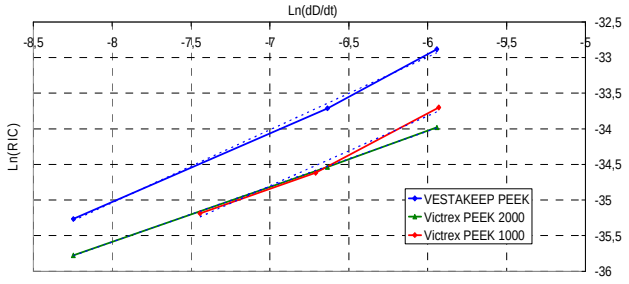

Fig. 10. Comparison of RIC between the three tested PEEK materials (radiation dose rate in Gy.s^{-1}): Victrex APTIV PEEK 1000, Victrex APTIV PEEK 2000 and Evonik VESTAKEEP PEEK

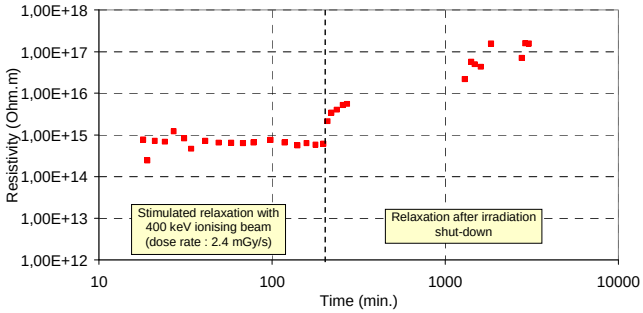

Fig. 11. Evolution of the induced resistivity during relaxation stimulated phase for APTIV 1000 PEEK samples (dose rate : 2.6 mGy.s^{-1})

3) ETFE materials

The experimental tests performed on the three ETFE based materials seem to reveal an increase of bulk conductivity with cross-linking process (see Table I). Colour (linked to additives injected in the material) seems as well to have a slight influence on the electric conductivity. The experimental tests performed with radiation dose rates do not allow extracting RIC on ETFE cables. However, an additional test performed at much higher radiation dose rate revealed that RIC on crosslinked ETFE cables is two decades lower than RIC on non-crosslinked ETFE : 5.10^{-15} $\Omega^{-1}.m^{-1}$ for crosslinked ETFE versus 10^{-13} $\Omega^{-1}.m^{-1}$ for non-crosslinked ETFE at a radiation dose rate equal to 660 mGy.s^{-1}. It is interesting as well to notice that RIC on non-crosslinked ETFE is 5 times lower than RIC measured on Teflon® FEP (see Table III). The main conclusion is that ETFE does not present any significant RIC for radiation dose rates applied in this study.

B. Conductivities of non-polymers

Bulk conductivities and RIC have been analysed on three different materials: two different borosilicate glasses (CMG and Borofloat) and alumina Al_2O_3 (96 % purity - provider : Goodfellow). Bulk conductivities have been assessed at three different temperature : room temperature, -80°C and -150°C. Instantaneous and delayed RIC has been assessed at low temperature (-150°C). Bulk conductivity of the three materials is strongly dependent on temperature, as we can see in Table IV.

TABLE IV. BULK RESISTIVITY ASSESSED AT DIFFERENT TEMPERATURES FOR THE DIFFERENT MATERIALS

	CMG (at ≈ 1.3.10^7 V/m)	**Borofloat (at 5.10^6 V/m)**	**Alumina (at 1000 min. relaxation)**
20°C	1.3 10^{14} Ω.m	1.4 10^{13} Ω.m	1.4 10^{16} Ω.m
- 80°C	1.7 10^{14} Ω.m	5.4 10^{14} Ω.m	6. 10^{16} Ω.m
- 150°C	2.1 10^{14} Ω.m	5.10^{15} Ω.m	2 10^{17} Ω.m

Thorough analysis allowed demonstrating that bulk conductivity of the three materials is steered at low temperature by variable range hopping process (VRH), meaning that charge transport occurs through hopping process from one trapping site to the other with no injection into the conduction band. It has actually been demonstrated that bulk conductivity is field dependent and could be written for the three materials with the following law :

$$\sigma=\sigma_0.(T_0/T)^{1/4}.\beta_1(E).\exp[-\beta_2.(T_0/T)^{1/4}] \quad (3)$$

for which β_1 and β_2 are energy ratios for tunnelling and hopping processes, T_0 is a reference temperature.

The test of validity of this formula on experimental data is a plot of $Ln(T^{1/4}.\sigma)$ versus $T^{-1/4}$ for a given electric field, which should produce a straight line. Fig. 12 presents this plot for CMG at -80°C and for electric field equal to 1.3 10^7 V/m. We can then notice clearly that conduction in CMG is ruled by VRH process at low temperature. The experimental behaviour is similar for Borofloat and alumina.

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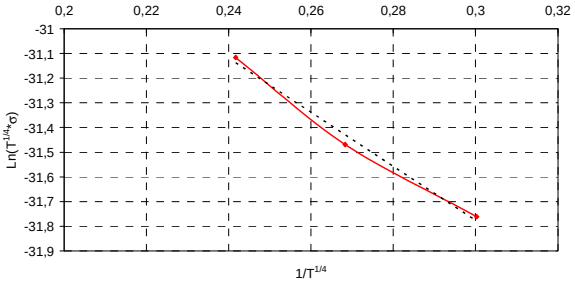

Fig. 12. Numerical fit with VRH model for thermal evolution of electric conductivity on CMG for an electric field equal to $1.3.10^7$ $V.m^{-1}$

At room temperature, we demonstrated that bulk conductivity of glasses is mainly steered by Poole-Frenkel effect (detrapping to extended states). It is important to note that evolution of charging rate as a function of temperature can not be ascribed to the sole evolution of conductivity: secondary electron emission yield seems indeed to decrease as temperature falls down (due to decrease of holes mobility with temperature), yielding then to higher charging rate at lower temperature, which is especially conspicuous for Alumina (see Fig. 13).

As seen in Fig. 14, experiments performed with 400 keV stimulation of potential relaxation demonstrate that both glasses do not present any significant RIC at low temperature: bulk conductivity overcomes RIC even for high radiation dose rates. Alumina presents noticeable RIC when working at high radiation dose rates (Fig. 15). However, RIC quickly fades away once the ionising beam is switched off: Alumina does not present any significant delayed RIC at low temperature.

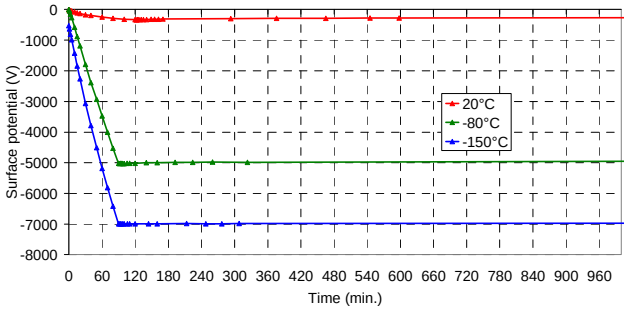

Fig. 13. Evolution of surface potential on alumina measured in SIRENE at room temperature, -80°C and -150°C

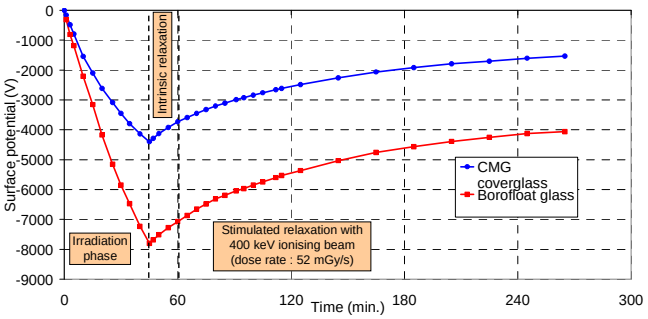

Fig. 14. Evolution of surface potential on CMG and borofloat at -150°C during irradiation, intrinsic and stimulated relaxation for RIC assessment at 52 mGy/s

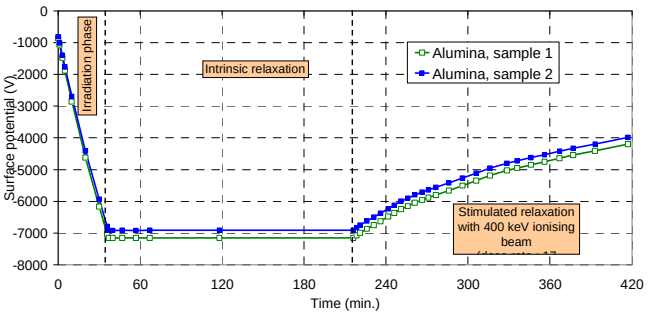

Fig. 15. Evolution of surface potential on Alumina at -150°C during irradiation, intrinsic and stimulated relaxation for RIC assessment at 52 mGy/s

IV. CONCLUSION

Bulk conductivity and Radiation Induced Conductivity have been assessed in different space used materials. The different extracted data can be implemented in conventional numerical tools for charging prediction in space environment. For polymers, we put into evidence strong discrepancies from one supplier to the other or from one grade to the other (for a given material type). It demonstrates that choice of material type, nature, composition and structure is of high importance for charge mitigation. For non-polymers, it has been observed that bulk conductivity was steered by complex mechanisms (hopping, electric field assisted detrapping) and that RIC can be discarded for borosilicate glasses while it may be significant for alumina.

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