

# Specific laboratory testing equipment & methodology for sputtering tests of electric propulsion materials

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**Abstract:** Specific laboratory testing equipment, which allows studying the Sputtering behavior of Electric Propulsion materials, has been designed. The versatility of the equipment allows studying the Sputtering behavior dependence with the ion energy, the angle of incidence and the temperature. To that end, a methodology consisting in measuring the eroded volume has been proposed, leading to a complete procedure of Sputtering Yield characterization. Preliminary tests with Argon and Xenon on Copper have been carried out at 5000 eV leading to validate the procedure as results fit with available experimental data. Moreover, a method to establish the current density distribution, as the reflex of erosion produced at normal incidence, has been developed. Finally, Xenon to Borosil (BN-SiO<sub>2</sub>) trials have been completed, validating the procedure for insulating ceramic materials, which are used in accelerating channels. Dependence with the angle at 5000 eV has been studied; trials at lower energies (1000 and 500 eV) have been accomplished and tests at high temperature have been successfully attempted. Results fit with the expectations.

## Nomenclature

$I$	=	Ionic current,
$j$	=	Ionic current density
$t$	=	Time under ion bombardment
$x$	=	Erosion depth
$A$	=	Area exposed to the ion beam

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## I. Introduction

**N**OWADAYS, the main cause of failure of Stationary Plasma Thrusters is the wear of the dielectric discharge chamber, due to Sputtering by Argon or Xenon Ions. Therefore, it is necessary to develop materials with improved resistance to ion bombardment. Unfortunately, up to now there is not clear and complete understanding of the plasma-chamber interaction, and no reliable models exist to predict degradation of the walls due to ion bombardment (Sputtering).

Up to now, the most common materials used for producing dielectric discharge chambers are Borosil (Boron Nitride and Silicon) and Alumina, because of their high Sputtering resistance. Regrettably, despite many tests have been carried out, results on the Sputtering behavior of different dielectric materials are not conclusive, leading to a lack of empirical laws to predict the sputtering response of the wall. Even if it is known that Sputtering resistance depends on many variables like chemical composition, particle size or manufacturing processes, those relationships are not yet established.

As the development of materials with improved Sputtering behavior is one of the ways to improve critical components and thus get Stationary Plasma Thrusters lifetime increased, materials with enhanced Sputtering resistance have become an aim pursued by the SPT manufacturers' community. Consequently, it becomes compulsory to develop a tool which allows studying the Sputtering behavior of Electric Propulsion Materials, in a practical, low time-consuming and economical way.

By this moment, the lack on conclusive results for the Sputtering behavior of Electric Propulsion materials has been caused by differences among test facilities and methodologies. The great majority of accomplished tests have been carried out using a whole thruster and a whole discharge chamber, which leads to unaffordable costs, big divergences among different testing facilities and not clear understanding of Sputtering erosion mechanisms. The way planned to mitigate these disadvantages is to set-up a specific laboratory testing equipment to carry out accelerated tests. This way, obtained Sputtering Yield data would help to build appropriate wall erosion simulation codes or to develop Sputtering simulation models for EP materials.

The approach made by INASMET-Tecnalia has been to design and set-up small and versatile equipment which allows sputtering a 1 inch sample with a focused ion beam. This way, parameters which influence the Sputtering behavior (Ion energies, temperature and incident angle) can be independently varied to carry out a complete characterization.

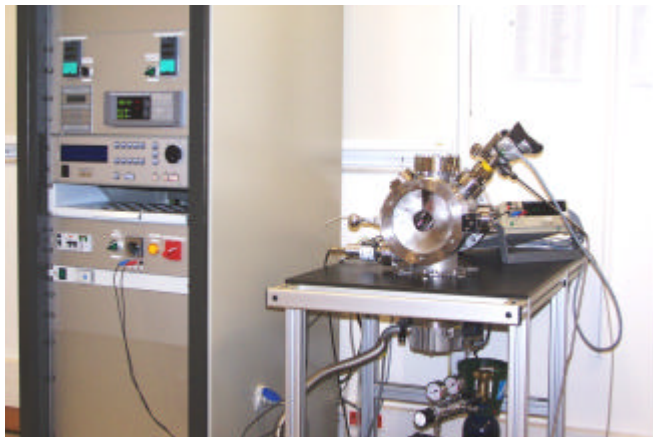
Only few authors<sup>1-4</sup> have accomplished Sputtering trials on BN-SiO<sub>2</sub> materials varying energies and incident angles, and there are not even yet temperature dependence conclusive results. Thus, the designed and already tested laboratory equipment can be considered at the cutting edge of the technical innovation in this field.

This article is organized as follows. First, a description of the specifically designed laboratory testing equipment is made. Then, experimental setup and procedure is described, followed by validation results from a reference metallic material. Finally, Xenon Sputtering on Borosil results are presented and discussed.

## II. Sputtering laboratory testing equipment (Experimental facilities)

The specifications followed to design the equipment were to provide capabilities to sputter a small sample of the wanted material, varying independently parameters such as ion energy, incident angle and temperature. In addition, the driving criterion was to do it in an economical way, to allow completely characterization of different materials without unaffordable costs. For that purpose, the system has been designed as a laboratory testing equipment and the main testing parameters are ion energy (200-500 eV), incident angle (0°-90°) and sample surface temperature (30-800°C).

The designed system consists on a small cylindrical vacuum chamber (150 mm in diameter x 250 mm length), pumped by a scroll and a turbo-molecular pump, capable of reaching 10<sup>-6</sup>



**Figure 1. Sputtering laboratory testing equipment (INASMET -Tecnalia).**

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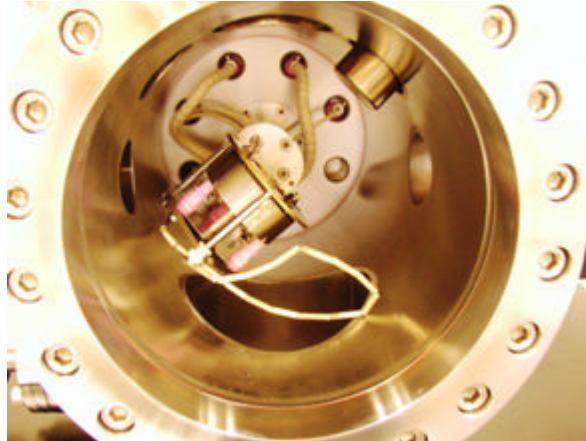
Torr during normal operation of the ion gun. Many Ultra High Vacuum ports are disposed all throughout the vacuum chamber in order to implement various elements. Five of them have the same focal point in the axis of the cavity, right where the centre of the exposed surface of the testing sample will be placed.

A SPECS IQE 11/35 ion gun is placed in one of the referred ports allowing a source-to-sample distance of 50 mm. It can be operated with different gases, and provides a focalized beam of 8-10 mm at the mentioned source-to-sample distance. It can supply up to 10-15 microamperes with a Gaussian beam profile, and the ion energy could be varied independently between 200 and 5000 eV.

Furthermore, the system is equipped with a sample holder which allows holding the sample in place for the Sputtering tests, placing it at different angles of incidence with respect to the ion beam, but also heating it for tests at elevated temperature. The sample is a disk of 1" in diameter x 5 mm thick, the sample temperature may be risen up to 600–800°C depending on material properties, and the angle of incidence with respect to the outer normal of the surface can be selected between -90° to 90°.

In addition, the equipment allows on-line monitoring the testing surface temperature by IR pirometry as well as with thermocouples placed wherever they are needed. Besides, the sample current is monitored (to know about the incident ion density) by a micro-ammeter and can be automatically registered in a computer by a LabView application. Some viewports are also present so that current test can be seen through them, and many blink ports are ready for implementation of further devices such as a QCM, or a bigger ion source.

Described equipment allows establishing the Sputtering Yield (ratio of eroded volume to ion current density), which is the parameter used to quantify the Sputtering behavior of different materials. To that end, the sample of the target material is sputtered under controlled parameters during a certain time. Then, the resulting eroded surface is measured using techniques such as AFM or profilometry. In addition, more information about Sputtering erosion mechanisms can be found out using microscopic conventional techniques such as SEM and EDS.



**Figure 2. Sample Holder**

### III. Experimental setup and procedure

Sputtering is described as the removal of atoms from a solid surface due to energetic particle bombardment, leading to erosion of the surface of the target materials<sup>5,6</sup>. The parameter which helps to quantify the Sputtering behavior of a given material is the Sputtering Yield,  $Y$ , which is defined as the mean number of atoms removed from the surface of a solid per incident particle. Such a definition is easy to understand for a monatomic material but is less comprehensive for composites, since atoms of different species are removed, and other aspects such as entire grain removal should be taken in account. To that end, Volumetric Sputtering Yield,  $Y_v$ , is defined as volume removed from the surface per amount of incident charge<sup>1,3</sup>. In the case of monatomic materials, it is easy to compare both magnitudes using Eq. (1), where  $\rho$  is the material density,  $N_A$  the Avogadro number,  $W$  the atomic weight and  $q$  the electric charge of an electron.

$$Y \left( \frac{\text{Atom}}{\text{Ion}} \right) = \frac{\rho \times N_A \times q}{1000 \times W} \times Y_v \left( \frac{\text{mm}^3}{\text{C}} \right) \quad (1)$$

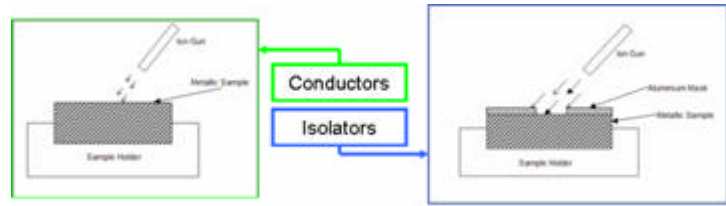
Generally speaking, the procedure proposed for estimating the Sputtering Yield consists in placing the target under ion bombardment during a certain time and then measuring the erosion depth produced by profilometry or AFM. Therefore,  $Y_v$  is directly obtainable from the Sputtering tests using Eq. (2), where  $V$  is the removed volume,  $Q$  the total incident charge, and  $dA$  the differential element of area exposed to the ion beam. In addition,  $\bar{x}$  and  $\bar{j}$  refer to the averaged values of the erosion depth and the ionic current density through the exposed surface.

$$Y_V = \frac{V_r}{Q_i} = \frac{V_r}{I \times t} = \frac{V_r}{\int j dA \times t} = \frac{V_r}{\bar{j} \times A \times t} = \frac{\bar{x}}{\bar{j} \times t} \quad (2)$$

The aim of the designed equipment & methodology is to characterize Sputtering behavior of Electric Propulsion Materials, which are normally insulators. However, because of the complexity of Sputtering experiments with insulators, and due to the lack of experimental data, a first validation with a reference material is needed. In addition, information about the ion current density distribution will be extracted from the analysis of the erosion produced on a metallic sample. To that end Xenon to Copper experiments have been carried out.

Sputtering on metals differs from Sputtering on insulators in one main concept. When a metal sample is bombarded by positive ions, electrons are emitted from the sample (field emitted electrons and secondary electrons). Thanks to the conductive character of metals, an electric current is established if the sample is grounded, so it can be easily registered by an ammeter. However, if the target is an insulator, no current can be established, and due to the

emission of electrons when ions impinge, a positive charged layer is created which reflects the incident ions disturbing the ion beam and even hindering Sputtering erosion. In order to avoid this effect, various solutions have been proposed, such as providing a heated thermionic wire surrounding the ion beam<sup>1,3,4</sup> or covering the sample by a thin film of a conducting material. If the film is holed, just where the ion beam is wanted to impinge onto the insulator, electrons from the conductive mask impede the charge build-up on the surface of the insulating material.



**Figure 3. Experimental layout for conductive and insulating materials.** The surface of the conductive material is facing directly the ion beam while the surface of the insulating material is covered by a holed thin Aluminum film.

Finally, masking technique has been adopted. This way, only a small area of the insulating material will be exposed to the ion beam, so the ionic current density distribution,  $\bar{j}$ , must be estimated to determine the fraction of the registered current which is received by the exposed area. Thus, the Volumetric Sputtering Yield on insulating Electric Propulsion Materials will be calculated using Eq. (3).

$$Y_V = \frac{V_{Exposed\_Area}}{\int_{Exposed\_Area} j dA \times t} = \frac{V_{Exposed\_Area}}{\bar{j}_{Exposed\_Area} \times A_{Exposed\_Area} \times t} = \frac{\bar{x}_{Exposed\_Area}}{\bar{j}_{Exposed\_Area} \times t} \quad (3)$$

#### A. Basic experimental procedure for Sputtering Yield determination

Before testing Electric Propulsion isolating materials, it has been decided to carry out trials on a conductive material (Copper) due to the following reasons. First, because it is needed to validate the procedure with a reference material, and second because these trials are needed to estimate the ionic current density distribution, as it is explained at the end of this section. Therefore, experimental procedure followed with conductive materials is described on the next paragraphs.

First of all, disks of 1" in diameter x 5 mm thick are prepared and polished achieving average roughness of about 0,1  $\mu\text{m}$ . After cleaning in an ultrasonic bath, the sample is fixed on the sample-holder and a thermocouple is put in contact with the upper surface of the sample. Then, it is waited until the pressure drops below  $5 \cdot 10^{-8}$  mbar. Afterwards, the angle of incidence is set by turning the calibrated wheel connected to the sample holder, the set-point temperature is set on a GEFTRAN PID which controls the heating of the sample, and the ion energy is established. Once the sputtering test is ready to be launched, a leak valve is opened to control the Argon or Xenon mass flow which is going to be ionized. When the pressure at the chamber is between  $3e^{-6}$  and  $3e^{-5}$  mbar (depending on the ion energy, and the desired ionic current), the ion gun is switched so the test is launched.

### 1. Determination of the eroded volume

After cleaning the sample, the sputtered surface profile is measured by an UBM 2010 Optical Profilometer. Many profiles are taken, along the X and Y axes, with an accuracy of 125 points/mm. The eroded area in each section is calculated by the UBM software, so the total eroded volume is calculated integrating those areas.

Such a profilometer analysis serves to get eroded volumes or average depths to calculate the Sputtering Yield as established in Eq (2). Moreover, this information is used to estimate the ionic current density distributions, without using faraday cup mobile systems<sup>7</sup>, which are complex methods often used to acquire this information.

### 2. Determination of the ionic current & ionic current density distribution

Due to the low-cost approach aimed for this tool, it was decided to obtain the ionic current using only one microammeter, and to estimate the ionic current density distribution as a reflex of the erosion produced on a metallic sample placed under ion bombardment at normal incidence.

This way, the sample is grounded by a microammeter so a sample current is registered. This current is the sum of the ionic current and current provoked by secondary electrons, so the last one must be subtracted in order to get the correct value. To that end, sample current is corrected, as in the Eq (4), using the Secondary Electron Yield, **SEY**, which is defined as the number of secondary electrons emitted per incident particle.

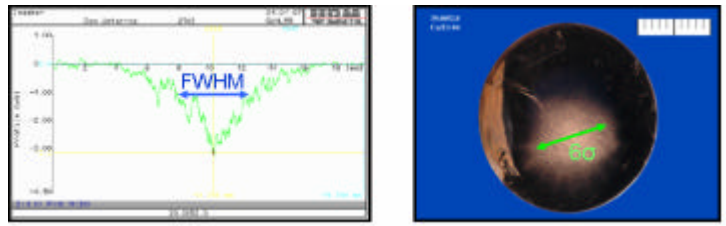


Figure 4. Estimation of the FWHM of the ion beam.

$$I_{IONIC} = \frac{I_{SAMPLE}}{1 + SEY} \quad (4)$$

Along with SPECS data, the ionic current distribution shows a Gaussian-like profile, whose Full Width Half Maximum, depends on various factors such as ionized gas, ion energy and source-to-sample distance. Thanks to the mathematical properties of a Gaussian-like distribution, all the distribution is known if the integral (total ionic current) and a dispersion parameter (FWHM) are known. Total ionic current is known for each experiment (see Eq. (4)) but, unfortunately, FWHM is only known for a few combinations of parameters.

Since the Sputtering erosion is proportional to the current density, it is thought that the erosion of a metallic sample must be the reflex of the current density distribution. Thus, FWHM is estimated from eroded profiles and from the eroded mark, as represented on Fig. (4).

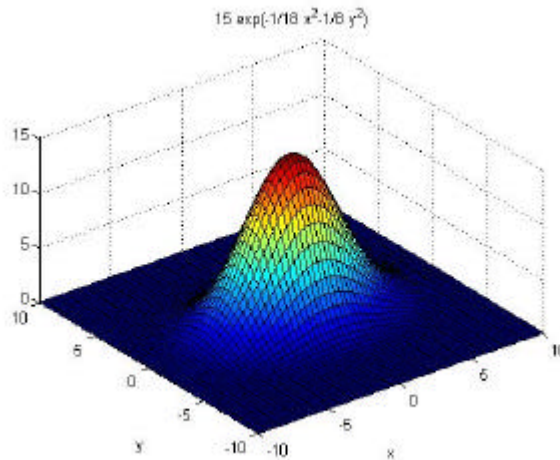


Figure 5. MATLAB simulated Gaussian-like distribution for angled incidence.

Note that FWHM and Typical Deviation follow the relationship  $FWHM \sim 2,354 s$ . On the other side, the value measured in the eroded mark is the total amplitude of the erosion, so it can be related with the typical deviation as the 99,7% of the erosion should be on the interval  $\mu \pm 3s$ . Therefore,  $s$  is the sixth part of the diameter of the eroded mark. This technique has been validated, as trials in conditions at which FWHM is known have been carried out and values calculated in this manner fit perfectly with SPECS data. Another aspect to take in account is that when the incidence is not normal, there will be a FWXM in x direction and another one along the y axis, and the distribution will have Gaussian-elliptic-like shape.



Once the total ionic current and FWHM are known for each combination of parameters, the current density distribution is modeled by the way shown on Fig. (5) and Eqs. (5) and (6), where (x,y) are the coordinates from the center of the ion beam,  $s_x$  and  $s_y$  are the typical deviations of the Gaussian function, and k the amplitude constant.

$$j(x, y) = k \exp \left[ - \left( \frac{x^2}{2s_x^2} + \frac{y^2}{2s_y^2} \right) \right] \quad (5)$$

$$k = \frac{I_{TOTAL}}{2ps_x s_y} \quad (6)$$

### B. Experimental procedure for insulators (Electric Propulsion materials)

The process is very similar to the one described on the previous section. The only difference is that, in this case, the sample will be masked by a holed Aluminum or Titanium foil. This time, the achieved average roughness is of about 0,6  $\mu\text{m}$  and exposure time was 2 days for 5000 eV and 4-5 days for 1000 and 500 eV.

To calculate the Sputtering Yield with Eq. (3), the average ionic current density of the unmasked zone is calculated using the previous model and the Eq. (7). The average step height is determined by profilometry.

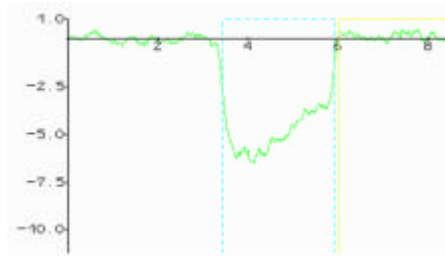


Figure 6. Step height measured by profilometry.

$$\bar{j} = \frac{\iint_{Unmasked\_Area} k \exp \left[ - \left( \frac{x^2}{2s_x^2} + \frac{y^2}{2s_y^2} \right) \right] dx dy}{A_{Unmasked\_Area}} \quad (7)$$

## IV. Results and discussion. Argon and Xenon Sputtering on Cu. Validation of the procedure.

Seven copper samples have been sputtered by Argon and Xenon ions at 5000 eV with incident angles of 0° and 45°. Exposure time was 8 hours, ion energy was set in 5000 eV, and sample currents of 11-12  $\mu\text{A}$  were registered. PID heater was in off mode, so temperature was around 30°C at the beginning of the test, and it increased only due to the ion bombardment. Experimental results were compared with data from other authors<sup>89</sup> and with data from TRIM simulations carried out by Iñaki Garmendia, from the Department of Numerical Analysis (INASMET-Tecnalia).

The first conclusion that can be extracted from those tests is that Sputtering Yield results fit with the expectations, so it can be affirmed that the equipment and the methodology are suitable to determine the Sputtering Yield, at least of metallic samples under bombardment of Argon and Xenon ions. Moreover, eroded profiles have

Table 1. Argon and Xenon to Copper Sputtering Yields.

Sample		Y (Atoms / Ion)		
		Inasmet	Yamamura	Simulation
Ar, 5000eV 0°, R.T.	CU02	6,426		
	CU03	4,948		
	CU04	6,193		
Average		<b>5,856</b>	<b>6,00</b>	<b>6,85</b>
Xe, 5000eV 0°, R.T.	CU05	6,128		
	CU06	7,559		
Average		<b>6,843</b>	<b>6,50</b>	<b>5,88</b>
Xe, 5000eV 45°, R.T.	CU07	12,354		
	CU08	12,665		
Average		<b>12,510</b>	<b>11,05</b>	<b>13,48</b>

also provided information on how to estimate the Gaussian-like current density distribution provided by the SPECS IQE 11/35 ion gun.

More trials will be necessary to completely validate the procedure but, as the objective was to study the behavior of Electric Propulsion Materials, efforts have been taken in this direction.

## V. Results and discussion. Sputtering on Electric Propulsion Materials

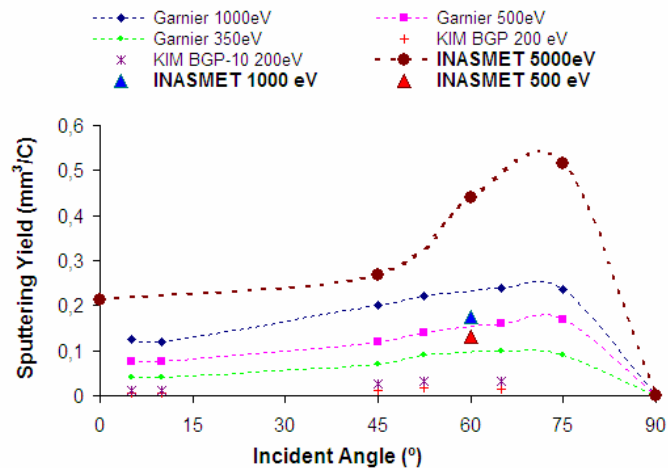
### A. Sputtering Yields

The planned Sputtering test campaign has been aimed to validate the procedure (equipment & methodology). For that purpose, several trials have been carried out varying Energy, Angle and Temperature. A deeper analysis has been made at 5000 eV and room temperature, varying the incident angle. Furthermore, a few trials have been accomplished at lower energies (1000 and 500 eV) and elevated temperatures (350 and 650 °C).

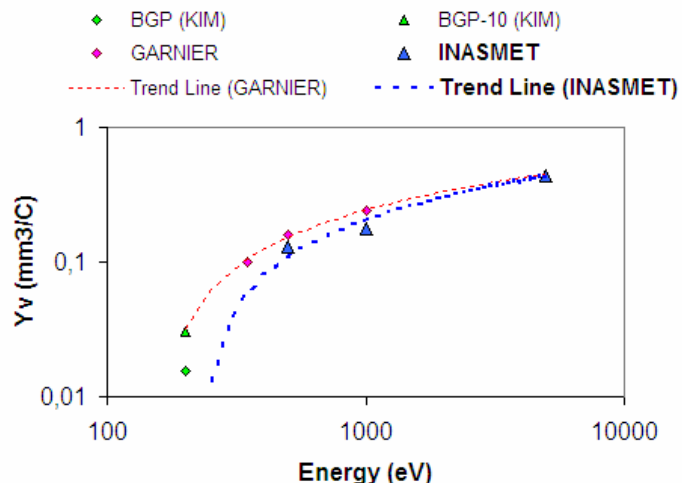
The Volumetric Sputtering Yield,  $Y_v$ , expressed in  $\text{mm}^3/\text{C}$ , is calculated relating the average depth of the eroded hole and the average current density received by the unmasked area, according to Eqs. (3) and (7). Results are represented in Figs. (6) and (7), where the Volumetric Sputtering Yield is represented first versus the angle of incidence and second versus the ion energy.

There exists an important lack of Sputtering Yield data on Electric Propulsion Materials such as Borosil, so only results from two authors<sup>1,2,3</sup> have been found out to perform a comparison for validating the procedure. Moreover, already published results have been carried out at different energies so they can not be directly compared and thus, there does not exist an attested reference value. In addition, BN-SiO<sub>2</sub> is a family of materials, whose properties may differ much from each other. Due to all the mentioned aspects, only tendencies and data extrapolations have been compared to determine if the procedure is valid to estimate the Sputtering Yield of Borosil materials.

First of all, it is found out that the Sputtering Yield increases with the incident angle, reaches its maximum around 60-70°, and drops abruptly for larger angles. It is thus corroborated that results obtained at INASMET-Tecalia show Sputtering Yield dependence with the angle which follows the same tendency than metallic materials<sup>8,9</sup> and BN-SiO<sub>2</sub> composites tested by Kim<sup>2</sup> and Garnier<sup>1,3</sup>.



**Figure 7. Xenon Sputtering on Borosil. Data comparison** *Sputtering Yield vs angle of incidence with respect to the surface normal, at 5000, 1000 and 500 eV*



**Figure 8. Xenon Sputtering on Borosil. Data comparison** *Sputtering Yield vs ion energy, at 60° of incidence*

Second, curve obtained for 5000 eV is far beyond curves at 1000, 500 and 350 eV<sup>1,3</sup>. That also follows logical trends, as Sputtering Yield increases with the energy. Furthermore, tests carried at 1000 and 500 eV can be directly compared with Garnier experiments. Those results enter between the expected ranges; they are approximately a 18% lower than the ones obtained by Garnier. If it is taken into account that ceramic materials present around 20% of variability in all their properties, and that BN-SiO<sub>2</sub> materials may differ much from each other, obtained results are completely successful. Therefore, it can be confirmed that the approach obtained with the designed laboratory testing equipment and the proposed methodology, is fairly acceptable.

Finally, when the Sputtering Yield is represented versus the energy in a double logarithmic scale (See Fig (8)), all the materials show a logarithmic-like trend between 10<sup>2</sup> and 10<sup>4</sup> eV<sup>8,9</sup>. Logarithmic trend lines have been drawn up based on results obtained by INASMET-Tecnalia and Garnier. As it is shown, results obtained at 60° fit perfectly with the expected trends. The same occurs for the rest of the tested incident angles.

## B. SEM Analysis

All the sputtered samples have been observed by Scanning Electron Microscopy in order to provide additional information about the surface topography. It has been checked that an abrupt step existed just on the limit between the masked and the unmasked area. It has also been observed that a preferential Sputtering exists in SiO<sub>2</sub> (see Fig. (9)), as BN grains outlast while “pools” appear where the amorphous Silica was before being sputtered. Finally, it has been detected that the resulting morphology depends on the incident angle, as it is shown on Figs. (9) and (10), because some kind of directionality, coinciding with the direction of the ion beam, appears in samples bombarded at 45 or 60°, contrary to the ones bombarded at 0°, where no directionality is found out.

According to SEM observation of sputtered samples, theories about the Sputtering erosion mechanisms in BN-SiO<sub>2</sub> materials may be established and a relationship between these mechanisms and material properties may be found out. Consequently, such an analysis will help to understand failure mechanisms in real thrusters.

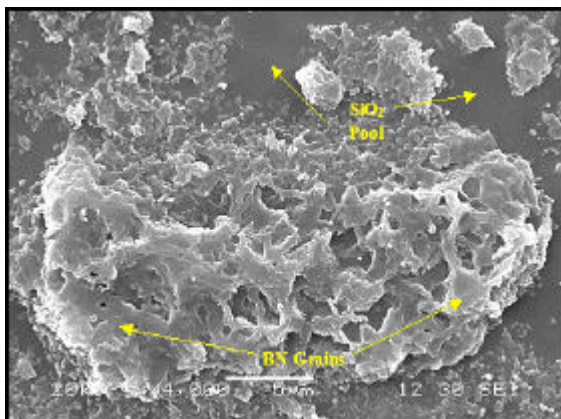


Figure 9. Sputtered Area (X4000). Xenon to Borosil, 5000 eV, 0°. INASMET-Tecnalia

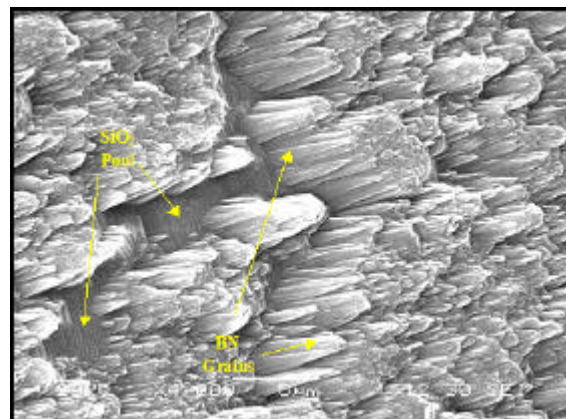


Figure 10. Sputtered Area (X4000). Xenon to Borosil, 5000 eV, 60°. INASMET-Tecnalia

## VI. Conclusions and future work

The specific testing equipment designed by INASMET to study the Sputtering behavior of Electric Propulsion Materials has been successfully mounted and tested, proving the correct functioning of the equipment. Besides, a methodology consisting on measuring the sputtered hole by profilometry and estimating ionic current density distribution as a reflex of the erosion produced, has been developed. Because of successful results on preliminary testing with Copper samples, it can be asserted that the designed equipment and the proposed methodology are suitable.

Moreover, several trials have been made on Borosil produced by INASMET-Tecnalia, placed under Xenon bombardment. First, an analysis of the incident angle dependence has been made at 5000 eV, leading to results which follow similar tendencies than the ones got by other authors. In addition, a few tests have been carried out at lower energies (1000 and 500 eV) and results are only a 18% lower than the ones obtained by Garnier<sup>1,3</sup>, what indicates that Sputtering Yields enter within the expected range. Besides, a few trials have been carried out at high



temperatures (350, 650°C) without conclusive data, but it is confirmed that the system can be used during large periods of time (4-5 days) at high temperatures, so it allows studying the temperature influence on the Sputtering behavior of Electric Propulsion Materials. It is obvious that more trials are necessary for a complete characterization of materials, but already obtained data is enough to affirm that ***the designed specific laboratory testing equipment and the proposed methodology are suitable to determine the Sputtering Yield of BN-SiO<sub>2</sub> composites, under Xenon Bombardment, so it will help to develop improved materials for Electric Propulsion accelerating channels.***

The realization of the present project has been the first approach carried out by INASMET-Tecnalia in Sputtering characterization, within the scope of developing better materials for ceramic discharge chambers for Stationary Plasma Thrusters. It has been demonstrated that the low-cost planned approach has been satisfactory. However, due to the precision required to distinguish between very similar materials, the system needs to be upgraded to gain accuracy. To that end, future efforts should be taken in carrying out more tests and in improving the procedure (equipment & methodology).

Some equipment improvements are being taken into consideration such as neutralizing the ion beam, investing in a more powerful ion gun, installing a mobile faraday cup system to directly measure the ion current, or using other techniques of measuring the sputtering yield (QCM, microbalance). Furthermore, more Xenon to BN-SiO<sub>2</sub> trials should be accomplished in order to completely evaluate angle and energy dependence of the Sputtering Yield. In addition, the effect of the temperature will be studied, as very few publications exist concerning this aspect, and the equipment is designed to introduce this variable. Moreover, erosion mechanisms will be studied by SEM and EDS, in order to establish a theoretical model about the Sputtering behavior of BN-SiO<sub>2</sub> composites, in order to predict failure mechanisms of real thrusters. Finally, the designed laboratory testing equipment and the developed methodology will constitute a tool which will be able to distinguish between similar Electric Propulsion materials. This way, it will be used to develop new materials and select among them the most resistant to Sputtering erosion, for implementation in Electric Propulsion devices.

### Acknowledgments

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