Real-Time RBS Analysis of Plasma Erosion in DIONISOS

by

Ethan E. Peterson

Submitted to the Department of Nuclear Science and Engineering in partial fulfillment of the requirements for the degree of Bachelor of Science in Nuclear Science and Engineering at the MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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Abstract

One of the primary scientific challenges still facing the development of commercial nuclear fusion reactors lies at the plasma-material boundary. Plasma temperatures greater than 10 million degrees Celsius (10 keV) require clever magnetic field configurations to confine the plasma near the center of the toroid. However, the materials directly surrounding the plasma, known as the first wall, will be in contact with a cooler plasma, closer to 5 eV, and must be able to withstand intense neutron radiation as well as high heat fluxes. It is still unclear how some proposed first wall materials such as tungsten and molybdenum will behave in environments with these plasmas. Scientists must provide evidence supporting the lifetime, fuel retention capabilities, and neutron resilience of these materials in order to assure their high quality performance inside fusion reactors for many years. As a result, scientists must better understand how plasmas interact with surfaces of materials. This project contributes to this endeavor by studying plasma erosion in real-time using a helicon plasma source and an ion beam analysis technique known as Rutherford backscattering spectroscopy (RBS) to determine target thickness and composition. Copper coated aluminum targets were subjected to helium plasmas of varying fluxes and ion energies and were analyzed in real-time with RBS to determine the copper layer thickness as a function of time. This analysis will provide the framework for studying fusion materials such as molybdenum and tungsten in the same way using hydrogenic plasmas. It is expected that the erosion rate will be proportional to the ion flux (a function of plasma density) and the sputtering yield (a function of ion energy), while being inversely proportional to the target density. The goal will be to develop a reliable method to characterize plasma regimes with reproducible, well-behaved flux profiles and use them to controllably erode samples, while performing real-time RBS analysis of the surface layer.

Thesis Supervisor: Dennis G. Whyte
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I am also extremely grateful to Dr. Graham Wright, for supervising my research over the past four years and providing invaluable advice in the writing of this thesis.

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Clean energy needs are becoming more and more apparent with the increasing consumption of fossil fuels accompanied by decreasing reserves. As a result, extensive research has been conducted to develop alternative energy sources. One such alternative is nuclear fusion [1]. Alternative energy strategies have been sought after for decades. Fusion energy research, specifically, has been conducted since the 1950s [2]. The overall objective of these alternative strategies is to reduce world-wide energy dependence on non-renewable resources, especially those with negative impacts on the environment. The potential for fusion energy to meet these desires and provide vast amounts of energy globally is the primary motivation for studying fusion systems [3]. Designing a fusion reactor has often been colloquially analogized to bringing a star to earth. Stars are the universe’s source of fusion power and to replicate such astrophysical conditions on Earth is no small feat. To achieve the state of self-sustaining fusion power production, known as ignition, there are two main paths: increasing the density, or increasing the temperature. Stars use their enormous mass to gravitationally confine an ionized gas known as plasma, which allows the density and pressure to be much higher than what we can obtain here on Earth. Consequently, our terrestrial reactors must opt to increase the temperature. Clearly, temperatures greater than the sun’s cannot be contained by any material object. As a result, leading fusion reactor technologies employ the use of strong magnetic fields inside a vacuum vessel to confine a plasma at very high temperatures [4]. The magnetic fields act as a bottle
to prevent the sides of the vessel from coming in contact with the core plasma, which can reach temperatures ten times greater than those in the sun.

The primary fuel consideration for fusion is a mixture of deuterium (D) and tritium (T), both isotopes of hydrogen, with one and two neutrons respectively [5]. When the D-T mixture is confined with magnetic fields and heated, it becomes plasma, and nuclear fusion can occur between deuterium and tritium. Each D-T fusion reaction releases a high energy neutron and an alpha particle. The alpha particle loses its energy in the plasma through Coulomb collisions in the plasma. This is beneficial because the alpha particles heat the electrons, which help sustain the plasma temperature [6]. The neutron loses energy in the surrounding wall, which provides heat to produce electricity. After the neutron has thermalized, it can be captured by lithium to breed more tritium, which is the very fuel the fusion process consumes [7]. This self-sustaining, fuel-breeding reaction is a very elegant process and is a major reason why fusion energy is so appealing. In addition, fusion reactors are not susceptible to meltdowns as are fission reactors since there are no positive feedback mechanisms, and the internal energy contained in the plasma at any given time is not very high due to its low density [6].

The elegance in the fusion fuel cycle, accompanied by the abundance of deuterium in sea water, its nonexistent carbon footprint and its inherent safety, are the primary motivating factors for the study of fusion systems and their application to power generation. There are still a few specific areas that stand in the way of commercial fusion reactor development. One of these areas is plasma-surface interactions, which describes how plasmas and material surfaces affect one another when in contact [8]. This is an extremely crucial field of fusion engineering.

Plasma is an extreme phase of matter that can be very disruptive to materials. It displaces atoms from their lattice points, implants ions, erodes material, and can transport that eroded material and deposit it at a new location. In addition, the neutrons released by fusion reactions can activate certain materials, making them radioactive, and causing many other types of material defects and changes in material properties like thermal conductivity [9]. Scientists have yet to prove the capability of
certain materials to withstand prolonged exposure to such fusion grade plasmas [10]. Such materials include tungsten and molybdenum, which are favored because of their high melting points. The specific plasma-material interaction that will be the focus of this thesis is the erosion of material due to plasma ion bombardment. This is an imitation of the behavior in the divertor region of fusion devices where the magnetic field lines intersect the surrounding material. Since the charged particles of a plasma follow the magnetic field lines, the divertor region bears the brunt of the energy deposition in the device [11]. This energy deposition is mostly in the form of energetic ions. As a result, it is important to understand the impact these energetic ions will have on the materials in the divertor after extended periods of operation [7]. Advances in understanding of plasma-surface interactions are a major step in bringing economically viable fusion power closer to implementation.

The helicon plasma source DIONISOS, which stands for Dynamics of IONic Implantation and Sputtering On Surfaces, is an apparatus at the MIT Plasma Science and Fusion Center specifically designed for studying plasma-surface interactions. The apparatus supports a target that can be rotated to many different angles, which is important for various ion beam analysis techniques. The target is then subjected to a plasma heated by radio frequency (RF) waves. The plasma ions interact with the atoms of the target, which can drastically change the surface as discussed above. The focus of this thesis is to demonstrate the capabilities of DIONISOS to measure plasma erosion of materials, in real-time, with an ion beam analysis technique known as Rutherford backscattering spectroscopy (RBS).
Chapter 2

State of Plasma-Materials Interaction Research

2.1 Historical Overview of Plasma Facing Materials

Ever since the conception of magnetic confinement techniques for thermonuclear fusion research in the early 1950s, the glaring issue of plasma material interactions has been an engineering and physics focal point. The presence of intense radiation fields, high thermal and particle flux, thermally induced stresses of disruptions and plasma contamination from wall impurities pose an unprecedented challenge to fusion engineers and material scientists that must be conquered to bring economic fusion power to fruition. In the first fusion experiments of the 1950s (mainly stellarators and pinches) the dominating issue was plasma impurities due to poor vacuum techniques and allowing carbon and oxygen to desorb from the walls of the devices and contaminate the plasma. These impurities cool the plasma significantly and induced crippling radiation losses in the core of the plasma [7].

With the development of tokamaks in the USSR in the early 1960s new vacuum techniques were being used to improve the purity of the plasma and plasma facing materials. These techniques included stainless steel vacuum vessels accompanied
by all metal seals, baking of vacuum components to remove moisture and gaseous impurities, and plasma discharge cleaning to remove surface impurities. Combining these advances with the improved confinement schemes of the tokamak gave rise to what was considered to be the first relatively hot and dense plasmas in the T-3 tokamak in Russia [7]. However, there were still problematic levels of impurities in the plasma including carbon, oxygen, and various metals.

To prevent the plasma from coming in direct contact with the vacuum walls and thus limiting the amount of impurities present, the concept of a limiter was introduced. A limiter is a structure that is placed inside the vacuum vessel at the edge of the plasma to control the shape of the last closed magnetic flux surface. These limiters were built from refractory material such as molybdenum in Alcator A at MIT and tungsten in ST at Princeton [7]. In the early 1970s the use of limiters to shape and control the magnetic flux surfaces led to the development of the divertor which is a region in the reactor where the magnetic field lines impinge on the material surface which allows charged particles to be exhausted. Charged particles including alphas from the fusion reaction and impurities in the edge of the plasma known as the scrape off layer (SOL) are transported along the magnetic field lines to the divertor where they are neutralized upon impact and can be pumped out [12]. The divertor is positioned far away from the core plasma as to prevent poor plasma performance from high impurity concentrations.

In the 1980s studies on divertor tokamaks showed that the presence of the divertor was able to reduced the temperature of the plasma in the divertor, which reduced the energy of the ions incident on the material, ultimately reducing the physical sputtering rate. With the introduction of neutral beam heating, much higher plasma temperatures and power fluxes were now accessible. This caused significant physical sputtering in devices with tungsten limiters such as PLT at Princeton. A switch was made to nuclear grade graphite as the limiter material because it did not melt at high temperatures (it undergoes sublimation) and carbon atoms would be fully stripped of electrons in the core plasma which would prevent radiation loss [7]. Throughout the 1980s many tokamaks adopted the graphite limiter/divertor technology, but it
was soon shown to have many undesirable properties. For example, in hydrogenic plasmas, carbon faces chemical sputtering rates many times greater than its physical sputtering rate. It was also shown to trap hydrogen very well which is bad for plasma performance and has poor structural properties when exposed to neutron radiation [13]. These problems led researchers to look for other candidate materials.

Beryllium was the next material to be considered due to its chemically inert nature in hydrogenic environments and its high thermal conductivity. However, its low melting point and high physical sputtering rate were generally considered to be even more problematic than the drawbacks associated with carbon limiters. Currently, carbon is a popular plasma-facing material in limiter devices where plasma temperatures are higher and densities are lower. On the other hand, divertor plasmas are known to have higher densities and lower temperatures, which makes metals attractive because the ion energies are set by the plasma temperature and may be below the physical sputtering threshold [7]. The high erosion rates expected in divertor plates made of carbon have steered the research of divertor materials back towards metals especially molybdenum as in Alcator C-Mod [14] and tungsten in ASDEX-Upgrade [15]. Both of these experiments have shown low net erosion rates in the divertor under normal operating conditions. However, disruptions are still of great concern because of the thermal stresses and highly concentrated power fluxes associated with them.

2.2 Plasma-Material Interaction Problems for Future Devices

The next step in fusion science is to build a reactor capable of producing significantly more power than it consumes to validate the viability of fusion reactors. This is the goal of ITER, which is currently under construction in Cadarache, France. To achieve this goal research has indicated that the scale of this reactor needs to be much larger than current experiments. Inevitably, a larger reactor implies larger energy content.ITER is projected to produce approximately 500 MW of fusion power whereas current
experiments are only on the order of a couple megawatts. Large size and higher power levels have many implications for plasma-material interactions issues. For instance, disruptions in ITER will be much more intense than in current devices, which can cause melting or other physical deformation of divertor materials [7]. ITER is also intended to be pulsed for a few hundred seconds at a time versus a couple seconds in most present machines. This increased pulse length will require better impurity mitigation strategies in order to achieve high plasma performance. As a result, the divertor region will have to be actively cooled and pumped to limit sputtering and to effectively remove impurities and helium ash as they are produced [10]. The long run time over many years of operation will also prove to be a challenge because of the net erosion rates expected for the graphite divertor plates in ITER. These may have to be replaced several times during operation, which is not desirable because it increases required maintenance time and will be a safety hazard due to the high tritium retention of carbon [16]. Finding a balance of high performance and duty factor in the approach to steady state operation will be the defining challenge for next step fusion devices. The crux of this issue is plasma-material interactions and the lifetime of plasma-facing components in these machines.
Chapter 3

Relevant Surface Physics

3.1 Physical Sputtering

Physical sputtering is a phenomenon involving the exchange of momentum between an incident ion and the atoms in a solid lattice. If the incoming ion has an energy significantly greater than thermal energies it will impact an atom in the lattice and initiate a chain of collision processes [7]. The collisions then propagate throughout the lattice and often back towards the surface. If a recoil atom has energy greater than the surface binding energy it can eject an atom from the surface. This is the dominant method of erosion observed for the helium-copper system studied here, when at constant temperature. In the case of copper, the surface binding energy is roughly 2-3 eV. The average number of ejected surface atoms (sputtered atoms) per incident ion is referred to as the sputter yield. The sputter yield for helium ions incident on copper is a strongly varying function of energy in the region of interest to this investigation (30-40 eV). A plot of the sputter yield for helium on copper as a function of energy can be seen in fig. 3-1. The data for fig. 3-1 was taken from the IAEA Aladdin particle-surface interactions database [17].

Erosion from plasma ion bombardment is particularly concerning in fusion systems such as tokamaks because the energetic charged particles like helium stream along the magnetic field lines and are exhausted where the field lines intersect the material surface of the divertor [12]. This causes erosion of the divertor much like in DIONISOS.
and can have many negative impacts on the system. For example, erosion of the
divertor produces impurities in the plasma and deforms the surface, which can alter
material properties such as thermal conductivity or hydrogen retention properties,
both of which are crucial to plasma performance. In order to study the erosion process
of plasma ion bombardment one can use Rutherford backscattering to measure the
erosion rate of a thin layer of material coated onto a substrate of a different material.

3.2 Rutherford Backscattering Spectroscopy

Rutherford backscattering is a form of ion beam analysis that yields information
about the target composition as a function of depth. As an ion of fixed incident
energy encounters the target surface it is slowed down via Coulomb collisions as it
passes through the target until it backscatters off an atom. The ion then slows down
again via Coulomb collisions as it leaves the target. Applying the conservation of
energy and momentum to the backscattering process, one will find that the angle of scattering will uniquely set the energy of the backscattered ion. A schematic view of this process can be seen in fig. 3-2.

Figure 3-2: Schematic view of RBS analysis with cross-sectional view of a copper coated target.

An energy spectrum collected at a given scattering angle will describe ions that penetrated the target to different depths and will give a depth profile of the material. This is useful because one can then observe the composition of the material as a function of depth since different elements will have different backscattering probabilities due to their relative cross-section, which is larger for higher Z materials. Higher cross-section materials will produce regions of higher count rate in the pulse height spectrum and can thus be identified at their respective depths. An example of this can be seen in fig. 3-3. There is a clear separation between the copper and aluminum plateaus. The width of the copper peak signifies how much energy was lost by an ion that backscattered off the deepest copper atom in the target which effectively measures the thickness of the copper layer on top of the aluminum substrate. One can also see that the copper cross-section is much higher than the aluminum cross-section because copper has higher Z. The front edge of the energy spectrum is also uniquely set by the species on the surface. This is because the energy of an incident particle
backscattering at a 90 degree angle off a copper atom at the very surface of the target is set uniquely by kinematics and can be used to identify the element at the surface. For example, the energy of a 1400 keV proton following a 90 degree scattering event off copper is 1356 keV, whereas off aluminum the energy is 1299 keV.

Figure 3-3: Energy spectrum differentiating the Al and Cu signals from a target with a 1.47 micron copper coating on aluminum substrate sampled with 1400 keV protons.
Chapter 4

Objectives

The objective of this thesis will be to measure erosion rates of helium plasmas on copper coated aluminum targets. This will mark the first instance of real-time erosion analysis and provide the proof of concept necessary for implementing this technique in fusion systems with hydrogenic plasmas and molybdenum or tungsten targets.

The first of these objectives is to determine the ion beam parameters necessary for RBS measurements. To do this a, 1.7 MV tandem accelerator will be used to produce ion beams for ion beam analysis. Reproduceable ion beams of a given species, energy, and spot size must be determined by tuning the accelerator steering and focusing elements through trial and error. Once the desired ion beams have been produced and accelerator parameters recorded, it will be a simple task to switch between ion beam techniques for different diagnostics.

The second objective is to characterize stable plasma regimes by finding stable plasma modes, with relatively uniform flux profiles that can be reproduced easily. This is accomplished by adjusting magnetic field strength, gas pressure, and RF power input to the helicon plasma source through trial and error. The quality of these plasma regimes will determine the accuracy of the measurements and reproducibility of the experiments.

Lastly, the copper coated aluminum targets will be placed in the DIONISOS chamber and subjected to these stable plasmas for a certain amount of time while simultaneously performing RBS analysis on them. The purpose of RBS analysis is to
determine the copper layer thickness as a function of time.
Chapter 5

Experimental Method

The DIONISOS experimental apparatus can be seen in fig. 5-1. To cause erosion of the target, it will be biased between -30 V to -40 V, which dictates the energy of the plasma ions that impact the target. The ions for ion beam analysis have much higher energies (e.g. 1-1.5 MeV), which means the acceleration they receive from the target bias is negligible. The ion beam will be incident on the target at 45 degrees and scatter into a solid state detector at 45 degrees, for a total scattering angle of 90 degrees, where it will deposit all of its energy. This RBS measurement will take place while the plasma is bombarding the target, and spectra will be collected at regular intervals until a satisfactory level of erosion becomes apparent. The data presented in this thesis were not taken under active temperature control, which proved to be a problem in the intense plasma regimes.

Once the proton deposits its energy in the detector, a signal proportional in height to the amount of energy deposited will be sent to an amplifier and then a multichannel analyzer where the signals will be sorted by height (energy) into different bins to produce a pulse height spectrum. Once these spectra are collected and calibrated, an ion beam analysis software called SIMNRA [18] will be used to determine the copper layer thickness. This will be done for all the spectra collected to obtain the time rate of change of the copper layer thickness. This is essentially the erosion rate and should be a function of the plasma ion energy, ion flux, target density and the sputtering yield, which determines how many atoms of material are sputtered by one incident
ion of a certain energy.

In addition, Langmuir double probes will be used as indicated in fig. 5-1 to measure the flux of ions in the plasma by collecting current. This flux will be recorded simultaneously with the RBS analysis to provide a consistency check between measured erosion rates and what is believed to occur theoretically. A sample RBS spectrum and SIMNRA fit from this investigation can be seen in fig. 5-3.

![Diagram of DIONISOS experimental apparatus](image)

Figure 5-1: Top-down view of DIONISOS experimental apparatus

### 5.1 Determine Ion Beam Parameters

To determine the ion beam parameters, beam species and energies must be chosen for RBS analysis. Since backscattering only occurs when the projectile is less massive than the target, protons and alpha particles will be used. Proton energies of 1000 keV to 1500 keV will be used. Alpha particles were intended for use as well to achieve optimal depth resolution, but accelerator difficulties prevented the attainment
of alpha particles at high enough energy. To produce these ion beams, the Cambridge Laboratory for Accelerator Study of Surfaces (CLASS) accelerator will be used in NW13-133 at MIT. The ion sources produce the protons or alpha particles, and the extraction voltage, steering magnets, and focusing lenses will be tuned to produce a roughly 1mm beam spot at the center of the target. This beam can then be steered by electrostatic deflectors to reach any point on the target. All of the parameters for each type of ion beam will be recorded for reproducibility.

5.2 Determine Stable Plasma Regimes

Stable plasma regimes are ones that do not have mode instabilities (flickering) and, for this study, have close to uniform flux profiles over a radial distance of roughly 10mm. These regimes are determined by tuning the magnetic field, gas pressure, and RF power input of the helicon plasma source and measuring the flux with a Langmuir probe at different radial positions. Once this flux is roughly constant in both radial position and time, the parameters of the plasma source will be recorded and reproduced for the experiments.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Plasma 1</th>
<th>Plasma 2</th>
<th>Plasma 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetic Field Coil Current (A)</td>
<td>85</td>
<td>155</td>
<td>155</td>
</tr>
<tr>
<td>Gas Pressure (Pa)</td>
<td>4.6</td>
<td>3.9</td>
<td>4.4</td>
</tr>
<tr>
<td>RF Power (W)</td>
<td>800</td>
<td>1100</td>
<td>1100</td>
</tr>
<tr>
<td>Flux (m⁻²s⁻¹)</td>
<td>3-6x10²¹</td>
<td>6-9x10²¹</td>
<td>9x10²¹</td>
</tr>
<tr>
<td>Electron Temperature (eV)</td>
<td>6</td>
<td>6</td>
<td>6</td>
</tr>
</tbody>
</table>

To verify the consistency of the plasma characteristics, double probe measurements of the ion saturation current were taken before and during the exposure. An example of the probe trace on axis in plasma 3 can be seen in fig. 5-2. The double probe trace of plasma 3 in fig. 5-2 is consistent with the ion saturation currents measured during the 4 experiments with plasma 3 shown in table 5.2.
Figure 5-2: Double probe trace of plasma 3 showing I-V characteristics, where the two data series "Up" and "Down" represent sweeping the voltage in the positive and negative directions respectively to show consistency

5.3 Data Acquisition and Analysis

The data for this investigation are in the form of RBS spectra. These spectra record the number of counts in different energy bins from backscattered protons (or alpha particles). While the target is being exposed to plasma, the ion beam will be incident on the target at a 45 degree angle and exit at 45 degrees into a solid state detector. Based on the composition of the target and the energy deposited in the detector by each proton, it can be determined how far into the target that proton went. These spectra can then be fit in SIMNRA [18], which makes theoretical predictions of the spectra using input parameters such as ion beam energy, beam current, scattering geometry, target composition, thickness and roughness, and detector type. It can also be used to iteratively fit the data and return estimated values for copper layer thickness. This is the primary function that will be used to determine the rate of change of the copper layer thickness and how that relates to the plasma parameters.

All Targets were 1 inch diameter, 2 mm thick aluminum substrates with a 1.5 micron copper coating. Targets 1, 2, and 3 were only coated with copper on one side and were not polished. Targets 9, 11, 15, 17, and 18 all had one polished side and
Figure 5-3: Sample RBS spectra (red points) of copper coated aluminum target taken with 1400 keV protons, using a 2 micron aluminum foil in front of the solid state detector, and subjected to helium plasma for 1530 seconds accompanied by a theoretical fit line produced by SIMNRA (blue line)

both sides were coated with a 1.5 micron layer of copper. A picture of a sample target prior to plasma exposure is shown in fig. 5-4. On the left is the polished face and on the right is the unpolished face. Targets 1, 2, and 3 looked like the unpolished face on one side and just aluminum on the other.
### Table 5.2: Experiment Parameters

<table>
<thead>
<tr>
<th>Target</th>
<th>Plasma Regime</th>
<th>Plasma Ion Flux (m$^{-2}$s$^{-1}$)</th>
<th>Avg. Target Current (mA)</th>
<th>Target Bias (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>3-6x10$^{21}$</td>
<td>-</td>
<td>-30, -35, -40 5min each</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>3-6x10$^{21}$</td>
<td>-</td>
<td>-30 to -39 (1V/3min)</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>3-6x10$^{21}$</td>
<td>-</td>
<td>-37</td>
</tr>
<tr>
<td>9</td>
<td>2</td>
<td>6-9x10$^{21}$</td>
<td>150</td>
<td>-35</td>
</tr>
<tr>
<td>11</td>
<td>3</td>
<td>9.26x10$^{21}$</td>
<td>388</td>
<td>-35</td>
</tr>
<tr>
<td>15</td>
<td>3</td>
<td>9.35x10$^{21}$</td>
<td>365</td>
<td>-32</td>
</tr>
<tr>
<td>17</td>
<td>3</td>
<td>9.35x10$^{21}$</td>
<td>345</td>
<td>-40</td>
</tr>
<tr>
<td>18</td>
<td>3</td>
<td>9.50x10$^{21}$</td>
<td>378</td>
<td>-32</td>
</tr>
</tbody>
</table>

Figure 5-4: Front (L) and back (R) view of a sample target prior to plasma exposure. The front side of the target shown on the left shows the reflection of the camera used to take the picture because the polished aluminum substrate made the copper coating smooth enough to act as a mirror.
Chapter 6

Results

6.1 Parameter Focusing

The initial intent of this investigation was to conduct RBS analysis with both protons and alpha particles and compare them. This was because the alpha particles would give a better depth resolution, due to their greater stopping power. However, accelerator difficulties prevented the production of high enough energies for the alpha particles to penetrate through the copper layer (roughly 4 MeV). Consequently, protons were used at 1200 keV with a beam current on target of approximately 10-20 nA. Three targets were analyzed with 1200 keV protons with the intention of finding an optimal target bias range.

Target 1 was subjected to Plasma 1 at a target bias of -30V for 5 minutes. There was no visible change in the energy spectra so the target bias was increased to -35V. When there was still no change after 5 additional minutes the target bias was increased to -40V where the copper layer was quickly eroded away. A finer target bias scan was deemed necessary and became the focus of target 2. Target 2 was also subjected to plasma 1 and the exposure commenced with a bias of -30V. The bias was then increased by -1V every 3 minutes to discover the point where there was noticeable yet relatively constant erosion. This point was determined to be around -37V. Spectra from target 2 were collected every 180 seconds for half an hour. Examples of the energy spectra collected from target 2 can be seen in fig. 6-1. Note that even after
720 seconds there was essentially no change in the copper peak width, indicating no erosion for target biases less than -35 V.

Figure 6-1: Sample energy spectra taken from target 2 during exposure taken at 360 second intervals

These energy spectra were then analyzed in SIMNRA to calculate the copper layer thickness at each time interval. This data yielded the erosion characteristics displayed in fig. 6-2, showing an erosion rate of 10-20 nm/min.

After the target bias scan involving target 2, a plasma exposure was planned for a constant target bias of -37V. This exposure involved target 3, which was, once again, subject to plasma 1. Energy spectra were taken every 90 seconds for 45 minutes. Sample energy spectra from target 3 can be viewed in fig. 6-3.

Similarly to target 2, the energy spectra from target 3 were analyzed in SIMNRA to calculate copper layer thickness as a function of time and hence the erosion rate. In fig. 6-3 one can see that the back edge of the copper peak moves to the left by a distance corresponding to approximately 40-50 keV every 360 seconds. This indicates that the protons are losing less energy in the copper layer as time goes on and signifies that the copper layer thickness is diminishing. Consequentially, the
erosion characteristics from target 3 are shown in fig. 6-4 and support an erosion rate of roughly 20 nm/min.

It is clear from fig. 6-3 that there is no distinct separation between the copper and aluminum signals as there were in fig. 3-3. In addition, it is apparent that the real-time erosion rates are relatively sporadic. This is possibly due to the aluminum substrate roughness, detector resolution, or energy straggling in the aluminum foil in front of the solid state detector. These low resolution spectra make it difficult to accurately fit them in SIMNRA which gives rise to inaccurate erosion statistics. To correct this in the next round of experiments, the aluminum substrates were to be polished prior to applying the copper coating, the proton beam energy would be increased to 1400 keV to reduce straggling, and a new detector would be used in an attempt to acquire better energy resolution.

Another concern that was raised about the experimental setup was the purity of the plasma. This was because the quartz tube containing the plasma appeared to be growing darker due to copper coating its inside. The end cap on the quartz tube where the gas feed through is was made of copper and sputtering in this area could have been causing copper to be carried into the plasma and deposited on the tube and, for that matter, elsewhere in the chamber, including the target. In order to improve the plasma purity, a molybdenum plate with a hole for gas flow was manufactured to cover the exposed copper on the end cap.

In addition to the molybdenum end plate, a new target current monitor with
Figure 6-3: Sample energy spectra taken from target 3 during exposure at 360 second intervals and -37 V bias

higher precision was installed to be able to more accurately monitor the current drawn at the target from the plasma. The purpose of this was to be able to monitor more closely the plasma behavior and prevent any significant mode changes that may not be visibly noticeable. These upgrades significantly improved the quality of the results from the subsequent erosion studies discussed in the next section.

6.2 Refined Attempts

The ion beam used for the following experiments was 1400 keV protons and the beam current on target was approximately 25nA. Five copper coated aluminum targets were eroded during this experiment and subject to two different plasma regimes and 3 different target biases. Both plasmas were helium, but were subject to different operating conditions. This produced two distinct modes with different, but relatively uniform flux profiles. Inevitably, there were some fluctuations in the plasma conditions between experiments. Therefore the subsections pertaining to targets 9, 11, 15, 17,
and 18 will include more detailed plasma characteristics if they differed substantially from the target regime.

### 6.2.1 Target 9

Target 9 was eroded using plasma regime 2 and took approximately 25 minutes to erode away completely at a target bias of -35V. Energy spectra were collected from target 9 every 90 seconds for fifteen minutes, when it was clear that the vast majority of the copper layer had been eroded. In fig. 6-5 one can see four representative energy spectra from target 9.

The increased resolution in comparison to the previous experiments was deemed due to the increase in ion beam energy and the new detector. This was because RBS spectra were collected from both the smooth and rough sides of target 9, which showed minimal differences. Nonetheless, the improved resolution was quite beneficial as it provided increased accuracy in fitting the spectra with SIMNRA. As a result, the calculated erosion rate was much less sporadic and can be seen along with the copper layer thickness of target 9 in fig. 6-6.

An interesting feature in the energy spectra of target 9 shows an increase in the aluminum count rate as the erosion increases. This increase in aluminum signal is accompanied by a decrease in the copper signal. This phenomena could likely be due to the 2mm (relatively large) beam spot diameter. If the beam were sampling copper
and aluminum at the surface of the target, the “copper” signal would be diluted due to the lower cross-section of the aluminum and the “aluminum” signal would increase because a high beam flux would be reaching it. This conclusion is also supported by the decreased separation in the energy spectrum at 810 seconds when compared to the preceding spectra. A picture of the target following plasma exposure is included in fig. 6-7 so the reader can see the damage caused by plasma ion bombardment over a time period as short as 15 minutes.

After target 9 was exposed to plasma regime 2, it was decided that it would be beneficial to study the erosion of a different plasma regime with different ion flux density. Plasma regime 3 was used for experiments with targets 11, 15, 17, and 18, while the target bias was varied.
Figure 6-6: Copper erosion characteristics from SIMNRA analysis of energy spectra taken from target 9

Figure 6-7: Front (L) and back (R) view of target 9 after exposure.

6.2.2 Target 11

Target 11 was eroded using plasma regime 3 and took approximately 5 minutes to erode away completely at a target bias of -35V. The intent of this experiment was to compare the results from target 9 to the erosion from a more intense plasma, but at the same ion energy. RBS spectra were collected from target 11 every 30 seconds for roughly 10 minutes. The count time was decreased to 30 seconds from previous experiments to see if better time resolution would improve the accuracy of the erosion characteristics. The decreased count time is partially why the beam current was increased from 10 -20 nA to 25nA. The only concern in increasing the beam current was pulse pile up which was observed to be negligible. Fig. 6-8 contains
energy spectra representative of the erosion of target 11.

![Sample energy spectra taken from target 11 during exposure at 150 second intervals and a target bias of -35 V](image)

Figure 6-8: Sample energy spectra taken from target 11 during exposure at 150 second intervals and a target bias of -35 V

The energy spectra collected from target 11 appear to show steady erosion at the rate of 40-60 nm/min until roughly seven minutes into the experiment when the copper signal disappears completely. This was an interesting result because previous experiments in this study indicated relatively constant erosion rates when the target bias was held constant. Perhaps -35 V on target was too great for this plasma regime. The next experiment would involve a lower target bias to see if this hypothesis was justified. The resulting erosion characteristics for target 11 can be seen in fig. 6-9.

As is clear from fig. 6-9, one can see a steady increase in the erosion rate as a function of time. This is supportive of the “run-away” erosion observed from the energy spectra. When target 11 was taken out of the vacuum chamber in preparation for the next experiment, it was extremely hot (approximately 300°C). It is possible that the great increase in target temperature caused the erosion to increase. Increasing the temperature of the atoms in the target would have decreased the energy necessary to liberate them from their lattice points. Since this is the case, it is reasonable to believe that higher temperatures would increase the sputter yield of helium ions on
Figure 6-9: Copper erosion characteristics from SIMNRA analysis of energy spectra taken from target 11

copper. Increasing the sputter yield would certainly increase the erosion rate. In support of this argument a picture of the front and back side of target 11 is displayed in fig. 6-10. It is clear that the front side reached extreme temperatures due to the bubbles and cracking on a previously smooth surface. It is also interesting to note that the back side (not exposed to plasma) of the target that was coated in copper prior to the experiment is greatly changed as well.

Figure 6-10: Front (L) and back (R) view of target 11 after exposure.

6.2.3 Target 15

Target 15 was eroded using plasma regime 3 and a target bias of -32V. Since the experiment with target 11 at -35V indicated much quicker erosion than expected the goal of target 15 was to demonstrate controlled erosion at a lower target bias for the
intense plasma mode (plasma 3). However, after two minutes of exposure the plasma mode abruptly intensified so that the target current exceeded 500mA, which was an increase of approximately 1.5 times. This lasted no longer than 10 seconds before corrections were made and plasma regime 3 was achieved again. Nonetheless, the entire copper layer had been destroyed. RBS spectra were collected every 30 seconds, but the abrupt plasma mode shift cut the experiment short. Samples of the RBS spectra collected are presented in fig. 6-11.

![Graph](image)

Figure 6-11: Sample energy spectra taken from target 15 during exposure at 60 second intervals with a target bias of -32 V

From the minimal energy spectra collected from target 15 before the copper layer disappeared, erosion statistics were computed from SIMNRA simulations. This data is shown in fig. 6-12.

Much like target 11, target 15 showed deterioration on both the front and back side of the target as evidenced in fig. 6-13.

The target deformation does not appear as severe as in target 11. This is probably due to the fact that the target did not have as long of an exposure time and was thus incapable of reaching the same temperatures. The rapid erosion in this case was most
likely due to the plasma mode change rather than significant temperature increase. Since the target current jumped by roughly a factor of two when the plasma shifted modes, the amount of sputtering that occurred would likewise have increased by a factor of two. However, this factor of two increase would not account for the practically instantaneous erosion of the remaining 1.4 microns of copper. There must be some event, other than simply the increase in ion flux that is causing the massive erosion of copper to occur. Since this experiment was inconclusive, another experiment with the same conditions was proposed, but with the intent of ensuring the stability of plasma regime 3 for the duration of the exposure. Target 18 was used during this experiment.
6.2.4 Target 18

Target 18 was eroded using plasma regime 3 and was eroded away in four minutes at a target bias of -32V. The plasma mode appeared to be stable for the duration of the experiment, which was the goal in light of the results from target 15. However, the same behavior was exhibited as in target 11. With no apparent plasma mode change the copper signal disappeared abruptly after only a few minutes. This indicated that it was not the target bias that was too high as the test with target 11 at -35V took longer to erode completely than this test at -32 V. At this point in the experimental procedure the target holder was quite hot and beginning a new experiment with a hot target holder may have sped up the erosion process by preemptively increasing the target temperature even prior to plasma bombardment. This would help to explain why the copper signal disappeared even quicker than the -35 V experiment with target 11. Present in fig. 6-14 are sample RBS spectra collected from target 18.

![Sample RBS spectra](image)

Figure 6-14: Sample energy spectra taken from target 18 during exposure at 90 second intervals and a target bias of -32 V

Erosion statistics for the first few minutes of target 18’s exposure were computed and are displayed in fig. 6-15. As was the case for targets 11 and 15, there is an increase
in erosion rate as time goes on. After two minutes of exposure there is about a 50% increase in erosion rate. Since the plasma ion flux did not change considerably in this time frame and the target bias was constant, this observed increase in erosion rate adds support to the hypothesis that the target temperature is beginning to dominate the erosion process after a few minutes of exposure rather than the target bias.

![Graph showing copper erosion characteristics from SIMNRA analysis of energy spectra taken from target 18](image)

Figure 6-15: Copper erosion characteristics from SIMNRA analysis of energy spectra taken from target 18

In fig. 6-16 one can see that the backside of the target is eroded as well as the front side, which is in agreement with previously mentioned hypotheses.

![Image showing front (L) and back (R) view of target 18 after exposure](image)

Figure 6-16: Front (L) and back (R) view of target 18 after exposure.

From the experiments involving targets 11, 15, and 18, the general consensus is that temperature control is necessary for accurate erosion measurement, especially in the more intense plasma regimes. Currently the laboratory is not equipped with such capabilities. Consequently this thesis does not investigate the effects of this pro-
posed improvement on the erosion analysis. At this point, time became a factor and without adequate temperature control most experiments following this point would be relatively fruitless. As a result, it was decided to test once more the maximum target bias range for reasonable a reasonable erosion rate. The target bias of -40V was revisited for a final test with target 17.

6.2.5 Target 17

Target 17 was eroded using plasma regime 3 and a target bias of -40V. Like target 15, target 17 was completely eroded in two minutes. However, this was not observed to be due to any change in plasma mode. As a result, only 4 spectra were collected, which show interesting characteristics. The energy spectrum at 60 seconds appears to have shifted to lower energies and the front edge of the copper peaks of the pre-plasma spectrum and 60 second spectrum no longer coincide. This made these spectra impossible to compare in SIMNRA and as a result no meaningful erosion rates were calculated from this experiment. In this subsection one will find samples of the RBS spectra collected during the experiment and a picture of the front and back of the target following plasma exposure. As was the case with targets 11 and 15, the copper coat on the back side of target 17 has deteriorated significantly as well. It was confirmed that a -40 V target bias is most likely out of the range for reasonable erosion rates and will most likely not be used in future experiments.

6.2.6 Summary

Here is presented a summary of the experiments that yielded erosion rates for some reasonable period of time. The average erosion rates for these experiments are presented along with the measured plasma ion flux, the plasma ion flux computed from the average erosion rate, and the measured sputter yield. Using the measured plasma ion flux and erosion rate, the sputter yield was calculated. For all experiments, the sputter yield was found to be in the 0.3% to 1.0% range which is in agreement with the sputter yields of 30-40 eV helium ions on copper as shown in fig. 3-1. It can also
Figure 6-17: Sample energy spectra taken from target 17 during exposure at 60 second intervals and a target bias of -40 V

be seen that the flux calculated from the ideal sputter yields and measured erosion rates is relatively consistent with the measured plasma ion flux.

Table 6.1: Average Erosion Rates

<table>
<thead>
<tr>
<th>Target</th>
<th>Plasma</th>
<th>Erosion Rate (nm/min)</th>
<th>Flux(_{LP}) (m(^{-2})s(^{-1}))</th>
<th>Flux(_{calc}) (m(^{-2})s(^{-1}))</th>
<th>Sputter Yield</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>1</td>
<td>15.74</td>
<td>3x10(^{21})</td>
<td>2.2x10(^{21})</td>
<td>7.4x10(^{-3})</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>19.8</td>
<td>3x10(^{21})</td>
<td>2.8x10(^{21})</td>
<td>9.3x10(^{-3})</td>
</tr>
<tr>
<td>9</td>
<td>2</td>
<td>30.3</td>
<td>-</td>
<td>3.75x10(^{21})</td>
<td>9.5x10(^{-3})</td>
</tr>
<tr>
<td>11</td>
<td>3</td>
<td>58.3</td>
<td>9.26x10(^{21})</td>
<td>7.5x10(^{21})</td>
<td>8.9x10(^{-3})</td>
</tr>
<tr>
<td>15</td>
<td>3</td>
<td>29.2</td>
<td>9.35x10(^{21})</td>
<td>5.75x10(^{21})</td>
<td>4.4x10(^{-3})</td>
</tr>
<tr>
<td>18</td>
<td>3</td>
<td>25.7</td>
<td>9.50x10(^{21})</td>
<td>9x10(^{21})</td>
<td>3.8x10(^{-3})</td>
</tr>
</tbody>
</table>
Figure 6-18: Front (L) and back (R) view of target 17 after exposure.
Chapter 7

Discussion

It is clear that the erosion rates measured during the experiments involving plasma regime 3 (targets 11, 15, and 18) are not consistent with the time that each sample took to erode. Erosion rates ranging from 30nm/min to 60nm/min should take roughly half an hour to an hour to erode a 1.5 micron copper layer completely. The only sample to exhibit this consistency was target 9, which was the only experiment corresponding to plasma regime 2. A question that rises from this discrepancy in plasma regime 3 is: what is causing the run-away erosion?

One possible answer to this lies in the more than factor of two increase in target current between plasma 2 and plasma 3. With target currents in the 350 mA range for plasma regime 3, the targets got quite hot since there is no active cooling in the target holder. It is possible that the extreme temperature of the target greatly affected the sputtering yield, causing the target to rapidly erode after only a few minutes of heating up. The increased thermal motion of the copper atoms will enhance the erosion due to increased vaporization.

This argument is also supported by the photos of each target. As was mentioned previously, the back sides of the targets subjected to plasma regime 3 are eroded as well as the front side, even though they were pressed tightly against the heating plate of the target holder. Since the back side of the target was not subjected directly to the plasma, yet eroded nonetheless, it is possible that the high temperature caused the copper coating to delaminate from the aluminum substrate. The aluminum disks
were polished and coated with copper. It is a reasonable hypothesis that the bond between coating and substrate would degrade to the point of delamination at high temperatures. Target 11 in particular shows very clear support of extreme temperatures due to the bubbles and cracks in the previously polished aluminum substrate. For future experiments, an actively cooled target holder is paramount to ensure controlled temperatures in all plasma regimes.

The in-situ erosion yields measured from the targets before the sample got to hot could be prolonged once an actively cooled target holder is implemented in DIONISOS. These in-situ erosion measurements provide valuable insight because they show directly that the erosion rate is constant until a catastrophic event occurs at high temperature, which would be, and was, lost in a time integrated measurement. Further in-situ erosion experiments could be conducted with proper temperature control more reliably. For example, a more extensive target bias scan in a fixed plasma regime would provide a better understanding of the net erosion rate as a function of incident ion energy. In addition, other plasma regimes can be used to gather more data on the effect of different flux values on the erosion rate. Hydrogen plasmas and argon plasmas could also be used to observe the effects of different species on the erosion process. For instance, erosion of copper in hydrogen plasmas may be dominated by chemical sputtering rather than physical sputtering. Aside from simply increasing the parameter space and collecting more data to compare with the erosion rates expected by theory, other interesting aspects about plasma erosion such as particle transport can be probed.

When an atom is sputtered off the surface of a target, the possibility exists that it could be ionized in the plasma and return to the surface where it could cause more sputtering or simply rejoin the material. If this is the case, it would be interesting to determine the effects of self-sputtering and how it depends on the plasma parameters. It would also be intriguing to establish if and where there is a preferred location of redeposition and why. DIONISOS is uniquely equipped to study these questions and others surrounding the transport of sputtered particles using spatial scans of the target with the method of real-time RBS analysis developed in this thesis.
Although this study did not incorporate fusion relevant materials directly, due to time constraints, the goal of developing this real-time RBS erosion measurement method is to be able to apply it to materials such as molybdenum and tungsten, two prime candidates for divertor plates in current and future fusion devices. Coating aluminum targets in thin layers of molybdenum or tungsten and subjecting them to hydrogen and helium plasmas in DIONISOS will allow researchers to simulate the erosion process in fusion machines like ITER. Applying the method of real-time RBS analysis established here will provide important insight into how plasma-facing components will fair in reactors, before they are even built. Experiments like this will also allow researchers to study plasma-material interactions in a much more versatile capacity than in large fusion machines. Multiple samples can be tested daily with real time RBS in DIONISOS, whereas in big devices samples have hundreds to thousands of plasma shots worth of exposure compounded on them and can only be tested when they are taken out of the reactor during maintenance periods. The incredible advantage of being able to study the plasma erosion process as it happens in DIONISOS will be crucial to furthering the field of plasma-material interactions research and improving fusion devices for the future.
Chapter 8

Conclusion

The experimental procedure for real-time RBS analysis of plasma erosion developed in this thesis provides a new diagnostic tool for the field of plasma-material interactions research. The benefits of this method include understanding of erosion processes on much shorter time scales than was previously possible and versatility of various target materials, plasma species, and plasma parameters. As a result, researchers will be able to gather large amounts of knowledge about plasma erosion, and plasma-material interactions in general, spanning a wide parameter space. Real-time RBS analysis of plasma erosion in DIONISOS will provide valuable research to the fusion community in the hopes of solving the issues associated with plasma-material interactions that are so problematic for plasma-facing component lifetimes. The only way to bring fusion power closer to reality is to improve the lifetime of these components one way or another so that reactors can be run essentially at steady state. This thesis aims to make contributions to furthering the knowledge of the processes which are so integrally related to this scientific and engineering challenge.
Bibliography


