

# Sputtering yield of potential ceramics for Hall Effect Thruster discharge channel

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**Erosion yields of three candidate ceramics (BN, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>) for electric propulsion applications have been measured and compared to simulations by the Onera simulation tool for ion sputtering - CSiPI. The influence of Xe<sup>+</sup> ion energy in the 200eV – 800eV energy range and of the angle of incidence in the 0 to 75° range have been investigated. Prediction of erosion yield by simulation is good with silica, but differences are observed on BN and Alumina. These differences have been attributed to grain detachment effects and sputtered atoms charge effects.**

## Nomenclature

<i>I</i>	=	Current
<i>V</i>	=	Voltage
<i>E</i>	=	Energy
<i>E<sub>s</sub></i>	=	Surface binding energy
<i>E<sub>B</sub></i>	=	Bulk binding energy
<i>Y</i>	=	Erosion Yield

## I. Introduction

**T**HE discharge Channel of Hall Effect Thrusters (HET) is a critical component. It has to be electrically resistive, and resistant to ion sputtering. Furthermore its electron emission characteristics govern some discharge plasma characteristics. Most of the Hall Effect thrusters developed all over the world use boron nitride (BN) or BN based materials (typically BN-SiO<sub>2</sub>) as ceramic material for the discharge channel. One of the great advantages of BN is that it is highly resistant to erosion under ion bombardment. Nevertheless, ceramic erosion remains the main life length limiting issue for this technology<sup>1</sup>. Efforts have been paid to characterize erosion resistance to ion bombardment of several candidate ceramics for HET discharge channel<sup>2-6</sup>. The work presented in this paper continues this effort.

Materials exposed to ion bombardment get eroded by sputtering, ie. the removal of target atoms displaced by the collision cascade generated by the impinging ions. This phenomenon is well established, characterized and

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documented<sup>7</sup>. In addition to sputtering, it is suspected that grain detachment under ion bombardment also plays a significant role in ion erosion.

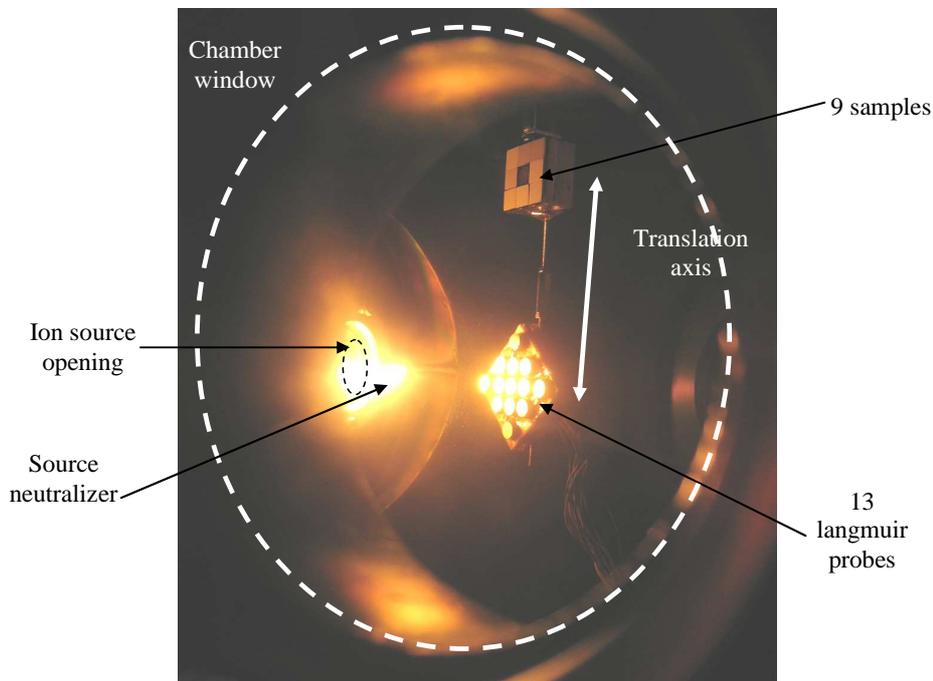
Erosion yields under Xe<sup>+</sup> ion bombardment have been measured using a mass loss technique for various materials, impinging energy and ion beam angle of incidence. In this paper result on BN, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> are presented and discussed. Erosion yields are compared with sputtering yield calculations using the Monte Carlo code CSiPI<sup>6</sup>.

## II. Experimental

Samples are 10mm x 10mm x 1mm ceramic blocs. The mounting of the samples was specifically developed to expose several samples simultaneously to the same ion beam. Samples are glued to a screw that is screwed on the sample holder. The sample holder is a 3x3 matrix that holds nine samples simultaneously. It is designed to limit sample contamination by sputtering products from the sample holder.

Erosion yields were measured by sample weighing before and after ion bombardment in the Onera ion erosion set up<sup>2</sup>. It is a vacuum chamber pumped by a 500L/min turbomolecular pump. The ion beam is generated by a Kaufman ion source (Ion Tech 3cm) that produces a neutralized monoenergetic ion beam (+/- 5eV). Mass loss were obtained by sample mass measurement using a Sartorius ME36S balance with weighing accuracy of +/-2 µg. Moisture desorption was managed by hot outgassing under primary vacuum. After moisture outgassing, exposure to air was limited to approximately two minutes when the samples are loaded in the erosion set up and two minutes again when the samples are unloaded from the erosion set up. Storage and weighing are performed under dry nitrogen atmosphere. Samples were exposed to a first erosion step to remove any possible initial sample contamination and to allow Xenon implantation that can impact sample mass variations.

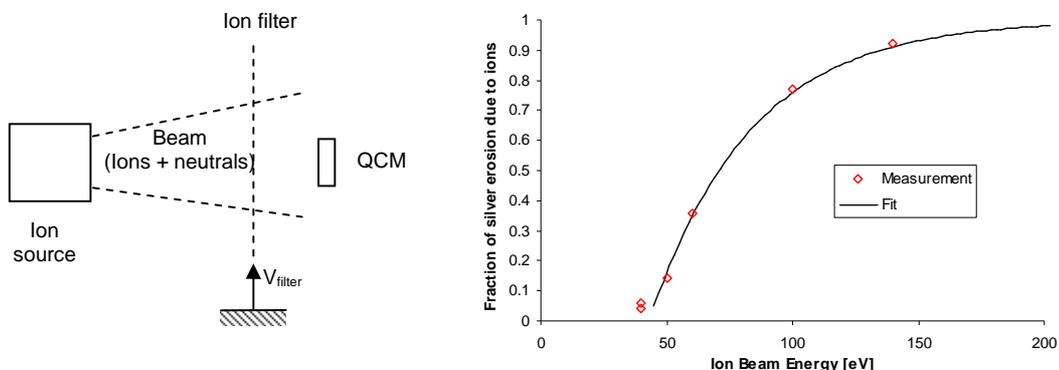
During the ion bombardment process, ion currents on samples were monitored using flat Langmuir probes facing the plasma. For this, samples are removed from the ion beam while Langmuir probes are moved inside the beam instead of the samples. This was periodically done to monitor ion beam current density drifts. The ion current density is obtained by mathematical treatment of the  $I(V)$  characteristic<sup>2</sup>. Typical ion current density was one hundred of µA/cm<sup>2</sup> and the drift could reach few percents during the whole erosion time (typically few hours).



**Figure 1: View of the sample holder loaded with nine samples and the Langmuir probes matrix.**

In such experiments, sputtering yield measurement can be altered by energetic neutrals generated by the ion source in the acceleration grid area by ions neutralization (charge exchange for instance). These fast neutrals are not

decelerated within the electrostatic sheath downstream the acceleration grid so that they can be significantly more energetic than ions when reaching the target material. Even if their flux on the target sample is low compared to the ion flux, their participation to erosion can be non negligible when ion energy approaches sputtering threshold energy. Erosion due to neutrals has to be deduced from raw erosion measurements to measure the ion sputtering yield. We characterized the participation of neutrals to silver erosion in our set up at low  $Xe^+$  energy. This measurement was performed by differential erosion rate measurement of a silver coating evaporated on a Quartz Crystal Microbalance (QCM). Erosion due to ions and neutrals were distinguished by ions elimination by a biased grid (see Figure 2-left). This grid is screened by two grounded grids. When filtration grid is biased above ions energy, erosion is only caused by neutrals. When it is not biased, erosion is due to neutrals and ions. By differential erosion rate measurement the relative participation of ions and neutrals was deduced. Figure 2-right presents the experimental participation of neutrals to the erosion of silver. Close to the sputtering threshold energy of silver, erosion due to ions becomes obviously negligible. This curve is only valid for silver since it strongly depends on the energy dependence of the eroded material. Nevertheless, this paper aims at investigating sputtering yields in the 200eV – 800eV energy range. The participation of neutrals to erosion is extrapolated to 2% at 200eV from Figure 2-right. Participation of neutrals to erosion was neglected in this study.



**Figure 2. Left: Schematic description of the diagnostic of neutral participation in erosion. Right: measured proportion of silver erosion due to ions in our set-up.**

### III. Simulations

Sputtering yield simulations were run to assess sputtering yields of the eroded materials. Simulations were run using the Onera simulation tool for ion sputtering, CSiPI<sup>6</sup> (Code de Simulation de la Pulvérisation Ionique). This is a Monte Carlo code similar to Tridyn<sup>8</sup> that uses the binary collision approximation with the ZBL interaction potential. The stoichiometry evolutions of the target surface under ion bombardment are treated. The kinetic of this evolution is not studied since the code is optimized to reach the surface equilibrium as rapidly as possible.

This type of simulation code needs input parameters that are the bulk binding energy and the surface binding energy. These input data are extracted from thermodynamical data<sup>6</sup>. This means that calculations are predictions since no sputtering input data is used. The classical modeling assumption is that bulk binding energy is null while erosion is limited by surface binding energy<sup>8</sup>. We consider that this assumption is correct for metals, (we propose a slight modification in ref. 6). For ceramic materials we consider the opposite assumption: cohesion of the material mainly results from bulk binding energy. For this we consider a simple molecular model of the target material where each atoms are characterized by one coordination value (e.g. 4 for Si, 3 for B, N and Al, and 2 for oxygen). We assume that atoms are bound to each other by localized chemical bonds. For instance each Si atom is bound to 4 O atoms in  $SiO_2$  while O atoms are bound to 2 Si atoms. The bound energy is determined by thermodynamically considerations

Target material	Metallic atom		O or N	
	$E_B$	$E_S$	$E_B$	$E_S$
$Al_2O_3$	23.4	0.02	15.6	0.02
BN	12.1	0.02	12.1	0.02
$SiO_2$	18.8	0.02	9.4	0.02

**Table 1. Bulk and surface binding energies in eV used for CSiPI calculations**

involving the heat of formation of the material. We consider a very limited surface binding energy  $E_s$  of 0.02eV. The data used for simulations are presented in Table 1.

#### IV. Results

Sputtering yields of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and BN at 200eV, 350eV, 500eV and 800eV under normal incidence Xe<sup>+</sup> bombardment are presented on Figure 1. The sputtering yields are expressed in mm<sup>3</sup>/C, that is the best unit to express the rate of ablation depth in a HET. Al<sub>2</sub>O<sub>3</sub> is the less sensitive material to ion erosion while SiO<sub>2</sub> is the most sensitive. The global trends for BN, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> are consistent with expected trends, except BN sputtering at 200eV. This measurement is surprisingly higher than at 200eV and even 500eV. Since all samples were eroded simultaneously, a problem due to the ion beam is excluded. Weighing was double checked. We believe that this correspond to a true effect that we will discuss in the discussion part.

Relative sputtering versus angle of incidence is presented for the same samples for 500eV Xe<sup>+</sup> ion bombardment. The trend is as expected for targets composed of low mass atoms compared to ion mass: The sputtering yield rapidly increases at high incidence. We note that this trend is not always visible. Especially roughness tends to flatten the incidence dependence of ion sputtering<sup>6</sup>.

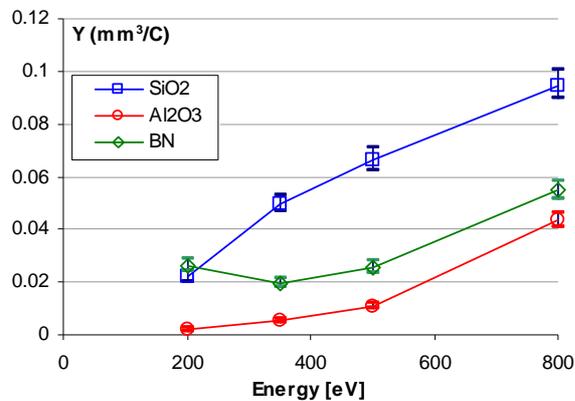


Figure 4. Sputtering yield of three ceramic materials as function of Xe<sup>+</sup> ion energy under normal incidence.

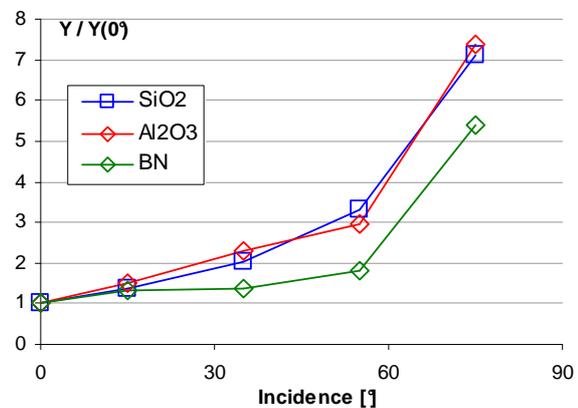


Figure 4. Normalized sputtering yield of three ceramic materials as function of the incidence of a 500eV Xe<sup>+</sup> ion beam.

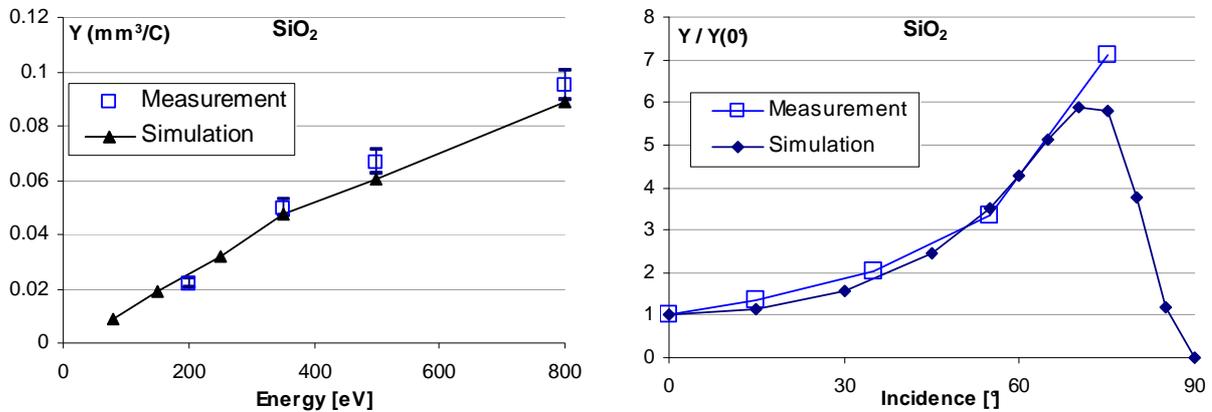
#### V. Discussion

Erosion measurements are compared to CSiPI simulations. Two main differences between measurement and simulations are expected:

- Erosion is due to sputtering (atomic emission stimulated by the impinging ions) and other mechanisms such as grain detachment. This second phenomenon is not treated by CSiPI.
- CSiPI consider ideal flat surfaces, while even a slight roughness can impact sputtering yields. The sputtering yield is only slightly impacted by roughness at normal incidence while it is much more impacted at grazing incidence. The key parameter of roughness is mainly the local slopes (at the nanometer scale). Slopes of few degrees tend to shift the maximum sputtering yield to higher incidences, while slopes of few tens of degrees tend to flatten the incidence dependence of the sputtering yield.

We compare simulation results with measurements and discuss the differences. Figure 5 presents silica erosion yield from experiments and simulation. The correlation between the two data is good. This is explained by the fact that silica is very close to the ideal material simulated by CSiPI: it was mirror polished and it is not composed of grains, so that grain detachment is not possible. Erosion at 75° is relatively higher than prediction by CSiPI. This can be explained by a limit of the simulation. We also highlight the fact that sputtering yield measurement at grazing incidence is highly sensitive to the relative angle of the ion current measurement system and samples. The quality of our angle management system could not achieve an angular positioning quality better than few degrees. This has a

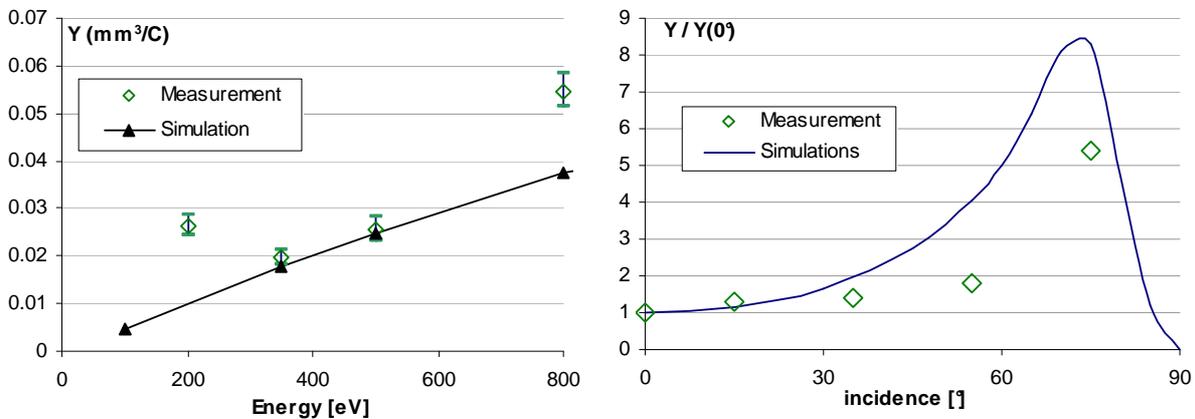
negligible effect up to 55° but is sufficient to explain the difference between measurement and simulation at 75° on Figure 5. The case of SiO<sub>2</sub> illustrates the capabilities of CSiPI to predict ion sputtering of ceramic materials.



**Figure 5. Comparison of erosion measurements with simulations by CSiPI with SiO<sub>2</sub>**

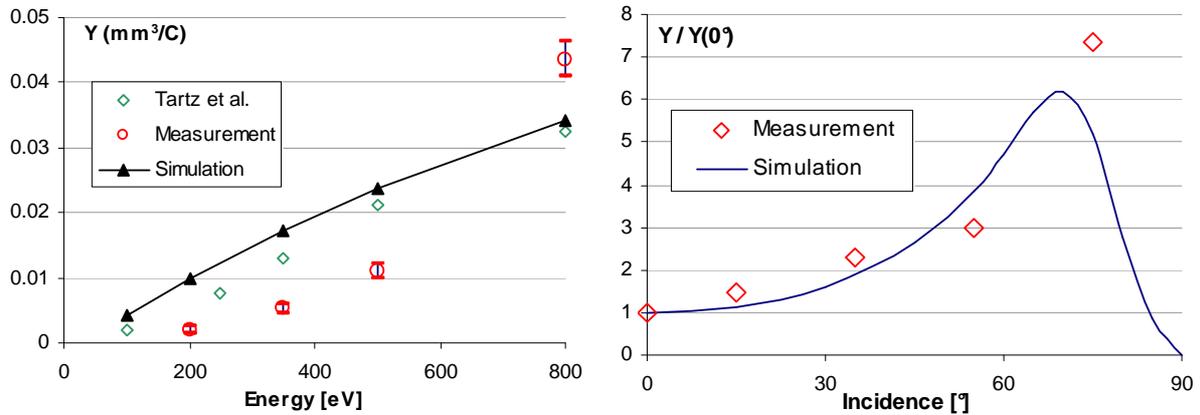
Figure 6 presents the similar comparison for BN. The correlation between simulations and measurements is not as good as with SiO<sub>2</sub>. On the energy dependence curve, the two intermediate points are properly predicted, while for the two extreme energies, experiment is above simulation. We interpret this fact by the fact that the BN samples were quite fragile. We suspect that the surface could get even more fragile after exposure to ion bombardment. Some grain detachment could have occurred during sample handling or during ion bombardment. We suspect that ion implantation could enhance grain detachment by accumulation at grain boundaries or by volume expansion of the implantation layer.

The angle dependence of BN sputtering is characteristic for a slightly rough surface.



**Figure 6. Comparison of erosion measurements with simulations by CSiPI with BN**

Figure 7 compares erosion from measurement and simulation on alumina. The prediction is clearly not correlated to measurement. Measured erosion is lower than predicted except at 800eV. We did not expect that erosion could be lower than prediction. Indeed roughness effects and grain detachment effects can not support this observation. Furthermore, since other samples are eroded simultaneously, any experimental artifact would have also impacted the other samples (BN and SiO<sub>2</sub>). In addition, comparison with measurements from Tartz et al.<sup>5</sup> gives a much better correlation with our simulation.



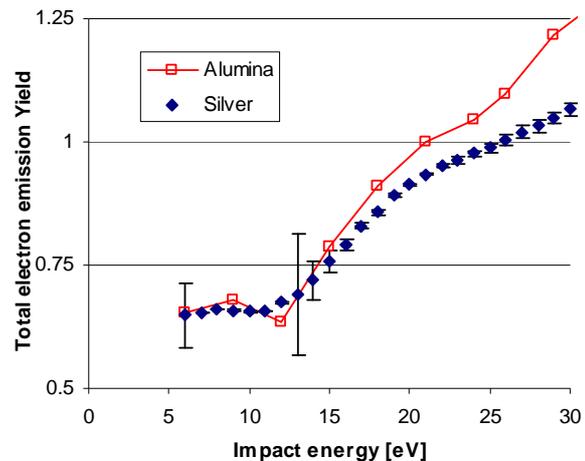
**Figure 8. Comparison of erosion measurements with simulations by CSiPI with  $\text{Al}_2\text{O}_3$ . Measurements from Tartz et al.<sup>5</sup> are also presented for comparison**

The main difference between their measurement conditions and our is ion beam neutralization. Our neutralizer is a heated filament. It is located next to the acceleration grid (we do not use any deceleration grid) in the middle of the ion beam. Floating potential is typically few volts below plasma potential. In ref. 5, neutralization is not conventional since the filament is out of the electron beam and electrons are emitted at 25eV. Furthermore the total electron current was set to get a null potential on a reference silver sample, with the assumption that the potential of the insulating target material would be the same. Nevertheless, as presented on Figure 8 electron emission under electron impact of alumina is higher at 25eV than the one of silver. Then it is expected that alumina during erosion is slightly positively biased. The secondary electron emission data must be handled very carefully since electron emission under electron impact of oxides depends on many parameters. Furthermore the target is not really alumina, but amorphous aluminum oxide with implanted Xenon. We finally conclude that alumina could be negatively biased compared to the plasma in our setup, while it would be positively biased in ref. 5.

Our next assumption is that aluminum atoms emitted by alumina under ion bombardment could be positively charged in an important proportion. In this case, in our erosion conditions  $\text{Al}^+$  ion would be recollected by the negatively biased surface, while they would be extracted from the positively biased surface.

The necessary assumption is that a non negligible fraction of aluminum atom are emitted charged and then recollected. We also precise that our measurements are consistent with those from ref 5. for  $\text{SiO}_2$  and BN. This could be explained the more ionic character of the Al-O bond compared to Si-O bond<sup>9</sup>, while BN is much more covalent. Aluminium cations in alumina are more positively charged than Silicium cations in silica<sup>9</sup>. This could explain the emission of ions instead of neutrals.

This discussion involves important assumptions that are not sufficiently supported. Nevertheless they are of high importance since ions could be injected in the discharge, and probably rapidly recollected by the discharge channel walls.



**Figure 7: Total electron emission yield under low energy electron impact on Silver (classical collection method) and Alumina (Kelvin Probe method)**

## VI. Conclusion

We have measured erosion yield of several ceramic materials that are potential HET wall materials. The influence of ion impinging energy in the 200eV to 800eV range was investigated. The influence of the angle of incidence has also been investigated. These measurements have been compared to predictive simulations. SiO<sub>2</sub> that is almost an ideal sample (mirror polished, no grains) leads to simulations very consistent with measurements. In opposition, BN erosion can be influenced by grain detachment effects and roughness effects. Alumina erosion was lower than expected. We suggest that this could be explained by the emission of charged Al<sup>+</sup> ions instead of Al atoms. These ions would be recollected by the negatively biased sample.

## Acknowledgments

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