

INVESTIGATION OF XENON ION SPUTTERING OF ONE CERAMIC MATERIAL USED IN SPT DISCHARGE CHAMBER

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Abstract :

Ceramics based on boron nitride are commonly used as insulating materials for the channel of Stationary Plasma Thrusters because of their good properties in terms of thermal and wear resistance. Nevertheless, the erosion of the edges of the channel is a factor which could limit thruster lifetime.

The erosion of BNSiO₂ ceramic is studied here in two separates and complementary devices. The first one is a vacuum chamber equipped with an ion source which allowed us to make exposures of ceramic samples with variable incidence and energy of xenon ions. The second one is a RF magnetron discharge chamber devoted to studies by Optical Emission Spectroscopy (OES) of the products eroded from the sample surface.

Introduction :

The Stationary Plasma Thrusters (SPT) using xenon presently under development, are to be used as western spacecraft propulsion devices in a near future. The erosion of discharge chamber walls, that could be a lifetime limiting physical phenomenon of Hall thrusters, remains to be investigated more in details, for general understanding of near wall physical processes. Sputtered material may contaminate spacecraft surfaces, and erosion also influences the thruster working parameters optimization. The walls of these discharge chambers are typically made of ceramic materials, the

composite ceramic made of boron nitride and silicon oxide (BNSiO₂) being commonly used as dielectric material in Russian SPTs.

The investigation of the sputter-induced erosion under low-energy xenon ions of this ceramic is reported here. The results of sputtering experiments with ion-beam energy ranging from 350 eV to 1 keV, surface analyses (Electron Spectroscopy for Chemical Analyses - ESCA, Secondary Ion Mass Spectrometry - SIMS, Scanning Electron Microscopy - SEM), and Optical Emission Spectroscopy (OES) with BNSiO₂ target materials are presented. Two methods for determining sputtering and erosion products were performed in relatively short periods of time. First, a high vacuum chamber equipped with an (xenon) ion source was used to conduct the sputtering experiments. And secondly, the erosion products of ceramic samples were analyzed by optical spectroscopy in a radio frequency magnetron discharge working in xenon.

It is found that the angular dependence of the sputtering yield is small due to a strong surface roughness, and the sputtering yield evolution (at normal incidence) with ion energy is linear. The surface irregularities of this kind of ceramic and its complex surface morphology are thought to play an important role in the surface evolution under erosion. The material surface composition evolution is also discussed from chemical analyses, and an attempt to relate optical emission line intensity of

sputtered atoms and molecular species to this surface composition evolution is made.

Description of the experimental apparatus at DESP :

Erosion tests are performed in a vacuum chamber made of a stainless steel cylinder of 500 mm in diameter and in height equipped with an ion source (Ion Tech, Inc.). An oil diffusion pump ensures the pumping and the vacuum limit is 10^{-6} hPa and the working (xenon) pressure rises up to $4 \cdot 10^{-5}$ hPa. The source delivers ions in the range 200 to 1200 eV with a maximum nominal current of 100 mA. An electron emitting filament provides the electrons neutralizing the beam.

The samples tested are ceramic discs (20 mm in diameter and 2 mm thick) used as target for sputtering experiments.

The sputtering yield of the material is determined by measuring the weight loss of the sample and the ion current density it collects (ref 1).

Two sample holders were constructed in graphite with several facets at various incidences to study the influence of ion incidence on sputtering yields. The sample holder is positioned into the plasma chamber on the source centerline (at 0.3 meter from the source) in order to limit the influence of the ion-beam dispersion on the yield.

The ion current density collected by each sample is evaluated in several steps : during a first step of calibration, planar Langmuir probes are directly positioned at sample locations on sample holder. Other Langmuir probes are located on each side of the sample holder and I-V characteristics are performed on each probe. An extrapolation is then made from ion current collected by the side probes during a sputtering experiment. The result provides the ion current collected by each sample taking into account the estimated potential of the ceramic samples (floating potential) and the presence of slow ions resulting from charge exchange between fast ions and residual neutrals (ref 2). The planar geometry of probes limits thus the

slow ion collection, which are not involved in the sputtering process.

Sputtering yield :

The sputtering yield thus obtained is measured as a function of the ion-beam incidence on the sample and the energy of xenon ions.

The results are presented on figures 1 and 2. As a comparison, the erosion yields measured on pyrolytic BN and SiO_2 (glass based on 99% silica) are also displayed on figure 2. This comparison indicates that BNSiO_2 erosion is governed by the element more resistant to impingement of Xe^+ ions ; the SiO_2 presence results in a slight increase of the global sputtering yield of the material as compared to BN.

Another observation is that the influence of the incidence angle is not very pronounced for BNSiO_2 and BN. This can be due to a strong surface roughness (of the order of $1 \mu\text{m}$) with a large amount of microscopic incidence angles. Concerning the SiO_2 , the surface state of this glass is rather smooth, with a roughness of the order of few nanometers.

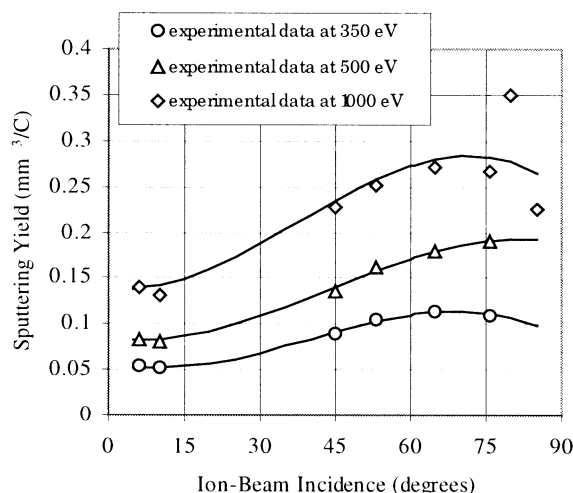


figure 1 : Sputtering yield of BNSiO_2 at 350, 500 and 1000 eV versus angle of ion incidence.

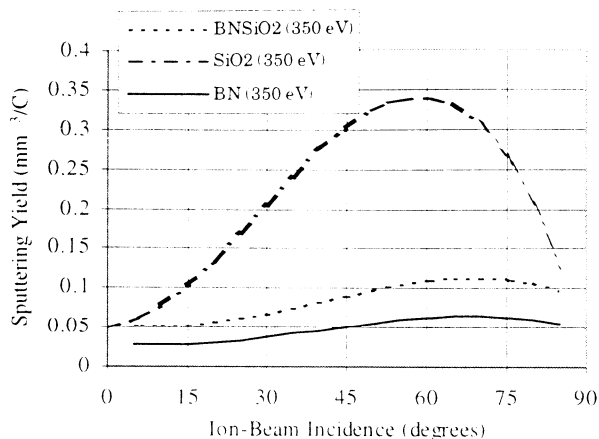


figure 2 : Sputtering yield of BNSiO₂, BN and SiO₂ at 350 eV, versus angle of ion incidence.

Surface analysis :

Surface characterization has been performed by ESCA, SIMS, SEM and non contact optically based measurements of surface roughness. These techniques gave data about stoichiometry, structure, surface morphology and surface profile.

	before erosion	after 83h
B	38%	17%
N	35%	14%
Si	9%	23%
O	18%	46%

table 1 : Elementary atomic composition before and after sputtering of the BNSiO₂

For example, table 1 gives values of percentages of each component detected on the surface of a non-eroded sample and of a sample exposed to 83 hours of sputtering under various incidences and various energy ions obtained by ESCA.

We first observe the equivalence between the values of B and N and also the fact that there is two atoms of oxygen for one atom of silicon. This correlation is maintained even after erosion. Nevertheless, the surface composition

changes totally after erosion since the silica, detected in increased proportion, becomes preponderant.

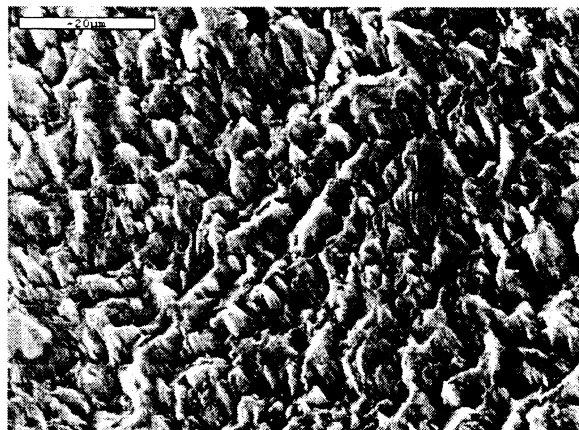


figure 3 : SEM image obtained with secondary electrons of BNSiO₂ surface after 83h of erosion.

The image obtained by Scanning Electron Microscopy (SEM) gives an idea of the surface topography after erosion. The surface indeed presents a pronounced roughness with the development of facets and conelike structures made of SiO₂ and containing BN at the top. The BN might be in the form of grains eroded less easily than the surrounded matrix of silica in whom they are included.

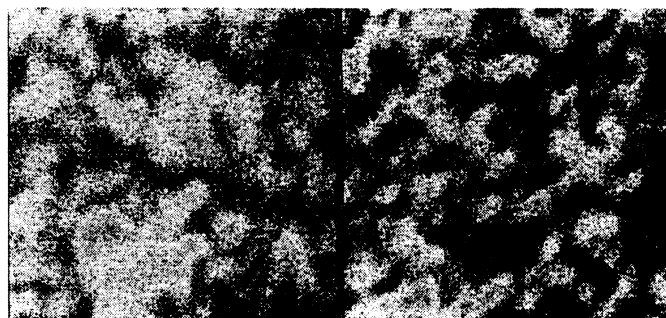


figure 4 : SIMS image of BNSiO₂ surface after 83h of erosion (B on l.h.s, Si on r.h.s.)

Figure 4 shows images obtained by SIMS (with incident ions O_2^+) on the surface of the sample after erosion. The first one presents the area where boron ions have been detected whereas the second picture concerns silicon areas. This result confirms the observation made by ESCA, i.e. the material is made of two separate components : BN and SiO_2 .

We saw on figures 1 and 2 that the sputtering yield of $BNSiO_2$ is governed by the erosion of the component more resistant to sputtering (BN). This "selective erosion" should thus induce an impoverishment in SiO_2 of the surface which is the opposite of the observation. The modification of the surface stoichiometry leads us to think that redeposition effects due to important microscopic slopes in the surface profile as seen on figure 3, like BN grain removals under Xe^+ bombardment might also occur as additional physical mechanisms.

Numerical simulation :

The erosion of this kind of multi-component ceramic was then interpreted through numerical simulations (figure 5). They consist in simulating BN grains (with a grain size of $10\ \mu m$) in a silica matrix with different erosion yield for each component. The initial surface relief is chosen flat, and evolves in consequence of erosion. Some topographic features are then developed during xenon ion bombardment, since the two components are not eroded at the same rate.

The surface after erosion obtained by numerical simulation presents irregularities similar to what was observed in SEM, with the less eroded component (BN) at the top of hillocks. The consequence of the differential erosion, is the development of surface features with strong microscopic slopes contributing to an important surface roughness. It is also observed that BN dictates the surface relief

evolution, slowing down the erosion process as early as a grain begins to be eroded. Moreover, the computed sputtering yield with respect to the ion-beam incidence corresponds to the experimental one (figure 6).

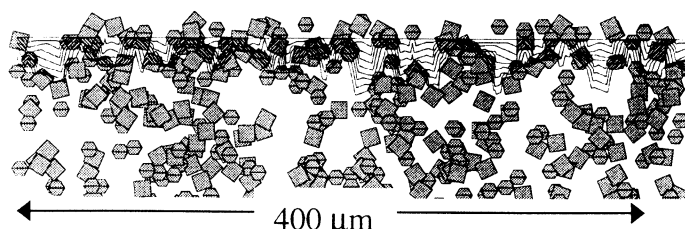


figure 5 : Simulation of $BNSiO_2$ erosion under Xe^+ bombardment at 350 eV, with the surface relief evolution during erosion.

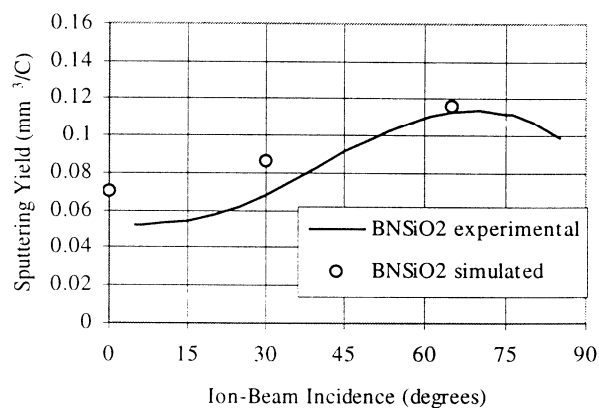


figure 6 : Experimental and simulated sputtering yield of $BNSiO_2$, at 350 eV, versus angle of ion incidence.

RF magnetron discharge

Another method was then used to determine erosion products on the ceramic. A RF 13.56 MHz magnetron apparatus is used to study the erosion of different ceramic and particularly $BNSiO_2$. The experimental set-up is presented on figure 7. The typical working conditions which are used for the magnetron discharge are the following : a pressure ranging from 5 to 100 mTorr and a discharge power

injected in the discharge and tuning circuit from 10 to 100W.

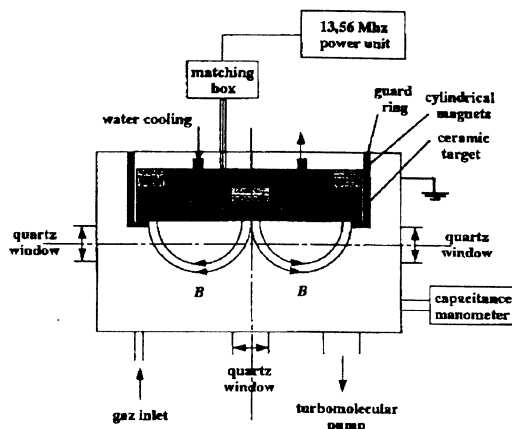


figure 7 : Schematic diagram of the RF magnetron apparatus.

The emission of the plasma is analyzed by a $f=150$ cm (THR Jobin Yvon) monochromator, a photomultiplier and a photocounting system in the visible and near-ultra-violet spectral range. An example of spectrum is reported in figure 8 for an Argon discharge (80mT, $P=100$ W) and a $BNSiO_2$ target. The resonant transitions of Boron ($^2P-^2S$), silicon ($^3P-^3D$) but also of the SiO ($A^1\Sigma - X^1\Sigma$) radical are observed between 220 and 250 nm. The reactions leading to this emission can be explained by a 2 step process : first, the boron and silicon atoms are ejected from the $BNSiO_2$ target by the Argon or Xenon ions accelerated through the sheath; secondly, the atoms are excited by electron collision in the plasma. The investigation of the emission when the discharge is pulsed, show that the temporal behavior of SiO is similar to the one of Si and B atoms. From this observation, it can be deduced that the molecules of SiO (or SiO_2) are directly ejected from the target and not formed by recombination of Si and O atoms in the volume.

In figure 9 are represented the variations of B, Si and SiO intensities versus RF power. On the same graph is reported the variation of the erosion rate. For one RF power, the erosion rate was

determined by measuring the erosion profile of the target after 10 hours. It can be seen that the erosion rate is proportional in these experimental conditions, to the intensities of B, Si and SiO lines. The optical emission spectroscopy OES can then be used as an real-time, in-situ, non-perturbing diagnostic of the ceramics sputtering.

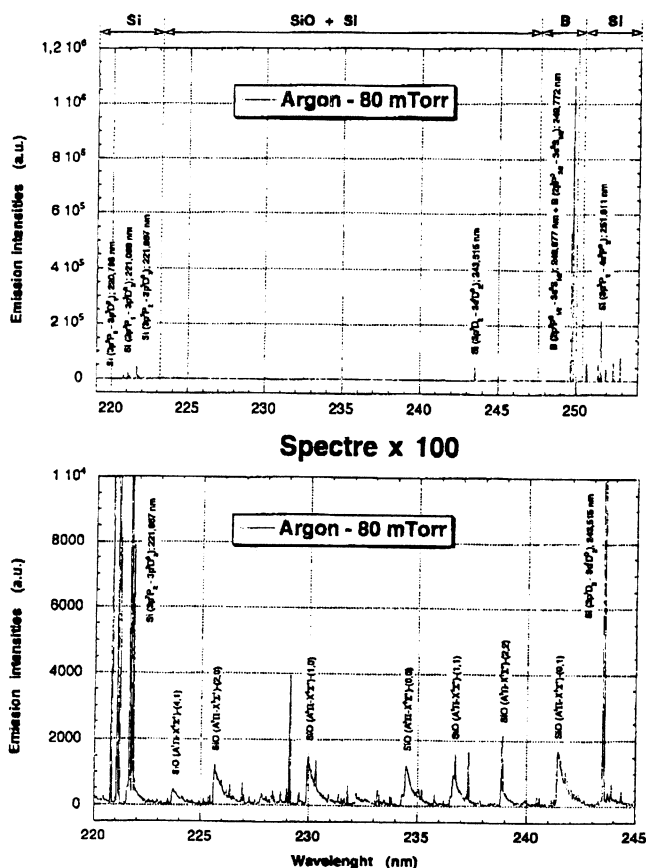


figure 8 : Emission spectra of the RF magnetron discharge Ar^+ ions – pressure 80 mTorr

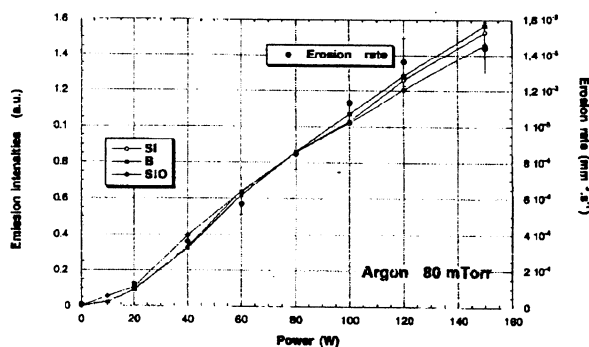


figure 9 : Correlation between the emission of the sputtered atoms/radicals and the erosion rate.

Conclusion :

A numerical model of multi-component ceramic erosion has been developed. The major experimental observations made on eroded samples are founded again. This model allows us to understand at best the erosion process of this kind of ceramic, made of two components which are differently eroded. This selective erosion leads to a strong surface roughness, which may have an influence on thruster operation.

Erosion products of the ceramic were observed in RF magnetron discharge. They can be involved in contamination of spacecraft equipments. This OES technique was used to study the sputtering of boron nitride based ceramics of an A3 type plasma thruster (ref 4). More recently, the spare-time evolution of the boron atoms sputtered from the ceramics of a SPT 100-ML (ref 3, 5) was studied by measuring the boron emission with a set of 16 optical fibers inside the channel of the thruster.

References :

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