Ion Beam Analysis of First Wall Materials Exposed to Plasma in Fusion Devices

PER PETERSSON
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Abstract

One major step needed for fusion to become a reliable energy source is the development of materials for the extreme conditions (high temperature, radioactivity and erosion) caused by hot plasmas. The main goal of the present study is to use and optimise ion beam methods (lateral resolution and sensitivity) to characterise the distribution of hydrogen isotopes that act as fuel. Materials from the test reactors JET (Joint European Torus), TEXTOR (Tokamak Experiment for Technology Oriented Research) and Tore Supra have been investigated.

Deuterium, beryllium and carbon were measured by elastic recoil detection analysis (ERDA) and nuclear reaction analysis (NRA). To ensure high 3D spatial resolution a nuclear microbeam (spot size <10 µm) was used with 3He and 24Si beams. The release of hydrogen caused by the primary ion beam was monitored and accounted for.

Large variations in surface (top 10 µm) deuterium concentrations in carbon fibre composites (CFC) from Tore Supra and TEXTOR was found, pointing out the importance of small pits and local fibre structure in understanding fuel retention. At deeper depths into the CFC limiter tiles from Tore Supra, deuterium rich bands were observed confirming the correlation between the internal material structure and fuel storage in the bulk.

Sample cross sections from thick deposits on the JET divertor showed elemental distributions that were dominantly laminar although more complex structures also were observed. Depth profiles of this kind elucidate the plasma-wall interaction and material erosion/deposition processes in the reactor vessel.

The information gained in this thesis will improve the knowledge of first wall material for the next generation fusion reactors, concerning the fuel retention and the lifetime of the plasma facing materials which is important for safety as well as economical reasons.

Keywords: Ion beam analysis, Microbeam, Plasma wall interaction, Deuterium, Beryllium, Carbon fibre composites, Divertor, Nuclear reaction analysis, Helium-3

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Paper III: I prepared the cut samples, planned the comparisons of the three surface treatments, handled the majority of ion beam measurements, data analysis and writing of the paper.

Paper IV: I participated in the ion beam measurements as well as the data analysis and writing of the paper.

Paper V: I contributed to the selection of samples and handled the majority of ion beam measurements, data analysis and writing of the paper.

Paper VI: I participated in the ion beam measurements as well as the data analysis and writing of the paper.

Paper VII: I prepared the samples for ion beam analysis and handled the majority of ion beam measurements, data analysis and writing of the paper.

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Papers not included

A In-situ measurements of carbon and deuterium deposition using the fast reciprocating probe in TEXTOR

B Fuel retention in carbon materials under ITER-relevant mixed species plasma conditions

C Interactions of diamond surfaces with fusion relevant plasmas
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D Laser-induced removal of co-deposits from graphitic plasma-facing components: Characterization of irradiated surfaces and dust particles
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Abbreviations and Symbols

CFC  Carbon-fibre composite
ELM  Edge localised mode
ERDA Elastic recoil detection analysis
IBA  Ion beam analysis
IFMIF International fusion materials irradiation facility
ITER International thermonuclear experimental reactor
JET  Joint European torus
LCFS Last closed flux surface
NRA  Nuclear reaction analysis
PFC  Plasma facing components
PFM  Plasma facing materials
PIXE Particle induced X-ray emission
PWI  Plasma wall interaction
RBS  Rutherford backscattering spectroscopy
SEM  Scanning electron microscopy
SIMS Secondary ion mass spectrometry
SOL  Scrape of layer
TEM  Transmission electron microscopy
TEXTOR Tokamak experiment for technology oriented research
ToF  Time of flight
TPL  Toroidal pump limiter

$M$  Mass of particle
$E$  Energy of particle
$\theta$  Scattering angle
$\phi$  Recoil angle
$K_{RBS}$ Kinematic factor for backscattering
$K_{ERDA}$ Kinematic factor for recoils
$I$  Ion fluence
$K_{prob}$ Probability constant
$V$  Interaction volume
$\rho$  Hydrogen concentration
$\Gamma$ Resonance width
1. Introduction and motivation

From fires to nuclear power stations, energy has always been central to human development and various novel sources have always been examined. Controlled thermonuclear fusion is one of the latest. Fusion, if realised under terrestrial conditions, can provide almost limitless supply of energy. Currently the international thermonuclear experimental reactor (ITER) [1] is driving the transformation of controlled thermonuclear fusion into a usable energy source.

Several different fusion reactions exist where light nuclei are fused together to heavier while releasing energy. The fusion reaction requiring lowest temperature is when two hydrogen isotopes deuterium (D) and tritium (T) react to produce a neutron (n), a helium nucleus \(^{4}\text{He}\) and energy:

\[
\text{D} + \text{T} \rightarrow \text{n} + ^{4}\text{He} + 17.59 \text{MeV}
\]

This reaction still requires plasma heating to over a hundred million degrees Kelvin or a kinetic energy of the particles of the plasma over 10 keV. To contain the plasma, strong magnetic fields can be used. Technical challenges associated with a reactor construction still exist and one of the main goals of ITER is to develop and test plasma-facing materials and components [2].

Tritium does not exist in sufficient quantities in nature and has to be produced inside the reactor structure. The proposed method to obtain tritium is to capture neutrons from a fusion reaction with a lithium (\(^{6}\text{Li}\)) blanket surrounding the reactor according to:

\[
\text{n} + ^{6}\text{Li} \rightarrow \text{T} + ^{4}\text{He} + 4.8 \text{MeV}
\]

The radioactivity of tritium complicates handling of all exposed reactor components and also poses a risk in case of accidents but the comparatively short half life of 12.3 years eliminates long-term hazards. Consequently it is important to have a good understanding of how the different hydrogen isotopes interact with plasma facing materials (PFM) and, where and if it is stored in the reactor walls. Part of fusion research has been and is increasingly focused on this field [3-8]. Because of the radioactive nature of


tritium most current fusion experiments use pure deuterium fuel which gives the following two reactions.

\[
\begin{align*}
D + D & \rightarrow H + T + 4.0 \text{MeV} \\
D + D & \rightarrow n + ^3\text{He} + 3.3 \text{MeV}
\end{align*}
\]

The large similarity between deuterium and tritium makes deuterium plasma an excellent choice in most experiments.

1.1 Aim of the thesis

The main objective of the present study is to improve the understanding of wall material modification by erosion process and accumulation of hydrogen isotopes by the use of ion beam methods. Special focus has been placed on optimising the methods for high lateral resolution and sensitivity.

The techniques have then been applied for quantification and precise mapping of hydrogen isotopes and first wall materials carbon and beryllium from reactors such as TEXTOR (Tokamak Experiment for Technology Oriented Research) located in Germany and JET (Joint European Torus) located in England as well as other sources.

1.2 Plasma confinement in a tokamak

The main issue of controlled thermonuclear fusion is to get a high plasma density at high temperature for a long period of time inside a reactor [9]. These three conditions are coupled. The triple product of plasma density, ion temperature and energy confinement time is used as a measurement of how “good” a particular plasma confinement is.

There are two efficient approaches to produce the aforementioned high triple product. The first, inertial confinement [10-12], relies on a very fast heating and compression of fuel e.g. by lasers focused on a small D-T pellet. This can give both high density \(10^3 \text{ g cm}^{-3}\) and temperature \(10^8 \text{ K}\), but short confinement time. The second approach, which will be applied in ITER, is magnetic confinement where the hot plasma is spatially controlled by strong magnetic fields.
Figure 1.1 Schematic drawing of the main coils and the resulting fields in a tokamak fusion reactor. This image is © Copyright Protected by ITER.

The most common arrangement for magnetic confinement is called a tokamak, a Russian acronym for “toroidal chamber with magnetic coils”, and a sketch of the torus, or doughnut, is displayed in Figure 1.1. The toroidal magnetic field generated by coils around the actual plasma is the stronger magnetic field used for the confinement. A weaker field, called the poloidal field, is generated by coils in the plane of the torus and prevents the plasma from drifting away. The poloidal coils also induce a large current in the plasma by acting as a transformer where the plasma forms the secondary coil. This induced current is also important for the confinement of the plasma. The overall effect of the different fields is closed surfaces of magnetic field lines along which the ions and electrons of the plasma move.

Inside the core plasma the magnetic field forms a series of closed magnetic flux surfaces and after the last closed flux surface (LCFS), also called separatrix, a scrape-off layer (SOL) is formed. Ions that reach the SOL will follow the field lines and are dumped into the wall. The dumping position can be controlled by terminating the field lines with so called limiters. An approach used in some reactors such as ITER is to terminate most of the field lines in a dedicated structure at the bottom or top of the tokamak referred to as a divertor. Limiters are also used in the divertor configuration for the initial face of the plasma experiment and to protect sensitive equipment.
A divertor configuration is preferred compared to only limiters as the divertor is further from the core plasma then limiters and released wall materials then will less likely contaminate the central plasma. A divertor also makes high confinement modes more accessible in which a transport barrier [13] is formed at the edge of the plasma allowing for higher plasma densities.

1.3 Plasma heating in a tokamak

To achieve the required high temperature, several methods of heating the plasma are used. The initial heat is generated by the “transformer current”, already mentioned, that passes through the plasma and raises the plasma temperature. This is referred to as “ohmic heating” and is efficient to heat the plasma to temperatures of a few keV. Plasma resistance decreases and the heating consequently drop when the temperatures increases further so to reach sufficient high temperatures auxiliary heating is required. One form is neutral beam injection [14] where atoms with a high velocity enter through the magnetic confinement into the plasma and deliver energy to the plasma. Radio frequency heating [15] is a second alternative of auxiliary heating where radio waves are used to heat the plasma. For a commercial, energy producing, fusion reactor it is important that alpha particles from the fusion reaction provide a large portion of plasma heating, since auxiliary methods consume large amounts of electricity.

1.4 Plasma-surface interactions

The ideal process of particle transport can be seen in Figure 1.2 where fuel is injected as gas near the plasma edge or shot in as a pellet. A small portion of the fuel also enters by the neutral beam injection. In both cases the atoms then become ionised and hopefully fuse together. When fuel ions or helium ash leak out of the confinement it is transported to the wall where it becomes neutral gas and is pumped away. In the divertor configuration ions that leave the plasma can be cooled because the field lines in the SOL twists several turns around the torus before connecting to the wall in the divertor giving a long apparent distance.
In reality, the interactions between particles leaving the plasma and the wall are far from ideal and some of the main processes are shown in Figure 1.3 [16].

There are two main mechanisms for the wall erosion by the ions from the plasma [17; 18]: physical sputtering and chemical erosion. In physical sputtering [19] kinetic energy is transferred from the impinging ion to atoms of the wall thus breaking bonds and causing removal of species by sputtering. Light elements are more easily sputtered then heavier and heavy incoming ions are more effective in sputtering than light ions. The effect of
the erosion can also be enhanced due to chemical reaction between the incoming ion and the wall; this is referred to as chemical erosion (or chemical sputtering). Chemical erosion of carbon is one of the problems in fusion devices with carbon PFM.

Some of the eroded wall atoms are re-deposited locally, i.e. in the vicinity of the erosion area. Another fraction of the materials is re-deposited further away and this can lead global material migration in a tokamak [20; 21]. Either mechanism can heavily modify properties of the PFM, both in the erosion and the deposition zones.

Fuel species are deposited together with eroded wall in a process called co-deposition [22]. Hydrogen isotopes can also diffuse into the bulk of the wall and be retained there. Part of the sputtered wall material is also transported to the core leading to plasma pollution and cooling by energy loss due to radiation. PFM with low atomic number are preferable as they will radiate less energy when ionised compared to ions with high atomic number.

Sometimes the higher than average amount of particles leaves the plasma. This can either be connected with a single event such as instability in the core or repeated smaller events that are primarily associated with edge localised modes (ELMs). This type of event can be highly damaging to the wall and could render the reactor un-functional by e.g. melting the dedicated plasma facing material.

In present-day devices the effect of neutron irradiation is normally fairly small in comparison to surface modification. Neutron-induced activation still has to be considered as it has an impact on safety issues. For a reactor-class device the neutron-wall interactions and the capture of neutrons are very important. It will probably be studied in more detail in the future in a special material test facility: IFMIF – International Fusion Materials Irradiation Facility.

In a thermonuclear fusion reactor the need to keep the plasma at a temperature over 100 million degrees K requires large amount of heating in excess of the energy produced in the nuclear reaction to replace the energy lost from the plasma. Most of the lost energy (~80%) will be in the form of neutrons that are absorbed in the blanket behind the first wall, but a significant remaining portion will be in the form of photons and energetic particles that interact with the wall. To handle this heat load one of the most important parameter for wall materials is heat conductivity.

Plasma is chemically and physically aggressive to all plasma-facing components [23]. The erosion rate has to be controlled because there are only very limited possibilities for in-situ repair and/or replacements of components. Therefore, the choice of wall material is also crucial for long term plasma operation.

Different carbon materials, such as graphite, have low atomic number and are resistant to heat. Consequently carbon-based materials are commonly
used in present-day fusion experiments. However, as already stated, carbon is chemically eroded by the plasma and this will increase the release of carbon from the walls. Hydrogen from the plasma will react with carbon and the loss of fuel and the erosion on the walls can be critical for the economy of a commercial fusion reactor. The accumulation of tritium in dust and deposits is also a safety concern because of the radioactivity [24].

Other materials than carbon are currently tested in JET [25-28] and suggested for the ITER design [29-31] where the part of the wall with least heat exposure will be constructed of beryllium that has a low atomic number and relative high heat resistance. Part of the divertor is planned to be made of tungsten that is even more heat resistant then beryllium and exhibits low sputtering yield. For the most exposed parts of the divertor an extra heat resistant form of carbon known as carbon-fibre composite (CFC) might be necessary to use since tungsten can melt at exceptional heat loads associated with disruptions and giant ELMs.

### 1.5 Tokamak installations

Besides JET and the future ITER there are several different fusion experiments in the world and summary of major parameters for some of the installations are presented in Table 1.1. All values should be considered as typical and changes due to experimental conditions and modifications. The most relevant for this work, TEXTOR[20; 32-35], JET[25; 33; 36-38] and Tore Supra[39-41], are presented in more detail in chapter 3. JT-60U[42], D-IID[43; 44], EAST[45; 46] and KSTAR[47; 48] are the largest tokamak in Japan, America, China and South Korea, respectively. ASDEX-Upgrade[49-52] is a European machine that tests different concepts such as all metal wall. Research are focused at supplying input to basic physics and to the construction of ITER[6; 29; 30] and its follower the demonstration fusion power plant (DEMO) on the path to a commercial power source.
<table>
<thead>
<tr>
<th>Tokamak</th>
<th>Major radius of torus (m)</th>
<th>Minor radius of torus (m)</th>
<th>Auxiliary Heating (MW)</th>
<th>Magnetic field on axis (T)</th>
<th>Plasma Current (MA)</th>
<th>Plasma Lifetime (s)</th>
<th>In operation (Year)</th>
<th>Wall materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEXTOR (Germany)</td>
<td>1.75</td>
<td>0.47</td>
<td>9</td>
<td>3</td>
<td>0.8</td>
<td>10</td>
<td>1983</td>
<td>Metal Graphite</td>
</tr>
<tr>
<td>JET (England)</td>
<td>3</td>
<td>1.25</td>
<td>50</td>
<td>4</td>
<td>7</td>
<td>60</td>
<td>1983</td>
<td>Beryllium CFC</td>
</tr>
<tr>
<td>JT-60U (Japan)</td>
<td>3.4</td>
<td>1</td>
<td>50</td>
<td>4.2</td>
<td>6</td>
<td>15</td>
<td>1985</td>
<td>Graphite CFC</td>
</tr>
<tr>
<td>D-IIID (USA)</td>
<td>1.67</td>
<td>0.67</td>
<td>26</td>
<td>2.2</td>
<td>3.0</td>
<td>5</td>
<td>1986</td>
<td>Graphite</td>
</tr>
<tr>
<td>ASDEX-Upgrade (Germany)</td>
<td>1.65</td>
<td>0.5</td>
<td>3.3</td>
<td>3.1</td>
<td>1.6</td>
<td>10</td>
<td>1990</td>
<td>Tungsten coated carbon</td>
</tr>
<tr>
<td>Tore Supra (France)</td>
<td>2.4</td>
<td>0.70</td>
<td>12</td>
<td>4.5</td>
<td>1.7</td>
<td>-1000</td>
<td>1998</td>
<td>CFC</td>
</tr>
<tr>
<td>EAST (China)</td>
<td>1.7</td>
<td>0.4</td>
<td>7.5</td>
<td>3.5</td>
<td>0.5</td>
<td>-1000</td>
<td>2006</td>
<td>Metal</td>
</tr>
<tr>
<td>KSTAR (South Korea)</td>
<td>1.8</td>
<td>0.5</td>
<td>15.5</td>
<td>3.5</td>
<td>2</td>
<td>20</td>
<td>2008</td>
<td>Graphite CFC</td>
</tr>
<tr>
<td>ITER (France)</td>
<td>6</td>
<td>2</td>
<td>110</td>
<td>5.3</td>
<td>15</td>
<td>420-</td>
<td>~2020</td>
<td>Beryllium Tungsten CFC</td>
</tr>
</tbody>
</table>
2. Ion Beam Analysis

Material characterisation by ion beam analysis (IBA) relies on interactions induced by ion beams produced by particle accelerators. When an ion from the accelerator interacts with atoms in a solid matrix, energy is transferred to the solid causing a slowing down of the incoming beam. Most of the energy is transferred via electronic excitations and is quantified by the electronic stopping power that describes the energy loss per unit path length. The ions can also interact with the nuclei of the solid and this mode of energy loss is consequently referred to as nuclear stopping and dominates at low velocities. For the fast ions used in ion beam analysis the electronic stopping power is dominant and can be seen as a friction force, gradually slowing down the particle. If the incoming ion and a nucleus of the sample collide, information about the sample can be obtained by analysing the mass and energy spectra of the resulting particles.

Figure 2.1 Different types of IBA techniques

IBA provides several different techniques, see Figure 2.1, useful for analysis of materials used in fusion technology [53]. This work focuses on examination of hydrogen isotopes accumulation in PFM, especially
deuterium retention and migration. For its characterization elastic recoil detection analysis (ERDA), Nuclear Reaction Analysis (NRA) and back scattering analysis are used [54]. In some cases a given ion beam can be utilised for simultaneous measurements with more than one technique. To demonstrate the different techniques simulated spectra of the same carbon substrate with a thin co-deposited film of H, D, C, Mo and W are shown along with sketches of the interactions in Figures 2.2, 2.3 and 2.5. The samples elements are chosen to represent a possible sample from a fusion device.

2.1 Rutherford back scattering spectroscopy

The most common IBA technique is Rutherford back-scattering spectroscopy (RBS). Light ions, normally helium or protons, are used as the primary ion beam and pure electrostatic (Coulomb) interaction is assumed to describe the collision between the incoming charged projectile and the target atoms (Figure 2.2a).

![Image](image_url)

Figure 2.2 (a) Principle sketch describing a RBS measurement showing the incoming particle with mass $M_1$ and the target atom with mass $M_2$ before and after the interaction. (b) Multi elemental spectrum for a 2 MeV $^4$He beam and 165° scattering angle ($\theta$) of the test sample. Hydrogen and deuterium cannot be observed in the spectrum since they have lower mass than the incoming beam.

The energy of the ion before and after collision is expressed by the kinematic factor $K_{RBS}$ for a specific target particle combination.
\[
\frac{E_{after}}{E_{before}} = \left( M_1 \cos \theta + \sqrt{M_2^2 - M_1^2 \sin^2 \theta} \right)^2 \left( M_1 + M_2 \right)^2 = K_{RBS} \quad (2.1)
\]

where the mass of the incoming ion is \( M_1 \), the target atom mass is \( M_2 \) and the scattering angle \( \theta \). The energy of the back-scattered ions can be measured with a detector. The likelihood of scattering (the scattering cross section \( \sigma \)) can be calculated using the Coulomb interaction between two point charges, and can also be experimentally determined for different target-beam combinations. Collisions deeper inside the sample will give scattered particles with lower energy compared to collisions close to the surface due to the stopping of ions on the way into and out of the sample. There are readily available methods and computer codes, such as TRIM [55] and SIMNRA [56] that combine theory and experimental data for simulating and interpreting spectra.

The mass of the incoming ion must be less than the target ion for backscattering to be possible. This is the reason why H and D are not visible in Figure 2.2b. Protons can be scattered backwards by deuterium, but at the relevant energies the Rutherford approximation of Coulomb interaction does not hold. As long as the reaction is elastic it has the same kinematic factor but a different cross section compared to the Rutherford approximation and can be referred to as enhanced proton scattering. For protons scattering on deuterium or tritium the measured cross section is higher than the calculated RBS cross section. The deuterium and tritium signals will, however, be weak due to the low \( K_{RBS} \), although the technique can still be useful in some situations.

### 2.2 Elastic recoil detection analysis

In elastic recoil detection analysis (ERDA) the recoiling ions are analysed instead of the backscattered particles and a principle view of the method and a typical spectrum can be seen in Figure 2.3.
Figure 2.3 (a) Schematic drawing showing particles before and after collision for ERDA with a foil in front of the detector to stop all but the hydrogen isotopes from entering the detector, and (b) a spectrum where a 15 MeV Si beam, 21µm mylar foil and 45° recoil angle is used for the test sample.

Energy and momentum are conserved in the interaction and the relation between energy of the recoiling target atom ($E_{\text{After}}$) and the incoming particle ($E_{\text{Before}}$) can easily be calculated when the mass of the incoming ion ($M_1$), energy, target mass ($M_2$) and the recoil angle ($\phi$) are defined:

$$K_{\text{ERDA}} = \frac{E_{\text{After}}}{E_{\text{Before}}} = \frac{4M_1M_2 \cos^2(\phi)}{(M_1 + M_2)^2} \text{ where } \phi < \frac{\pi}{2}. \quad (2.2)$$

As $\phi < \pi/2$ the target needs to be tilted relative to the incoming beam as indicated in Figure 2.3 (or a thin target can alternatively be used).

An alternative recoil detection method is time-of-flight (ToF) ERDA [57]. In a ToF-E setup both the velocity, via time of flight measurement and the energy of the target are measured and consequently both recoil particle mass and depth can be calculated. Start and stop signals for a timer is generated when the recoiled particle passes thin foils and with the known distance between the foils the velocity is calculated. The energy of the recoil is measured with a standard solid state energy detector after the last foil.
2.4 An ERDA Time-of-flight spectrum measurement for deuterium implanted into carbon. The different elements in the sample can be seen as curves in the spectrum.

With ToF-E detection separation of hydrogen isotopes is easy due to the large relative difference in mass. The low probability that a hydrogen ion will be registered in the thin foils however means that long measuring times are needed and that quantitative analysis is difficult. The long distance needed for the ToF tube (~0.5m) gives a small solid angle for detection and consequently longer measure times. Heavy ions are required for all atoms to get sufficient energy to leave the sample and in Figure 2.4 40 MeV $^{127}$I are used as the primary beam.

2.3 Nuclear reaction analysis

Nuclear Reaction Analysis (NRA), in contrast to RBS and ERDA, is based on specific nuclear reactions between the incoming ion and target nuclei. This means that different nuclear reactions have to be used depending on the elements investigated. For nuclear reactions to occur the incoming ion has to have a sufficiently high energy.
Most nuclear reactions used for NRA are in fact fusion reactions where two light elements are combined to form new particles and the release of energy.

One important reaction is where a $^3$He ion reacts with D to form a proton and a $^4$He particle. A shorthand form to write this is: $\text{D}(^3\text{He},p)^4\text{He}$, where the particles inside the parenthesis are the incoming ion and the particle used for the analysis. The energy spectra for the particles from that reaction and from $^{12}\text{C}(^3\text{He},p)^{14}\text{N}$ are seen in Figure 2.5. Energy is released in the reactions so the protons will have higher energy than the incoming beam and the spectra will, therefore, have low disturbing background noise. At low energies the background can also be reduced by a foil that stops backscattered $^3$He ions.

In the field of fusion the determination of deuterium content is important and the reaction $\text{D}(^3\text{He},p)^4\text{He}$ is widely used. In this reaction 18.4 MeV of energy is released and the protons will have energy of about 12 MeV after the reaction. The cross section, seen in Figure 2.6, has a maximum around 0.6 MeV but by using higher beam energy the deuterium content can be probed at greater depths.
Figure 2.6 Nuclear reaction cross section for the \(^2\text{D}(^3\text{He},p)^4\text{He}\) reaction [58-61]

\(^3\text{He}\) ions can also react with carbon according to: \(^{12}\text{C}(^3\text{He},p)^{14}\text{N}\). The \(^{14}\text{N}\) nucleus that is formed can have several energy levels and different amount of kinetic energy is then available for the emitted protons. The corresponding distinct peaks in the energy spectrum as seen in Figure 2.5b are labelled \(C_{p0}\), \(C_{p1}\) and \(C_{p2}\) to show the specific reaction. The cross sections for the reactions can be seen in Figure 2.7.
$^3$He can also be used to measure the amount of beryllium and the corresponding cross sections for two different reaction channels are displayed in Figure 2.8. The reaction has a Q value of 10 MeV and $p_0$ and $p_1$ protons will have energies of about 10 and 8 MeV, respectively.

Figure 2.8 Nuclear reaction cross section for the $^9$Be($^3$He,p)$^{10}$B [63] reactions in the 150° direction.
These types of reactions can also be observed by the characteristic gamma rays emitted when the involved nuclei decays. Another available advantage in nuclear reaction analysis is the use of resonances, where the reaction has a considerably enhanced cross section in a small energy interval. The $^6_1H(^{15}_{14}N, \gamma ^4_{4}He)^{12}_{6}C$ [64] reaction for hydrogen has an extraordinary narrow resonance ($\Gamma=1.8$ keV) at 6.385 MeV. By scanning the energy of the nitrogen beam the hydrogen content can be probed with high sensitivity and depth resolution.

2.4 Particle induced X-ray emission

In particle induced X-ray emission (PIXE) the interaction between the beam and the target electrons is explored. In this process the primary ion beam ionise the target atom and the subsequent relaxation give rise to characteristic X-rays.

Using high resolution detectors the different X-ray peaks can be separated and the atomic elements identified. X-ray energies depend solely on the electron configuration; PIXE can therefore not be used to separate isotopes. Protons are normally used as incoming particle as they give high cross section for x-ray formation. PIXE is well suited for microbeam applications as a proton beam requires lower magnetic field to be focused and each incoming ion can produce a significant amount of X-ray photons.

2.5 Hydrogen release during IBA measurements

Ion beam techniques are generally considered as non-destructive. The energy transferred during the interaction between the incoming ion and sample material can, however, affect the target composition [65-67]. This process called “detrapping” is most pronounced for light elements in the sample such as hydrogen. In order to estimate the hydrogen concentration in a sample before irradiation by the probing beam, knowledge of the release mechanism and models for interpolating the original hydrogen concentration are necessary. This is especially important in a microbeam application where the high current density of the focused beam can in some instances cause a dramatic impact on the sample composition during an IBA measurement.

In literature, several different models for hydrogen release during ion irradiation of carbon matrixes have been proposed. Phenomenological descriptions of the observed data that use one or several exponential functions have been suggested [65; 68]. Although this approach describes the data quite well, no physical interpretation is normally assigned to the parameters.
A model for hydrogen release from amorphous carbon has been proposed by Adel [69]. He assumed that the incoming ion breaks hydrogen bonds along its track in the sample. These free hydrogen ions can be retrapped in the sample or react with other free ion to form a hydrogen molecule if the two ions are within a characteristic volume. The hydrogen molecule will then diffuse and be released from the sample. This process can be described by the differential equation:

$$\frac{d\rho}{dI} = -K_{\text{prob}} \rho (\rho V - 1)$$

(2.3),

where $I$ is the ion fluence, $V$ the interaction volume and $K_{\text{prob}}$ a probability constant for the reaction. By solving this differential equation the initial hydrogen concentration $\rho_0$ is given:

$$\rho(I) = \left( V + \frac{1}{\rho_0 - V} e^{-KI} \right)^{-1}$$

(2.4).

In order to validate Adel’s model we made ERDA measurements with three different ion beams. In Figure 2.9 the yield of deuterium in an amorphous carbon film is shown as a function of ion fluence where a strong dependence on ion species and ion fluence can be seen. Three different fits using Adel’s model can be seen in Figure 2.9 which describes the fluence dependency for

Figure 2.9 Normalised yields of deuterium as a function on ion fluence for an a-C:D film measured by ERDA with three different ions beams. The data is fitted according to Adel’s model.
the different incoming ions well. Significant decrease in yield for the two heavier ions is observed while the lighter He ion shows no loss of deuterium on this fluence scale.

We collect data in list mode, which records each event and allows for monitoring of the hydrogen yield as the measurement progress. Decrease has been observed during measurements with $^3\text{He}$ and can be seen with the nuclear microbeam if a stationary focused beam is used and can then be compensated for. Figure 2.10 is an example of one such measurement.

![Graph](image)

Figure 2.10 Normalised yields of deuterium as a function of ion fluence for a deposited film from JET. The measurement was performed by NRA with a 2.5 MeV $^3\text{He}$ beam and the data fitted according to Adel’s model.
3. Equipment

3.1 Fusion experiments

In this thesis materials exposed to plasma in several fusion experiments and plasma simulators in Europe and USA have been studied. For a brief introduction some basic data for some of the world’s tokamaks are presented in Table 1.1. Below the most relevant for this thesis is presented in more detail.

Figure 3.1 Toroidal view inside the TEXTOR vacuum vessel: (1) Inconel® liner, several types of limiters (2)-(4) and, (5) antennas for auxiliary plasma heating with radio frequency waves. This image is © Copyright Protected by TEXTOR.

TEXTOR

Toroidal experiment for technology oriented research (TEXTOR), see Fig 3.2, is medium sized tokamak (7 m³ plasma volume) located in Jülich, Germany. The tokamak is equipped with several different types of diagnostics and probes that make it suitable for performing plasma material interaction experiments. As can be seen in Figure 3.1 the cross section is circular with
several different limiters. Samples can be inserted in the plasma edges by probes for exposure during parts of a single plasma shoot. Materials can also be mounted on various transfer systems with special holders for test limiters and probes. They are positioned in the scrape-off layer for exposure to a single plasma pulse or during several days of operation.

Figure 3.2 Toroidal view inside the JET vacuum vessel: (1) divertor, (2) inner wall guard limiters, (3) outer poloidal limiters, (4) antennas for radio frequency heating. The left part shows the configuration from 1999-2001 and to the right the configuration from 2002-2004. This image is © Copyright Protected by EFDA JET.

JET

Joint European Torus (JET) (see Figure 3.2) located at Culham Science Centre outside Oxford, UK is currently the world’s largest fusion experiment with a plasma volume of 80 m$^3$. The facility has plasma parameters that are the closest to the future ITER experiments compared to other current experiments. Most material for analysis is collected from the interior during the major shutdowns after long experimental campaigns. In Figure 3.3 part of the divertor is shown with the most exposed part of the plasma facing components (PFC). The divertor has been changed several times to test different configurations and plasma operation scenarios while the basic structure and most of tiles have remained the same from 1998 until 2009, when the refurbishment of the wall started in order to install beryllium and tungsten plasma facing components for the ITER-Like Wall Project[28].
Figure 3.3 Schematic of the different tiles that made up on poloidal cross section of the MkII Gas Box divertor used between 1998-2001. Strike point positions, where most of the energy is deposited, on the vertical targets (Tiles 3 and 7) is shown in gray. Other strike points are also possible. The numbers 1-8 refer to different tiles. The size of the divertor is about a half meter. This image is © Copyright Protected by EFDA JET.

Tore Supra

The size of Tore Supra is in between that of TEXTOR and JET; see Table 1.1. The machine has super conducting magnetic coils that allow for plasma pulses several minutes long. During the long pulses so much energy is delivered to the PFC that an active water-based cooling is needed to protect the wall from over heating. As seen in Figure 3.4 Tore Supra is a limiter machine without a divertor. The main and the largest power handling components is the toroidal pump limiter (TPL) seen in the bottom of the image.

Figure 3.4 Inside of Tore Supra with several different types of limiters marked. This image is © Copyright Protected by Tore Supra.
PISCES

PISCES-A is a steady-state linear-plasma simulator facility located at the University of California at San Diego (USA) used for the study of interaction between plasma and materials. The schematic of the experimental set-up is shown in Figure 3.5. Plasma is generated and then accelerated towards target. High fluxes of D ions impinging on a sample can be produced.

![Figure 3.5 Schematics of the PISCES-A plasma source](image)

ITER

The International Thermonuclear Experimental Reactor (ITER) currently under construction in Cadarache (Southern France) is the target for much of the fusion research today. The main goal is to produce more than 10 times more fusion energy than supplied by the heating system. ITER is also a scientific and technological experiment and especially the plasma-facing components in the divertor is an area for great research. In Figure 3.6 the different materials suggested for plasma facing components to handle the extreme requirements are marked: beryllium in the main chamber wall, tungsten on the baffle and the dome, whereas carbon fibre composites (CFC) would be used for the lower part of vertical targets where the greatest power loads will be deposited. This approach is planned for the initial phase of operation with hydrogen fuel. An all metal wall is presently assumed in order to avoid large tritium inventory during the D-D and D-T phase.
3.2 Equipment for ion beam analysis

A beam of well defined mono energetic ions is required when ion beam techniques are applied for analysis. To produce this beam an accelerator system is needed along with several beam line components, target chamber, sample holders and a data acquisition system. IBA cannot be performed inside the tokamak reactor vessel instead materials first have to be retrieved from the interior of the fusion reactor. Afterwards, they can be directly mounted in the surface analysis station. In some cases sample preparation is needed e.g. by cutting, polishing etc.

The purpose of the accelerator facility is to produce a stable beam of well defined ions in terms of mass, energy, charge, suitable for the material and IBA technique to be used. A drawing of the entire laboratory system in Uppsala, Sweden is depicted in Figure 3.7. The major units are: injector system (1); tandem accelerator (2) and beam lines with surface analysis stations for various types of measurements with a large beam size spot (3) and a microbeam (4).
Figure 3.7 Drawing of the tandem laboratory with the accelerator. The main components are further described below: 1 injector, 2 accelerator, 3 and 4 target beam lines used in fusion research.

Ion source

The role of the injector system (1) is to form a primary beam for the accelerator. The set-up comprises several types of ion generation units and analysing magnets/electrostatic deflectors. The negative ion beam for the accelerator is generated in the ion source and pre-accelerated to 50-100 keV.

For gaseous elements at room temperature, e.g. H₂, a negative ion beam is formed in a duoplasmatron were the gas is heated to form a plasma. The negative ions are extracted from the plasma boundary by an applied electric field gradient. For helium it is not possible to produce negative ions directly. Instead, the duaoplasmatron produces positive ions that are converted into negative ions by a charge exchanger. In the charge exchange channel incoming positive ions pass a vapour of potassium where electron transfer occurs.

Ions from solid materials are produced by sputtering, where a primary beam of Cs⁺ hits the solid target, eject atoms and simultaneously transfer an electron to the sputtered atoms. The negative ions are pre accelerated and analysed before entering the acceleration section. Different ion optical elements like lenses, slits and dispersive components are installed along the beam-line to control the ion transport. In total the Uppsala accelerator facility has two duoplasmatrons and two sputter ion sources.
5 MV pelletron accelerator

The negative ion beam is then steered and focused into the central beamline of the accelerator and accelerated by the positively charged terminal (up to 5 MV) toward the middle of the high voltage insulating pressure tank, marked as 2 in Figure 3.7. Inside the terminal the ions pass a so called stripper, consisting of a gas cell or a thin foil. The stripper removes electrons from the ion by ion electron collision creating positively charged ions that are now repelled from the terminal. By converting the charge of the ions, the high potential is used twice to elevate the ions kinetic energy.

The charge needed to keep the terminal and the stripper at a high potential is generated by a chain consisting of metal cylinders of a few cm (pellets) insulated by nylon rods that runs along the beamline inside of the pressure tank. In order to prevent sparks, the high voltage terminal is housed in a pressurised tank filled with insulating gas, SF₆.

The energy of the accelerated ions is determined by a well calibrated magnetic field were only ions with the correct energy and mass to charge ratio have the required curvature to physically pass through the 90° magnet. Another magnet is then used to direct the beam to a specific beam line.

Large sample experimental chamber

There are six different experimental stations in the experimental hall of the Tandem Laboratory at Uppsala University and two have been used in this thesis. The beam line marked as 3 in Figure 3.7 has two scattering chambers where samples can be installed for IBA. The first chamber is equipped both with a ToF telescope for ERDA as well as surface barrier detectors for RBS. The next chamber on this beam line is designed to handle a large variation of sample sizes and is often used for analysis of larger samples from fusion experiments. The sample can be moved both linearly as well as rotated.

Inside the chamber there are presently two solid state detectors mounted, one with a 150 µm active detection depth and one thick detector with an active depth of 1500 µm. The thick detector is needed to capture the high energy protons formed during some NRA measurements.

A LabVIEW control system has been developed as part of this work for positioning of the samples. The program can also control the data acquisition system which gives the possibility to measure large series of sample points automatically.

Microbeam

The ion beam used for analysis normally has a beam diameter of about 1 mm. This is a practical beam size that can be easily obtained using standard types of ion optical devices combined with a collimator in front of the
sample. However, the existence of very small samples and the need to have a good spatial resolution across the sample surface makes smaller beam spots necessary. It is difficult to manufacture collimators for ion beams in the µm range and the loss of incoming ions through such a small collimator would make measuring times unreasonably long. By focusing the beam the loss of beam current can be compensated for by increased intensity of the beam. The requirements on the collimator are reduced by placing it in front of the multi lens system that forms a de-magnified image of the collimator on the sample.

Figure 3.8 The end section of the microbeam line. Components along the beam line are: collimators, the scanning coils, the three quadropoles and the target chamber with a microscope for viewing.

To focus MeV ion beams to small dimensions, magnetic quadropoles are most commonly used, although electrostatic quadropoles, and/or superconducting solenoids are less common alternatives. Magnetic quadropoles have high focusing power, but only in one plane, and therefore quadropoles are normally installed in pairs to allow for focusing in both horizontal and vertical planes. For maximum demagnification more than two quadropoles can be combined [70].

Two arrangements that have been frequently applied in different labs are the so-called Oxford triplet and the Russian quadruplet [70] and in Uppsala a commercial Oxford triplet is installed (Figure 3.8). In Figure 3.9 a schematic view of the ion optics in the microbeam setup is shown as calculated with the PRAM software [71]. The trajectories of selected ions in the x, or horizontal plane, and the y, or vertical plane are shown in red and green, respectively. The magnetic quadropole triplet is seen in the figure at a
distance of about 6.5 meters from the first collimator while a second collimator is present at about 6 m from the first. This collimator prevents aberrations in the image by removing ions too far from the optical axis.

Figure 3.9 The ion trajectory from the object collimator at 0 m to the target at 7 m red paths is in the x plane and green in the y plane

The configuration shown in the figure gives high demagnification but requires high magnetic fields. Therefore, it can only be used for ions with low magnetic rigidity. To use the quadropoles for heavier ions a second configuration can be chosen (Figure 3.10). This allows the focusing of heavier ions at higher energy, but lower demagnification will be obtained. To achieve this mode the orientation of the last quadropole is changed by reversing the current in this coil.

Figure 3.10 Low demagnification ion path. The only different compared to the situation described in Figure 3.9 is the strength of the magnetic field and, consequently, the ion path.
With the high demagnification configuration, the demagnification achievable is a factor of 85 in the horizontal plane and 23 in the vertical. To achieve a very small beam spot on the target, i.e., with diameters < 10 µm, a well designed collimator is needed. In the Uppsala microbeam line beam spots of less than 10 µm have been achieved for both helium and carbon beams.
4. Results and discussion

The aim of this chapter is to provide an overview of technical achievements and scientific results obtained in studies of plasma-facing materials exposed in several tokamaks and in a plasma-wall interaction simulator. First, crucial steps in the calibration of the microbeam system are reported. This is followed by a summary of the most important results that improved the understanding of fuel accumulation and migration in the materials. For more details about the microbeam and hydrogen release see papers I and II. The fuel retention in CFC is described in papers VI and VII and the results of the deposited layers from JET can be found in papers II-V.

4.1 Microbeam spot size

Two different methods are used to determine the size of the focused beams. For larger beam spots an optical microscope is used for visual determination of the spot size. This is done by comparison to a grid with known dimensions. For smaller beam spots, mapping of backscattered ions from a grid are used. Normally, copper or gold nets for transmission electron microscopy (TEM) are used because they have well defined edges in a large range of dimensions.

The resolution of the 9 MeV $^{12}$C$^{+3}$ (0.1 nA) ion beam used in Figure 4.1a was determined by studying the projections from the marked regions, see Figure 4.1b. The vertical and horizontal resolution was determined to 5 µm and 8 µm respectively. The resolution can be further improved by decreasing the object collimator which limits the beam current and thus increase the data acquisition time.

Maps were made of samples implanted with a dose of $2 \times 10^{16}$ D/cm$^2$ with both ERDA and NRA. With both techniques regions were deuterium had been implanted could be separated from regions that had been masked off during implantation, but with limited resolution. For a single large beam spot higher sensitivity is possible but to produce maps higher concentrations is preferable for good resolution.

In summary, a beam spot of less than 10 µm has been achieved and then applied in studies reported in the following sections.
Figure 4.1 Backscattered carbon ions from a gold TEM grid (a). The resolution of the focused beam is determined by the width of the edges I and II in figure (b).

4.2 Ion beam induced hydrogen release

In quantified analysis of materials all factors influencing the accuracy must be recognised and assessed. Apart from thermal heating of the material, release may be caused by the deposited energy from the electronic stopping, see chapter 2.5. Ion-induced detrapping is subsequently expected to be more pronounced for Si ions than for He. The observed fast initial decrease followed by a stable phase that is described in 2.5 can also be seen in Figure 4.2. The sample has been exposed to the plasma and the deposited layer can be seen on top of the tile. The deposited layer is a mixed matrix containing carbon, beryllium, oxygen and stainless steel components together with deuterium fuel and, also a significant amount of hydrogen probably from water vapour. Along with the plot of the experimental data is also a fit to the data by Equation 2.4.

\[
\rho(I) = \left( V + \frac{1}{\rho_0 - V} e^{-KI} \right)^{-1}
\]  

(4.1)

The fit was calculated using the non linear simplex search method [72] minimising the square of the error \([V=5.9, \rho_0=0.040, K=0.3861, r = 0.99]\).
Figure 4.2 Concentration of deuterium as a function of the fluence for a 6 MeV $^{28}$Si$^{3+}$ ion beam. The solid lines represent a fit of equation 4.1 to the experimental data.

The effect of the primary ion beam on the original hydrogen profile was also investigated for vitreous carbon and studied as a function of ion fluence. Different beam fluxes were obtained by varying the ion beam spot size (0.15 – 3 µA/mm²) as well as scanning the beam over a large area.

Total yields for the hydrogen from the surface region of vitreous carbon are plotted in Figure 4.3 as a function of the ion fluence. The sample was moved ~2 mm between runs to avoid interference between subsequent measurements. The hydrogen close to the surface shows a decline for all different doses that is similar in shape to predictions by Adel’s model.

The variation of the hydrogen concentration in the sample for different ion doses exhibits the same dependence for a scanned and a stationary beam spot, so Adel’s model is appropriate for use with a focused ion beam both in stationary and scanned mode. This is in agreement with Adel’s assertion that all interaction takes place along the track of a single incoming ion and is independent of current density. For the measurements presented here all data has been collected in list mode that allows for monitoring e.g. the hydrogen yield as a function of time or ion fluence but for a light projectile such as helium the decay is often small as can be seen in Figure 2.9.

**In summary:**
Adel’s model has successfully been used to describe hydrogen detrapping.

No extra effects due to the increased current density of a focused beam have been observed.
Figure 4.3 The integrated yields of hydrogen as a function of the ion fluence for several densities of the beam current.

4.3 Fuel inventory in carbon fibre composites

In most present-day tokamaks PFC are made of carbon fibre composites (CFC). For such materials to be considered for future devices of a reactor-class a detailed understanding of the fuel inventory in CFC brand used in fusion machines is essential.

Carbon fibres are manufactured by first producing a polymer fibre which is then heated until carbonised. Dependent on the original fibre the resulting carbon fibre will have different properties [73; 74]. For CFC materials polyacrylonitrile (PAN) and petroleum pitch are used as precursors. The different fibres is then be known as ex-PAN and ex-pitch fibres and can either be woven into 2D sheets that are combined or needled together directly in a 3D structure to form the bulk material. The third step to form the CFC is to create a carbon matrix around the fibres by graphitising a carbonaceous gas or liquid. The finished material will then have both high strength and heat conductivity. Material properties can be tailored to demand with respect to thermo-mechanical performance by the choice of fibre type and structure.

The study of deuterium retention and distribution was carried out for carbon fibre composite NB41 manufactured by Snecma Propulsion Solids. This 3D-type composite consists on average of 77% ex-pitch fibres. The different fiber types were clearly visible by eye both before and after
exposure of the samples. Samples of NB41 were exposed to high flux deuterium plasmas in PISCES-A at normal angle with the total ion fluence of $3 \times 10^{21}$ D/cm$^2$, the surface temperature was 470 K.

After the exposure in PISCES-A, the fiber structure was still visible by the naked eye but a thin amorphous carbon layer formed by the bombardment of deuterium ions was visible as a darkening on top of the material. Figure 4.4 shows a map of deuterium content as measured with the microbeam. Two regions with distinctly different D content can be distinguished. They correspond to two different types of fibres, ex-PAN and ex-pitch.

Figure 4.4 Deuterium distribution (in at./cm$^2$) recorded with µ-NRA in the 8 µm thick surface layer of the NB41 sample exposed in PISCES-A: Different retention is observed in the two types of CFC constituents: ex-pitch and ex-PAN.

The difference in the D concentration is present both at the surface and deeper into the sample. Two reasons for the observed difference may be considered: density of respective fibres and surface roughness. PAN-type fibres are less dense (1.75-1.8 g/cm$^3$) than ex-pitch fibres (2 g/cm$^3$) [22], taking this into account greater D content could be expected in the less dense PAN fibres. The contradictory result speaks to the importance of sample roughness. Surface features in Figure 4.4 indicate the larger roughness, seen in Figure 4.5, of the broken fibres of the ex-pitch region compared to the smoother surface in the ex-PAN region where the fibres run along the sample surface.

The overall retention of the deuterium in a CFC material should still be possible to give good average values by using a beams spot larger then the needled structure, or by averaging several different measurements points.
Figure 4.5 Details of (a) the ex-pitch region with the fibre coming out of the sample and (b) the ex-PAN region with the fibre in the plane of the surface.

Figure 4.6 (a) shows a pattern of the deuterium distribution in the surface layer of CFC exposed in TEXTOR. In this case no direct link to the fibre structure can be made, probably due to more factors influencing the plasma-wall interaction in a tokamak compared to a simulator. This shows the importance of combining simplified experiments to study one factor at a time with larger experiments for an overall picture. In Figure 4.6 (b) the relative carbon yield also shows large variations. There are spots with concentration minima for both D and C. This can possibly be attributed to the presence of small cavities or pits in the fibre structure in the erosion zone. In such regions metallic plasma impurities can be accumulated, as discussed in [75].

In summary:

CFC exposed to plasma in PISCES-A shows variation in a deuterium distribution that can be linked to the fibre structure.

CFC exposed in TEXTOR shows both deuterium and carbon distributions to be inhomogeneous, but no direct link to the fibre structure can be made.

Figure 4.6 Elemental mapping with μ-NRA: (a) Deuterium distribution and content and (b) relative Carbon distribution in the erosion zone on a CFC probe exposed in TEXTOR
4.4 CFC limiter tiles from Tore Supra

Tiles (25 x 22 mm²) made of carbon fibre N11, SNECMA from the toroidal pump limiter (TPL) in Tore Supra were studied. The tiles position on the TPL is identified by two numbers denoting the finger (F, toroidal direction) and the individual tile (T, poloidal direction) with different plasma conditions for different positions of the limiter (see Figure 4.7).

![Figure 4.7](image)

Figure 4.7 Part of the toroidal pump limiter of Tore Supra. The numbers denoting the tile position is shown as well as different deposition region.

To give a better comparison of different tiles, the deuterium signal has been normalised with the intensity of protons from the p₀ and p₁ reaction channels with carbon.

![Figure 4.8](image)

Figure 4.8 Map of the relative surface deuterium to carbon yield for a µ-NRA measurement for tile F4/T19
A mapping recorded with µ-NRA of the D content on the plasma-facing surfaces of a tile from the deposition zone of the TPL is shown in Figure 4.8. The differences in the D content over the micro-region under examination reach over 10. On the contrary, in the erosion zone, the content is lower and fairly uniform. However, in some spots higher concentration is found. This may be attributed to the retention in small cavities or pits which act as tiny shadowed areas protecting implanted or co-deposited species from the direct plasma impact, as presented and discussed earlier [24; 25; 75; 76].

With the broad $^3$He$^+$ beam five measurements were made: in the middle and in four positions located 10 mm apart from the tile center. Figure 4.9 show the deuterium depth profiles and variations can be seen both in the near surface layer and deeper into the sample. For the depth calculation the density of 1.6 g/cm$^3$ was assumed.

![Figure 4.9 Depth profiles for deuterium as a function of depth for 5 five points at the F4/T19 tile.](image)

The deuterium content further into the sample was investigated by microbeam analyses of the cross section; see Figure 4.10. The plasma-facing surface was coated with a hard paint [77] to block the surface deuterium and prevent contamination. On the graph the position of the plasma-facing surface is not shown; it is located outside of the plot at 0 μm.
There is one characteristic feature seen in Figure 4.10 and it is also found on other cross-sections: deuterium is detected in bands approximately 100 μm wide located roughly parallel to the plasma-facing surface. The belts are found up to the depth of 2 mm. These “well structured” distributions seem to be independent on the tile location of the toroidal pump limiter and the systematic observations indicate a relation between the internal material structure and fuel storage in the bulk.

Figure 4.11 Scanning electron micrograph of the cross-section obtained by cleaving Tile F4/T19 from the region with thin deposits on the TPL.
Three-dimensional N11 CFC has very high porosity. Low magnification microscopy image as seen in Figure 4.11 proves the existence of gaps (Region A in central part of the image with fibres parallel to the image plane) separating the bundles of fibres (denoted as Regions B). The width of the gap and the bundles is similar, around 100 - 150 μm. It is difficult to say with a high degree of confidence whether the deuterium resides in the bundles (perpendicular to the image plane) or on side surfaces of the fibres which are parallel to the image plane, i.e. in Region A.

In Summary:
The surface deuterium concentration of the tiles of the Tore Supra TPL shows variations both in the lateral distribution as well as the depth profile.

In-depth fuel migration is detected. Deuterium is found in belts of deuterium roughly parallel to the original surface at depth up to 2 mm.

4.5 Deposited layers in JET

During operation thick deposited layers are formed in the JET divertor and after several years of operation the layers can be several hundreds of micrometers thick in some locations. Such layers are hard to analyse using Secondary Ion Mass Spectrometry (SIMS) or normal IBA methods, but by applying microbeam analysis on cross sections depth profiles can be produced.

Cross-sectional samples for microscopy and microbeam analysis were cut out from divertor tiles, placed into cold mounting epoxy and polished as described in [23]. Figure 4.12 shows the SEM images of the cross section
after cutting from the tile (a) as well as an optical microscope image (b) of the layer after polishing.

![Deuterium distribution on a cross section for a polished sample](image1.png)

**Figure 4.13** Deuterium distribution on a cross section for a polished sample

To test the effects of the polishing process a polished sample was first analysed and then part of the top layer was cut off and the sample was reanalysed.

![Cross section with deuterium distribution for the same area as in Figure 4.13 but the top surface cut away](image2.png)

**Figure 4.14** Cross section with deuterium distribution for the same area as in Figure 4.13 but the top surface cut away

The deuterium maps Figure 4.13 and Figure 4.14 are from the same area on the sample, the only difference being that in Figure 4.14 the originally
polished surface has been cut away and a small change in alignment. The layer structure, with high concentration of deuterium near the interface to the CFC and near the original surface of the layer, remains intact when the sample is polished and cut.

Figure 4.15 Beryllium distribution on a cross section for a polished sample. The epoxy cladding adds some background in the region above the deposited layer.

Similarly, Figure 4.15 and Figure 4.16 show the mappings of beryllium.

Figure 4.16 Beryllium distribution on a cross section for a polished sample after cutting away the top surface. The epoxy cladding adds some background in the region above the deposited layer.
The results show that the main features in the elemental concentration profiles are preserved through the polishing and cutting operations. However, there appears to be a systematic difference in the concentrations, and some beryllium and deuterium may be removed by polishing. It also maintains the layer structure very well for studies with optical microscopy [78]. If more accurate concentration values are required, it is preferable to make the analysis on a raw, unpolished cross section, or to cut off the polished surface before analysis.

In Figure 4.17 elemental maps for Be and D as well as depth profiles for a sample from tile 6 (exposed in 1998-2007), position 7 are shown and compared to a photograph.

Two areas can be distinguished in the photograph. The lower part, close to the original CFC surface, shows a clearly layered structure, both in the visual appearance and in the Be distribution, whereas the upper part also shows vertical variation. There is very little deuterium in the region close to the CFC. The different peaks in the Be depth profile are quite narrow, suggesting that they were formed in a short time span or due to isolated events.

In Figure 4.18 the depth profiles for position 6 on the sloping part of tile 6 shows a similar structure with 5 peaks as can be seen in Figure 4.17d for sample 7 of tile 6 that also is from the sloping part. Along with this plot there
are also plots for samples at this position but from tiles exposed at different periods.

Figure 4.18 (a) Deuterium and (b) Beryllium depth profiles for position 6 on the sloping part of tile 6 for three different time periods. Both 1998-2007 and 1998-2001 start at zero representing the interface. For the sample from 2001-2004 the profile starts at 195 µm (marked by the dashed line) the thickness of the layer from the sample exposed in 1998-2001 at this position.

Comparing Figure 4.17d and Figure 4.18a the sample is thicker in pos 7 compared to position 6 but there are still is 5 peaks in the beryllium concentration, although not as clearly visible as in Figure 4.18a. Overall the maximum concentrations are less in pos 6 compared to pos 7, in part this may be due to lower concentrations but another factor is that the resolution of the beam averages the beryllium concentration to a larger volume than the actual layers. For beryllium there is quite a good agreement for the different
periods whereas for deuterium no such agreement is observed. This indicates that beryllium is deposited and stays in place whereas deuterium is more mobile in the material which is to be expected but for a more conclusive answer samples collected during the rebuild of JET for the ITER like wall should help answer if and for which element an archaeological investigation is possible.

The layer cross sectional cuts and elemental mapping with microbeam provides reliable absolute composition of the deposited layers and allows for depth profiling even of the thickest layers, which are more than 500 μm thick. This makes the method a valuable complement to SIMS depth profiles and ion beam analysis from the surface.

Earlier investigations have shown comparatively low deposition in the outboard channel of the divertor (tile 6) with the exception of the sloping part, thick deposits in the inboard divertor channel (tile 4) and in particular high levels of deuterium retention in the thick deposits in shadowed areas of tile 4 [37; 79]. This corresponds well with what has been observed in these measurements.

The visual microscopic appearance of the layer structure is also qualitatively well reproduced in the elemental distribution, in the areas where there is a clear modulation of the layer composition, such as in Figure 4.17b and d. When comparing the layer thicknesses on samples exposed 1998-2001, 2002-2004 and the whole period 1998-2007 respectively the missing last period would have to show an increased net deposition from 4-7 nm per shot to about 15 nm for position 6 on tile 4. In order to find a closer correspondence it would be necessary to make more detailed statistics on types of pulses, in particular on pulses with different strike point positions [37; 79].

In Summary:

Depth profiles of deuterium and beryllium has been produced via scans of cross sections of deposited layers.

The effect of the polishing on the cross section was found to be limited and can be eliminated by cutting of the top surface.

The cross section reveals a layered structure but also lateral variations in deuterium and beryllium concentration.

For beryllium but not deuterium depth profiles can be constructed by adding samples from different periods.
5. Summary and conclusions

The work presented in the thesis has concentrated on studies of materials facing dense deuterium plasmas in controlled fusion devices such as JET, TEXTOR and Tore Supra tokamaks and a simulator of plasma-wall interactions (PWI). The main objective was to determine the content and distribution of deuterium in materials.

The study was carried out both for wall materials retrieved from fusion devices after long operation periods and, for probes exposed to plasma in dedicated series of experiments. Accelerator-based ion beam analysis (IBA) has been used as it offers a set of efficient tools for absolute quantitative determination of the elemental composition in the surface region. To ensure sensitive and selective tracing of light elements, especially hydrogen isotopes, nuclear reaction analysis (NRA) was chosen as a key method.

The nuclear microbeam system has been fully brought to operation for heavier ions by reconfiguration of the focusing magnet and adopted for NRA and elastic recoil detection analysis (ERDA). Lateral resolution of less than 10 μm has been achieved. All those upgrades allows for comprehensive studies of the deuterium with standard and micro-NRA.

The major contribution of this work to fuel retention studies is the detection of significant inhomogeneity on a micro-scale of deuterium distribution both in the surface layers and in the bulk plasma-facing components for the improved understanding of fuel inventory and erosion and deposition processes. Microbeam has for the first time been used for studies bulk concentration on cross-sections obtained by cleaving and applied for broad studies of plasma-facing materials from many different devices, thus allowing for comparisons of fuel retention in various substrates, especially in carbon fibre composites.

Limiter components from Tore Supra were examined. This resulted in: (a) the identification of micro-regions rich in deuterium on surfaces from the erosion zone; (b) mapping of bulk distribution of fuel in narrow bands (100-150 μm) located parallel to the tile surface. The accumulation in bands is most probably attributed to the porous internal structure of the N11 composite and the bulk concentration is rather low thus not being decisive for the overall fuel inventory.

Fuel retention in the NB41 carbon composite (ITER reference material) was determined after exposures at TEXTOR and PISCES. At micro-scale the deuterium retentions were strongly related to structure of fibres: the region
with ex-pitch fibres has more deuterium at both the surface and at deeper depths compared to ex-PAN.

Unique measurements were performed on cross-sections of thick co-deposits from the JET divertor to determine distribution of deuterium, beryllium and carbon. The distribution is dominantly laminar but also shows other structures. This type of information improves understanding of the history of plasma-wall interaction in JET. Special attention has been paid to the effect of the polishing by comparing several different surface treatments and polishing appear to have some effect on the surface but this can be reduced by cutting of part of the sample surface if needed. For Beryllium some part of the deposition history can be seen by comparing samples from similar positions but different time periods, but for Deuterium the different time periods does not add up.

The scanning nuclear microbeam has been demonstrated as a versatile instrument in studying 3D deuterium concentration profiles in carbon materials at a μm scale.
Fusion (sammanslagning av lätta atomkärnor) förser solen med dess energi och är en mycket intressant framtida energikälla. På jorden försöker man åstadkomma fusion genom att hetta upp två olika isotoper av väte, deuterium (D) och tritium (T), så mycket att de blir ett plasma, d.v.s. samma tillstånd som materian har i solen. I plasmat kan D och T slås ihop och bilda neutroner (n), helium ($^4$He) och energi enligt formeln:

$$D + T \rightarrow n + ^4He + 17.59\text{MeV}$$

För att hålla plasmat på plats så kan vanliga material inte användas eftersom plasmat skulle förstöra dem. Istället så hålls plasmat på plats i reaktorn av starka magneter. Med hjälp av denna magnetisolering så kan man ha ett plasma som är många miljoner grader varmt på ett avstånd mindre än en meter från material vid rumstemperatur.


Även om väldigt stark magnetisk inneslutning används så läcker en del av plasmat ut och reagerar med de olika väggmaterialen. Om material från väggarna kommer in i plasmat kan de kyla plasmat och därmed försvåra fusionsreaktionen, i synnerhet om det är tunga element inblandade. Detta gör att väggmaterialen har väldigt tuffa krav på sig och för tillfället används olika former av kol i de flesta experiment, framförallt grafit och kolfiber. Dessutom så används beryllium och volfram i en del experiment och är främst tänkta att användas i ITER. Beryllium är relativt värmetåligt, dock inte lika bra som kol och volfram, som också är tåligt för plasmaerosion. Kol är bra eftersom det inte smälter och om det kommer in i plasmat så ger det bara mindre problem. Det stora problemet med kol är att det lätt eroderas av plasmat och återdeponeras tillsammans med joner från plasmat. När tritium
används i reaktorn så blir dessa avlagringar radioaktiva och kontaminerar väggarna samt minskar den mängd bränsle som är tillgänglig för fusion.

I den här avhandlingen är syftet att etablera en bättre rumslig upplösning och känslighet för jonstråleanalys av material som utsätts för typiska plasmaförhållanden. För att undersöka hur de olika materialen beter sig kan man antingen mäta på delar av väggarna som man plockar ut efter flera års exponering och får på så sätt en inblick i påverkan under denna tid, eller så kan man föra in en sond i reaktorn för att få en ögonblicksbild.

![Diagram of three different types of ion beam methods]

**Figure 6.1 De tre olika typer av jonstrålemetoder som har använts**

Tre typer av jonstråleanalys har använts, tillbakaspridning (Rutherford Back Scattering, RBS), elastisk rekylanalys (Elastic Recoil Detection Analysis, ERDA) och kärnreaktionsanalys (Nuclear Reaction Analysis, NRA). I RBS så studsar en lätt jon mot en atom i provet och utifrån hur mycket energi jonen har efter att ha lämnat provet kan massan och kollisionsdjupet beräknas. RBS passar bäst för tunga element i en lättare matris, t.ex. volfram i kol.

Med ERDA använder man istället en tyngre jon och kan på så sätt slå ut atomer ur provet. Med hjälp av ett detektorsystem, t.ex. löptidsdetektor och energidetektor, så kan man få fram både djup och atomslag av atomerna i provet. ERDA lämpar sig bäst för lätt element i en medeltung matris t.ex. väte i kol. NRA utnyttjar specifika kärnreaktioner t.ex. D(3He,p)4He där protonen får en hög energi och ger en signal som är fri från störningar.

Med hjälp av dessa metoder kan man få mycket bra information från en eller några mikrometers djup. Detta är tillräckligt för korta exponeringstider och tunna avlagringar på väggar, men eftersom avlagringarna kan bli flera 100 µm tjocka, efter år av avlagringar, är det inte alltid tillräckligt. Då kan man istället dela provet och analysera tvärsnittet med en fokuserad jonstråle och detta har genomförts med proverna från JET, och ett exempel kan ses i
Figure 6.2 med både deuterium och beryllium fördelningen som jämförs med ett mikroskop bild.

Figure 6.2 Deuterium (a) och beryllium (b) fördelningen från den plasma exponerade väggen i JET som var exponerad 1998-2007. I (c) visas en mikroskop bild samma prov som visas i (a) och (b). I (d) så visas koncentrationsprofilerna från (a) och (b) som en funktion av avståndet till det ursprungliga substratet.


Mätningarna och metoderna som presenteras i denna avhandling syftar till att användas vid konstruktionen av nästa generations fusions rektorer framför allt gällande vägg materialens livstid och upptag av bränsle.
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