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Experimental test facilities for representative characterization of space used materials

Thierry Paulmier, Bernard Dirassen, Mohamed Belhaj, Virginie Inguimbert, Denis Payan, Nicolas Balcon

***Abstract*— Dedicated experimental facilities have been developed at ONERA and CNES (Toulouse, France) for the electrical qualification of space used materials and the extraction of the different physical parameters steering their charging behaviour under electron irradiation. These facilities allow working in a large energy range between few eV up to several MeV, a large temperature range (down to -150°C and up to 600°C depending on the facility) and at high vacuum level. This paper aims at presenting the technical characteristics of these different facilities.**

Keywords— Irradiation facilities, Spacecraft Charging, Bulk conductivity, Radiation Induced Conductivity, Charge Transport, Secondary Electron Emission

I. Introduction

Spacecrafts and their component materials are submitted in planetary magnetosphere to high particle radiation flux and extreme environment that could lead to important and hazardous electrical charging levels. The need for assessing external and internal charging levels and predict any risk of electrostatic discharge led to the development of several codes for the simulation of space plasma / spacecraft interaction. Experimental characterisation is moreover necessary for space material qualification, which implies the characterization of their charging behaviour through ground experimental tests under representative electron irradiation spectrum. For numerical prediction, it is important as well to extract the different key physical parameters steering charge process, such as intrinsic and radiation induced conductivity (surface and bulk), permittivity, secondary electron emission (yield and emission spectra). Dedicated experimental facilities have therefore been developed at ONERA and CNES (Toulouse, France) for these different tasks. These facilities allow working in a large energy range between few eV up to several MeV, a large temperature range (down to -150°C and up to 400°C depending on the facility) and at high vacuum level. This paper aims at presenting the technical characteristics of our different relevant facilities. These different facilities allow qualification of space used materials (conductors, insulators, semi-conductors) and the extraction of the different physical parameters steering their charging behaviour under electron irradiation.

For thorough assessment of bulk intrinsic conductivity and the analysis of ionisation and ageing recovery effects, it is also of high importance to store the irradiated materials for a long period of time. Storage has to be performed under high vacuum level without any vacuum break-up during the sample transfer to avoid any possible air recovery at atmospheric pressure. Dedicated transfer unit and storage facility have also been developed for the above purpose.

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II. THE SIRENE EXPERIMENTAL FACILITY

The irradiation test facility SIRENE (Fig. 1), funded by CNES and installed at ONERA (Toulouse, France) allows charge characterization under GEO-like electron irradiation spectrum. Fig. 2 presents the electron beam spectral characteristics of the SIRENE facility with an energy spectrum (Kp>5) approaching that of the geostationary charging environment.

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 250 pA/cm^2 and 50 pA/cm^2 but can be raised respectively up to 1 nA/cm^2 and 200 pA/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).

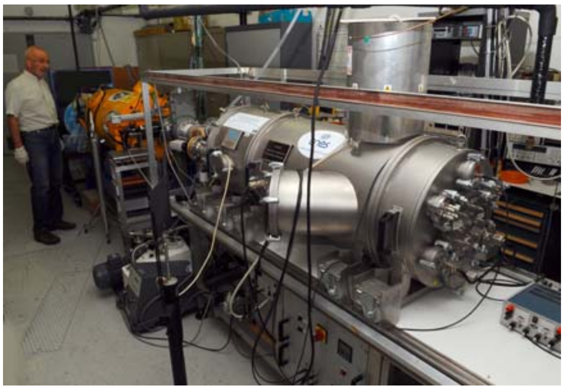

Fig. 1. View of the SIRENE facility

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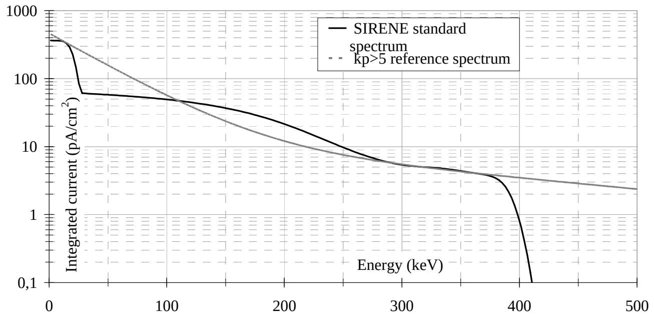

Fig. 2. SIRENE standard spectrum and reference KP>5 spectrum

This facility allows realistic assessment of voltage built up in geostationary orbit. Its flexibility allows the simulation of extreme environments different than the geostationary one (eg, MEO, LEO or other planetary radiation environments). This facility is widely used as well for the characterisation of radiation induced conductivities of space materials in vacuum thanks to the use of a penetrating 400 keV electron beam which stimulates the potential decay during the relaxation phase without inducing any bulk charging. Using this high energy electron beam and a high incoming flux on the sample (up to 5 $nA.cm^{-2}$), we are able as well to perform radiative ageing on the material. Space materials, such as polymers (Kapton, Teflon, ...), adhesive or coverglasses, can then be aged in SIRENE with equivalent flight durations equal to several months or years. Thanks to these different experimental specifications (bulk conductivity and RIC characterisation, temperature effect) and its spectrum flexibility, the SIRENE experimental facility is highly relevant for space material qualification and extraction of physical key parameters for charging prediction.

III. THE GEODUR IRRADIATION FACILITY

GEODUR is a radiation test facility allowing the study of satellite internal and surface charging, evaluation of RIC of thick materials and sample radiative ageing using 400 keV to 2 MeV monoenergetic electrons. It is equipped with a 2 MeV Van de Graaff electron accelerator and a double scattering system for the production of a distributed electron spectrum in the energy range [200 keV – 1 MeV]. It is instrumented with a contact-less electrostatic probe and current measurement systems for the characterisation of internal charging behaviour of space elements. The temperature of the sample holder can be controlled in the range [-180 °C, +250 °C] allowing to reproduce the temperature variations of materials on flight. A pumping system allows experiments at vacuum of around 10^{-6} hPa. Transfer of charged samples from GEODUR to the storage facility SPIDER (or to SIRENE or DEESSE facilities) can be performed under vacuum with the STRASS unit transfer (see next section)

Fig. 3. View of the GEODUR facility

IV. THE SPIDER STORAGE FACILITY AND STRASS TRANSFER UNIT

A. *Technical details*

The SPIDER experimental test facility enables storing irradiated dielectric materials under vacuum (up to 10^{-6} hPa) for a long period of time (up to several months) 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]). This facility can be coupled with a new existing transfer unit STRASS (System for Transfer from SIRENE to SPIDER) that enables displacing, under vacuum, dielectrics irradiated samples from one test facility to an other one without any vacuum break-up (that would lead to sample discharging or annealing of radiation effect). We can then measure conductivities as low as 10^{-20} $\Omega^{-1}.m^{-1}$. This test facility enables as well studying evolution of delayed radiation induced conductivity or photo-conductivity for a long period of time or the relaxation of physical ageing process (for instance, through the adjustment of relaxation time between two successive irradiation steps).

The measurement method that shall be described in the following section is based on the analysis of surface potential relaxation after irradiation shut-down. Through the potential relaxation kinetics, we are then able to extract a bulk conductivity value. However, for high resistive material, the intrinsic relaxation kinetics is not steered, during the first minutes or hours, by the sole effect of bulk conductivity. Polarisation, surface induced conduction and relaxation electron emission [1] process can take place and rule at great level the relaxation kinetics, leading then to a high value for the apparent conductivity and to a temporal decrease of this effective parameter that are not realistic [2]. The intrinsic conductivity assessed at the beginning of relaxation of Teflon® FEP sample is then equal to 10^{-16} $\Omega^{-1}.m^{-1}$. This value is far too high and not realistic in comparison with the value provided in the handbooks (10^{-19} $\Omega^{-1}.m^{-1}$). This relaxation method is however an optimized method to extract real conductivity but the condition for high resistive materials is to

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store and monitor relaxation of these materials under vacuum for a long time to get rid off the processes ruling relaxation during its first few hours. For polymer materials, the time for bulk conductivity to prevail over the other processes can reach several days or weeks. The motivation for developing the SPIDER facility was therefore to be able to store the samples that have been irradiated in SIRENE to be able to extract accurate values of bulk conductivities. The second motivation was to equip this facility with different analytic equipment (potential and current measurements, non-contact PEA) so as to be able to study charge transport in the material and understand, through crossed analysis in SIRENE and SPIDER, the physical mechanisms steering the different processes underlying charging and relaxation electrical behaviour.

SPIDER facility (Fig. 4) enables to store under vacuum (10^{-6} hPa) at room temperature dielectrics samples previously irradiated in SIRENE. It is a cylindrical vessel with 260 litres volume capacity equipped with rotative and motorized cylindrical support case able to receive up to 12 sample holders (which can interlock in the cylindrical case). SPIDER is equipped with two high vacuum system unit (each unit is composed of a primary vacuum pump coupled to a turbo-molecular pump) to allow redundancy and avoid then any vacuum break-up in case of failure of one pumping unit. Each pumping system is equipped with pneumatic vacuum valve and automatic start-up system to switch off automatically the failed pumping system and start up simultaneously the second pumping system. Irradiated samples can then be stored for several months or years to study the charge relaxation processes and charge transport in dielectrics materials. We are then able assessing bulk and surface intrinsic conductivity with high precision using the potential decay method. The facility is equipped with a Kelvin potential probe installed on a motorized support allowing scanning all samples placed on each sample support. Each sample holder is placed in front of the potential probe by automatic motorized rotation of the cylindrical support case: all samples placed in SPIDER can be scanned automatically with defined scanning frequency thanks to the controlled displacement of the cylindrical case and the potential probe. Control of the different translation and rotation movements within SPIDER and potential measurements are performed with a Labview program installed in a dedicated PC. The potential measurement data are stored and classified automatically in the computer for each sample and scan. Two different sample holders are available: one conventional sample holder on which planar samples can be fixed and one PEA sample holder on which it is possible to connect two PEA (Pulse ElectroAcoustic) measuring heads for non-contact PEA measurements. SPIDER is therefore equipped with a PEA exciting non-contact electrode that can be placed at few millimeters (the distance is controlled with good precision) from both samples placed on both PEA heads. Both types of sample holder are equipped at their back with 12 pins sub-D connectors to perform electric current measurements or any other electric operation.

Fig. 4. SPIDER facility and its transfer unit STRASS installed on top.

The Kelvin potential probe as well as the exciting PEA electrode are placed on dedicated vacuum lock (coupled with manual vacuum valve) to allow changing or repairing these measurement systems (in case of failure or damage) without any vacuum break-up within SPIDER. The STRASS transfer unit is placed at the top of SPIDER and connection between both facilities is performed through a dedicated vacuum lock equipped with two vacuum valves.

It was indeed important to avoid any vacuum break-up for the transfer of samples between SIRENE and SPIDER, since this process would induce sample discharging and recovery effect. The STRASS unit facility has been developed for this purpose. This facility enables to move irradiated samples from SIRENE to SPIDER (and the reverse as well). It is a cylindrical vessel with 13 liters volume capacity equipped with a mobile secondary vacuum pumping system (primary vane pump coupled to a turbo-molecular pump) to reach vacuum level down to 10^{-6} hPa. The transfer unit is composed of a telescopic vertical shifting system with locking/unlocking device to pick up and disconnect samples from the different facilities (SPIDER - SIRENE), store and maintain them in this unit for vacuum transfer and then connect them to another facility.

The major equipment used for the evaluation of bulk intrinsic and delayed radiation induced conductivity is the Kelvin potential probe. Kelvin probes used in SIRENE, GEODUR and SPIDER are +/- 20 kV potential probe (reference: TREK 3455ET) connected to a Trek electrostatic voltmeter (reference: TREK 341B). This measuring system has a sensitivity of 1 V, a lateral resolution equal to around 10 mm when the sample / probe distance is equal to 8 mm. Measured potentials as a function of probe location on samples is controlled and recorded on dedicated PCs. All surface potential measuring system have been calibrated with reference potential source (standard ISO 9001 reference instrument calibrated by external reference companies) available at the DESP/ONERA metrology laboratory.

Surface potential measurements could be coupled to leakage and displacement electric measurements for redundancy on

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the evaluation of charge diffusion kinetics during the relaxation phase. Electric current measurements are usually performed with pico-ammeters or with current voltage converters designed at ONERA and coupled with multimeters. These electric current measurement systems have been calibrated using current calibrated source (standard ISO 9001 reference instrument calibrated by external reference companies) available at the DESP/ONERA metrology laboratory.

B. Use of spider for intrinsic conductivity assesment

Different experimental tests have been performed for the extraction of bulk intrinsic conductivity of space used polymers in this SPIDER facility. Samples have first been irradiated in SIRENE facility with low energy (20 keV) electron beam up to a given surface potential. They have then been transferred to SPIDER with the STRASS facility. No electrostatic discharge are detected on the samples during this transfer. The surface potential can be recorded every day and month on the different samples. Surface mapping of the charging potential can then be performed in SIRENE, as seen in Fig. 5. The slight well observed at the center of the sample is due to the fact the electron beam is deflected to the sample edges when the sample get highly charged. This is not observed for smaller samples.

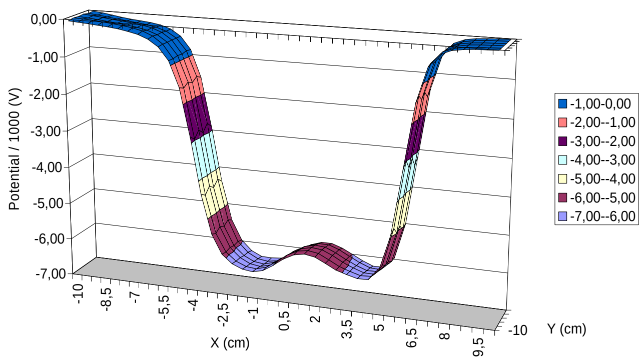

Fig. 5. Mapping of surface potential measured in SPIDER on Teflon® FEP sample irradiated with 20 keV electron beam up to -6350 V.

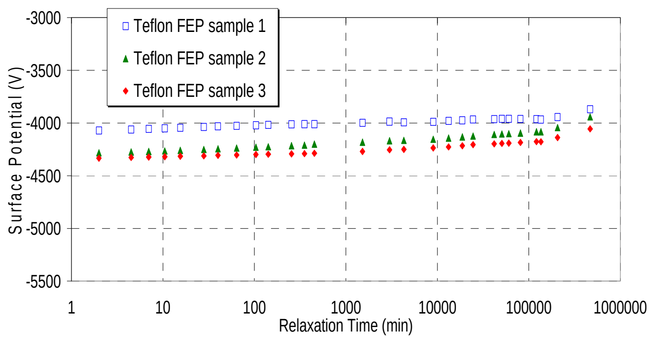

Fig. 6. Evolution of maximum potential on Teflon FEP samples during 325 days relaxation

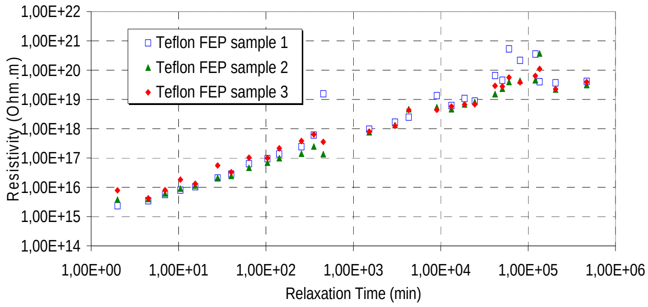

Fig. 7. Evolution of the apparent intrinsic bulk resistivity assessed on Teflon FEP samples

Fig. 6 presents the evolution of the maximum surface potential as a function of the relaxation time. We can notice that the relaxation kinetics gradually decreases to reach a constant level after 7800 hours (325 days) of relaxation. After 13 days of relaxation, the potential drop is then constant equal to around 2 V per 24 days, which would lead to intrinsic bulk conductivity equal to around 7.10^{-20} $\Omega^{-1}.m^{-1}$. The apparent conductivity is assessed using the potential decay method. The model of a capacitor relaxing its charge through a resistance is applied. The time constant of the discharge (RC) is determined and ultimately the conductivity:

$$\sigma=\varepsilon.dV_s/dt/V_s \qquad (1)$$

for which σ is the bulk electric conductivity, ε the relative permittivity of the material, and V_s is the surface potential of the material.

Fig. 7 presents the evolution of the assessed bulk resistivity as a function of the relaxation time. We can notice on this figure a first increase of this apparent resistivity due to the fact that other processes (polarization, electron emission and radiation induced surface conductivity) steers the potential drop. After 13 days of relaxation, the apparent resistivity reaches a steady state value corresponding to the real intrinsic apparent conductivity of Teflon® FEP. We have demonstrated as well that focusing the electron beam used for charging at the center of the sample allows annealing the effect of radiation induced surface conduction [3], which is proved by a decrease in potential drop kinetics during the first hours of relaxation (in comparison with a sample irradiated on its overall surface).

C. use of spider for ageing relaxation processes

The SPIDER facility can also be used to study ageing and ionization effect on charging properties of space materials and to discriminate these two mechanisms. A 127 µm thick Teflon® FEP sample has been submitted to successive irradiations under multi-energetic electron beam (20keV, 250pA/cm2 +400keV, 50pA/cm2). The dead time between both irradiation steps has been adjusted: we have applied three

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different dead times. Fig. 8 presents the evolution of surface potential measured on Teflon® FEP during the first irradiation step and the second irradiation step performed after 3 hours, 1 week or 1 month following the first irradiation. The objective of this study was to analyse the long-term effect of ionisation or ageing on the electric properties of polymers. For 1week and 1 month relaxation, the irradiated sample has been transfer from SIRENE to SPIDER between both irradiation steps. We can first notice that the charging potential is similar for all irradiations, which tends to suggest that ageing due to structure evolution is not significant at this dose level (up to 10^3 Gy). Ionisation effect may prevail over ageing one. We can however notice dramatic evolution of the charging profile with the relaxation time. For a relaxation time equal to 3 hours, the surface potential tends to reach quite quickly a quasi-equilibrium state. This has been ascribed to the fact the concentration of free holes kept up a high level due to the high trapping time. At the same time, holes generation in the Valence band due to ionisation effect by the 400 keV electron beam, is quickly compensated by holes trapping since holes density in the Valence band is high (which increases then trapping probability). The charging profile is drastically different for 1 week and 1 month irradiation. For high relaxation time, density of free holes is low but trapped electrons (due to the first ionisation step) act as recombination centres which tend to reduce holes generation in the valence band and RIC.

Ionisation and its relaxation effect should therefore be taken into account in the analysis of the experimental results and especially for devising the experimental protocol for parameters extraction. The SPIDER facility enables therefore to study long-term ionisation and ageing effect through studies for which long relaxations are involved.

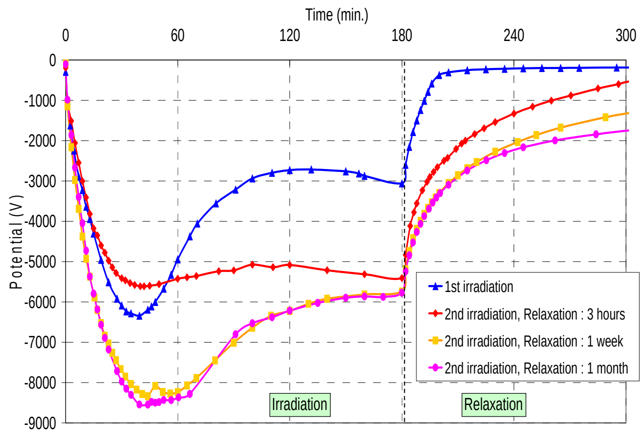

Fig. 8. Evolution of charging surface potential on FEP under successive irradiations with (20 keV, 250 pA.cm^{-2} + 400 keV, 50 pA.cm^{-2}) and variation of relaxation duration between both irradiations : 3 hours, 1 week or 1 month

V. The DEESSE and CELESTE irradiation facilities

Two facilities (DEESSE and CELESTE) are available at ONERA and are intended to study the electron emission proprieties of solids under electron and photon irradiation. These facilities are mainly used for applications that regards electrical charging [4], Hall Thruster technology [5] and multipactor effect [6]. The multipactor is an undesirable phenomenon, occurring on some sensitive RF compounds of satellites. DEESSE as well as CELESTE are equipped with electrons guns covering the energy range of 1 eV to 22 keV, an ion gun (50 eV to 5 kev), a VUV photon source and a high resolution hemispherical electron energy analyzer. The facilities are equipped with Faraday cups, Kelvin probe and an electron collector. The working pressure is in the 10^{-9} -10^{-10} mbar range. The incidence angle of the electrons, photons or ions could be adjusted from the normal to the grazing incidence. The sample holder temperature can be controlled from room temperature to 600°C. DEESSE is especially designed to allow the transfer under High vacuum conditions (10^{-6} mbar) of samples from or to other ONERA facilities (SIRENE, GEODUR and SPIDER). The measurements that are ordinarily performed in DEESSE or CELESTE are the total electron emission yield, the electron backscattering yield, the electron emission energy distribution, and angular distribution of the emitted electrons.

Typical results are shown in Fig. 10. A short electrons pulses (typically few µs) are used to measure the total electron emission yield shown in Fig. 12a [7]. The measurement of the energy distribution (Fig. 12b) brings a lot of useful details, as the mean electron emission energy and the relative proportions of elastic backscattered electrons and secondary electrons. These details are of high importance in multipactor and Hall thruster modelling.

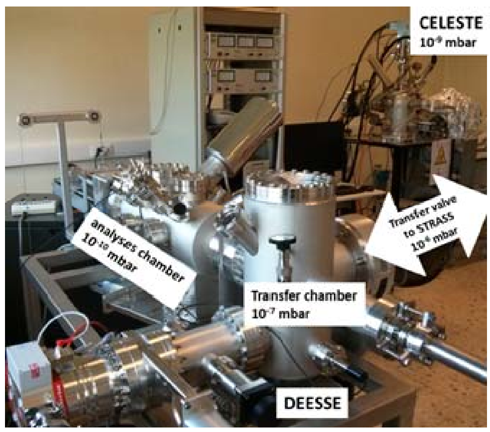

Fig. 9. Picture of the two Onera facilities dedicated to the electron emission studies.

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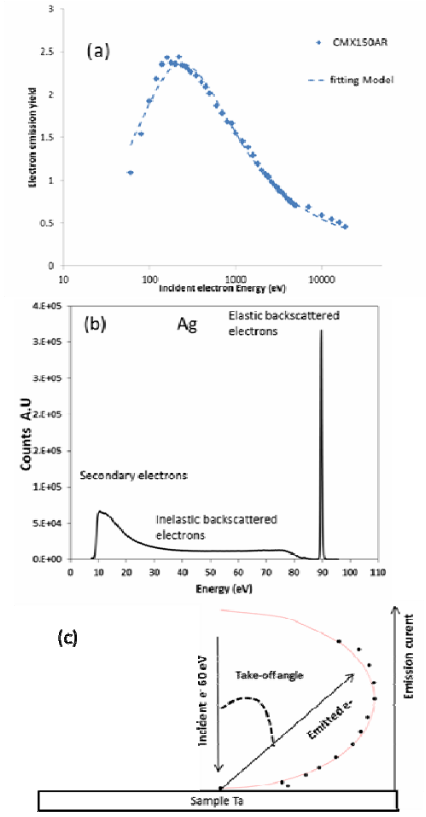

Fig. 10. Typical experimental results obtained in DEESSE and CELESTE. (a) Total electron emission yield measured on CMX150 AR. (b) Energy distribution of electron emitted from silver sample irradiated at 90 eV and biased at -9 V.(b) angular distribution of emitted electrons from tantalum sample irradiated at 60 eV at normal incidence.

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REFERENCES

[1] T. Paulmier, B. Dirassen, M. Belhaj, Denis Payan, N. Balcon, "Relaxation Electron Surface Emission from Space Used Polymers", IEEE Transactions On Plasma Science, 41, 12, 2013, pp 3416-3421

[2] L. Levy, T. Paulmier, B. Dirassen, C. Inguimbert, and M. Van Eesbeek, "Aging and Prompt Effects on Space Material Properties", IEEE Trans. Plasma Sci.", 36, 5 (2008) 228-2237

[3] R. Hanna, T. Paulmier, M. Belhaj, B. Dirassen, D. Payan, N. Balcon, “Characterization of intrinsic and induced lateral conduction in space dielectric materials”, J. Appl. Phys., 115, 063707 (2014)

[4] M. Belhaj, T. Tondu, V. Inguimbert: Effect of the incident electron fluence on the electron emission yield of polycrystalline Al 2O 3. Applied Surface Science 01/2011; 257(10):4593-4596.

[5] T Tondu, M Belhaj, V Inguimbert: Electron-emission yield under electron impact of ceramics used as channel materials in Hall-effect thrusters. Journal of Applied Physics 01/2011; 110(9):093301-093301.

[6] V.E. Semenov, M. Belhaj, E. Rakova, J. Puech, M. Lisak, J. Rasch, E. Laroche: Preliminary results on the Multipactor effect prediction in RF components with ferrites. 14th International Vacuum Electronics Conference - PARIS – 2013.

[7] T. Paulmier, M. Belhaj, B. Dirassen: Charging Properties of New Materials - FR1 - Final Report RF 7/19285 DESP 2013.