CHARGING BEHAVIOR AND AGEING EFFECT ON SPACE USED CERAMICS

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ABSTRACT

This study allowed demonstrating that thermal and surface treatments as well as alumina coating lead to significant decrease of the charging level on BN ceramics. Dedicated experimental in-situ techniques revealed the initiation of surface electrostatic discharges that tend to reduce the charging levels through neutralization processes but also age the alumina coating after a significant dose level. We can indeed observe a degradation of the electric properties of the ceramic system under irradiation with a shift toward higher charging kinetics. Degradation of the alumina coating as well as contamination process have been identified and confirmed through post-irradiation analysis (Raman, XPS). Based on this thorough experimental study, a physics model has been developed describing charging, discharging and ageing mechanisms occurring on rough ceramics surfaces under critical electron flux.

1. INTRODUCTION

Technical dielectric materials and ceramics are used in different high technology industrial areas [1, 2] and especially for spacecraft applications. On satellite, these materials are submitted to extreme conditions due to space environment. To cope with this issue, these ceramic insulators must have remarkable electrical and thermal properties. Boron Nitride (BN) and Aluminum Oxide (Al_2O_3) are especially used because they combine good electrical insulation and high thermal conductivity. However, in space environment, BN and Al_2O_3 used inside spacecraft application are exposed to critical radiative constraints. Under these conditions, these insulators are thus irradiated by electrons with high energies and flux. The charged particles are trapped in ceramics leading to high electric field and to the possible occurrence of electrostatic discharges. These phenomena may induce degradations and destructions of materials and embedded systems.

It is therefore of high importance to understand the physical mechanisms steering the charging behavior and the ageing processes of ceramics materials under high energy irradiation. The dielectrics have been irradiated and characterized at ONERA Toulouse (DESP) in an irradiation chamber called CEDRE (Chambre d'Etude De Revêtement Electrisés). A parametric study was performed to assess influence of incident energy and flux, temperature, coating, annealing and ionizing dose, on charging and relaxation kinetics of these ceramics. Radiative ageing of electric and charging properties has been studied as well.

2. EXPERIMENTAL SET-UP

The CEDRE facility is one of the many high vacuum electron irradiation test chambers localised at ONERA Toulouse. CEDRE means in French: "Chambre d'Etude De Revêtements Electrisés". In fact, this facility has the capability to be flexible and easily adjustable to characterize dielectrics surface potential with several instruments. It is therefore possible to assess the charging and discharging of the spacecraft materials in space representative conditions. The vacuum level during tests is approximately equal to 5.10^{-7} hPa. Chamber vacuum pumping is performed thanks to a dry pump group in order to limit contamination. An electron source is available to irradiate samples controlling the flux and the energy of the electron beam. Another source such as a UV lamp can be easily installed if necessary. A ceramic heating element sustains the sample holder which can be whether floating or grounded. This experimental assembly developed at ONERA allows controlling the temperature in the range between 20°C and 400°C. A rotating cube allows the installation of this assembly. The electron flux can be quickly measured before surface potential characterizations. A Kelvin Probe is also installed in CEDRE to assess the charging and relaxation kinetics. A CCD camera is used for visual check of observable phenomena during tests. The used electron gun (Kimball, Model EGPS-4212B) allows having reproducible irradiation conditions. It provides a mono-energetic electron beam (energy spread ≈ 0.5 eV) with energy range between 1 keV and 20 keV and energy stability of ±0.01 % during one hour. Generally, the used beam is continuous and its flux ranges between 0.05 $nA.cm^{-2}$ and 100 $nA.cm^{-2}$.

Thus, the experimental method usually applied in CEDRE to study the charges transport in dielectrics is to implant incident electrons with flux and energy that are calibrated in order to measure their surface potential with a Kelvin Probe (KP). The continuous beam is focused ($\Phi < 3$ mm) or defocused ($3 \text{ mm} < \Phi < 50$ mm, depending of the energy) in order to study the different conduction mechanisms (surface and bulk conductivity). The surface of the irradiated samples is often greater than 20mm x 20mm, because of spatial resolution of the KP. The heating element can be used to assess the temperature influence on the conductivity. It can also allow the bake-out of samples during the pumping phase before testing or to neutralize it after testing. Instruments such as the Kelvin Probe or the Repulsive Electron Potential Analyser (REPA) [3] and data processing are automatically controlled by computer using GPIB interfacing and a DAQ card under LabVIEW control.

The studied materials are industrial samples (~1 x 1 x 10 mm) of pyrolytic hexagonal boron nitride (BN), alumina-coated boron nitride (BN/Al_2O_3), and annealed alumina-coated boron nitride (an-BN/Al_2O_3).

3. EXPERIMENTAL PROTOCOL

The experimental protocol was divided into two main steps :

- Step 1 : irradiation test of pristine materials

The three different materials (described in section 2) have been irradiated with electron beam at low fluxes (few $nA.cm^{-2}$) to study their charging behaviour as a function of the incident energy (from 5 to 20 keV). Evolution of the surface potential along the irradiation and relaxation phases has been monitored with the KP and REPA method.

- Step 2 : radiative ageing of the materials

To study the evolution of their electric properties with the received radiation dose, the materials have been irradiated with high electron fluxes so as to apply an acceleration factor and observe any alteration of the charging behaviour. Evolution of surface potential along the radiative ageing process has been monitored with the KP method.

4. EXPERIMENTAL RESULTS

4.1. Electrical behaviour of pristine materials under irradiation

Fig. 1 presents the evolution of the equilibrium potential on the three different materials as a function of the applied incident energy. The dotted line presents the limit in energy below which the electrons are implanted in the top alumina layer. We can first notice a significant increase of the potential with energy : as electrons go deeper in the material, they tend to get trapped in deeper defects in the BN (and it has been demonstrated that alumina present a higher conductivity than BN). The most interesting point lies on the differences from one material to the other : we can indeed observe that the presence of Al_2O_3 coating tends to reduce the absolute charging potential. Annealing of the sample induces a further decrease of this potential. Dedicated experimental protocols demonstrated that these characteristics could not be ascribed to the sole effect of conductivity and secondary electron emission yield variation.

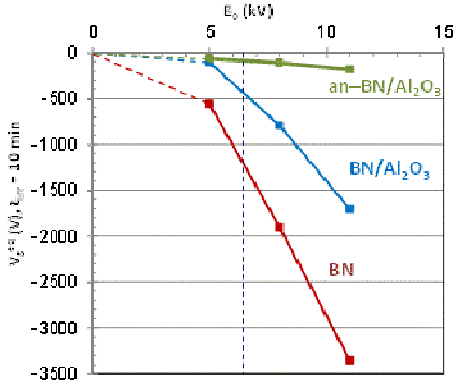

Figure 1. Evolution of the equilibrium surface potential (measured with the KP method) of BN, BN/Al_2O_3 and an–BN/ Al_2O_3, as a function of the incident energy after 10 min under electron irradiation (10 $nA\cdot cm^{-2}$, room temperature, defocused beam)[4]

Additional measurements performed with the REPA method on the annealed alumina-coated boron nitride (Fig. 2) demonstrated that this material is submitted to strong variations on its surface potential that have been ascribed to the occurrence of partial discharges at the surface of the material. These discharges are due to the inhomogeneity of the alumina coating at the surface of the sample that generates potential differences between alumina and boron nitride parts (due to differences in secondary electron emission yield and electron implantation).

These discharges further material desorption and the generation of plasma that tends to partially neutralise the surface and therefore reduce the absolute surface potential.

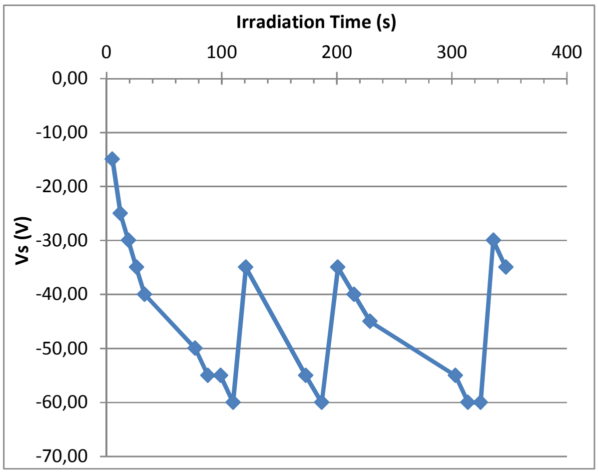

Figure 2. Evolution of surface potential measured with the REPA method on an–BN/Al_2O_3 irradiated at 10 keV and 1 nA.cm^{-2}

4.2. Electrical ageing of coated BN under high radiation dose

Fig. 3 presents the evolution of the surface potential along the radiative ageing process of the annealed alumina-coated boron nitride sample irradiated at high fluxes. We can observe two main phases : a first phase during which the surface potential gradually rises up and a second phase characterised by a steep rise of the absolute surface potential with the increasing radiation dose. Analysis of the irradiated material by Raman spectroscopy (Fig. 4) and XPS revealed a noticeable degradation of the alumina coating and the formation of a carbon contamination deposit at the surface of the sample. From these measurements and observations, the first stage has been assigned to the influence of carbon deposit that reduces the amount of electrostatic surface discharges and then induces less neutralisation and an increase of the absolute surface potential. During the second stage, contamination competes with alumina reduction which tends to reduce the electron emission yield of the sample and therefore increase the absolute surface potential.

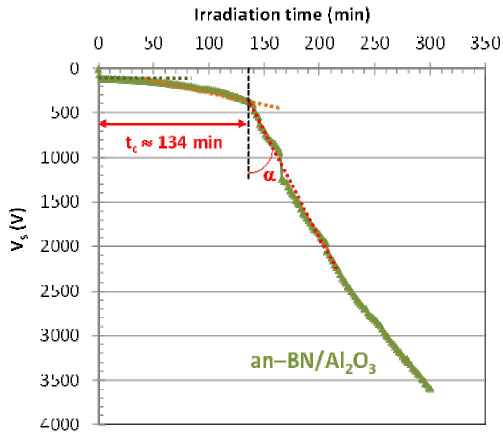

Figure 3. Temporal evolution of the surface potential of an–BN/Al_2O_3 under critical irradiation (20 keV, 750 nA·cm^{-2}, T°C room, focused beam)[4]

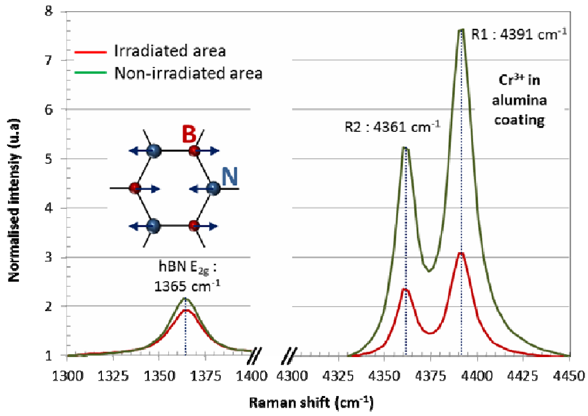

Figure 4. Raman spectra measured at 532 nm incident wavelength on pristine and aged an–BN/Al_2O_3

5. CONCLUSION

The irradiation tests performed on BN, BN/Al_2O_3 and an–BN/Al_2O_3 have demonstrated that the alumina coating and especially the annealing thermal treatment under vacuum result in a reduced surface potential of the BN substrate. The main physical mechanism responsible of low surface potential observed in the alumina coated samples is due to partial surface discharges that regulate the surface potential of an-BN/Al_2O_3 sample through neutralisation process.
An important electrical aging test of an–BN/ Al_2O_3 was performed. The carbon contamination as well as alumina reduction have been brought into evidence. Both processes induce a decrease of secondary electron emission yield of the heterogeneous surface composed of alumina and boron nitride. In conclusion, several steps of the elaboration process, such as coating and annealing treatment, can be applied to limit the charging of an insulator used on satellites. However, these

treatments and the elaboration parameters have to be optimized in order to increase the life-time of these technical ceramics irradiated under critical conditions.

6. ACKNOWLEDGMENTS

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7. REFERENCES

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