Summer 6-2015

Electrical Properties of Annealed and Coated Boron Nitride under Electron Beam Irradiation

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THE FRENCH AEROSPACE LAB

UtahStateUniversity MATERIALS PHYSICS GROUP

retour sur innovation

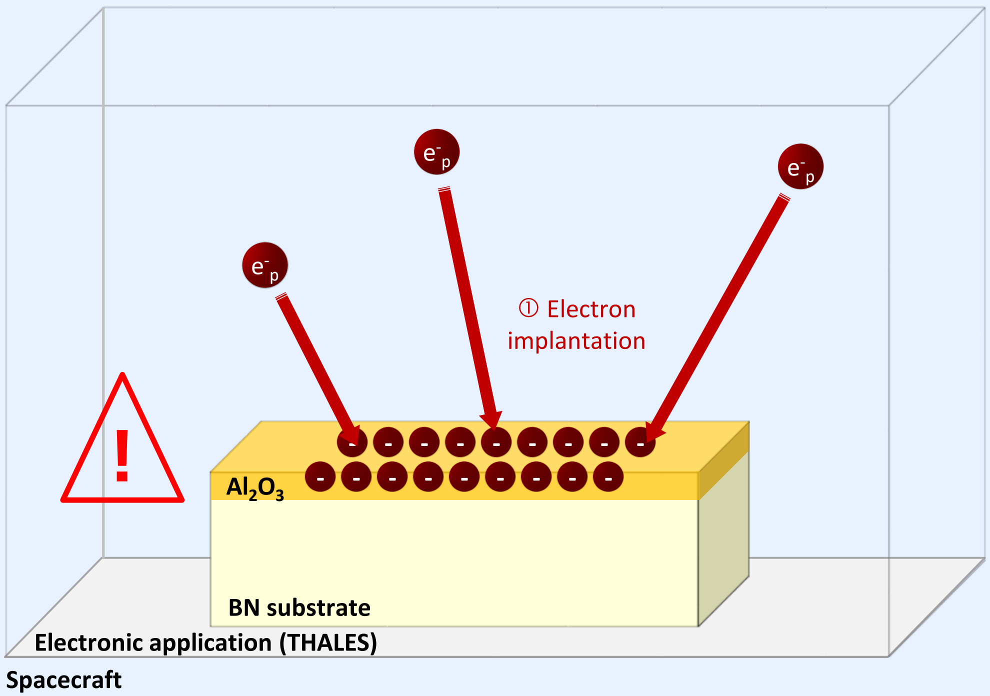
13th International Symposium on Materials in the Space Environment (ISMSE-13)

22 - 26 June 2015 – Pau, France

Cifre

THALES

Context & motivation



Context

Insulator materials such as **boron nitride (BN)** are exposed to electron radiation during the operation of THALES's electronic application

Issue

- Charging of BN under irradiation
  ⇨ The application efficiency decreases
- **Alumina coating** on BN to limit charging
  ⇨ Properties degrade over time

Motivation

- Identify the physical mechanisms of charging and degradation in materials
- Optimise the electrical properties of ceramics to limit these phenomena

Outlines

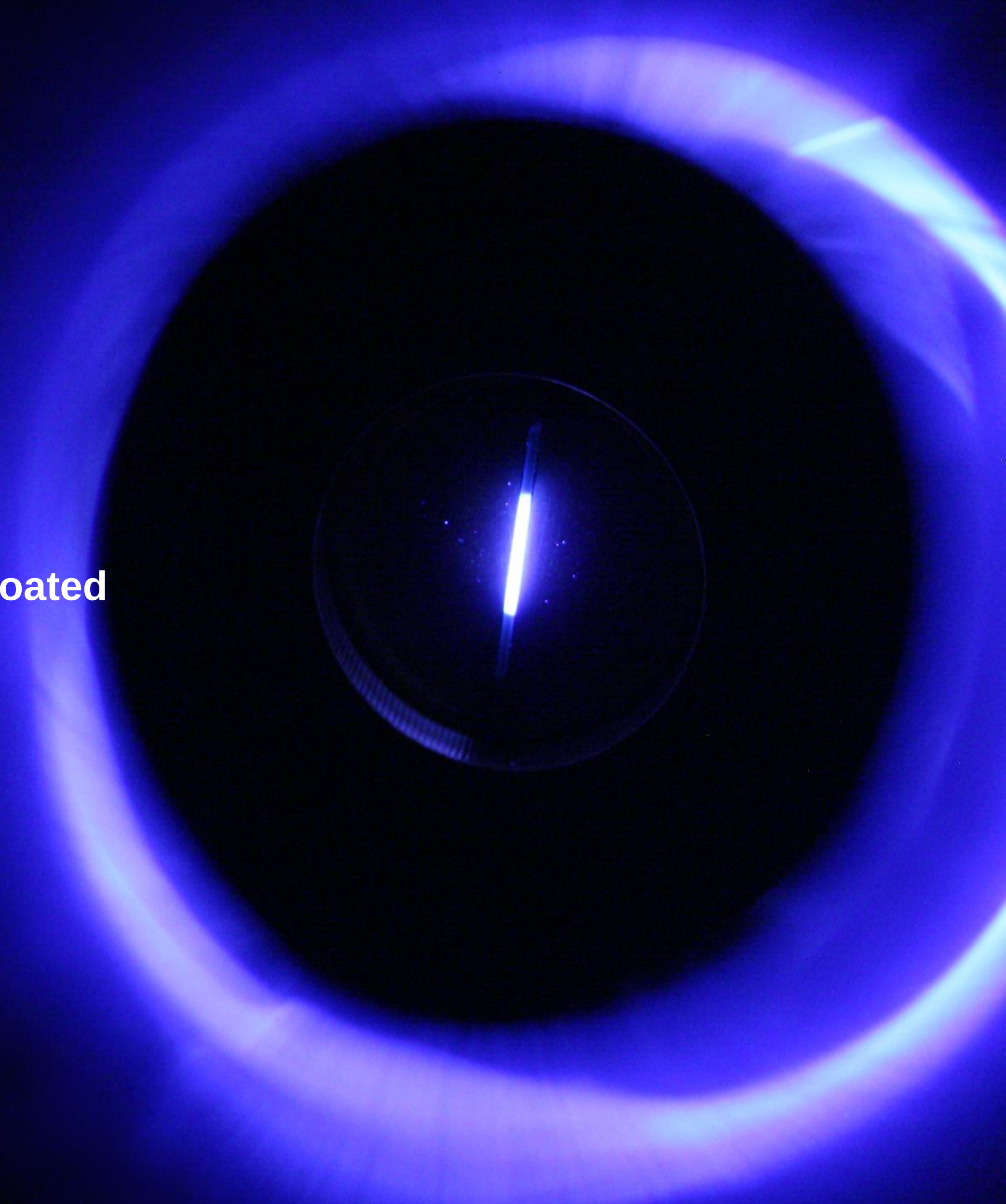
➊ **Scientific approach**

➋ **Experimentation**

➌ **Charging study of annealed and coated BN**

➍ **Aging study of annealed BN/Al_2O_3**

➎ **Conclusion & outlooks**



2014 - MPG (USU) / ONERA
Luminescence of BN under electron irradiation

Charging study of new industrial samples

Parametric study under electron beam :

- Energy
- Flux
- Temperature
- BN substrate
- Alumina coating
- Annealing treatment

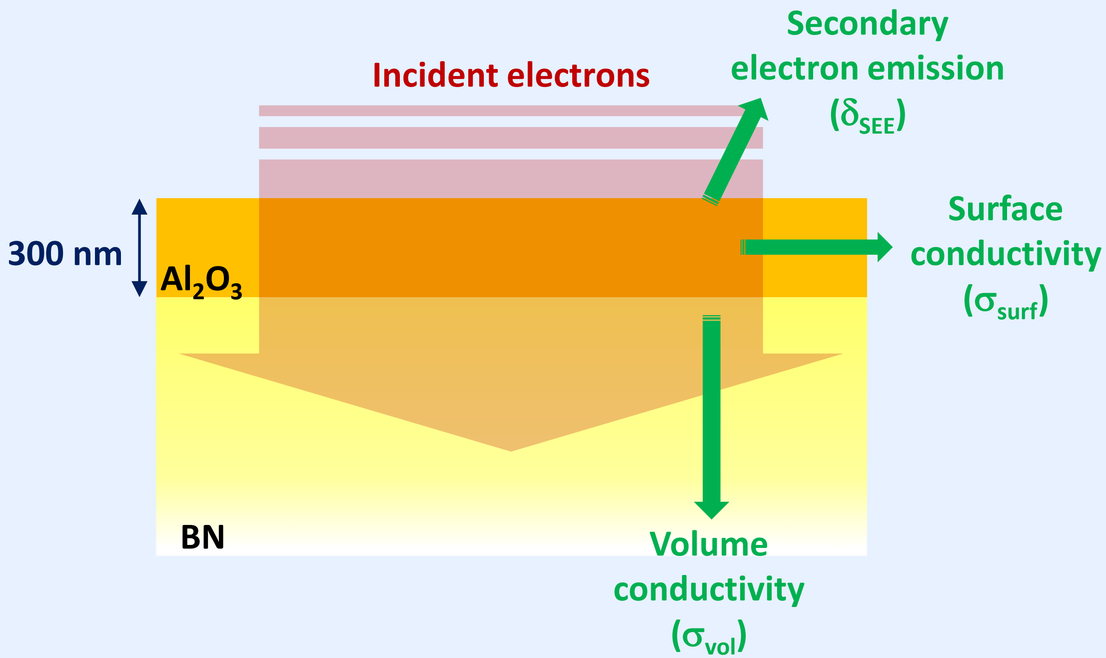
Aging study of the annealed BN/Al_2O_3

Under critical electron irradiation :

- High flux
- Long time

⇨ High dose

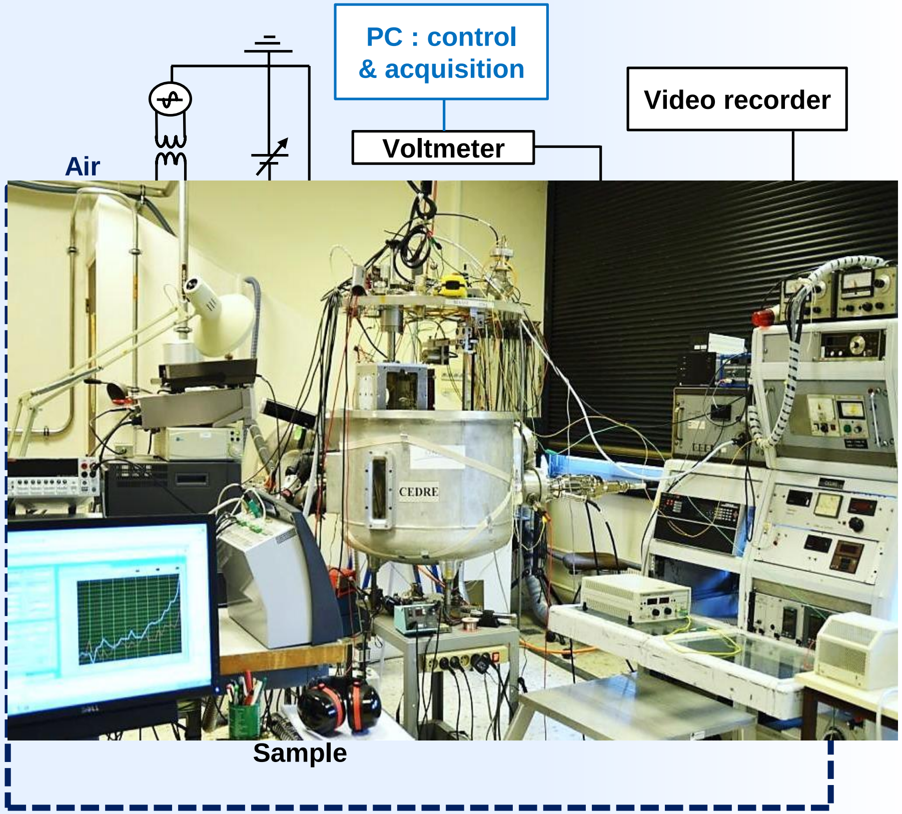
Dark conductivity study of the industrial ceramics



CEDRE facility and method

Study the charging and relaxation kinetics and the aging in representative conditions

- Secondary vacuum (P ~ 5 10^{-7} hPa)
- 20 °C < T < 400 °C
- Electron gun : 1 keV < E_i < 20 keV
  50 pA.cm^{-2} < J_i < 100 µA.cm^{-2}



Surface potential (V_s) measurement with an electrostatic probe (Kelvin Probe – KP)

CVC chamber and method

Study the dark conductivity in representative conditions

- Secondary vacuum (P ~ 1 10^{-5} hPa)
- -150 °C < T < 200 °C

Constant Voltage Conductivity (CVC) method[1] –

70 V < V < 470 V

I(t) > 10^{-16} A

$$\sigma^{CVC}(t) = \frac{I(t) \times d}{S \times V}$$

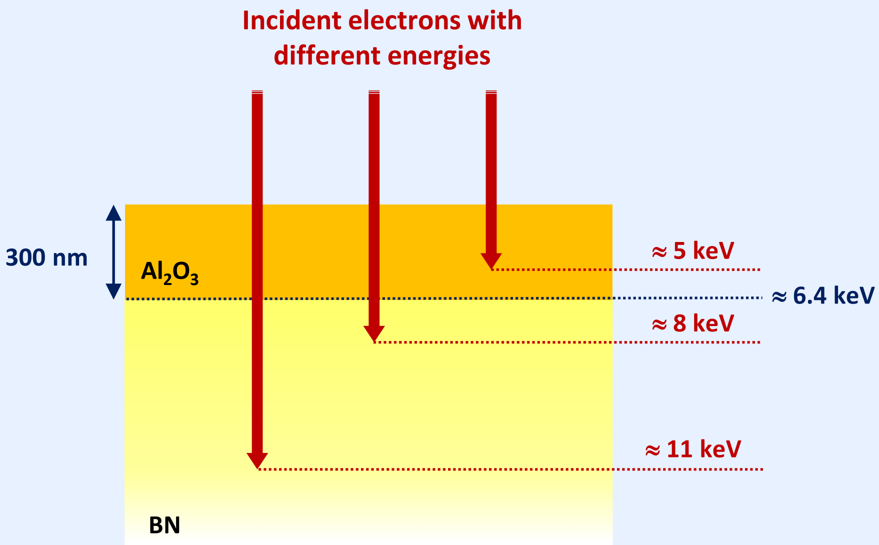
[1] J. Brunson, « Hopping Conductivity and Charge Transport in Low Density Polyethylene », PhD Thesis, Utah State University, Logan, UT, USA, 2010.

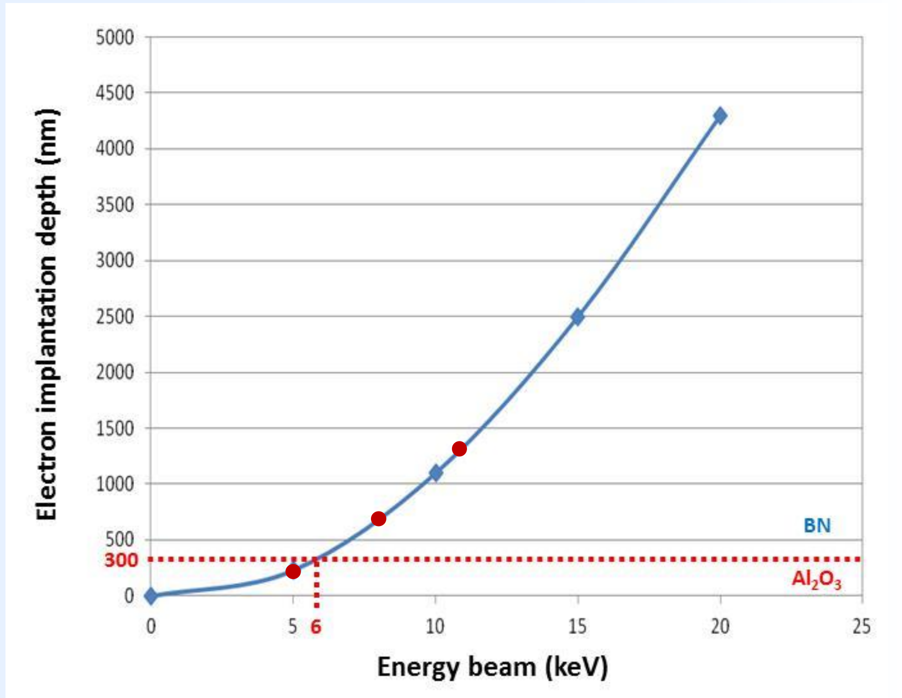
Irradiation phase

Aim: Identify the influence of coating and thermal treatments on the surface potential regulation of BN

3 configurations of unused industrial ceramics:

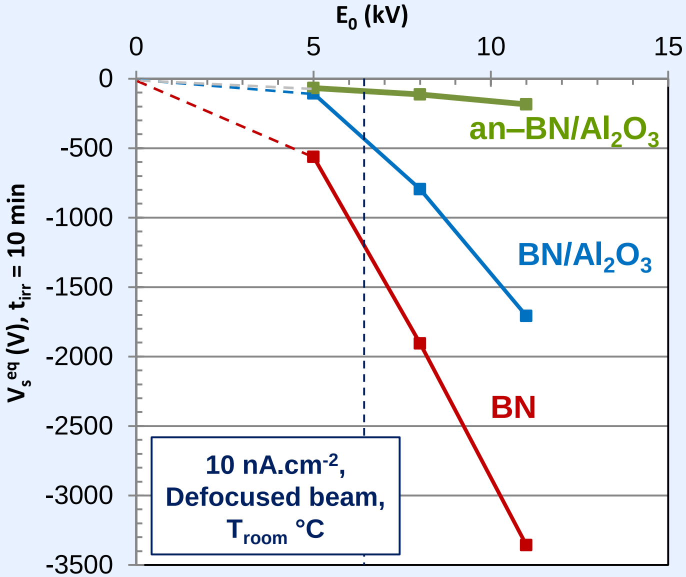
- Boron nitride substrate - **BN**
- BN with alumina coating - **BN/Al_2O_3**
- Annealed BN/Al_2O_3 - **an–BN/Al_2O_3**

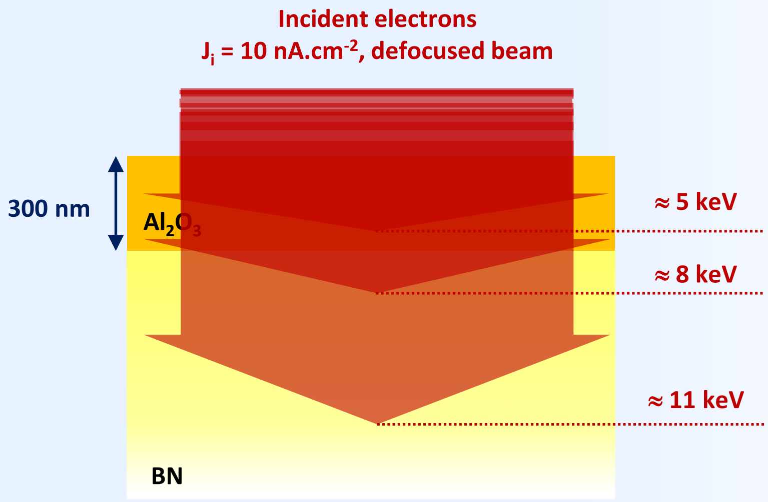




The energy for a penetration length of 300 nm in Al_2O_3 is ≈ 6.4 keV (Monte-Carlo simulations)

Irradiation phase





E_i = 5 keV (non-penetrating beam for the coating) : No charging in coated BN samples

$E_i \geq$ 8 keV : Critical and low charging in BN and coated BN, respectively but the V_s is limited in an-BN/Al_2O_3

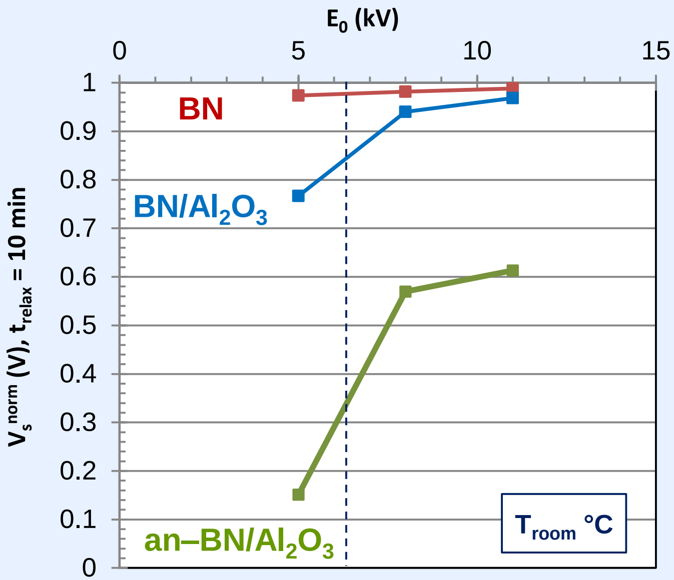
⇨ The alumina coating decreases the surface potential through its higher δ_{SEE}[2] than that of BN[3][4]

⇨ The annealing treatment under vacuum limits the V_s :

- Defects generation has been determined which may increase the conduction mechanisms
- Hypothesis: decrease of the critical threshold of surface discharges

[2] P. H. Dawson, « Secondary Electron Emission Yields of some Ceramics », J. Appl. Phys., vol. 37, no 9, p. 3644, 1966
[3] L. G. Sherstnyov et Al., Report of the Moscow Energetic Institute, (87) N° 68091701, 1969.
[4] B. V. Prokofiev, « Pyrolytical Boron Nitride as a Window Material for High Power Microwave Electron Devices », IEEE, 2010

Relaxation phase



$$V^{norm}_{s(10\,min)} = \left(\frac{V_{s(10)}}{V_{s(0)}}\right) \equiv \left(\frac{Q_{s(10)}}{Q_{s(0)}}\right)$$

After E_i = 5 keV : Charge relaxation from alumina coating and especially in the annealed one

After E_i ≥ 8 keV : Low relaxation rates even though some charges are evacuated in the annealed sample

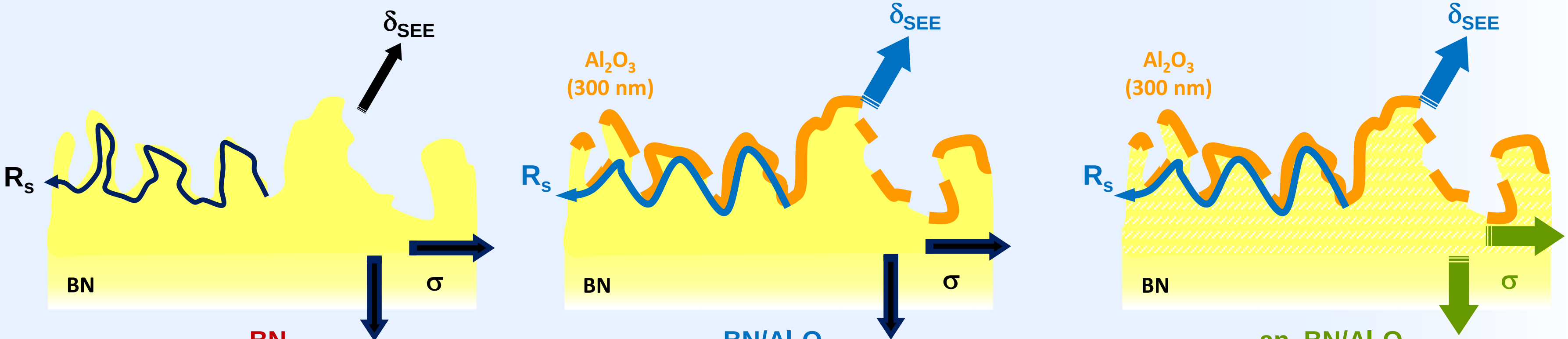
Intrinsic conductivities of industrial samples determined through CVC method

BN	BN/Al_2O_3	an–BN/Al_2O_3
$(0,5 \pm 0,1).10^{-15}$ S.m^{-1}	$(0,2 \pm 0,1).10^{-15}$ S.m^{-1}	$(8 \pm 1).10^{-15}$ S.m^{-1}

⇨ The annealing treatment increases the total conductivity (by 25 – 30)

Discussion

- **Influence of surface state, coating and thermal treatment :**



BN
Roughness ≈ 0.8 µm ⇨ high R_s
Low δ_{SEE}
Low σ

⇨ High critical V_s

BN/Al_2O_3
Roughness ≈ 0.6 µm ⇨ lower R_s
Higher δ_{SEE}
Low σ

⇨ Lower critical V_s

an–BN/Al_2O_3
Roughness ≈ 0.6 µm ⇨ lower R_s
Higher δ_{SEE}
Higher σ

⇨ Lower V_s

But σ and δ are too low to limit V_s under high energy and flux

- **Charging characterisation of an–BN/Al_2O_3 under continuous irradiation with the REPA method[5] :**

Partial discharge phenomenon has been identified (unmeasurable with the KP method)

Does this annealing treatment allow limiting the charging potential after a long time exposure ?

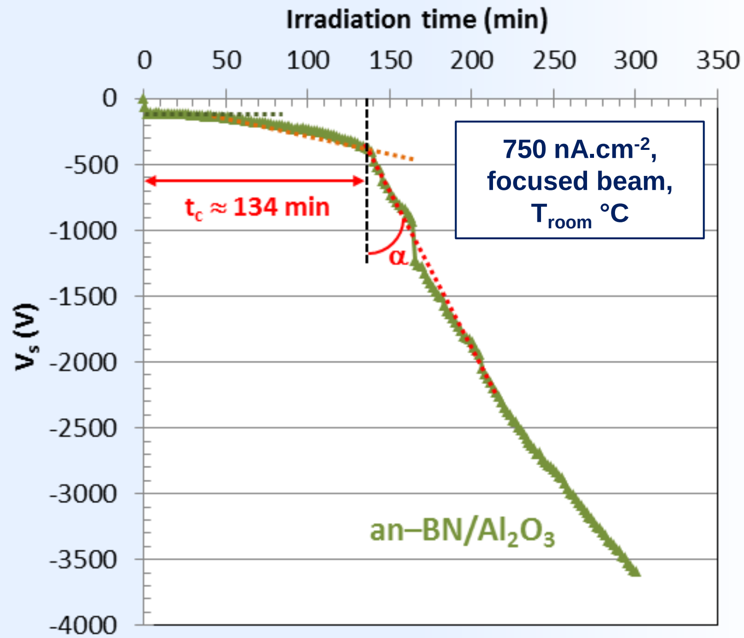
[5] K. Guerch et Al., « Characterisation of charging kinetics of dielectrics under continuous electron irradiation through real time electron emission collecting method », Nucl. Instrum. Methods Phys. Res. Sect. B Beam Interact. Mater. At., vol. 349, 2015

Electrical aging under high electron flux

Aim: Accelerate the material degradation to reproduce the critical exposure in spacecraft over time

3 regimes :

- Temporary equilibrium (0 < t < 50 min)
- Transitory (50 min < t < critical time t_c = 134 min)
- Critical (t > t_c)



Critical dose corresponds to the sudden electrical aging beyond t_c :

- $D_c(Al_2O_3) \approx 7.4\ 10^7$ Gy
- $D_c(BN) \approx 1.3\ 10^8$ Gy

$$D_{c(x)} = \frac{J_i}{q_e} \times \frac{t_c}{\rho_m} \times \frac{E_i}{R(E_i)}$$

Flux and Energy affect the electrical aging :

$T_c \propto 1/J_i$

$\alpha \propto 1/E_i$

Electrical aging irreversible and σ tends towards that of BN :

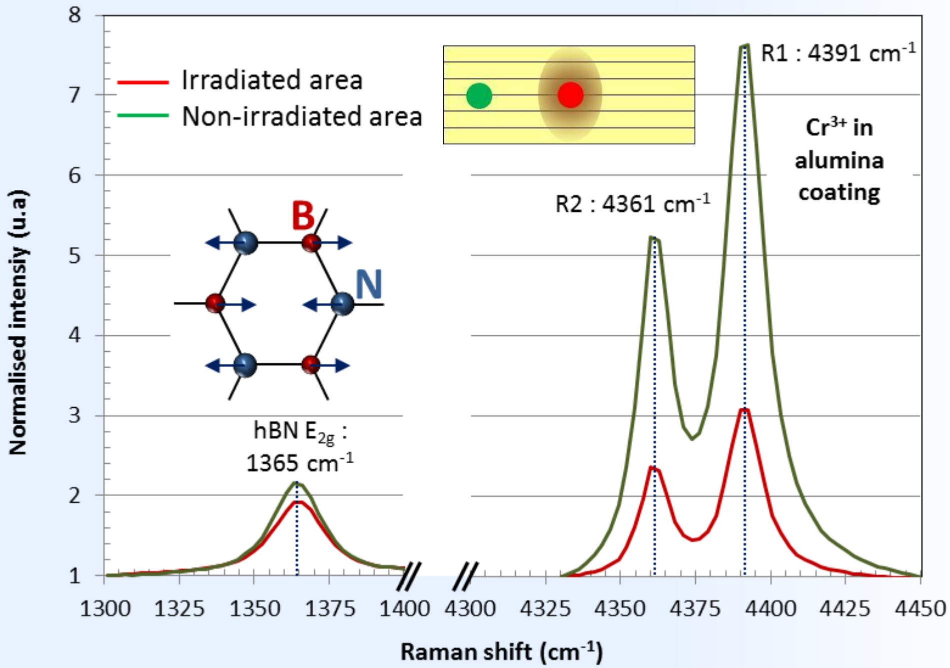
an–BN/Al_2O_3	$(8 \pm 1).10^{-15}$ S.m^{-1}
an–BN/Al_2O_3 (aged)	< $(2 \pm 3).10^{-15}$ S.m^{-1}

What is the chemical degradation process that leads to this electrical aging ?

Chemical degradation

RAMAN Spectroscopy (CIRIMAT) :

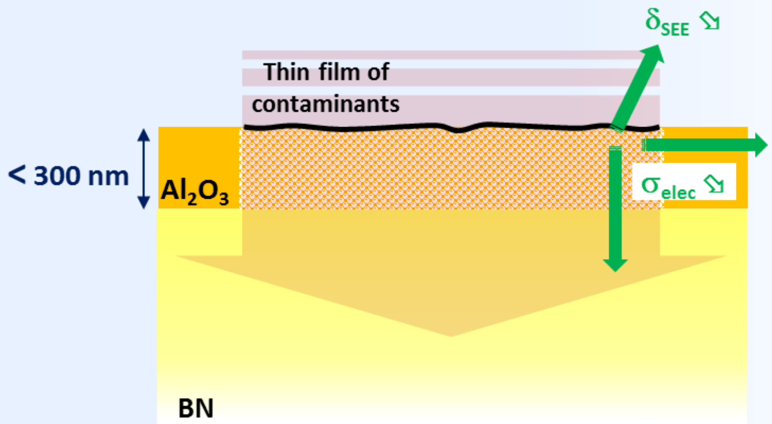
Laser 532 nm, 600 tr/mm



- ~ 1365 cm^{-1} : E_{2g} phonon mode of hBN
- ~ 4361 et 4391 cm^{-1} : Cr^{3+} impurities (R2 & R1) in annealed alumina coating

In the irradiated area (deteriorated):

- Decrease of species concentration in coating and/or decrease of coating thickness
- Effect of a contaminant thin film[6] [7]



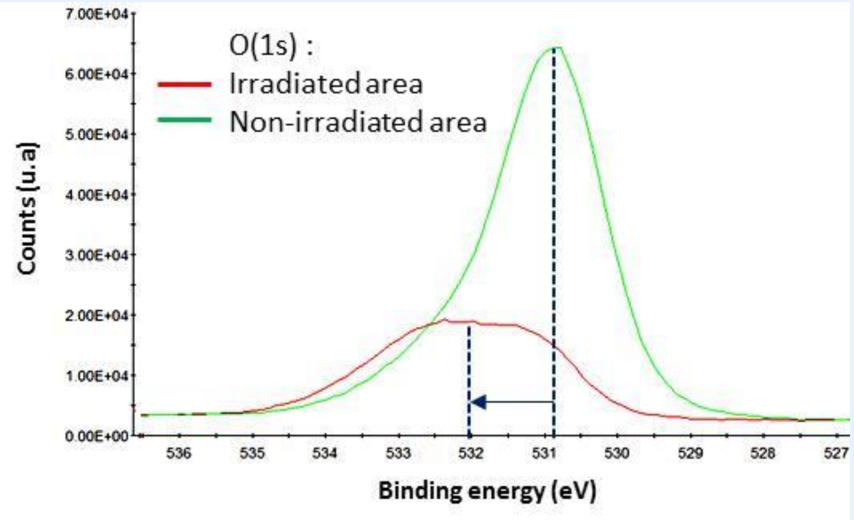
[6] J. E. I. Rau, « The effect of contamination of dielectric target surfaces under electron irradiation », *Appl. Surf. Sci.*, vol. 254, nº 7, p. 2110-2113, 2008.
[7] R. E. Davies and J. R. Dennison, « Evolution of Secondary Electron Emission Characteristics of Spacecraft Surfaces », *J. Spacecr. Rockets*, vol. 34, nº 4, p. 571-574, 1997.

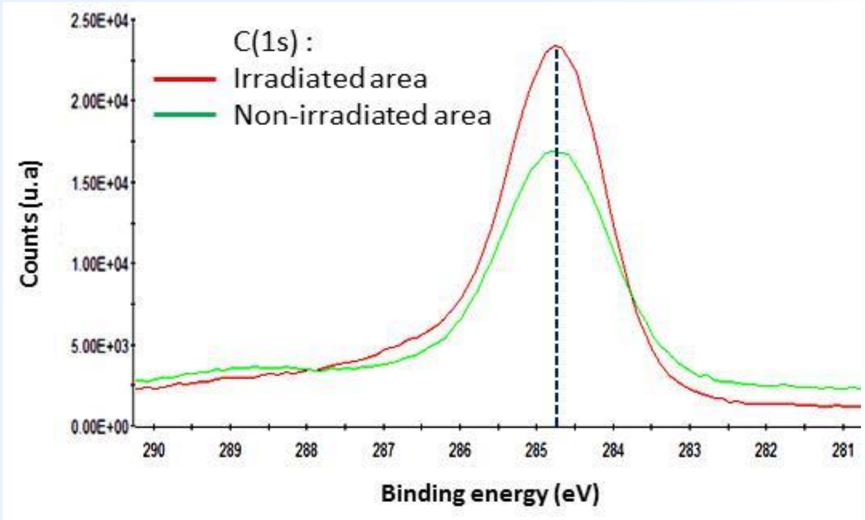
Chemical degradation

XPS (CIRIMAT) :

Monochromatic source

(ray Al Kα - 1486.6 eV), Area$_{analysed}$ = 400 µm





- Decrease of bonds Al(2p) and O(1s)

Signals decreased by 53 % and 70 % respectively

- Chemical shift (1 eV) of the oxygen environment

- More bonds C-C in the irradiated area (signal increased by 41 %)

⇨ **Chemical evolution of insulator especially in annealed Al_2O_3 coating = electrical properties deterioration**

⇨ **Important contamination which is favoured under electron radiation (depending on flux)**

Charging mechanisms

- **Critical charging of BN substrate** for the electronic application (in the beam energy range which is used)
- **The alumina coating decreases the charging** through higher δ_{SEE} and the lower roughness (R_s decreases)
- **The annealing treatment limits V_s** because the electrical conductivity increases (and partial discharges)

Degradation mechanisms

- **Electrical aging** (decrease of δ_{SEE} and σ) of an–BN/Al_2O_3 sample after a critical dose
- **Degradation & contamination processes** especially of the annealed alumina coating

Optimisation of industrial coating process

- High roughness ⇨ Heterogeneous coating **= properties deterioration**
- **Substrate roughness & coating thickness** should be optimised to increase the life time of coated ceramics

Outlooks

- Study the **deterioration evolution** as a function of flux and energy to optimise our degradation model
- Study the electrical aging under electron radiation **as a function of the coating thickness**

Thank you for your attention

Acknowledgment:

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Partial discharges characterisation through the REPA Method