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Electrical properties of annealed and coated boron nitride study under electron beam irradiation

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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

1. Scientific approach

2. Experimentation

3. Charging study of annealed and coated BN

4. Aging study of annealed BN/Al₂O₃

5. Conclusion & outlooks
Scientific approach

Charging study of new industrial samples

- Energy
- Flux
- Temperature

- BN substrate
- Alumina coating
- Annealing treatment

Aging study of the annealed BN/Al₂O₃

- High flux
- Long time

⇒ High dose

Dark conductivity study of the industrial ceramics

- Incident electrons
- Secondary electron emission ($\delta_{SEE}$)
- Surface conductivity ($\sigma_{surf}$)
- Volume conductivity ($\sigma_{vol}$)

BN

Al₂O₃

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

- Secondary vacuum (P \sim 5 \times 10^{-7} \text{ hPa})
- 20 \degree \text{C} < T < 400 \degree \text{C}
- Electron gun: \(1 \text{ keV} < E_i < 20 \text{ keV}\)
  \(50 \text{ pA.cm}^{-2} < J_i < 100 \mu\text{A.cm}^{-2}\)

Surface potential \((V_s)\) measurement with an electrostatic probe (Kelvin Probe – KP)
Study the dark conductivity in representative conditions

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

Constant Voltage Conductivity (CVC) method\(^1\) –

\[70 \text{ V} < V < 470 \text{ V}\]
\[I(t) > 10^{-16} \text{ A}\]

\[
\sigma_{\text{CVC}}(t) = \frac{I(t) \times d}{S \times V}
\]

Charging study of annealed and coated BN

**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/Al₂O₃
- Annealed BN/Al₂O₃ - an–BN/Al₂O₃

The energy for a penetration length of 300 nm in Al₂O₃ is ≈ 6.4 keV (Monte-Carlo simulations)
Charging study of annealed and coated BN

Irradiation phase

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

$E_i \geq 8 \text{ keV}$: Critical and low charging in BN and coated BN, respectively but the $V_s$ is limited in an-BN/Al$_2$O$_3$

- The alumina coating decreases the surface potential through its higher $\delta_{\text{SEE}}$ 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

Charging study of annealed and coated BN

**Relaxation phase**

\[ V_{\text{norm}}^{s(10 \text{ min})} = \left( \frac{V_{s(10)}}{V_{s(0)}} \right) \equiv \left( \frac{Q_{s(10)}}{Q_{s(0)}} \right) \]

After \( E_i = 5 \text{ keV} \): Charge relaxation from alumina coating and especially in the annealed one

After \( E_i \geq 8 \text{ keV} \): Low relaxation rates even though some charges are evacuated in the annealed sample

Intrinsic conductivities of industrial samples determined through CVC method

<table>
<thead>
<tr>
<th></th>
<th>BN</th>
<th>BN/Al(_2)O(_3)</th>
<th>an–BN/Al(_2)O(_3)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>((0,5 \pm 0,1).10^{-15} \text{ S.m}^{-1})</td>
<td>((0,2 \pm 0,1).10^{-15} \text{ S.m}^{-1})</td>
<td>((8 \pm 1).10^{-15} \text{ S.m}^{-1})</td>
</tr>
</tbody>
</table>

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

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

  ![Diagram showing roughness and critical voltage](image)

  - BN
    - Roughness ≈ 0.8 μm ⇒ high $R_s$
    - Low $\delta_{\text{SEE}}$
    - Low $\sigma$
    - $\Rightarrow$ High critical $V_s$
  
  - BN/Al$_2$O$_3$
    - Roughness ≈ 0.6 μm ⇒ lower $R_s$
    - Higher $\delta_{\text{SEE}}$
    - Low $\sigma$
    - $\Rightarrow$ Lower critical $V_s$
  
  - an–BN/Al$_2$O$_3$
    - Roughness ≈ 0.6 μm ⇒ lower $R_s$
    - Higher $\delta_{\text{SEE}}$
    - Higher $\sigma$
    - $\Rightarrow$ Lower $V_s$
  
    But $\sigma$ and $\delta$ are too low to limit $V_s$ under high energy and flux

- **Charging characterisation of an–BN/Al$_2$O$_3$ under continuous irradiation with the REPA method**[^5]:

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

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Aging study of an–BN/Al₂O₃

**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 $t_c$:
- $D_c(\text{Al}_2\text{O}_3) \approx 7.4 \times 10^7$ Gy
- $D_c(\text{BN}) \approx 1.3 \times 10^8$ 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 $\sigma$ tends towards that of BN:

<table>
<thead>
<tr>
<th>Material</th>
<th>Current Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>an–BN/Al₂O₃</td>
<td>$(8 \pm 1) \times 10^{-15}$ S.m⁻¹</td>
</tr>
<tr>
<td>an–BN/Al₂O₃ (aged)</td>
<td>&lt; $(2 \pm 3) \times 10^{-15}$ S.m⁻¹</td>
</tr>
</tbody>
</table>

What is the chemical degradation process that leads to this electrical aging?
### Aging study of an–BN/Al$_2$O$_3$

**Scientific approach**

**Experimentation**

**Charging study of annealed and coated boron nitride**

**Conclusion & outlooks**

#### Chemical degradation

**RAMAN Spectroscopy (CIRIMAT)**:

*Laser 532 nm, 600 tr/mm*

- $\sim 1365$ cm$^{-1}$: $E_{2g}$ phonon mode of hBN
- $\sim 4361$ 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$^6$ $^7$

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Aging study of an–BN/Al$_2$O$_3$

Chemical degradation

XPS (CIRIMAT):

Monochromatic source

(ray Al K$_\alpha$ - 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$_2$O$_3$ coating = electrical properties deterioration

⇒ Important contamination which is favoured under electron radiation (depending on flux)
## Conclusion & outlooks

### 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 $\delta_{\text{SEE}}$ 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 $\delta_{\text{SEE}}$ and $\sigma$) of an–BN/Al$_2$O$_3$ sample after a critical dose
- **Degradation & contamination processes** especially of the annealed alumina coating

### Optimisation of industrial coating process
- High roughness $\Rightarrow$ 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**
Experimentation

Scientific approach

Charging study of annealed and coated boron nitride

Aging study of an BN/Al$_2$O$_3$

Conclusion & outlooks

Thank you for your attention

Acknowledgment:

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Partial discharges characterisation through the REPA Method

![Graph showing the relationship between irradiation time and potential difference.](image)