A Novel Measurement of Net Erosion and Plasma-Material Interaction in Plasma Thrusters

by

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B.S., Aeronautical and Astronautical Engineering, Purdue University (2014)

Submitted to the Department of Aeronautics and Astronautics in partial fulfillment of the requirements for the degree of Master of Science in Aeronautics and Astronautics at the MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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Abstract

The lifetime of plasma thrusters is usually limited not by depletion of the propellant supply, but by erosion of the magnetic circuit from plasma particles bombarding the walls of the acceleration channel leading eventually to catastrophic engine failure. As erosion is the primary failure mode of these systems, understanding the erosion physics, plasma-material interactions (PMI), and the relationship between them is of utmost importance for extending the lifetime of high power and other next generation engines. A new measurement technique has been developed that uses lithium depth markers implanted in a thruster wall material. Nuclear Reaction Analysis (NRA) on the implanted material gives net erosion measurements while simultaneous Rutherford Backscattering Spectroscopy (RBS) quantifies the plasma retention and contamination in the surface layers. This insight into the surface composition and PMI is one of the major advantages of this technique when compared to other measurement methods. As an initial assessment of this technique’s applicability for plasma thrusters, it has been applied to samples of Boron Nitride exposed to the plume of a Xenon fueled BHT-200 Hall thruster and the results are presented.

Thesis Supervisor: Dennis G. Whyte
Title: Professor and Head, Nuclear Science and Engineering
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Finally, shout-out to all of my friends that I’ve met along the way from Purdue, MIT, Tokyo, and everywhere else. Thanks for the great memories and I hope to make many more in the future with all of you.
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Chapter 1

Introduction

Electric propulsion is a type of propulsion that utilizes electric power as the sole or a supporting means of producing thrust. Compared with the typical chemical powered propulsion, electric propulsion has better performance in terms of the specific impulse, allowing the same amount of propellant to give a larger change in impulse to the vehicle. It is also capable of smaller impulses, making it ideal for a spacecraft’s pointing requirements. However, it produces orders of magnitude less thrust than chemical propulsion and is therefore only viable in space for station-keeping, orbit-raising, interplanetary trajectories, and other low thrust maneuvers. For an idea of scale, the Space Shuttle Main Engine running off of liquid hydrogen and oxygen produces 2.3 MN of thrust in a vacuum with a specific impulse of 450 s [1] while a typical gridded ion thruster has a thrust of around 20 mN, which is comparable to the weight of a penny, and a specific impulse of 2400 s [2]. But of course there are a variety of electric propulsion devices with varying performance and popularity.

Electric propulsion is classified into three types depending on how they utilize their electric power: electrothermal, electrostatic, and electromagnetic. Electrothermal systems enhance chemical propulsion devices by adding additional energy to the propellant flow through electric means. Common examples are resistojets, where an otherwise typical chemical thruster has a resistor dissipating heat and adding energy to the propellant flow, and arcjets, where the flow passes over an extremely hot electric arc and is heated. Electrostatic thrusters, as their name implies, accelerate charged
particles electrostatically. The two most popular examples of these are gridded ion engines, where ions are accelerated through a large potential difference between biased grids, and Hall thrusters, where ions are created by an applied magnetic field and accelerated out of a channel by a potential due to the Hall effect. The firing of a Hall thruster designed and built at the MIT Space Propulsion Lab (SPL) is shown in figure 1-1. Electromagnetic thrusters utilize the magnetic force to accelerate charged particles and thus have higher thrust densities, but are still in the research and development phase due to low efficiency. Two interesting examples are Pulsed Plasma Thrusters (PPT) or Magnetoplasmadynamic (MPD) Thrusters.

The research in this thesis focuses on applications to electrostatic thrusters because these engines have the largest specific impulses levels and are the most viable and promising options for most current space propulsion needs. More specifically, this research focuses on applications to SPT type Hall thrusters because of their simplicity, open design, and availability in university research. SPT type Hall thrusters are one of the two popular variants of Hall thrusters, the other being TAL type Hall thrusters. The primary difference is that TAL thrusters have a short acceleration region with conductive metal channel walls, while SPT thrusters have a long acceleration region with channel walls insulated by an appropriate material, like boron nitride (BN) [3] [4].

1.1 Motivation

Although they produces small thrust, electric propulsion systems are are able to impart a large impulse to a vehicle and change its velocity, a parameter called $\Delta V$, by firing continuously for long periods of times, over 10,000 hours for many thrusters. The lifetime of these thrusters is usually limited not by the failure of an electronic component or the depletion of the propellant supply, but by the erosion of critical components due primarily to charged particle impacts. For gridded ion thrusters, the components limited by erosion are the accelerating grids and the discharge and neutralizer cathodes [5]. Hall thruster design is concerned about the erosion of the
channel walls that protect the critical magnetic circuit components. Once the magnetic circuit has begun to erode and alter the magnetic field topology, the thruster is said to have reached a "soft failure" and drops in performance before eventually being unable to fire at all. Erosion also sputters high atomic number (Z) material that is ejected into the plume, eroding spacecraft components that are hit by the plume and generating deposits that undesirably alter spacecraft surface properties [6].

Due to the fact that erosion is the primary life limiting factor of plasma thrusters and can have considerable effect on the operation of a spacecraft, it warrants substantial research to understand how to mitigate the effects. Most designs attempt to mitigate erosion through material selection by choosing materials with low sputtering yields but also possessing good heat conduction to allow for higher power operation and high strength to survive launch vibrations. Recent work in Hall thruster design has focused on mitigating erosion (to the degree that they are essentially erosion-free) through clever magnetic circuit design that reduces the two main causes of erosion, the ion flux to the walls and energy of the ions near the wall [7], through a technique called magnetic shielding [8] [9] [10]. This promising zero-erosion development allows
for interesting design configurations that were not previously possible that improve performance or reduce the cost of a thruster by replacing insulating, expensive BN rings with conducting graphite, for example, that allows for higher power operation from the better heat transfer properties [11]. In the case of gridded ion thrusters, however, there is not currently an erosion-free thruster design and the lifetimes are determined primarily by the operating plasma temperature and density conditions.

Furthermore, there are a few trends in the space industry that have direct effects on electric propulsion and are guiding the future of the field. Recent improvements in power generation allows for higher power operation of propulsion systems. Also, there has also been a surge in popularity of nanosatellites and microsatellites, so a lot of research and development in electric propulsion has been put into scaling down thrusters. For Hall thrusters, higher power operation and scaling down thrusters require higher temperature and density plasmas, magnifying the causes of erosion [12] [13]. Furthermore, the same gridded ion thruster’s lifetime, for example, drops by a factor of about 4.5 just by operating at a power level that is just 2 times higher [2].

Due to the advances in erosion mitigating designs and the trends towards higher power and more destructive operating conditions, there must also be improvements in validating these erosion mitigating designs through accurate erosion measurements.

1.2 Overview of Erosion Measurement Methods

Erosion is handled through either physical testing or simulations. A variety of erosion and sputter yield measurement methods are available including weight loss [14], coordinate measuring [10], quartz crystal microbalance [15], radioactive tracers, mass spectrometry, and rutherford back scattering. Optical methods are also popular and include optical emission spectroscopy [16] [17] [18] [19], laser induced fluorescence [6], laser absorption spectroscopy [20], image reconstruction techniques [21], and cavity ring-down spectroscopy [22]. A useful erosion measurement method is one that is cheap and robust while having quick, accurate, spatial resolution; in addition, it
should be independent of surface diffusion and be noninvasive to preserve the bulk material properties of the original target material and not alter the plasma-material interactions to ensure trustable measurements under realistic erosion conditions. Additional positives would be the ability to distinguish between erosion and deposition while tracking any contaminants on the surface.

The cost of full lifetime test firings are expensive due to the fact that thrusters have to fire for thousands of hours while consuming expensive Xenon and other consumables like liquid nitrogen for cryogenic pumps. For this reason, a lot of effort has been put into improving thruster simulation codes like HPHall and HPHall-2 from MIT or Hall2De from JPL by adding in erosion models to eliminate the need for full lifetime testing [23] [24] [25] [26] [27] [28]. These models are still being improved upon by better modeling the physics and by using improved sputter yield data for more specific types of BN and in the low energy range of interest for sputtering from plasma particles in Hall thrusters [29] [30]. Thruster simulation codes are cheap, quick, and have recently been able to approximate thruster performance with a high degree of accuracy. In order to validate these erosion models in the first place, however, accurate physical erosion measurements are needed.

One example of how critical it is to maintain the material properties of the target is demonstrated with the “multilayer coating method.” This method creates a material with different layers of thruster material like BN with thin layers of metals, like silver, interspersed through the bulk ceramic acting as depth markers [31]. As the BN erodes and exposes the silver, spectroscopy is used to measure the emission of the metal particles and an erosion rate is inferred by measuring the frequency of these emission peaks. While this method has quick resolution times and provides spatial resolution capability, the multilayered coated chips have different material properties than bulk BN and can lead to erosion rates atleast 4 times higher than in normal Hall thruster operation.
1.3 Outline of Research

This research is a joint project between the Plasma Science and Fusion Center (PSFC) and the Space Propulsion Laboratory (SPL) at MIT due to importance of erosion and plasma-material interaction (PMI) in both fusion and electric propulsion applications. SPL has vacuum facilities capable of testing a variety of electric propulsion devices, and PSFC has the Cambridge Lab for Accelerator and Surface Sciences (CLASS) which is capable of performing the required erosion and PMI diagnostics. The research presented in this thesis builds onto previous work developing and validating this lithium depth marker erosion and PMI measurement technique described in detail in section 3 for aluminum and tungsten samples [32] by extending the method for eventual use in Hall thrusters by analyzing samples of BN, which is the most common insulating material used in Hall thruster acceleration channels. The BN studied in this research is Boric Oxide (BO) grade BN, which is not normally used but has similar material properties as the grades used in Hall thrusters.

This work started with samples of BN exposed to the plume of a Xenon fueled Busek BHT-200 Hall Thruster. The net erosion in these samples was measured using an NRA spectra and the corresponding RBS spectra showed a high Z contamination after plasma exposure. Due to proton RBS’s limited atomic resolution, other methods to identify the high Z contaminant were used such as X-ray Photoelectric Spectroscopy (XPS) and heavy ion RBS. XPS gave atomic composition measurements in the first few nanometers of the surface of the samples. No high Z contaminant was detected which indicates the mystery element lodged itself deeper in the material than XPS can detect; however, XPS did show a significant enhancement of carbon on the surface of samples from ion irradiation during lithium implantation and the following proton irradiations for NRA and RBS characterization. Various samples of BN and other materials irradiated by ions under different conditions were analyzed by XPS to determine the cause of the unforeseen carbon enhancement. Heavy ion RBS with helium ions was used on one of the plasma exposed samples to provide better atomic resolution to distinguish between some of the possible high Z contaminants.
1.3.1 CLASS Facilities

This research was mostly carried out at the CLASS lab in PSFC. This facility has sputter and RF plasma ion sources connected to a 2 MeV accelerator that has two possible pathways to direct the ion beam. One is to a sample target chamber for ordinary implantation and irradiation experiments, and the other is to the Dynamics of ION Implantation and Sputtering Of Surfaces (DIONISOS) experiment which allows for simultaneous plasma exposure and beam analyses. Some sections of the beamline are shown in figures 1-2, 1-3, 1-4, and 1-5.

Figure 1-2: Helium plasma from the RF Plasma Source for alpha particle irradiation

Figure 1-3: Beamline for normal irradiations
Figure 1-4: DIONISOS

Figure 1-5: A 2 MeV particle accelerator
Chapter 2

Relevant Physics

2.1 Sputtering

Sputtering is a process wherein surface atoms are ejected from a surface due to energetic particle impacts. The incoming particles collide with the surface and either backscatter or imbed themselves into the material, where they then become trapped or desorb from the surface. Although there are many possible types of incident particles, ion bombardment is the primary cause of erosion in plasma thrusters. Other than erosion of the surface, ions also cause emission of electrons and photons and cause radiation damage that changes the surface topography [33]. In the incident energy range of interest for plasma thrusters in the low eV to keV range, sputtering is due mainly to incident ions transferring energy to the surface particles who are either ejected immediately in the "single knockon regime" or continue to collide with other neighboring particles in the "linear cascade regime". If the energy is transfered to one of the surface particles and the energy normal to the material's surface is greater than the surface binding energy, the atom will be ejected. This process is called physical sputtering, in comparison to chemical sputtering where incident ions form a chemical bond with the target material and can either increase or decrease sputtering. The sputtering threshold is the minimum energy of incident particles that causes sputtering, usually between 10 and a few 100 eV.

Erosion is calculated with the sputtering yield, a variable representing the average
number of sputtered atoms per incident particle. Another important parameter for plasma thrusters is the secondary emission yield, defined as the average number of electrons emitted per incident ion; larger secondary electron emission (SEE) reduces the average electron temperature of a plasma and reduces the performance [34]. The sputter yield is dependent upon the energy of the incident particle, the angle of impact, and the masses of the target and incident atoms. Sputter yields for atomic targets are well defined, but those for multi-atomic targets, like BN, are still under investigation [23]. Sputtering yield will increase with incident energies above the threshold energy before reaching a maximum and decreasing because higher energy collisions will penetrate deeper into the surface and will not transfer as much energy to the surface layers. In addition, if particles impact normal to the surface, they will also penetrate deeper while particles that impact almost parallel to the surface are either repelled by surface forces or do not transfer energy efficiently to the surface, so there is an incident angle around 50 to 80 degrees where sputter yield is a maximum. In multi-atomic targets, certain atoms are preferentially sputtered causing surface roughness [35]. For this reason, sputter yield for multi-atomic materials are usually given in units of $mm^3/C$ representing a volume of sputtered material per incident ion. The grade and type of target BN also affect the sputter yield as they are formed in different crystal structures or contain different binders [36].

The details of the sputter yield are important for erosion simulations, but for experimental erosion measurements it is just important to know that sputter yield and thus erosion depends on the type of target material and the type and energy of incident particles. So in plasma thrusters, erosion would depend on the plasma facing surface material and the temperature, density, and type of plasma (usually Xenon or Argon).

### 2.2 Plasma Sheath

Compared to the behavior of a plasma in free space, the plasma near a wall must satisfy the wall boundary conditions. The region near the wall where plasma
conditions change from the free space to the wall conditions is called the "sheath."

For a wall of insulating material enclosing a plasma, electrons will arrive to the wall quicker than ions and build up an excess of negative charge on the wall [37]. This potential then repels low energy electrons and attracts all ions, accelerating them to the wall across this potential drop. Therefore, ions impacting the walls in plasma thrusters have the energy from the plasma, but also additional energy from being accelerated by the sheath before impacting the wall. The potential drop is given by $\phi$ in equation 2.1, where $k$ is Boltzmann’s constant, $T_e$ is the plasma’s electron temperature, $e$ is the elementary charge, $m_i$ is the ion mass and $m_e$ is the electron mass [38].

$$\phi = \frac{kT_e}{e} \ln\left[\sqrt{\frac{2m_i}{\pi m_e}}\right]$$  \hspace{1cm} (2.1)

This equation shows that high temperature, heavy ion plasmas will have higher energy collisions to walls and most likely increase erosion. The concept of magnetic shielding discussed in section 1 aims to reduce the electron temperature near the walls, reducing the energy of ions impacting the wall to around the sputtering threshold energy limit.
Chapter 3

Lithium Depth-Marker Technique

3.1 Summary

The general version of this technique relies on an implanted species to act as a depth marker by providing a means for a high energy nuclear reaction whose resulting photon’s or particle’s energy can be measured. This physical process will work for any material and for any implanted species with relevant nuclear reactions that produce particles of sufficient energy to pass through the bulk material. The methodology and validation of the technique using implanted lithium on aluminum and tungsten samples is described in reference [32]. Lithium is used for a few specific reasons: its nuclear reaction with protons has a large cross-section for quick resolution times and also produces two high energy alpha particles (∼9 MeV) that can be easily distinguished from lower energy backscattered particles and reactions. Also, lithium has a low atomic number, so it produces very few vacancies when implanted into a target material and thus retains the bulk material’s properties which is one important requirement for realistic erosion measurements.

Proton irradiation of the lithium implanted target provides the ability to perform nuclear reaction analysis (NRA) and rutherford backscattering spectroscopy (RBS) simultaneously. Through NRA, the energy peak of the high energy nuclear reaction can be compared before and after plasma exposure and the shift in this peak is proportional to the change in depth of the lithium, or net erosion. Through RBS,
the surface composition is monitored and any deposited materials or contaminants are identified and their concentrations quantified. Another quality of this technique is that the proton beam can be scanned across target surface for spatial resolution measurements.

3.2 Lithium Implantation and Characterization

The required implantation energy is determined by how deep the lithium should be implanted to be far enough away from the surface to not be eroded away, but not so deep that the resulting high energy particles cannot be distinguished from lower energy particles. The Stopping and Range of Ions in Solids ("SRIM") program and its included Transport of Ions in Matter ("TRIM") program was used to simulate a beam of lithium being implanted into a BN target with mean density $2.1 \text{g cm}^{-3}$. The actual density of BN depends on the type of BN used for implantation, but this value is representative of a general BN and is sufficient for initial validation of this technique on BN samples.

The simulated range distribution of 5000 3300 keV lithium ions being implanted into this BN target is shown in figure 3-1.

The mean range is 6.25 microns deep with a roughly gaussian distribution. The concentration of lithium ions in the layers of the target at the implanted depth is determined by the beam fluence or can be fit from the experimental NRA spectra. The depth of the implanted layers controls the energy of the detected high-energy particles and the concentration of the implanted lithium controls the number of NRA counts for a given fluence. For the lithium implantations carried out in these experiments, the lithium beam was rastered over the entire sample surface to maintain constant lithium concentration for spatial resolution from the ensuing proton irradiation. The effects of lithium implantation on a sample are shown in figure 3-2. Lithium implantation darkens the material, except the areas where clips held the sample.
Figure 3-1: Range distribution of 3300 keV lithium ions implanted into a 50-50 split of BN with a density of $2.1 \text{ g cm}^{-3}$.

Figure 3-2: A BN sample after plasma exposure. The dark color is still present because the lithium was implanted deep enough to not be eroded.
3.3 Experimental Setup

The NRA and RBS spectra from proton irradiation of the lithium implanted samples are obtained from two silicon detectors. The NRA detector has maximal surface size, as the cross-section for the lithium-proton nuclear reaction is the limiting factor on resolution time and has a thin, 25 micron aluminum foil placed over the face to screen out the lower energy back scattered particles and only allow the high energy nuclear particles through. The RBS detector is of the same type, but has a thick piece of aluminum foil with a small slit to reduce the flux of detected backscattered particles and reduce pulse pileup.

The resolution of the peak from the NRA data determines the resolution of the net erosion measurement. There are a few factors that affect the energy straggling of the peak. One is that particles arrive to the detector from different solid angles, so each passes through different amounts of BN and aluminum foil before being detected. Another cause is from thickness variations in the aluminum foil. This thickness variation is about 10%, so particles passing through the 19.5 $\mu$m foil may pass through any thickness between 17.55 and 21.45 $\mu$m. Another source is from the range distribution of the lithium shown previously in figure 3-1 that causes the nuclear particles to be born at different depths in the target, causing detection of different energy particles from the same nuclear reaction.

The samples were set up so that the beam was normal to the target surface, and the detector geometry was set so that the scattering angle was 150 degrees and the exit angle was 30 degrees. This is another parameter that can be validated by the simNRA software, discussed in the next section.

3.4 NRA and RBS Spectra Fitting

After implanting, the depth of the lithium and surface makeup can be characterized by irradiating the sample with protons and analyzing the resulting NRA and RBS spectra. A 1200 keV beam of protons is used for NRA and RBS analysis be-
cause this energy corresponds to the maximum in the nuclear cross-section for quicker resolution times.

The experimental spectra data are compared with simulated spectra through a program called “simNRA” developed by the Max-Planck-Institut für Plasmaphysik. The simulated data is created by defining an experimental setup including the type of beam, beam energy, detector geometry, detector resolution, and other parameters. The sample target is created by patching together multiple layers that can have separate thicknesses and atomic compositions, allowing the user, for example, to define a target composed of thin surface layers that may contain contaminants over a bulk material interspersed with thin layers of lithium at a specific depth. The program also allows for defining the RBS and other non-RBS, like nuclear reaction, cross-sections for each element in the target. The relationship between simulated and experimental data of course validates the experimental setup, but more importantly, it provides the ability to model the composition and thickness of different layers in the target sample before and after plasma exposure, providing a net erosion measurement from the NRA fits and quantifies the plasma-material interaction by quantifying surface concentrations of contaminants or imbedded plasma ions.
Chapter 4

Applying Technique to Plume-Exposed Samples

4.1 NRA

The first application of this technique to a plasma thruster was placing lithium implanted samples of BN in the plume of a Xenon fueled BHT-200 Hall Thruster. While the plume environment is obviously less intense than the environment of concern inside the Hall thruster, this type of exposure nevertheless allows the method to be validated for materials and plasma energies of interest for Hall thrusters. Three different samples were exposed to the plume, from here on out referred to as BN1, BN2, and BN3. BN1 was implanted for 250 minutes at 4.35 MeV for a lithium depth of \( \sim 7.35 \) microns. BN2 and BN3 were implanted at 3.3 MeV for a lithium depth of \( \sim 6.25 \) microns. BN1 was exposed to the plume for 1.5 hours at 30.7 cm from the thruster exit, while BN2 was exposed for 15 minutes and BN3 for 2 hours at 15.7 cm from the thruster exit. The exact details of this plume exposure are discussed in reference [39].

The detectors output number of counts per channel, and each channel corresponds to a certain energy of the detected particle depending on the amplifier settings. Amplifier setting is chosen for maximum energy resolution for each analysis. The energy per channel is different for NRA and RBS due to the foils, and depends on the type of
experiment as the energy of backscattered particles depends on the energy and mass of the beam, so an energy calibration is needed for each different experiment.

For NRA, the energy calibration was obtained by placing thin aluminum foils of varying thicknesses over different areas of a detector. A polonium-210 source gives off constant 5.3 MeV alpha particles that are detected by the detector through either 19.5 microns, 12.5 microns, 7 microns of aluminum foil or detected directly without any material impeding the particles. The resulting spectra is shown in figure 4-1.

![Polonium Energy Calibration](image)

Figure 4-1: Detected alpha particles emitted from Polonium 210 source through various aluminum foil thicknesses

The four peaks shown in figure 4-1 along with the energies of these alpha particles passing through thin layers of aluminum calculated by TRIM yield the data shown in table 4.1. This data is used to make the quadratic energy calibration fit shown in equation 4.1 that is used as a parameter in simNRA to generate the simulated spectra to fit to the experimental spectra.

$$E = -212 \text{ keV} + 8.76 \text{ keV/channel} - 0.00230 \text{ keV/channel}^2$$  \hspace{1cm} (4.1)

The NRA spectra from the center of the BN1 sample before and after plasma exposure is shown in figure 4-2a, and the NRA spectra from the centers of the BN2 and BN3 samples before and after exposure are shown in figure 4-2b.
Table 4.1: Energy calibration data from polonium source detected through foils of varying thickness using an amplifier setting of 25

<table>
<thead>
<tr>
<th>Foil Thickness [μm]</th>
<th>Energy [keV]</th>
<th>Channel Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>19.5</td>
<td>1230</td>
<td>127</td>
</tr>
<tr>
<td>12.5</td>
<td>3040</td>
<td>332</td>
</tr>
<tr>
<td>7</td>
<td>4120</td>
<td>549</td>
</tr>
<tr>
<td>0</td>
<td>5300</td>
<td>699</td>
</tr>
</tbody>
</table>

Figure 4-2: Comparison of pre-exposure and post-exposure NRA data at the center of BN1, BN2, and BN3

The BN1 sample was implanted under different conditions so the NRA spectra cannot be compared directly against the BN2 and BN3 samples. The pre-exposure spectra for BN1 has smaller counts, so although the statistics are not as strong as in the other spectra, it is still possible to compare the peaks with larger error; however, there was no measurable erosion, which is why the BN2 and BN3 samples were placed closer to the thruster exit during exposure.

The pre-exposure NRA spectra is essentially the same for the BN2 and BN3 samples as they were both implanted under the same conditions and to the same depth, so they can be compared against each other to see how exposure time affects net erosion. For the shorter exposrer time with the BN2 sample, there is a small but
noticeable shift in the peak heights. This shift is much more pronounced for the longer exposed BN3 sample, indicating more erosion for BN3 than BN2.

For a quantitative net erosion value, the simulated spectra must be compared before and after plasma exposure using simNRA. The shift in peak height is achieved by modifying the depth of the lithium in the target while keeping all other parameters the same. An example of a fit to NRA data is shown in figure 4-3, which is the fit for the center spot of the BN3 sample after exposure with experimental data in red and the simulated data in blue.

![Figure 4-3: BN3 post-exposure simulated NRA spectra fit with experimentally obtained spectra](image)

The spatial resolution of the technique was tested by characterizing different areas of the sample surface by positioning the proton beam at different locations. The differences in energy peaks after plasma exposure for different locations on the BN1, BN2, and BN3 samples are shown in figures 4-4a, 4-4b, and 4-4c, respectively.

The slight differences in peak position for the different locations indicates that there are differences in the net erosion at each location. The quantitative erosion values at different locations on the BN2 and BN3 samples inferred from simNRA before and after plasma exposure are shown in table 4.2.
Figure 4-4: Comparison of post-exposure spatial NRA data for BN1, BN2, and BN3

Table 4.2: Net erosion at different locations on the BN2 and BN3 sample due to exposure to a plasma plume.

<table>
<thead>
<tr>
<th>Net Erosion [nm]</th>
<th>BN2</th>
<th>BN3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Center</td>
<td>230</td>
<td>1380</td>
</tr>
<tr>
<td>y+3 mm</td>
<td>598</td>
<td>1520</td>
</tr>
<tr>
<td>y-3 mm</td>
<td>230</td>
<td>1200</td>
</tr>
<tr>
<td>x+3 mm</td>
<td>322</td>
<td>1290</td>
</tr>
<tr>
<td>x-2 mm</td>
<td>138</td>
<td>736</td>
</tr>
</tbody>
</table>
BN2, which was exposed to the plume for 15 minutes, shows lower net erosion
than BN3, which was exposed for 2 hours. The erosion for BN3 is about a factor of 4
or 5 larger even though the exposure time was 8 times longer, showing that erosion is
a dynamic process that may be nonlinear with time. To confirm the nonlinearity, at
least one more data point from a new sample would be needed. Also, the magnitude of
erosion is different across the face of a sample due to the different plasma fluxes seen
by different locations on the sample. The y+3 mm position obviously was exposed
to the most extreme fluxes, while the x-2 position was exposed to the least extreme
fluxes. The technique is successfully able to resolve spatial differences in erosion on
the face of a sample.

To verify that this measurement technique provides accurate erosion measure-
ments, it is good to validate the measured erosion with predicted erosion. For the
BHT-200 plume for the operating conditions used, the current density 15.7 cm down-
stream of the thruster exit on-axis was measured to be $100A/m^2$ [40]. Also, in the
near field plume just past the cathode exit plane, the average ion velocity and en-
ergy is 14,750 m/s and 186 eV [41]. Furthermore, at the operating condition, the ion
species fraction is 0.955 Xe1, 0.037 Xe, and 0.008 Xe3 down the centerline [42], but
the current density accounts for all charged species of xenon already. These values
yield a relation for erosion given by equation 4.2, where $Y_{\text{normal}}$ is the normal sputter
yield, $\Delta h$ is the net erosion, and $t$ is the exposure time.

$$\Delta h [mm] = Y_{\text{normal}} [mm^3/C] \times 100 [A/m^2] \times t [s]$$ (4.2)

Using the measured erosion values and exposure times and rearranging using con-
sistent units, the calculated sputter yield required is given in table 4.3.
Table 4.3: Calculated sputter yield using equation 4.2 and measured erosion values from table 4.2.

<table>
<thead>
<tr>
<th>$Y_{normal} [mm^3/C]$</th>
<th>BN2</th>
<th>BN3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Center</td>
<td>0.0026</td>
<td>0.0019</td>
</tr>
<tr>
<td>y+3 mm</td>
<td>0.0066</td>
<td>0.0021</td>
</tr>
<tr>
<td>y-3 mm</td>
<td>0.0026</td>
<td>0.0017</td>
</tr>
<tr>
<td>x+3 mm</td>
<td>0.0036</td>
<td>0.0018</td>
</tr>
<tr>
<td>x-2 mm</td>
<td>0.0015</td>
<td>0.0010</td>
</tr>
</tbody>
</table>

Sputter yield data on BN for low eV xenon ion bombardment shows the normal sputter yield to be somewhere between 0.001 and 0.05 $mm^3/C$ at ion energies around 190 eV [23] [43]. The erosion measured from this technique, then, lies within the range of sputter yield data found in literature and differences may be due to the grade of BN used or errors in the NRA energy calibration that can be fixed by using a more consistent calibration technique.

### 4.2 Proton RBS

During plasma exposure, heavy plasma ions are being accelerated by the plasma sheath to high energies and colliding with the plasma facing surfaces. Heavy plasma neutrals also collide with these surfaces, but at much lower energies that may be below the sputter threshold. The impact of these heavy plasma particles cause significant sputtering by kicking out surface atoms and sometimes burying themselves in the surface.

This plasma retention process has a significant effect on the erosion, as the sputtering mechanisms for like-like particle collisions (plasma on imbedded plasma) is different than for different mass particle collisions (plasma on surface material atoms). Understanding the dynamic plasma retention, i.e. the rate that the trapped plasma concentration changes during and not during active plasma exposure, gives essential
information for understanding erosion mechanisms. One possible explanation for the nonlinear erosion rate seen in the BN2 and BN3 samples is plasma retention. As the Xenon ions imbed themselves in the surface, they could reduce the average sputtering yield of the surface and shield the BN as they are preferentially sputtered over other atoms in the surface. BN2 had a shorter exposure time, so it was not able to build up a sufficient concentration of xenon in the surface layers, while the longer exposed BN3 sample could build up this protective layer.

Another energy calibration is needed for proton RBS as the spectra were obtained using a higher amplifier setting. Rather than using sources like was done for NRA, the proton RBS energy calibration was obtained from directly fitting the various features in simNRA. The resulting quadratic fit is given by equation 4.3

\[
E = -70 \text{keV} + 1.995 \text{keV/channel} - 0.00011 \text{keV/channel}^2 \tag{4.3}
\]

The RBS component of this method is used for monitoring surface elements and contaminants. Figure 4-5 illustrates the qualitative change in the RBS spectra before and after plasma exposure, and also between the samples that were exposed under different conditions.

![Pre and Post-Exposure Proton RBS](image)

Figure 4-5: Comparison of proton RBS data at centers of the BN1, BN2, and BN3 samples with pre-exposure baseline from the BN3 Sample
The primary difference in the spectra due to plasma exposure is the appearance of the high Z contaminant with the diffusion tail. This should be trapped xenon, but it is difficult to rule out other high Z elements from this low energy proton RBS. It was initially thought to be tungsten from the cathode being sputtered onto the sample, but other methods of confirming this element to be xenon were undertaken and are discussed further in later sections. Furthermore, although the sample face is eroding, any sputtered tungsten itself would not necessarily be eroded away by the xenon plasma as the other elements would be sputtered much more easily than tungsten would be.

Figures 4-6a and 4-6b show the post-exposure proton RBS data for the various locations on the BN2 and BN3 samples, respectively.

![BN2 Proton RBS Spatial Comparison](image1)

![BN3 Proton RBS Spatial Comparison](image2)

Figure 4-6: Comparison of post-exposure spatial proton RBS data for BN2 and BN3

For each location on the BN2 or BN3 samples, the xenon tails are just about the same. The BN3 sample exhibits similar characteristics to the BN2 sample, however the xenon diffusion tail is much broader and also has a higher peak, indicating that the xenon diffused deeper into the surface and also has a higher concentration. This higher diffusion is also supported by thermocouple measurements of the samples during exposure that measured higher temperatures for the BN3 sample. The quantitative concentrations of xenon were obtained by fitting simulated data with simNRA to the various RBS spectra and including small concentrations of xenon in the upper surface
layers. Example RBS fits for the center locations of the BN2 and BN3 samples from simNRA are shown in figures 4-7a and 4-7b, respectively.

Figure 4-7: Simulated proton RBS spectra fit with experimentally obtained spectra for center location of BN2 and BN3

The simulated data matches the experimental spectra fairly well except for over-shooting the boron signal and the hump on the boron plateau which may due to non-rutherford reactions. However, it can easily match the xenon tail to give the xenon concentration as a function of depth, which is illustrated for each of these samples and locations in figure 4-8.

Figure 4-8: Comparison of xenon retention in the surfaces of BN2 and BN3

BN3 has a maximum xenon concentration in the very top surface layer of around 2 atomic percent, and diffuses around 1.6 microns into the surface. BN2 has a lower
xenon surface concentration of about 0.7 atomic percent and only diffuses about 0.8 microns into the surface. This BN3 sample was then left alone for around 11 months as other parts of this research were investigated, and then reanalyzed with RBS to understand the passive xenon outgassing rate. The comparison of RBS spectra from two points on the BN3 sample after 11 months is shown in figure 4-9.

![BN3 Proton RBS Comparison, Time](image)

Figure 4-9: Comparison of proton RBS Data of same BN3 sample 11 months apart.

After 11 months sitting around, the xenon signal has decreased significantly in strength, indicating that it has outgassed out of the sample. This is a good indicator that this impurity is infact xenon as xenon does not bind easily with other elements and will readily outgas from a surface and eliminates the possibility of the impurity being tungsten, as tungsten would bind with the surface elements. Figure 4-10 compares representative xenon concentrations from the original analysis on BN2 and BN3 with this decreased signal xenon from 11 months of outgassing.

The sample now only has around 0.6 atomic percent at the surface and diffuses about 1.2 microns into the material, comparable to the conditions from the BN2 sample originally. RBS is successfully able to detect contaminants, but is also able to quantify the xenon retention in the surface that gives interesting insight into the plasma material interaction and gives the opportunity to investigate how plasma retention affects net erosion.
Figure 4-10: Comparison of xenon retention in the surface of the BN3 sample after 11 months with the original concentrations

4.3 Alpha Particle RBS

Heavy ion RBS was used on two of the plasma exposed samples to see if the increased resolution from heavy ions would increase confidence in the assertion of the high Z contaminant being xenon. The analysis was performed at two beam energies of 1200 keV and 3600 keV. The 1200 keV analysis is the same beam energy as our proton RBS, but did not resolve the xenon, so a 3600 keV analysis was performed. Figure 4-11a shows the alpha RBS spectrum for BN3 at 1200 keV beam energy while figure 4-11b shows the comparison between the alpha RBS spectra from BN1 and BN3 at 3600 keV beam energy. The missing peak in the high energy section of the BN1 spectra is the signal from xenon.
The energy calibration for these RBS fits was constructed from RBS performed on a few samples with well-defined surface layer thicknesses and elements. These samples included silicon with a thin chromium surface layer, a sample of silicon with a thin gold surface layer, and a sample of silicon with thin surface layers of sulfur, calcium and tin. These various spectra are shown in figure 4-12.

The energy of the backscattered particles is given by simNRA and the channel number corresponding to this energy is taken directly from figure 4-12. The energy
calibration data is shown in table 4.4 and the resulting quadratic energy fit is given by equation 4.4.

Table 4.4: Energy calibration data for use in heavy ion RBS fit for an amplifier setting of 50

<table>
<thead>
<tr>
<th>Element and Beam</th>
<th>Channel Number</th>
<th>Backscattered Energy [keV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr, 1200 keV Proton</td>
<td>287</td>
<td>1115</td>
</tr>
<tr>
<td>Au, 1200 keV Proton</td>
<td>303</td>
<td>1177</td>
</tr>
<tr>
<td>S, 3600 keV α</td>
<td>589</td>
<td>2300</td>
</tr>
<tr>
<td>Ca, 3600 keV α</td>
<td>652</td>
<td>2550</td>
</tr>
<tr>
<td>Cr, 3600 keV α</td>
<td>712</td>
<td>2700</td>
</tr>
<tr>
<td>Sn, 3600 keV α</td>
<td>844</td>
<td>3150</td>
</tr>
</tbody>
</table>

\[ E = -213\, keV + 4.95\, keV/channel - 0.00115\, keV/channel^2 \]  \hspace{1cm} (4.4)

An example of the alpha particle RBS fit is shown in figure 4-13, which is the fit for the BN3 sample for a 3600 keV beam.
The fit for this heavy ion RBS is extremely dependent upon the energy calibration, which is why so many data points were used to construct the calibration equation. However, while the fit matches the experimental data in the low Z region reasonably well, it fails at matching oxygen and other high Z elements, including the xenon which is the reason for performing heavy ion RBS in the first place. One explanation for why these heavy ion fits do not match the xenon peak is that there has been a buildup on the surface burying the xenon. An experiment to confirm this would be heavy ion RBS of a sample immediately following plume exposure and doing new fits for a situation where there is definitely no surface buildup. While heavy ion RBS does allow sufficient resolution to distinguish between xenon and other high Z elements like tungsten, the actual fit fails to confirm the identity of the high Z element. Nevertheless, this increased the confidence in the mystery element being xenon.
Chapter 5

Effects of Ion Irradiation on Surface

X-ray Photoelectric Spectroscopy (XPS) is a surface analysis instrument that measures atomic concentrations and chemical information in the first ~10 nanometers of a surface and distinguishes between atomic species well. The PHI Versaprobe II Scanning XPS Microprobe shown in figure 5-1 from MIT’s Center for Material Science and Engineering (CMSE) was used to try to confirm the identity of the high Z surface impurity of our xenon plume-exposed BN samples. The results, however, did not measure any high Z impurity, but instead showed a very high carbon concentration on the surface that returned to normal levels if the upper surface layers were sputtered away by an argon gun.

Three BN samples were initially analyzed with XPS: a plain baseline sample, a sample that had been implanted with lithium and characterized by proton irradiation, but not yet exposed to a plasma, and an implanted, characterized, and plasma exposed BN sample. Tables 5.1 and 5.2 present the results from XPS of these three samples before and after sputtering, respectively.

Table 5.1: Atomic concentrations on surface of BN samples pre-sputtering

<table>
<thead>
<tr>
<th>Atomic %</th>
<th>B</th>
<th>N</th>
<th>C</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline</td>
<td>44.6</td>
<td>40.4</td>
<td>10.3</td>
<td>4.6</td>
</tr>
<tr>
<td>Unexposed</td>
<td>16.7</td>
<td>12.7</td>
<td>57.4</td>
<td>12.7</td>
</tr>
<tr>
<td>Exposed</td>
<td>13.8</td>
<td>8.3</td>
<td>55.0</td>
<td>21.1</td>
</tr>
</tbody>
</table>
Table 5.2: Atomic concentrations on surface of BN samples after 14 minutes of sputtering from a 2 keV 2 $\mu$A argon gun

<table>
<thead>
<tr>
<th>Atomic %</th>
<th>B</th>
<th>N</th>
<th>C</th>
<th>O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline</td>
<td>47.9</td>
<td>37.7</td>
<td>3.2</td>
<td>9.6</td>
</tr>
<tr>
<td>Unexposed</td>
<td>45.8</td>
<td>34.9</td>
<td>5.5</td>
<td>12.5</td>
</tr>
<tr>
<td>Exposed</td>
<td>37.7</td>
<td>21.3</td>
<td>22.4</td>
<td>17.0</td>
</tr>
</tbody>
</table>

Ensuring that the surface is free of any contaminants before plasma exposure is critical for the method to accurately measure realistic plasma-material interactions. If there is a carbon rich surface layer because the measurement technique deposited it on the surface obscuring the clean BN surface, then this contaminated layer would be the first to be eroded by a plasma, but would give erosion measurements that are inherently false compared to the erosion environment in the real systems because of the sputtering yield's relationship with atomic composition. For this reason, the carbon enhancement issue must be solved for this method to be of any practical use.

There is a larger carbon enhancement on samples after the implantation step, so the carbon contamination must have been introduced through ion irradiation from either lithium implantation or proton irradiation. Further probing into this unfore-
seen phenomenon by irradiating new fresh samples with protons and performing the same XPS analyses to determine the atomic concentrations on the surface showed that proton irradiation is a source of carbon enhancement. Lithium implantation is also a source of the contamination and was confirmed from implanting a sample of molybdenum and analyzing it with XPS.

The proton irradiation on BN shows distinct discoloration of the entire irradiated surface area, with an even more distinct discoloration from the proton beam. The proton beam spot on BN is well defined and figure 5-2 shows a BN sample during proton irradiation.

![Figure 5-2: A BN sample lights up as it is irradiated by protons.](image)

Figure 5-3 shows the comparison of various irradiated samples with the backside of a sample; the entire sample face is darkened with extreme darkening on the beam spot. The darkening may be related to the carbon enhancement, which is one reason why it was not encountered during initial validation of this technique on aluminum and tungsten targets. This discoloration is not as noticeable on metal samples (figure 5-4), so there were no obvious signs of proton irradiation affecting the surface. Lithium implantation also darkens the surface, so any other discoloration after this implantation step would not be visible. Also the surface atomic compositions for these BN samples were initially carried out with the intention of identifying the xenon, so the team carrying out the previous validation did not accidentally stumble upon this contamination as they were not concerned with tracking the plasma retention after exposure. Furthermore, the darkening is present throughout the top layers of ion ir-
radiated samples. Figures 5-5a and 5-5b show the sample before and after 10 minutes of sputtering with an argon gun over a 2x2 mm area around analysis location 6.

Figure 5-3: Effects of proton irradiation on the discoloration of BN targets compared with the backside of a BN sample, which has the normal, bright white color.
Figure 5-4: Effect of alpha irradiation on the discoloration of an aluminum sample. No discernible discoloration outside of the beam spot.

Figure 5-5: BN surface before and after sputtering from a 2kV 2μ A Argon gun

Interestingly, the carbon concentration was enhanced over the entire irradiated surface area, not just on the beam spot. This carbon enhancement was even present behind the sample holder clips, although to a lesser degree. XPS analysis on the back side of one of the irradiated samples showed normal levels, indicating that the enhancement could not be purely because of the vacuum environment or vacuum
impurities. More samples irradiated by protons under a variety of conditions were analyzed with XPS to try to determine if there was any link between the enhancement and vacuum quality, beam fluence, beam irradiation time, or sample material.

Vacuum quality in the target chamber was improved by fixing small leaks and pressure in this section improved from the 5e-6 torr range ("High Pressure") to 1e-6 torr ("Low Pressure") and then to 6e-7 torr ("Super Low Pressure"). Samples with various irradiation times and beam fluences were analyzed. Also, samples of stainless steel, aluminum, and molybdenum were analyzed to investigate whether BN's porosity or other material properties were a cause. The molybdenum sample was implanted with lithium, but was not irradiated by protons. There were also samples of BN and aluminum that were irradiated with alpha particles rather than protons. For each sample, various locations on surface were analyzed with XPS such as on the beam spot, off of the beam spot, backside, and behind the sample holder clips for some samples. The resulting XPS spectras for representative BN, stainless steel, aluminum and molybdenum samples are shown in figures 5-6, 5-7, 5-8, and 5-9, respectively.

Figure 5-6: The XPS spectra of the BN sample irradiated for 60 minutes with 85 nA of proton current in the best vacuum conditions.
Figure 5-7: The XPS spectra of the stainless steel sample irradiated for 60 minutes with 85 nA of proton current in the best vacuum conditions.

Figure 5-8: The XPS spectra of the aluminum sample irradiated for 30 minutes with 400 nA of alpha current in the best vacuum conditions.
Figure 5-9: The XPS spectra of the molybdenum sample implanted with lithium in the best vacuum conditions.

In each of the spectras, there are strong carbon and oxygen peaks with various other distinct peaks corresponding to the sample material and other contaminants. The carbon and oxygen peaks are much stronger than the underlying sample material’s peaks, indicating that carbon and oxygen are the dominant species on the surface. In the BN and moly samples, for example, there are zinc, calcium, and silicon contaminants, but these are less than 1 atomic percent and would probably be present regardless of proton irradiation from storage or other causes. In the stainless steel spectra, however, the silicon peaks are much stronger and correspond to larger silicon concentrations of about 15 atomic percent that is believed to be from a thin layer from manufacturing. For the metal samples, the metal signatures are also very weak, meaning that there is hardly any of the bulk material on the surface. Also note that although the molybdenum sample was implanted with lithium, the lithium is not detected by XPS because the implantation depth is deeper than the ~10 nanometer depth resolution of the device.

The summary of the effects of proton irradiation fluence, time, and pressure on carbon enhancement for various sample materials are illustrated in figures 5-10, 5-11, 5-12, 5-13 and 5-14.
Figure 5-10: Carbon surface concentrations versus irradiated beam fluence for BN samples

Figure 5-11: Carbon surface concentrations versus irradiated beam fluence for Metal samples

Looking at the fluence of irradiated particles gives insight into the net energy transferred from the beam to the sample, as every incoming particle imparts the same
energy to the sample. Some of the points may have the same fluence because they are different locations on the same sample, but others will have the same fluence from irradiating for longer or shorter at lower or higher beam currents. For the most part, the carbon enhancement is higher directly on the beam spot than at other locations on the sample, with a few outliers. From looking at the effect of fluence on carbon concentration, there is no obvious correlation.

Furthermore, it is interesting to see that the stainless steel and the molybdenum also had high concentrations of carbon, indicating that the porosity of the BN is not to blame. In fact, the metal signatures were extremely small on the metal samples, like less than 4% of molybdenum on the molybdenum sample, indicating that there may be a small layer of carbon covering up the metal surface underneath. BN, on the other hand, still showed significant traces of boron and nitrogen. Also, even different locations on the same sample had a varying carbon enhancement levels, indicating that enhancement may be an effect of a diffusion process.

Figure 5-12: Carbon surface concentrations versus beam irradiation time for BN samples
While the irradiation time gives insight into the net energy transferred to the sample, it also gives information about the conduction of heat transferred through the sample if this contamination is a thermal diffusion effect. If the same fluence of particles hit the target in a smaller time frame, then the target will have a higher power absorption and a higher average temperature. The same general trend of higher carbon enhancement on the beam spot holds in this case as well. While the metal samples showed little to no trace of the original sample material, the BN samples still showed significant traces of boron and nitrogen. There may be a saturation limit on the carbon enhancement, which for BN looks to be around 65% because even the samples that were irradiated for 1 hour and 45 minutes at a relatively large beam current of 400 nA had around this level of carbon enhancement. The huge fluence from these few samples is larger than the total fluence from the multiple NRA and RBS characterizations at various locations of any of our plasma-exposed samples. However, it is clear that there is a large spread of values for the same irradiation time and thus there is no obvious correlation with irradiation time.

To prove that the enhancement is not due to vacuum impurities, the vacuum in
Figure 5-14: Carbon surface concentrations as a function of vacuum chamber pressure

the target chamber was improved by about an order of magnitude and new samples irradiated under these conditions were analyzed. The results show that there is high carbon enhancement even from vacuum conditions that are more than sufficient.

Also, a 2x2 mm area around the beam spot of one of the BN samples was sputtered away by an argon gun and XPS measurements were taken after each round of sputtering, giving the depth profile shown in figure 5-15. The carbon concentration starts at around 65% and eventually reaches nominal levels around 10% after many minutes of sputtering. To convert from sputtering time to sputtering depth would require the sputter yield for Argon on BN. The oxygen concentration stays low throughout the surface layers, but the boron and nitrogen concentrations develop a difference of about 10% even though they start at basically the same concentrations. This may be due to preferential sputtering of the nitrogen atoms by the argon beam; this same sputtering physics should apply to the xenon plasma as well as discussed in previous sections.
Potential oxygen enhancement was also investigated for the same variety of samples and conditions and is visualized in figures 5-16a, 5-16b, 5-17a and 5-17b. It was shown that there was no significant enhancement of oxygen, only carbon, on the BN samples. The stainless steel samples have relatively large oxygen surface concentrations due to oxidation, which would be present even in baseline metal samples.

Figure 5-15: Atomic concentration depth profile on beam spot of BN sample irradiated for 1 hour and 45 minutes at 400 nA of alpha particle current.

Figure 5-16: Oxygen surface concentrations as a function of irradiated beam fluence and irradiation time for BN.
A possible explanation of the growth of carbon on the surface from ion irradiation may be due to dissociation of hydrocarbons present in the sample chamber from the oil based pumps by thermal cracking and secondary electron emission from samples [44]. To investigate this possible cause, the roughing pump responsible for the sample chamber had its oil filter replaced and a mass spectrometer was moved into the sample chamber to survey for high mass contaminants corresponding to hydrocarbons. A 3 MeV 400 nA alpha particle beam was used to irradiate various samples to look for changes in the mass spectra. A BN and an aluminum sample were irradiated and the resulting mass spectra are shown in figures 5-18 and 5-19, respectively.
The dominant signals are at mass 2, 18, and 28 for hydrogen, water, and nitrogen, respectively. There are also weaker signals at higher masses, like 44 which is CO2 and others which may be hydrocarbon signals, but these molecules have partial pressures below about 1e-9 torr and are hard to resolve from the instrument noise. The XPS results discussed earlier included these samples and they are comparable to the other samples.
Chapter 6

Results and Conclusions

6.1 Overview

The lithium depth marker technique using NRA and RBS to measure net erosion and monitor surface contaminations has been successfully applied to samples of BN exposed to the plume of a xenon fueled BHT-200 hall thruster. The net erosion in the samples was shown to vary across the sample face in accordance with varying plume conditions, with locations of maximum and minimum net erosion. RBS of the exposed samples showed that a high Z element identified as imbedded xenon diffused throughout the surface layers, and characterizing the RBS spectra quantified the depth and concentration of this plasma retention.

Initial attempts to identify the high Z element as xenon using XPS indicated a larger problem of high levels of carbon contamination due to ion irradiation from both the lithium implantation and the proton irradiation from the NRA and RBS characterization steps. Further XPS analysis on a variety of samples showed that the carbon contamination was essentially independent of ion irradiation time, ion fluence, sample material, and vacuum quality. The cause is thought to be due to hydrocarbon cracking from leaking pump oil, which is not an inherent flaw of the measurement technique itself. Heavy ion RBS using helium was also carried out as another means to identify the xenon, but a more accurate energy calibration would be needed for a confident conclusion from this data.
6.2 Future Work

Future work will focus on better characterizing the xenon retention physics and applying the technique to more realistic thruster conditions. Further characterization of xenon retention can be accomplished by investigating the xenon saturation limit in BN and whether it is a function of plasma temperature by exposing more samples to various plasma conditions in the thruster or in DIONISOS and characterizing them with RBS. Applying the technique to realistic thruster conditions would require placing samples closer to areas of high erosion in the thruster, like near the thruster exit. Even better would be placing coupons of implanted BN samples in the thruster wall near these high erosion zones that could be inserted and removed for NRA and RBS characterization.

There is still ongoing work in investigating the inadvertent carbon enhancement from ion irradiation discussed in section 5. There are plans to investigate the discoloration that is present throughout the upper surface layers by exposing untouched BN samples to the plume and seeing if this introduces discoloration. Other plans to look into the carbon enhancement issue include XPS analysis of samples irradiated in the presumably clean DIONISOS chamber and replacing the oil pumps with dry pumps, but replacement costs are an inhibiting factor.
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