Plasma-Facing Components in Tokamaks: Studies of Wall Conditioning Processes and Plasma Impact on Diagnostic Mirrors

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Abstract

Understanding of material migration and its impact on the formation of co-deposited mixed material layers on plasma-facing components is essential for the development of fusion reactors. This thesis focuses on this topic. It is based on experiments performed at JET and TEXTOR tokamaks. The major objectives were to determine: (i) fuel and impurity removal from plasma-facing components by ICWC in different gas mixtures, (ii) fuel and impurity transport connected to ICWC operation, (iii) plasma impact on diagnostic mirrors. All these issues are in line with the ITER needs: mitigation of co-deposition and fuel inventory, and the performance of first mirrors in long-term operation. The novelty in research is demonstrated by several elements. In wall conditioning studies, tracer techniques based on injection of rare isotopes ($^{15}$N, $^{18}$O) were used to determine conclusively the impact of respective gases. Also, a new approach to ICWC was developed by combining global gas balance studies based on mass spectrometry and the use of multiple surface probes exposed to discharges and then studied ex-situ with accelerator-based techniques. Impact of plasma on diagnostic mirrors was determined after exposure to the entire first experimental campaign in JET-ILW.
List of Papers

This thesis is based on the work presented in the following papers:

I *Impact of ion cyclotron wall conditioning on plasma-facing components at TEXTOR*
   A.G. Carrasco, S. Möller, P. Petersson, D. Ivanova, A. Kreter, M. Rubel, T. Wauters

II *Self-consistent application of ion cyclotron wall conditioning for co-deposited layer removal and recovery of tokamak operation on TEXTOR*

III *Nitrogen removal from plasma-facing components by ion cyclotron wall conditioning in TEXTOR*

IV *An overview of the comprehensive First Mirror Test in JET with ITER-Like Wall*
My contribution to publications

**Paper I:** I was involved in the planning of the experiment and I actively participated in the entire experiment. I was involved in the measurement of deuterium and oxygen-18 from TEXTOR tiles. I performed the analysis of data. I wrote the paper and presented the results at the 14th International Conference on Plasma-Facing Materials and Components for Fusion Applications, Jülich, Germany, May 2013 (Poster).

**Paper II:** I was involved in the planning of the experiment and I actively participated in the entire experiment. I was involved in the measurements of deuterium and oxygen-18 from laboratory-prepared samples.

**Paper III:** I was involved in the planning and performing of the experiment. I was involved in the measurements of co-deposited impurities. I performed the analysis of data. I wrote the paper and presented the results of the 21st International Conference on Plasma Surface Interactions, Kanazawa, Japan, May 2014 (Poster).

**Paper IV:** I was involved in the reflectivity and surface analysis measurements and analysis of the data.
Acknowledgements

I would like to express my gratitude to my supervisor Marek Rubel for his continuous support and encouragement. I would like to thank my co-supervisor Per Petersson for all his help and guidance. I really enjoyed working together.

It has been a great pleasure to work with Tom Wauters and Sören Möller during the ICWC experiments at TEXTOR. I would like to thank them for their advice and patience. In England, Anna Widdowson and Charlie Ayres were very helpful with all organizational issues and little problems that come up when you work in another laboratory. I would like to thank Göran Possnert and Jonas Åström for their help in the Tandem Accelerator Laboratory in Uppsala. I would like to thank Håkan Ferm for his help in the design and construction of holders for our experiments.

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Finalmente, quiero agradecer a mis amigos, a Leska y especialmente a mi familia por todo su apoyo y cariño.
Acronyms

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Definition</th>
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<tbody>
<tr>
<td>ALT II</td>
<td>Advanced Limiter Test II (TEXTOR)</td>
</tr>
<tr>
<td>AUG</td>
<td>ASDEX Upgrade</td>
</tr>
<tr>
<td>CFC</td>
<td>Carbon Fibre Composite</td>
</tr>
<tr>
<td>DED</td>
<td>Dynamic Ergodic Divertor (TEXTOR)</td>
</tr>
<tr>
<td>DEMO</td>
<td>Demonstration reactor of a power plant</td>
</tr>
<tr>
<td>ELM</td>
<td>Edge Localised Mode</td>
</tr>
<tr>
<td>EPS</td>
<td>Enhanced Proton Scattering</td>
</tr>
<tr>
<td>ERDA</td>
<td>Elastic Recoil Detection Analysis</td>
</tr>
<tr>
<td>FMT</td>
<td>First Mirror Test</td>
</tr>
<tr>
<td>GDC</td>
<td>Glow-Discharge Cleaning</td>
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<tr>
<td>IBA</td>
<td>Ion Beam Analysis</td>
</tr>
<tr>
<td>ICF</td>
<td>Inertial Confinement Fusion</td>
</tr>
<tr>
<td>ICR</td>
<td>Ion Cyclotron Resonance</td>
</tr>
<tr>
<td>ICWC</td>
<td>Ion Cyclotron Wall Conditioning</td>
</tr>
<tr>
<td>ILW</td>
<td>ITER-Like Wall (JET)</td>
</tr>
<tr>
<td>ITER</td>
<td>International Thermonuclear Experimental Reactor</td>
</tr>
<tr>
<td>JET</td>
<td>Joint European Torus</td>
</tr>
<tr>
<td>LHD</td>
<td>Large Helical Device</td>
</tr>
<tr>
<td>LCFS</td>
<td>Last-Closed Flux Surface</td>
</tr>
<tr>
<td>MCF</td>
<td>Magnetic Confinement Fusion</td>
</tr>
<tr>
<td>NRA</td>
<td>Nuclear Reaction Analysis</td>
</tr>
<tr>
<td>PFC</td>
<td>Plasma-Facing Component(s)</td>
</tr>
<tr>
<td>PFM</td>
<td>Plasma-Facing Material(s)</td>
</tr>
<tr>
<td>PWI</td>
<td>Plasma-Wall Interactions</td>
</tr>
<tr>
<td>LOS</td>
<td>Line-of-Sight</td>
</tr>
<tr>
<td>RBS</td>
<td>Rutherford Backscattering Spectroscopy</td>
</tr>
<tr>
<td>RGA</td>
<td>Residual Gas Analysis</td>
</tr>
<tr>
<td>RF</td>
<td>Radio Frequency</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscopy</td>
</tr>
<tr>
<td>SIMS</td>
<td>Secondary Ion Mass Spectroscopy</td>
</tr>
<tr>
<td>SOL</td>
<td>Scrape-off Layer</td>
</tr>
<tr>
<td>TDS</td>
<td>Thermal Desorption Spectrometry</td>
</tr>
<tr>
<td>TEXTOR</td>
<td>Toroidal Experiment for Technology Oriented Research</td>
</tr>
<tr>
<td>ToF</td>
<td>Time-of-Flight</td>
</tr>
<tr>
<td>XPS</td>
<td>X-ray Photoelectron Spectroscopy</td>
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Chapter 1

Introduction

Energy is essential in our lives. It is used to heat and light houses, to transport goods and people, to power industry and household appliances. More than 80 % of the world energy supply comes from burning fossil fuels [1]. Fossil fuels are non-renewable and affect negatively the environment by releasing green-house gases and other air pollutants such as sulphur oxides [2]. We need to reduce their use to meet a growing energy demand with alternative and more sustainable options. Nuclear fusion stands out as a promising candidate for future energy generation. It offers many advantages: non-emission of green-house gases, power production independent of weather conditions, reduced land-use and virtually unlimited fuel supply [3].

Nuclear fusion is a reaction where two lighter atoms combine to form a heavier one. This process is accompanied by a release of energy when the reacting atoms have lower masses than iron. A fusion reaction considered for energy production is that between deuterium (D) and tritium (T) to produce an alpha particle ($^4$He) and a neutron (n):

$$ D + T \rightarrow ^4He + n + 17.6 \text{ MeV} $$

This reaction only takes place in plasmas with temperatures over a few million degrees. The 17.6 MeV of energy released are distributed between the alpha particle and the neutron according to their mass ratio. Under magnetic fields, the alpha particle is confined and it transfers its energy to the background plasma through Coulomb collisions. This is necessary to keep the plasma hot and to compensate for energy losses. The fast neutron escapes the plasma volume and it is slowed down in the blanket, where its kinetic energy is turned into heat and then into electricity by conventional technologies.

The most critical issue for a fusion power plant is to prove fuel self-sufficiency [4]. As pointed out above, the two fuel species are deuterium and tritium. Deuterium is the stable isotope of hydrogen and it is obtained from regular water [5]. It has two nucleons and an isotopic abundance of 0.015 %. Tritium is the radioactive isotope
of hydrogen. It has three nucleons and a half-life time of about 12 years. There is
not enough tritium available in nature for technical applications. At the moment
it is produced in heavy-water fission reactors [6]. In future power-plants, neutrons
from fusion reactions will be used to breed it in a lithium-based blanket according
to:

\[ ^6\text{Li} + n \rightarrow ^4\text{He} + T + 4.8 \text{ MeV} \]
\[ ^7\text{Li} + n \rightarrow ^4\text{He} + T + n - 2.8 \text{ MeV} \]

\(^6\text{Li}\) is the most advantageous isotope because unlike \(^7\text{Li}\), it reacts with neutrons
at low energies below 1 MeV. To ensure enough tritium production a neutron mul-
tiplier is necessary to compensate neutron losses to unsuccessful breeding reactions
or to uncovered parts of the blanket (e.g. diagnostic ports). Possible candidates
are beryllium or lead.

1.1 Confinement concepts

To produce efficient fusion-power there are three main parameters to consider:
temperature, density and energy confinement time. The temperature must be high
enough for ions to overcome their electrostatic repulsion and fuse. The density
is connected to collision frequency of fuel species. The energy confinement time
is a measure of how long a plasma takes to dissipate its energy when all heating
sources are switched-off. To achieve ignition (i.e. fusion power output with no
external heating) the product of these three parameters has to be greater than
3 \times 10^{21} \text{ keV s m}^{-3}. This condition is known as the Lawson criterion and it is
derived from a power balance argument [7]. There are two approaches to satisfy
the Lawson criterion: inertial confinement fusion (ICF) and magnetic confinement
fusion (MCF).

In ICF powerful lasers or particle beams heat a small fuel pellet by delivering
megajoules of energy in a few nanoseconds. This produces a micro-implosion that
compresses the center of the pellet to high densities and temperatures, initiating
fusion. Confinement is provided by the own fuel inertia that resists expansion [8].
The largest ICF machine is the National Ignition Facility [9]. This experiment
was designed to achieve ignition and it has recently obtained an energy gain fac-
tor greater than one [10]. Key challenges for ICF reactors are technology-related.
These are target manufacturing cost, repetitive target ignition and construction of
a reaction chamber to extract fusion power and to breed tritium [11].

In MCF charged particles are confined in a magnetic field. This restricts their
motion across field lines but not along them. To cope with end losses and desta-
balizing drifts, the field lines are closed on themselves forming a torus and twisted
around the toroidal axis. There are two main designs to provide such magnetic
configuration: the stellerator and the tokamak.

The stellerator uses external currents to shape the magnetic topology of plasma.
It does not need a toroidal current and, therefore, it may operate in steady-state. As
Table 1.1: Comparison of characteristic parameters of several tokamaks. The minor radius is an average between the horizontal and vertical radii.

<table>
<thead>
<tr>
<th></th>
<th>TEXTOR</th>
<th>AUG</th>
<th>JET</th>
<th>ITER</th>
</tr>
</thead>
<tbody>
<tr>
<td>Major radius [m]</td>
<td>1.8</td>
<td>1.7</td>
<td>3.0</td>
<td>6.2</td>
</tr>
<tr>
<td>Minor radius [m]</td>
<td>0.5</td>
<td>0.7</td>
<td>1.7</td>
<td>2.0</td>
</tr>
<tr>
<td>Magnetic field [T]</td>
<td>3.0</td>
<td>3.1</td>
<td>3.5</td>
<td>5.3</td>
</tr>
<tr>
<td>Plasma current [MA]</td>
<td>0.8</td>
<td>1.6</td>
<td>4.8</td>
<td>15</td>
</tr>
<tr>
<td>Auxiliar heating [MW]</td>
<td>9</td>
<td>27</td>
<td>38</td>
<td>40</td>
</tr>
<tr>
<td>Plasma lifetime [s]</td>
<td>10</td>
<td>10</td>
<td>20</td>
<td>400</td>
</tr>
<tr>
<td>Wall materials</td>
<td>C</td>
<td>W</td>
<td>W &amp; Be</td>
<td>W &amp; Be</td>
</tr>
</tbody>
</table>

disadvantages, stellators have complex three-dimensional structures that complicate understanding of plasma physics, diagnostic location and device manufacture. The largest stellators in the world are Large Helical Device in Japan [12] and Wendelstein 7-X, currently under construction in Greifswald, Germany [13].

The tokamak is the most advanced design for a future fusion reactor. Its name is a Russian acronym that comes from “toroidal chamber with magnetic coils”. The main feature of tokamaks is the induction of a plasma current to produce a poloidal magnetic field. Plasma itself acts as the secondary winding of a transformer. This means that the reactor cannot work in steady-state unless non-inductive techniques are used to drive current [14]. Table 1.1 presents a summary of basic characteristics of several tokamaks.

1.2 Towards fusion electricity

It is complex to answer how far we are from producing profitable fusion energy. After more than fifty years of research, important advances have been achieved [15]. The triple product, which determines fusion plasmas performance, has been increased by more than six orders of magnitude. A remarkable success were the D-T campaign at TFTR in 1994 [16] and at JET in 1997 [17], where up to 16 MW of fusion power were released in a controlled way. However, there is a long way to the construction of DEMO, the demonstration of a commercial power plant based on fusion. The intermediate step is the International Thermonuclear Experimental Reactor (ITER), under construction in Cadarache, France. ITER will be the largest tokamak in the world. It is expected to confirm viability of fusion and to test technologies and materials. Most of research effort in fusion is focused in this project with several tokamaks around the world working on ITER-related issues: JET [18], DIII-D [19], ASDEX Upgrade (AUG) [20], Tore Supra [21], TEXTOR [22], JT-60U [23] or NSTX [24] are some examples.
CHAPTER 1. INTRODUCTION

This thesis focuses on material migration and its impact on the formation of co-deposited mixed material layers on plasma-facing components. This work is based on studies of wall conditioning processes and plasma impact on diagnostic mirrors. The experiments were performed in tokamaks. They were mainly based on the exposure of pre-characterized surface probes and their subsequent ex-situ analysis by accelerator-based techniques.

1.3 Fusion devices

Experiments presented in this thesis were performed at two different tokamaks. Ion cyclotron wall conditioning studies on fuel removal and impurity transport were performed at TEXTOR, whereas the First Mirror Test project was carried out at JET. They are briefly introduced below.

**TEXTOR** (Toroidal Experiment for Technology Oriented Research) was a limiter tokamak operated for 30 years until December 2013 at the Institute of Energy and Climate Research of Forschungszentrum Jülich, Germany [25]. Probe transfer systems allowed exposure of large-size samples at different radial positions. This made TEXTOR specially suited for plasma-wall interaction studies [26]. A toroidal view into the vessel is shown in Figure 1.1. The liner was made of a Ni-based alloy and the limiters were made of graphite. The toroidal bumper limiter was on the high field side. It protected the Dynamic Ergodic Divertor (DED), which was a system to improve control over plasma edge [27]. The Advance Limiter Test II (ALT-II) was a toroidal belt pump limiter for investigation of particle confinement and recycling. It was located on the low field side. The main poloidal limiter was on top and bottom of the vessel.

**JET** (Joint European Torus) is currently the world’s largest fusion facility. It is operated at the Culham Center for Fusion Energy, England. JET has a divertor configuration which allows to experiment with advanced operational scenarios. It has recently changed its plasma-facing components (PFC) to operate with same materials as planned for ITER: beryllium and tungsten [28]. The ITER-like wall (ILW) project is a test to demonstrate acceptable tritium retention levels and ability to operate with high power with that mix of materials [29]. PFC are shown in Figure 1.2. The main chamber is made of bulk beryllium tiles for the limiters and Be-coated inconel elsewhere. The neutral beam shine-through area is covered with W-coated carbon fibre composites (CFC) to increase power handling capability. In the divertor, tiles are made of W-coated CFC with exception the load bearing tile that is made of bulk tungsten.
1.3. FUSION DEVICES

Figure 1.1: Toroidal view inside TEXTOR: 1) toroidal bumper limiter, 2) main poloidal limiter, 3) ALT-II, 4) niquel-based liner.

Figure 1.2: View from inside the JET vacuum vessel after ILW installation works: 1) inner wall guard limiter, 2) remote handling robot, 3) outer wall guard limiter, 4) divertor, 5) ion cyclotron resonance heating antenna.
Chapter 2

Plasma-wall interactions

The study of plasma-wall interactions (PWI) comprises all processes involved in the exchange of mass and energy between the plasma and the surrounding wall. The driving force is the assessment of material migration, tritium inventory and dust formation. These issues have a large impact on the economy and safety of the machine and their study is on the priority list in fusion research [30, 31]. Understanding of PWI is complex and it requires an interdisciplinary approach based on experimental evidence, theoretical knowledge and computational models. Experiments are performed in fusion devices, for example JET [32], TEXTOR [33], Tore Supra [34], AUG [35], LHD [36], and in linear simulators of PWI such as PISCES [37], MAGNUM-PSI [38] and JUDITH [39].

PWI research also includes investigation and development of first-wall materials. Selecting a candidate is not an easy task considering the demanding requirements:

(i) compatibility with high-vacuum,
(ii) good thermal conductivity to evacuate heat loads,
(iii) high thermal resilience to withstand transient events (e.g. disruptions),
(iv) resistance to erosion to extend life-time of components,
(v) low fuel inventory and neutron activation to minimize safety risks.

Compatibility among all in-vessel components is also essential. At the moment, there is no material or compound fulfilling all demanded specifications. For the last decades, research has been focused on carbon with many carbon-wall experiments. However, this material shows erosion rates and resulting fuel inventory levels which are not acceptable for a fusion reactor. As a result of a detailed assessment, ITER will use a combination of beryllium and tungsten [40].

It is desirable to localize PWI in a specific region of the wall to minimize possible damage to other parts of the vessel that are more vulnerable. One option is to use a material structure protruding from the wall to intercept particles at the plasma edge. This material structure is known as limiter. The innermost part of the limiter defines the last-closed flux surface (LCFS). The plasma region between the LCFS and the wall is called scrape-off layer (SOL). The other option is to exhaust
escaping ions to a remote region called divertor by means of auxiliary magnetic coils. This alternative is more advanced because the back stream of impurities to the plasma core is reduced and it is possible to pump them out. Figure 2.1 presents the concepts of a limiter and a divertor.

2.1 Plasma-facing materials for ITER

The main objective of ITER is to deliver ten times more power than it would consume: 500 MW of output power using 50 MW of auxiliary heating. From a technological perspective, it will test integrated operation of fusion reactor components [41]. There are essential differences between present-day devices and ITER. Pulse length (400 s) will bring the problem of material erosion and fuel inventory to a new level, and high-energy content of the plasma (350 MJ) might lead to melting and damage of the wall under disruption events or edge localized modes (ELMs), thus affecting material life-time. There is no experience in such conditions so all designed components have to be tested [42].

Figure 2.2 shows distribution of plasma-facing materials in ITER: beryllium for the blanket and tungsten for the divertor [43]. Also, different designs of tritium-breeding blanket modules will be placed in three equatorial ports to test their breeding efficacy [44]. All wall components will be actively water cooled using a copper-based heat sink. Table 2.1 summarizes thermo-physical properties of beryllium, tungsten and carbon fibre composites (CFC).

The blanket has two purposes: (i) provide a boundary for the plasma during transient events, and (ii) isolate the vacuum vessel and other external systems from heat loads and neutron fluxes. The area of the blanket is 700 m$^2$ and it is divided into 440 modules. Each module consists of a shielding block and a first-wall panel. Shielding blocks are made of stainless steel and they provide nuclear protection.
Table 2.1: Comparison of properties of beryllium, tungsten and carbon [42].

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<thead>
<tr>
<th></th>
<th>Be</th>
<th>CFC</th>
<th>W</th>
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<tbody>
<tr>
<td><strong>Atomic mass [amu]</strong></td>
<td>9</td>
<td>12</td>
<td>184</td>
</tr>
<tr>
<td><strong>Thermal conductivity (\lambda) [(\text{W m}^{-1} \text{K}^{-1})]</strong></td>
<td>190</td>
<td>200–500</td>
<td>150</td>
</tr>
<tr>
<td><strong>Melting point [K]</strong></td>
<td>1560</td>
<td>2500 (subl.)</td>
<td>3695</td>
</tr>
<tr>
<td><strong>Thermal expansion ([10^{-6} \text{K}^{-1}])</strong></td>
<td>11.5</td>
<td>0–4</td>
<td>4.5</td>
</tr>
<tr>
<td><strong>Thermal capacity ([\text{J kg}^{-1} \text{K}^{-1}])</strong></td>
<td>1825</td>
<td>709</td>
<td>134</td>
</tr>
<tr>
<td><strong>Major advantage</strong></td>
<td>low Z</td>
<td>shock resistance</td>
<td>low erosion</td>
</tr>
<tr>
<td><strong>Major disadvantage</strong></td>
<td>melting</td>
<td>chemical erosion</td>
<td>high Z</td>
</tr>
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</table>

First-wall panels face the plasma. They are made of beryllium and individually shaped to minimize heat loads [45], which are expected to go up to 5 MW/m² during start-up and ramp-down.

Beryllium was selected because it has low atomic number, which leads to low radiation losses in the core plasma, and it has good gettering ability for oxygen impurities, thus improving the quality of vacuum. Another important point is the operational experience with this material, especially in JET [46, 47]. The main disadvantage of beryllium is its low melting temperature of 1550 K. This might be a problem during transient events such as ELMs or disruptions. Other shortcomings are toxicity of beryllium dust and neutron-induced brittleness.

The divertor is responsible for exhausting most of the heating power and the removal of helium ash. The area of the divertor is 150 m² and it is divided into 54 cassettes fully removable by remote handling. The initial plan was to start with a tungsten and carbon divertor during non-active operation and then change to a full-tungsten divertor during active operation. The purpose was to optimize flexibility in the start-up phase and reduce fuel retention in the D-T phase. However, it has been decided to start directly with a full-tungsten divertor to reduce costs. The power handling demand for the strike points is 10 MW/m² in steady-state conditions.

The selection of tungsten is motivated by a high melting temperature of 3695 K and a high threshold energy for sputtering by deuterium (200 eV). The weaknesses of tungsten are its brittleness below the ductile-to-brittle transition temperature (420 - 670 K) and the reduction of the strength and hardness above the recrystallization temperature (1300 - 1600 K) [48]. Additionally, activation by neutron irradiation leads to transmutation and helium production, modifying tungsten properties [49]. Carbon will not be used in ITER but it is a relevant first-wall material. Carbon advantages are a good thermal conductivity and high thermal shock re-
2.2 Material migration

PWI results in erosion of plasma-facing components. Erosion is caused by either excessive heat loads or flux of particles to the wall. Large heat loads might melt or sublimate the material whereas impinging particles cause physical sputtering and chemical erosion. Eroded species are either pumped out or ionized in the plasma. Ionized impurities travel in the plasma until they are eventually re-deposited, or co-deposited with other species somewhere in the wall. This process is called migration. It is important because it modifies morphology and properties of PFC [50–52].

Migration patterns are complex and difficult to predict. They depend on the operation scenario and differ from one machine to another. However, global migration studies show general trends. The main chamber of the tokamak is an erosion zone. The divertor presents an asymmetric pattern with net deposition in the inner divertor and net erosion or neutrality in the outer divertor. This picture is based on post-mortem tile analysis, tracer-injection experiments and probe measurements among others [53–55].

Deposition or implantation of impurities away from their original location may lead to the formation of mixed-material layers. Modified surfaces present different properties with respect the original plasma-facing material (PFM). More specifi-
cally, physical and chemical erosion, thermal conductivity and fuel retention properties may be affected. For this reason, potential mixing of materials in ITER are studied [56–58]. The most important erosion processes are described below.

**Physical sputtering:** It is a process where atoms from the outermost layers of a solid are ejected from the material as a result of momentum transfer from incoming particles. The sputtering yield is the average number of knocked-out atoms per projectile. It depends on: projectile energy, incidence angle, mass ratio projectile-target, and surface-binding energy. The energy of the incident particle is given by the plasma edge temperature and the sheath potential. For any given combination of projectile-target the sputtering yield falls to zero below certain energy, known as threshold energy [59, 60]. Figure 2.3 shows the sputtering yield by deuterium on different plasma-facing materials.

**Chemical erosion:** It takes place when a projectile and a target atom react chemically forming a volatile compound and leaving the solid. In contrast to physical sputtering, chemical erosion occurs only for certain projectile-target combinations. It depends on: surface temperature, energy and flux of the projectile, and material condition. Chemical erosion is the dominant erosion process in carbon-wall machines. Carbon reacts with hydrogen isotopes producing hydrocarbons. They leave the wall and they are co-deposited forming carbonaceous layers. Carbon also reacts with oxygen impurities forming oxides [62].

![Graph showing sputtering yield by deuterium on different materials](image)

Figure 2.3: Sputtering yield by deuterium under normal incidence on different fusion-relevant materials. Carbon is chemically eroded at low energies [61].
Melting, evaporation and brittle destruction: During transient events, PFC will receive extreme power loads in very short time scales. For example ITER expects disruptive power loads in the order of GW/m$^2$. These events have different impact on materials. Metals might melt. The molten layer will then be subject to gravitational and electromagnetic forces. It will either be ejected into the plasma, or drift and recrystallize changing its previous characteristics. The behaviour of carbon is different because it sublimates. This is beneficial because topology remains unchanged. The disadvantage of carbon is the ejection of debris produced by brittle destruction when power-loads exceed values around 1 GW/m$^2$ [63].

2.3 Tracers

The term “tracer” denotes an agent introduced in minute amounts into a system so that its subsequent distribution can be followed from some distinct property. In studies of material migration exotic gases are injected from a well-defined source to study their transport and retention on plasma-facing components, for instance WF$_6$ [64] or rare isotopes: $^{13}$C [65], $^{15}$N [66], $^{18}$O [Papers I, II], natural abundances 1.12 %, 0.21 % and 0.36 % respectively. Another approach is the use of marker tiles. These may either contain traces of a rare isotope (e.g. $^{10}$Be [67], natural abundance below 0.01 %) or have a multilayer structure combining elements with high and low atomic number [68].

It is advantageous or even necessary to use tracers in order to distinguish different effects. For instance, it is not possible to discriminate between species retained during plasma exposure from those adsorbed on the surface under contact with air during the probe transfer to a surface analysis station. In Papers I, II and III tracers $^{18}$O and $^{15}$N were used to tackle this issue. Previously, methods for $^{15}$N analysis were developed and applied in TEXTOR [69] and AUG [70].

2.4 Tritium inventory

In-vessel tritium retention is limited in ITER to 700 g for safety reasons. When this limit is reached, operation must be stopped and conditioning tasks are performed to remove trapped fuel. Therefore, control and monitoring of tritium inventory is important to optimize the machine operation time [71–73]. Research intensity on fuel retention was boosted after D-T experiments at JET and TFTR showed that tritium accumulation was a serious concern [74,75]. Since then, several studies have been performed to understand the underlying mechanisms of fuel retention [76,77] and to assess their impact on machines with carbon [78–80], tungsten [81] and ITER-like walls [82–84]. Fuel retention is usually studied by gas balance measurements and ex-situ analysis of PFC [85].

When extrapolating fuel retention levels in current carbon machines to ITER, the limit is reached in less than few hundred discharges. Predictions for carbon and other wall materials are shown in Figure 2.4. For ITER materials (Be and W)
tritium retention level is almost two orders of magnitude lower than for a carbon wall. The most relevant fuel retention mechanisms are co-deposition, implantation, and trapping in neutron-induced defects.

**Co-deposition:** impurities eroded from the wall are co-deposited with hydrogen isotopes forming fuel-rich layers on PFC and shadowed areas. This is the main mechanism responsible for fuel retention in carbon-wall machines. In TEXTOR, typical film-growth rates were between 1 and 3 nm s\(^{-1}\) [86]. Co-deposits build up in shadowed areas, where re-erosion and transport by plasma is no longer possible.

**Implantation:** particles bombarding the wall penetrate the material, where their energy is reduced to thermal levels by means of elastic and inelastic collisions. This process is followed by diffusion and trapping. Typical energies of fuel particles reaching the wall are between tens to hundreds eV and they can go up to the keV range for charge-exchange neutrals. For these energies the implantation range in beryllium or carbon is in the order of tens of nm. Implantation depends on diffusion and recombination coefficients, as well as in the concentration of trapping sites. It is the dominant fuel retention mechanism in tungsten [87,88].

![image](image_url)

Figure 2.4: Prediction of tritium retention in ITER for different material combinations [73].
Neutron-induced defects: Fuel retention is enhanced under neutron irradiation because it damages the material increasing trapping sites for hydrogenic species. Additionally, beryllium may also transmute into tritium or helium. Tritium produced in the bulk material can then be trapped in helium bubbles or microscopscopic defects of the material [89].

2.5 Dust

The term “dust” refers to loose particle-aglomerates with sizes ranging from tens of nanometres to milimitres. Dust and small debris may originate from PWI processes, but also from installation works during shutdowns. Common production mechanisms are flaking of re-deposited layers, melting of material followed by droplet splashing, arcing or fine debris produced by brittle destruction of carbon [90,91].

Dust production is and important safety issue for ITER [92,93]. If there was an accidental release of dust from the machine, particles of tungsten and carbon co-deposits would be a source of radioactivity, and beryllium a source of toxicity. In case of water or air leak, hydrogen production and related pressure build-up could lead to an explosion in reaction with dust. From an operational point of view, dust can also degrade diagnostics and pumping systems.
Chapter 3

Wall conditioning

In fusion science and technology, the term “wall conditioning” refers to the modification of the wall-surface state. Wall conditioning provides effective means to reduce plasma impurities and control over fuel recycling. In present-day machines, it is used to recover from torus vents and to assist plasma start-up, especially after leaks or disruptions [94]. In ITER, wall conditioning will also be used in the mitigation of tritium retention. A wide variety of techniques have been developed and they are briefly presented.

3.1 Baking

Baking is based on heating of the wall surfaces to induce thermal desorption of water and other adsorbed species. The length of the process may be in the order of days and it leads to better vacuum conditions and less plasma impurities. Baking is usually performed after a period in which air was in contact with the wall, for instance in-vessel interventions. In ITER it will have the additional purpose of releasing tritium trapped in beryllium and tungsten co-deposits. Planned baking temperatures are 240 °C for the blanket and 350 °C for the divertor [95].

3.2 Thin film deposition

Coating of the wall with low-Z elements results in improved plasma performance due to reduction of plasma impurities. Protective layers are produced by glow-discharge in a through-flow of a reactive gas. Typical film thickness is between 50 and 100 nm. Duration of the conditioning effect depends on the operation scenario and properties of the coating, but it is usually within few hundred seconds of plasma exposure.

First conditioning coatings were performed in TEXTOR using methane (CH$_4$) as reactive gas in a process called carbonization [96]. In later experiments, methane and diborane (B$_2$H$_6$) were mixed to reduce chemical erosion of carbon layers and to
improve oxygen gettering [97]. Boronization was used in other tokamaks with the same positive results [98]. Diborane is toxic and explosive so other less hazardous compounds based on boron such as trimethylborane \((\text{B(CH}_3)_3)\) [99] and decaborane \((\text{B}_{10}\text{H}_{14})\) [100] were tested. Wall coating with silane \((\text{SiH}_4)\) was investigated to increase edge radiation and to reduce oxygen impurity level [101,102].

In future steady-state machines, thin-film deposition techniques previously described cannot be applied due to their short lifetime. An alternative is to coat the wall by injection of reactive gases during a tokamak discharge. The species that will form the protective layer are screened at the edge of the plasma and they are eventually deposited on the wall [103]. Conditioning by lithium pellet-injection follows a similar principle [104]. The disadvantage of these methods is that only plasma-wetted areas are covered. Beryllium evaporation was regularly used in JET to coat the walls [105]. Figure 3.1 shows the gas composition of the vacuum vessel of JET before and after beryllium evaporation. The quality of vacuum improves with a significant reduction of partial pressures of hydrocarbons, water and oxygen by about one order of magnitude [106].

![Figure 3.1: Mass spectrum from the residual gas analysis before and after beryllium evaporation. A strong reduction of the partial pressures of hydrocarbons, water and oxygen is observed [106]](image)

### 3.3 Glow-discharge cleaning

Glow-discharge cleaning (GDC) is the most effective method for surface impurity removal and it is routinely applied in almost all fusion devices. It is operated by introducing various electrodes into the vacuum vessel and then excite them with a power source to initiate and sustain the discharge. Electrodes serve as anodes and the grounded vessel walls serve as cathodes. This arrangement produces a flux
of ions to the wall used for conditioning [107]. Glow-discharge plasmas have low temperatures and densities (around 10 eV and $10^{14}$ m$^{-3}$, respectively) so the desorbed particles are not promptly ionized and re-deposited before they are pumped out of the system. GDC is unstable under magnetic fields. Therefore, it cannot be used for inter-shot conditioning in machines with super-conductive coils, where magnetic field is turned off only during maintenance periods.

When the working gas for the glow-discharge is hydrogen or deuterium, wall impurities are released by reduction of metal oxides and hydrogenation of carbonaceous deposits. Chemical erosion is the dominant process so the removal rate depends on flux density to the wall. Hydrogen is also applied to change the wall isotopic ratio [108]. Oxygen is considered to erode carbon co-deposits through the formation of carbon oxides [109]. However, in future D-T installations oxidative techniques will not be allowed due to formation of tritiated water. Tritiated water is corrosive and it has detrimental effects on the performance of the tritium plant. GDC in inert gases, e.g. helium, desorbs impurities by physical sputtering. It is also observed that carbon surfaces release carbon monoxide and hydrocarbons under helium bombardment, effect known as particle-induced desorption [110].

### 3.4 Ion cyclotron wall conditioning

Ion cyclotron wall conditioning (ICWC) is based on low temperature plasmas produced and sustained by ion cyclotron resonance (ICR) heating in reactive or noble gases [111–113]. The purpose of ICWC is to improve performance of tokamak operation. This is done by reducing the influx of desorbed impurities from the wall and by controlling fuel recycling, including mitigation of fuel inventory. It is considered as an alternative to GDC for inter-shot conditioning in ITER due to its compatibility with toroidal magnetic field. The technique has been tested in several tokamaks: Tore Supra [114], TEXTOR [115], JET [116], AUG [117], HT-7 [118].

Discharge initiation results from collisional absorption of radio-frequency (RF) power by electrons. Electromagnetic waves in the ICR frequency band do not propagate in vacuum in present-day machines so only the region close to the antenna absorbs RF power and becomes ionized. When electron density builds up, plasma waves propagate extending the ionized volume and energy deposition further in the vessel. Ion resonant energy absorption occurs in the part of the vessel where antenna frequency is a multiple of the ion gyration frequency. Charge-exchange reactions with ions in the resonant band lead to fast neutral fluxes to the wall [119].

Although plasma production and sustainment are different in GDC and ICWC, both methods owe their conditioning effect to the production of a controlled flux of particles to the wall. The impact of a working gas based on reactive or inert species is the same for both techniques, as described earlier in section 3.3 on GDC. A summary of the characteristic ICWC parameters is given in Table 3.1. Ion fluxes parallel to the toroidal magnetic field decrease exponentially from the innermost part of the limiter structures, so conditioning efficiency is in principle higher there.
Table 3.1: Summary of characteristic ICWC parameters. Parallel (∥) and perpendicular (⊥) refer to the toroidal magnetic field

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Parallel (∥)</th>
<th>Perpendicular (⊥)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electron temperature (eV)</td>
<td>1 - 10</td>
<td></td>
</tr>
<tr>
<td>Electron density (m⁻³)</td>
<td>10¹⁶ - 10¹⁸</td>
<td></td>
</tr>
<tr>
<td>Fast neutrals (keV)</td>
<td>1 - 10</td>
<td></td>
</tr>
<tr>
<td>Pressure (Pa)</td>
<td>10⁻³ - 10⁻¹</td>
<td></td>
</tr>
<tr>
<td>Heating power (kW)</td>
<td>10 - 800</td>
<td></td>
</tr>
<tr>
<td>Coupled power (kW)</td>
<td>10 - 350</td>
<td></td>
</tr>
<tr>
<td>Magnetic field (T)</td>
<td>0.2 - 4</td>
<td></td>
</tr>
<tr>
<td>Ion flux (m⁻²s⁻¹)</td>
<td>10¹⁹</td>
<td>10²¹</td>
</tr>
<tr>
<td>ICR range (MHz)</td>
<td>1 - 50</td>
<td></td>
</tr>
</tbody>
</table>

Electron densities are higher than in GDC. Therefore, desorbed impurities are not as easily exhausted. To reduce their ionization and re-deposition ICWC may be operated in a pulsed mode. Pulse and cycle length are typically 0.5 - 2 s and 10 - 20 s respectively [120].

**Surface analysis of PFM exposed to ICWC**

Until recently, main experimental activities aiming at the characterization of fuel removal and impurity retention by ICWC have been based on mass spectrometry. This is a global approach and cannot provide local understanding of how ICWC affects wall surfaces. For that purpose, a detailed surface analysis is needed. In Papers I, II and III, surface analyses of PFC were performed for the first time to determine directly the impact of ICWC discharges on wall materials. Specific aims were to assess: (i) fuel removal efficiency, (ii) poloidal and toroidal uniformity of fuel removal and (iii) retention of species used in the gas mixture, e.g. oxygen and nitrogen.

In one of TEXTOR experiments [Papers I, II], three probe-transfer systems were used to insert materials for plasma exposure: so-called test limiter locks from the bottom (LL-1) and from the top (LL-3) of the machine, and fast probe manipulator in the equatorial plane. Samples were exposed at different poloidal and toroidal locations to enable assessment of uniformity in fuel removal. Locations of the systems are shown in Figure 3.2 (a) and (b). Positions of two ICR frequency antennae are also indicated. The probes are presented in Figure 3.2 (c)–(f) where, respectively, an example of a DED tile with the regions of the cut-outs marked, equatorial probe and two sides of the holders used in limiter locks are shown. On holders at LL-1 and LL-3 (equipped also with Langmuir probes) a DED tile and a large number of laboratory-prepared samples were installed. The size of the holders, their step-like construction and the size of cut-outs (4 x 5 and 2 x 8 cm) allowed for exposures at a range of radial positions.
Figure 3.2: (a) Top view of TEXTOR. Locations of sample holders, antennae and gas inlet are indicated; (b) poloidal cross-section with position of the probes marked: equatorial holder in the lower magnetic field side and top and bottom holders; (c) DED tile with the region of the cut-outs marked; (d) equatorial holder with installed specimens; (e) rear side of the holder for the limiter locks with installed DED tile cut-outs and laboratory-prepared samples; (f) front side of the holder.
The samples were exposed to ICWC discharges in different gases: hydrogen, deuterium and oxygen–helium mixture using oxygen-18 as a marker. An important contribution of this work to fuel retention and removal studies is that, for the first time, the examination was carried out with surface analysis methods to assess changes in the deuterium content in tokamak co-deposits. Comparative experiments performed in hydrogen and in deuterium indicated the isotope exchange to be the main mechanism for the deuterium content reduction. The impact of oxygen-assisted discharges on the reduction of fuel content was less effective than that obtained with hydrogen plasma. The most probable reason is connected with differences in the operation conditions. Experiments with oxygen-18 tracer gave evidences that species used for wall cleaning are partly retained in PFC.

On the other TEXTOR experiment [Paper III] efficiency of ICWC in the removal of nitrogen from plasma-facing components was assessed. Nitrogen-15 marker was used to identify nitrogen retained during plasma operation. Carbon, tungsten and TZM samples were mounted on test limiters and exposed to nitrogen loading discharges followed by ICWC cleaning in deuterium and helium. Gas balance studies based on mass spectrometry were performed to complement surface analysis of the samples. Results showed that ICWC contributes to the removal of nitrogen, but most of the injected gas still remains in the vessel. The material migration induced by ICWC leads to deposition of impurities away from their original location.
Chapter 4

Analysis methods

4.1 Ion beam analysis

Ion beam analysis (IBA) methods involve the use of a MeV ion beam to obtain information of the elemental composition and depth profile of a target material in the near-surface region. Beam ions transfer their energy to the target material by electron excitation and nuclear collisions. This process may result in the emission of particles and radiation. Analysis of energy spectrum of emitted species provides the information. Most frequently used IBA methods are shown in Figure 4.1. They constitute an important tool in studies of plasma-facing components to determine material modification by erosion-deposition processes [121–124].

![Figure 4.1: Summary of the beam-target interaction processes](image-url)
IBA studies presented in this thesis were performed in the Tandem Accelerator Laboratory in Uppsala [125]. The major systems required to produce a well-defined mono-energetic ion beam are: ion source, accelerator and analysing magnet. The ion source produces a negative ion beam with energies in the keV range from either gaseous elements (duoplasmatron) or solid materials (cesium sputtering). In the accelerator, a high voltage terminal up to 5 MV accelerates ions twice by changing their electrical polarity in a stripper gas, usually nitrogen or argon. Ions leave the accelerator with energies in the MeV range. An analysing magnet filters those particles with unwanted energies or mass-to-charge ratios. The beam is steered towards the analysis chamber and focused by collimators on a target sample.

Ion beam analysis methods used in this thesis are briefly explained below and their main parameters are summarized at the end of the chapter in Table 4.1.

**Rutherford backscattering spectroscopy**

Rutherford backscattering spectroscopy (RBS) is based on the analysis of the energy spectrum of ions elastically backscattered from the near-surface region of a solid material. This method is used to determine composition of medium and heavy elements in a sample. The remaining fraction of the initial ion energy after collision is defined as the kinematic factor. The kinematic factor measures the efficiency of the momentum transfer in the collision. It depends exclusively on the masses of the interacting particles and the scattering angle. The kinematic factor establishes that the lower the mass of a target element, the lower the energy of the backscattered ion. It also establishes that projectiles only scatter backwards from elements with higher masses than their own.

The scattering probability is given by the Rutherford cross-section. It is proportional to the square of the atomic number of the target atom. This means that the sensitivity of the method is higher for heavier elements. Rutherford formula deviates from actual measurements at high energies. Deviation is caused by the presence of short-range nuclear forces. To avoid this effect the energy of $^4\text{He}^+$ beams is usually limited below 2.5 MeV.

Impinging ions lose energy when they go through a material due to electron excitation and collisions with atomic nuclei. Therefore, particles backscattered from an element at some depth will have less energy than particles backscattered from the same element on the surface of the material. Knowing the stopping power of a material for the incoming ion allows the measurement of depth profiles. Probed depth for a 2 MeV $^4\text{He}^+$ beam on a carbon substrate is approximately 2 $\mu$m.

Figure 4.2 shows an RBS spectrum obtained with a 1.8 MeV $^4\text{He}^+$ beam from a graphite collector probe exposed in TEXTOR during neon-assisted discharges. It is possible to identify: molybdenum from a test limiter, silicon from wall conditioning, metals from the liner, carbon from the substrate, oxygen and neon. The position of the peaks is determined by the kinematic factor, and the concentration of the species is determined by the scattering cross-section. Molybdenum and oxygen peaks have similar number of counts. However, scattering cross-section for molybdenum is
around 30 times higher than for oxygen. This means that there is more oxygen than molybdenum in the surface of the sample.

Enhanced proton scattering (EPS) is a variation of RBS. It uses a proton beam to benefit from the higher cross-sections induced by nuclear resonances. In studies of plasma-facing materials, it is used to measure carbon isotopes and beryllium.

**Elastic recoil detection analysis**

In elastic recoil detection analysis (ERDA) information from the target material comes from the analysis of the energy of recoiled particles. Kinematics of the collision are derived from energy and momentum conservation and the Rutherford formula is assumed for the recoil cross-section. Target material is typically irradiated at shallow incidence angles (about 20 °C) so this method is specially suited for smooth surfaces such as mirrors. $^4\text{He}^+$ beams with energies about 2 MeV are used to measure hydrogen isotopes. A foil is placed in front of the detector to stop or slow down scattered projectiles and recoils heavier than deuterium.

Light elements up to uranium are measured with heavy ion beams (e.g. iodine, gold) to minimize scattering of the primary beam into the detector. Depth and mass information of recoils may overlap in the energy spectrum. To avoid this, velocity and energy of particles are measured independently using a time-of-flight (ToF) system [126]. Depth profiles are determined from the stopping power of projectiles and recoils in the material. For example, information depth with a 36 MeV iodine beam goes up to half a micrometer with 2 - 20 nm depth resolution.
Figure 4.3 shows a ToF-energy spectrum from a graphite tile exposed in TEX-TOR to nitrogen-seeding discharges followed by ICWC in deuterium and helium [Paper III]. A 36 MeV \(^{127}\text{I}\)\(^{8+}\) ion beam was used. Each banana-shaped cloud of points corresponds to a different mass, so isotope separation is possible for light elements. This characteristic shape comes form the quadratic relationship between the velocity and the energy. The main goal of the experiment was to determine efficiency of ICWC to remove nitrogen from wall components. Nitrogen-15 was used as a marker, showing remaining traces after ICWC operation: \(2 \times 10^{16}\) cm\(^{-2}\).

**Nuclear reaction analysis**

Nuclear reaction analysis (NRA) is a method to determine composition of light elements and isotopes in a sample. It is based on the analysis of the products of nuclear reactions induced by the ion beam. The kinematic factor depends, in addition to the mass and energy of the interacting particles, on the variation of energy in the reaction. There is no analytical cross-sections for most nuclear reactions so they are measured experimentally [127]. Depth profiling may be performed. Its determination is based on the loss of energy of ions in the material or by scanning the beam energy when the cross-section presents a sharp resonance (width below 100 eV). \(^3\text{He}\)\(^{+}\) beams with energies around 1 - 3 MeV are extensively used in studies of PFM to determine deuterium, carbon and beryllium content [128]. Their respective nuclear reactions are \(D(\text{^3He}, p)^4\text{He}\), \(^{12}\text{C}(\text{^3He}, p)^{14}\text{N}\), \(^9\text{Be}(\text{^3He}, p)^{11}\text{B}\), where the first two terms denote reactants and the last two the products.
4.2. MASS SPECTROMETRY

Figure 4.4: NRA spectrum from a DED tile from TEXTOR

Figure 4.4 shows an NRA spectrum from a graphite DED tile retrieved from TEXTOR. The study was performed using a 2.8 $^3\text{He}^+$ ion beam [Paper I]. The purpose was to pre-characterize carbon and deuterium content before exposure to ICWC plasma. Products of induced nuclear reaction have in general higher energies than backscattered species so there is no overlapping in the energy spectrum. Information depth was up to 5 $\mu$m with 100 nm depth resolution. Peaks in the spectrum correspond to nuclear reactions with carbon, deuterium and boron isotopes from regular boronization.

4.2 Mass spectrometry

Mass spectrometry is a method to determine relative pressure of different gases in a mixture. It is performed with a residual gas analyser (RGA). The RGA consists of three parts: ion source, mass filter and detector. The gas to analyse is ionized in the ion source by electron bombardment. Then it is transported to the mass filter by focusing lenses. There are different types of mass filters. The one used in this study is the quadrupole separation system. An oscillating electric field is applied to four parallel rods so that only ions with a specific mass-to-charge ratio can pass through and reach the detector. The detector provides a current signal proportional to the number of ions detected. The main problems in the analysis of mass spectra are to discriminate species with the same mass-to-charge ratio and to know the different molecule fragmentation patterns. Mass spectrometry is applied to monitor time-evolution of impurities or fuel isotopic ratio during plasma operation. It is also used in gas-balance studies, thermal desorption spectroscopy (TDS) and to detect
leaks in the vacuum vessel [129].

Figure 4.5 shows the partial pressure evolutions of helium (M = 4), oxygen-18 (M = 18), CO (M = 28, 30) and CO$_2$ (M = 44, 48) in the $^{18}$O/He-ICWC experiment [Paper II]. $^{18}$O was used to facilitate mass spectrometry and post-mortem surface analysis. Its injection was feedback controlled on the total pressure to compensate wall pumping. The helium background was used to provide better pressure control on discharge initiation. CO and CO$_2$ outgassing increases during the pulse and it peaks after discharge termination. Exhausted particles per unit of time are proportional to the product of pressure and pumping speed.
Table 4.1: Summary of the IBA methods used in this thesis. The drawing at the bottom shows the geometry of the measurements (incidence and detection angle)

<table>
<thead>
<tr>
<th>Method</th>
<th>Isotope</th>
<th>Beam</th>
<th>Probed depth [µm]</th>
<th>Remarks</th>
</tr>
</thead>
</table>
| RBS    | Mo, W   | 2.0 MeV \(^4\)He\(^+\) | 1-2     | Sensitivity: \(10^{13}\) atoms cm\(^{-2}\)  
Depth resolution: 10-20 nm  
Poor mass resolution for high-Z elements |
| ToF-ERDA | H to Si | 36 MeV \(^{127}\)I\(^{8+}\) | 0.2-0.5 | Sensitivity: \(10^{14}\) atoms cm\(^{-2}\)  
Depth resolution: 1-2 nm  
light isotope separation  
Poor energy resolution in rough surfaces |
| NRA    | D, C    | 2.8 MeV \(^3\)He\(^+\) | 3-5     | Sensitivity D: \(10^{15}\) atoms cm\(^{-2}\)  
Sensitivity C: \(10^{17}\) atoms cm\(^{-2}\)  
Depth resolution: 100 nm  
Ion-induced detrapping |
Chapter 5

First Mirror Test at JET

Many diagnostics in ITER will rely on optical systems to access plasma while maintaining neutron shielding. This is done by using metallic mirrors to guide the light through a labyrinthine path across the shielding block. Optical stability of mirrors is therefore essential to ensure reliability of diagnostics [130]. Plasma-facing mirrors will undergo modification due to plasma-wall interaction processes. Erosion by impinging particles will change roughness and chemical composition of material, while migration and co-deposition of plasma impurities may lead to formation of coating layers on the mirrors surface. Both situations result possibly in degradation of reflectivity. There is an ongoing research on fusion experiments to assess the behaviour of the so-called first mirrors and to elaborate solutions to prolong their lifetime. Some examples are the works at JET [131], TEXTOR [132, 133], DIII-D [134,135], Tore Supra [136], or HL-2A [137].

The First Mirror Test (FMT) project is carried out at JET on request of ITER design team. It has delivered so far results from three different campaigns. Campaign I (2005-2007) [138,139] and Campaign II (2008-2009) [140] with carbon walls, and Campaign III with ITER-like walls (2011-2013) [Paper IV]. The next set of mirrors will be retrieved on January 2015. The research program involves: (i) selection of the material for mirrors, (ii) production of mirrors and their carriers for in-vessel installation, (iii) optical pre-characterization, (iv) exposure in different locations of the first wall of JET for a complete operational campaign, (v) comprehensive post-mortem analyses based on optical and surface-sensitive techniques, (vi) and development and assessment of cleaning methods to recover initial properties of the mirror.

About 80 mirrors were manufactured and tested up to now. Their design was a cube of 1 cm$^3$ with a small protruding leg for positioning in the carrier. Selected materials were polycrystalline molybdenum, stainless steel 316L and rhodium-coated molybdenum. Rhodium coating was 1 μm thick produced by magnetron sputtering [141]. Carriers are composed by two detachable plates with three or five channels as shown in Figure 5.1(a). Different slots allowed positioning of mirrors.
Chapter 5. First Mirror Test at JET

Figure 5.1: a) one of the two detachable plates of a mirror carrier. The slots allow positioning of mirrors at different distances from the mouth of the channel, b) JET divertor with location of mirrors marked in red.

at different distances from the mouth of the channel, from 0 to 4.5 cm. Carriers were located in the outer wall mid-plane and in the divertor. Figure 5.1(b) shows the different positions in the divertor marked in red: inner leg, outer leg and under load bearing tile.

5.1 Characterization of mirrors

Reflectivity of mirrors is determined before and after exposure to plasma. The measuring system must comply with work procedures for samples retrieved from JET, i.e. contaminated with tritium and beryllium. Even with carbon walls, mirrors were potentially contaminated from regular wall conditioning by beryllium evaporation. After retrieval, measurements are performed in a glove-box. To minimize its size and reduce equipment cost, spectro-photometers and integrating sphere are separable. Spectro-photometers are placed outside the controlled area and the integrating sphere and the contaminated samples inside the glove-box. Total and diffuse reflectivity is determined in the visible and near infra-red wavelength range: 400-1600 nm.

A comprehensive set of surface analysis techniques was used to determine elemental composition, chemical nature, depth profile, and topography of the mirrors. Composition was measured with ion and X-ray beam techniques: (i) RBS for tungsten, (ii) NRA for deuterium, beryllium and carbon (iii) EPS for beryllium, carbon, and tungsten, (iv) and ToF-ERDA for all isotopes. X-ray photoelectron spectroscopy (XPS) was used to determine chemical bindings in the surface layer up to 3 nm. Depth profiling was performed complementing ToF-ERDA in the first 200 nm and secondary ion mass spectroscopy (SIMS) up to 2 micrometers. Topography of mirrors was determined with scanning electron microscopy (SEM).
5.2 Summary results

With carbon-walls, mirrors were exposed up to 80 hours of plasma operation. Reflectivity of all divertor mirrors decreased by 80 - 90 % because of thick flaking co-deposits on the surfaces. Thickness of deposited layers exceeded 20 µm in some cases and they were composed primarily by carbon and fuel species. In the outer wall, reflectivity of mirrors with larger solid angles to the plasma was reduced by only 5 - 10 %. Erosion by impact of charge-exchange neutrals inhibited growing of co-deposited carbonaceous layers and therefore optical degradation of the sample. When comparing results from stainless steel and molybdenum mirrors they follow similar trends. Rhodium-coated mirrors, that initially had the highest reflectivity, showed after exposure similar reflectivity as the molybdenum mirrors even in erosion dominated areas. This is explained by modification of the first 10 - 30 nm surface layer due to implantation and diffusion of impurities.

In the first campaign in JET-ILW, mirrors were exposed to 18.9 hours of plasma operation. Reflectivity of divertor mirrors showed again a significant decrease of 50 - 85 %. However, this time there was not flaking and the deposited layers were thinner than with carbon walls, below 700 nm. Main co-deposited elements were carbon, deuterium and beryllium. All outer wall molybdenum mirrors suffered minimal changes in their reflectivity. Even those located deep in the channels with small solid angles to the plasma did not have deposits, unlike with the carbon wall. Like in previous campaign, rhodium-coated mirrors did not show better reflectivity than molybdenum mirrors after exposure to plasma.

5.3 Cleaning techniques

Results previously presented highlight the need of methods to mitigate reflectivity degradation of mirrors. Photonic methods may be used to remove co-deposits but this requires knowledge on their composition and thickness to set up laser parameters to avoid damage of treated surfaces. Photonic methods were tested on JET mirrors with beryllium-containing deposits and they did not provide satisfactory results [142]. Replaceable protective filters are ruled out because they would be promptly degraded by gamma and neutron irradiation. The most promising solutions are the use of shutters to limit exposure time and periodic molybdenum evaporation in the vicinity of mirrors to coat damaged surfaces. Also cassettes with replaceable mirrors are considered. In such case, ex-situ methods to clean mirrors, e.g. polishing or ultra-sound cleaning, have been tested and demonstrated their effectiveness [143].
Chapter 6

Summary

In this section the main scientific results are summarised. The study is based on experiments in tokamaks. It has been focused on material migration and its impact on the formation and properties of co-deposited mixed material layers on plasma-facing components, including test diagnostic tools. The major objectives were to determine: (i) fuel and impurity removal from plasma-facing components by ICWC in different gas mixtures, (ii) fuel and impurity transport connected to ICWC operation, (iii) plasma impact on diagnostic mirrors. All these topics are fully in line with the ITER needs: control and mitigation of co-deposition and fuel inventory and, the performance of first mirrors in long-term operation.

ICWC studies were performed at the TEXTOR tokamak. The novelty in the study is twofold. Tracer techniques ($^{15}$N, $^{18}$O) were used to determine precisely the impact of respective gases in ICWC. The other point was a new approach in ICWC by combining global gas balance studies based on mass spectrometry and the use of multiple surface probes exposed to discharges and then studied ex-situ with ion beam analysis methods.

The test of mirrors was performed in JET during the entire first experimental campaign in JET-ILW. The most important result is the decrease of deposits formation on mirrors in the tokamak with metal walls, in comparison to JET-C. Molybdenum mirrors on the main chamber wall retained their high reflectivity. This result allows some optimism regarding the mirror performance in ITER. Based on this, a mirror test in the ITER-like diagnostic geometry has been planned in JET.

**Paper I: Fuel removal and impurity retention by ICWC**

An important contribution of this work to wall conditioning studies is that, for the first time, the examination was carried out with surface analysis methods to assess the changes in the deuterium content in tokamak co-deposits. Experiments were performed in three different gases: hydrogen, deuterium and the tracer oxygen-18. Probe surfaces were exposed from three different locations in the machine (top,
bottom and mid-plane) to assess uniformity of the conditioning effect. Isotope exchange was identified as the main mechanism for deuterium content reduction. ICWC in deuterium showed the highest efficiency to erode carbon co-deposits under the considered experimental conditions. $^{18}$O retention was uneven. The mid-plane probe showed higher oxygen content levels than the top and bottom probes.

**Paper II: Recovery from an impurity loaded wall by ICWC**

ICWC in oxygen was applied to erode carbon co-deposits. Large oxygen retention prevented subsequent ohmic discharge initiation. After multi-pulse ICWC operation in deuterium and helium, it was possible to establish again a stable ohmic discharge. Determination of oxygen, carbon and fuel retention and removal were performed by gas phase analysis of exhausted gases. After recovery, oxygen characteristic radiation levels showed only a slight increase by a factor of 1 - 1.5 times.

**Paper III: Nitrogen removal by ICWC**

Gas balance studies based on mass spectrometry and surface analysis of probes were performed to determined nitrogen retention and removal by ICWC cleaning in deuterium and helium. Nitrogen-15 was used as a marker. Gas balance results showed that about 25 % of the retained nitrogen was removed after the conditioning discharges, whereas surface analysis of the plates based on ToF-HIERDA showed an increase of the deposited species after the cleaning. This indicates that during ICWC operation on carbon devices, nitrogen is not only pumped out but also transported to other locations on the wall.

**Paper IV: First Mirror Test**

This work is a continuation of the First Mirror Test project started at JET in 2003. Molybdenum mirrors were exposed to the entire first campaign in the JET-ILW. Mirrors located in the main chamber wall retained their reflectivity. However, mirrors in the divertor region reduced their initial reflectivity by about 50 - 80 % due to co-deposits of mainly beryllium, carbon and deuterium. Some of the mirrors were coated with rhodium to test performance of different materials. Co-deposition and material mixing in the surface layer led to similar final reflectivity for both types of mirrors.
References


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