# ACCELERATOR TECHNIQUES AND NUCLEAR ATA NEEDS FOR ION BEAM ANALYSIS OF WALL MATERIALS IN CONTROLLED FUSION DEVICES

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### Abstract

A brief overview of ion beam analysis methods and procedures in studies of materials exposed to fusion plasmas in controlled fusion devices with magnetic confinement is presented. The role of accelerator techniques in the examination and testing of materials for fusion applications is emphasised. Quantitative results are based on robust nuclear data sets, i.e. stopping powers and reaction cross-sections. Therefore, the work has three major strands: (i) assessment of fuel inventory and modification of wall materials by erosion and deposition processes; (ii) equipment development to perform cutting-edge research; (iii) determination of nuclear data for selected ion- target combinations. Underlying physics, advantages and limitations of methods are addressed. A note is also given on research facilities with capabilities of handling radioactive and beryllium-contaminated materials.

### 1. INTRODUCTION

Energy research driven by the quest for effective sources and means of electricity production is crucial for sustainable development. Despite distinct progress in energy-saving technologies and increasing number of installations based on fossil-free sources, the demand for electricity generation is ever growing to ensure functioning of transport, lighting, tele-communication and all branches of industry which require stable high-power supply. Simultaneously strong emphasis is on the safe and environmentally sound means of energy generation, while the production volume may be limited by the access to natural resources, currently available technologies, climate and, also by political situation.

Development of future technologies like Generation IV nuclear reactors and controlled thermonuclear fusion has a long history. In both cases, integrated efforts in science and technology are directed towards the construction and operation of reactor-class facilities. Controlled fusion is a multidisciplinary field encompassing plasma and ion physics, remote handling (RH) and radiofrequency (RF) technologies, nuclear physics and chemistry, demanding civil engineering, radiation protection and countless aspects of materials science and engineering: from the composition and structure of concrete for a base of a reactor containment to the detailed characterisation of the plasma-facing wall: plasma-facing materials (PFM) and components (PFC); both abbreviated jointly in the following as PFMC. The surface state of the latter class of materials is studied mainly by accelerator-based methods commonly called ion beam analysis (IBA).

In the interdisciplinary field of fusion research, the role of particle accelerators is at least five-fold: (i) ion beam analysis (IBA) of materials retrieved from vacuum vessels of controlled fusion devices; (iii) ion-induced simulation of neutron radiation effects in surfaces of solids; (iii) provision of nuclear data for ion-material interactions; (iv) ion-induced neutron generation for the material irradiation facility; (v) high current units in the neutral beam injection system for plasma (deuterium and tritium: D and T) heating. The first three aspects will be presented in the following sections with a focus on the role of accelerator techniques in the examination and testing of materials for fusion applications. Quantitative results can only be obtained using highly advanced laboratory equipment and combined with robust sets of nuclear data, i.e., stopping powers and reaction crosssections. Therefore, the work has three essential strands: (a) assessment of fuel inventory and modification of PFMC composition by erosion and deposition processes; (b) equipment development to perform cutting-edge research; (c) determination of nuclear data for selected ion-target combinations.

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#### 2. CONTROLLED FUSION AND PLASMA-WALL INTERACTIONS: IMPACT ON MATERIALS

The goal of controlled thermonuclear fusion is to harness energy that powers stars: reactions of light nuclei characterised by Q values of several MeV and high reaction rates:

$D + D \rightarrow T (1.01 \text{ MeV}) + H (3.03 \text{ MeV})$	(1a)
$D + D \rightarrow {}^{3}\text{He} (0.82 \text{ MeV}) + n (2.45 \text{ MeV})$	(1b)
$D + T \rightarrow {}^{4}\text{He} (3.52 \text{ MeV}) + n (14.06 \text{ MeV})$	(2)
$D + {}^{3}\text{He} \rightarrow {}^{4}\text{He} (3.67 \text{ MeV}) + \text{H} (14.69 \text{ MeV})$	(3)
o of Reactions 12 and 1h is around one. Deuterium f	inal ic

the branching ratio of Reactions 1a and 1b is around one. Deuterium fuel is used in most present-day devices, but the Q value (17.58 MeV) and the cross-section of the D-T process [1,2], favors that mix of hydrogen isotopes as a fuel for a reactor in a future power station. Reaction 3 has a significantly lower cross-section than Reaction 2 and, currently cannot be considered because of a very limited availability of <sup>3</sup>He.

Two major schemes of fusing nuclei have been developed. Inertial confinement fusion (ICF) uses high power photon (laser) [3] or ion beams [4] focused on a small (~1 mm in diameter) D-T containing pellet placed in a vacuum chamber of a few meters in diameter. Magnetic confinement fusion (MCF) based on plasmas generated and maintained by magnetic fields of a few T in toroidal systems [1,5]. The latter scheme exploits two reactor concepts: tokamaks [1,6] and stellarators [7]. In either case, both ICF and MCF, under terrestrial conditions the fusion plasma must be surrounded by walls of a vacuum vessel and, the energy released must then be absorbed by wall structures: 20% related to <sup>4</sup>He ( $\alpha$  particles) by PFMC [8], while the neutron energy (80% of the total) is to be transferred to a <sup>6</sup>Li-enriched blanket where the conversion to heat and tritium production will occur [9,10]. The energy confinement time ( $\tau_E$ ) of particles of (up to 1.0-1.5 s) is shorter than the plasma discharge time.

Consequently, particles escape the plasma and impinge on the wall. These are electrons, ions at different ionisation states and charge exchange neutrals (CXN). In addition, there are neutrons generated in fusion reactions as well as electromagnetic radiation with a broad spectrum from RF down to hard gamma and X-rays. They are decisive for what is called plasma-wall interactions (PWI) which involve a huge range of processes: physical sputtering, chemical erosion, reflection, implantation, gas retention, desorption, melting, boiling, splashing, arcing, cracking, ionisation, recombination, compound formation, activation and consequential transmutation [8,11-13]. All of them are dynamic arising from atomic, molecular and nuclear physics and chemistry.

A scheme of interactions is shown in Figure 1. Eroded species are immediately ionised and travel along the magnetic field lines. Eventually, if not pumped out, the migrating species are re-deposited in a close or distant location with respect to the place of origin. Re-deposition involves atoms of different elements originally eroded from the wall. It is a simultaneous co-deposition in which also fuel atoms are included. As a result, mixed material layers are formed. The composition and other properties of such deposition zones significantly differ from those of the original substrates. Thermo-mechanical incompatibility between the substrate and co-deposit may lead to flaking and spalling-off of the layer thus forming dust which constitute a major operational issue if large amounts of dust are formed and, if such particles contain considerable fraction (a few atomic %) of fuel atoms, especially radioactive tritium or neutron- activation products.

In short, PWI comprises all processes of energy and mass exchange between the plasma and the surrounding surfaces. As a result, the plasma and the wall are modified with serious consequences for reactor operation. The plasma gets contaminated and loses energy, while properties of PFMC and some crucial tools for plasma diagnosis (mirrors and windows) are changed. This has an impact on the material lifetime and fuel inventory thus for the reactor safety. However, plasma-wall interactions are unavoidable but also necessary. The wall provides vacuum conditions indispensable for operation, removes heat and – only under D-T reactor conditions - ensures final thermalization of helium ash to enable its pump out and, absorption of energetic neutrons in the blanket for tritium productions and power generation.

In-vessel materials must be, in the first place, compatible with vacuum and strong magnetic fields, while the list of requirements for PFC candidates comprises in addition: high thermal conductivity ( $\lambda$ ), i.e. over 150 m<sup>-1</sup> K<sup>-1</sup>, resilience to thermal shocks, low erosion yield by plasma species, low sorption of hydrogen isotopes to limit fuel inventory, high melting (T<sub>m</sub>) and boiling (T<sub>b</sub>) points, low-Z to minimise plasma energy losses by impurities, low erosion rate, low affinity to fuel and to oxygen impurities towards the formation of volatile products, affinity to oxygen impurities towards their gettering to form solid oxides, low neutron-induced activation. None of the known substances has properties fulfilling such requirements especially that some of them are contradictory. Therefore, the material selection is based on the approach that properties should change as little as possible under plasma impact.



FIG. 1. Plasma-wall interactions: schematic illustration of erosion-deposition processes.

The major materials of interest for PFC are beryllium (Be), carbon (C) in the form graphite or carbon fibre composites (CFC), and tungsten (W). In addition, molybdenum (Mo) is important as material for so-called first mirrors, i.e., plasma-facing materials for optical diagnostics. Crucial advantages and drawbacks of respective wall materials are compiled in Table 1, while very detailed characteristic can be found in [8]. Graphite and several types of CFC have been used in toroidal devices since seventies of the 20th century because of their excellent power handling capabilities. Issues related to the erosion rates and the formation of fuel-rich co-deposits were known, but their dramatic seriousness was recognised after full D-T campaigns in TFTR [14,15] and JET [16-21] operated with carbon walls: nearly 30% of the injected tritium was retained in the vessel, especially in the remote areas of the divertor, i.e., places shadowed from the direct plasma line-of-sight. Such locations are very difficult to reach by any cleaning method [18,20,22]. No efficient means of fuel removal have been developed and the use of carbon in a D-T fusion reactor had to be reconsidered [22-27]. A large scale-test with all-metal walls was decided at the largest tokamak in the world: the Joint European Torus (JET) [28-30]. Carbon PFC were removed and replaced by solid Be limiters and Be coatings on the main chamber wall [31,32], while W components (bulk metal and coatings on CFC tiles) were installed in the divertor [32-34]. The operation of JET with the ITER-like wall (JET-ILW) started in 2011 and, it was clearly shown that the elimination of carbon sources resulted in a significant decrease of fuel retention [35-42] and dust generation [43-48]. In a consequence, the ITER Organisation decided to abandon carbon components in the divertor and prepare for operation with Be panels in the main chamber and tungsten in the divertor [49].

Element	Advantages	Drawbacks/Limitations	Remarks
С	Low-Z. Resilience to thermal shocks and no melting $\lambda$ of some CFC > 300 W m <sup>-1</sup> K <sup>-1</sup> .	Chemical erosion by fuel atoms, C <sub>x</sub> H <sub>y</sub> formation, high erosion rate and fuel inventory in co-deposits.	PFMC in most tokamaks [8,14- 21,27,50-55] and stellarators [56-58] because of excellent power handling capabilities.
Be	Low-Z, no chemical erosion	Low $T_m$ and high sputter yield.	Used in JET-ILW in the main chamber wall [29,31,59]; the same decided for ITER wall [49].
W	High T <sub>m</sub> and low sputter yield by fuel	High-Z, risk for plasma contamination and disruptions. Activation and transmutation.	ASDEX Upgrade wall and divertor [60-61]. JET-ILW divertor [29,32-34,62]; the same decided for ITER divertor [49].
Мо	High T <sub>m</sub> and low sputter yield by CXN	High activation.	Tested candidate for first mirrors in ITER diagnostic systems [63-67]

TABLE 1. Key properties of C and metals as wall materials and diagnostic components.

The major research objectives are to determine: (i) the lifetime of PFMC, (ii) in-vessel fuel accumulation, i.e., to obtain quantitative mapping of the distribution of D and T, (iii) quantity and properties of dust with particular emphasis on the identification of sources, generation pathways and fuel content, (iv) plasma impact on diagnostic components which are crucial for plasma characterisation and machine protection. All of them are decisive for reactor economy and safety. As such, these are key points in the licensing process. Conditio sine qua non for conclusive studies is the access to materials (specimens from diverse locations: wall tiles, probes, dust) retrieved from the vacuum vessel after experimental campaigns. Research requires a huge variety of material characterisation methods which directly implies the access to laboratories with relevant apparatus, competent research teams and – in many cases – capabilities and certificates for handling radioactive materials: T-contaminated and activated.

# 3. ANALYSIS METHODS AND INSTRUMENTATION

### 3.1 Challenges and Solutions Analysis: Needs and Methods

Over the years, more than fifty different material characterisation techniques have been used in the PFMC research: ion, electron, neutron and optical spectroscopies, methods based on probing solids with magnetic field, sound waves, mechanical force or thermal means applying either a steady temperature rise or shocks by flash heating. The variety of probing ('signal in') and detection means ('signal out'), their broad energy spectrum and a range of physical processes involved in the interactions create a huge number of "signal in - signal out" combinations, and – by this – research opportunities. Nearly every combination may actually be applied in a certain area of material characterization. However, only most efficient, methods for analyses of PFMC are mentioned in the following, i.e., techniques capable of sensitive and selective quantitative determination of the content and distribution (in-depth and lateral) of a wide range of elements and, in many cases, their particular isotopes present in the examined materials. Capabilities for mapping surface species on large areas on the tiles (e.g., 10x20 cm) are also required in many cases. Compositional analyses must cover a broad range of species which constitute wall and diagnostic components, fusion fuel and gases injected for auxiliary plasma heating, plasma edge cooling, disruption mitigation, wall conditioning or as markers (tracers) in material migration studies. As a result, the list extends from H, D, T, <sup>3</sup>He, <sup>4</sup>He and other noble gases (Ne – Xe), isotopes of Li, Be, B, C, N, O, F to heavier species such as Al, Si to Cr, Fe, Ni and then to W, Re, even Au is to be taken into account. The role and origin of respective species in the reactor is addressed in Table 2 in which also the information on relevant analysis methods is conveyed.

The requirement for lateral mapping and depth profiling of such diverse compositions are met by IBA methods. Their detailed description with physics basis can be found in [68,69], while the role in PFMC analysis has been addressed in overview articles [70-72]. IBA is based on the irradiation of a solid target with an ion beam and then detection and analysis of energy and/or mass spectra of signals emitted from the surface: reflected primary ions, products of nuclear reactions, recoiled atoms, photons (from visible to X and gamma rays), sputtered species such as secondary ions (monoatomic and molecular) and neutrals. Dependent on the ion (type, energy) – signal combination there is a number of methods governed by different underlying physical processes.

- Rutherford Backscattering Spectrometry (RBS) mainly with  ${}^{4}\text{He}^{+}$  in the 1.5 3 MeV energy range.
- Non-Rutherford Enhanced Proton Scattering (EPS) with  $H^+$  in the 0.5 2.5 MeV range.
- Nuclear Reaction Analysis (NRA) a huge variety of analytical capabilities using low-Z ion beams: mainly <sup>3</sup>He<sup>+</sup> (0.6 – 6 MeV) and H<sup>+</sup>, but also D<sup>+</sup>, <sup>12</sup>C, <sup>15</sup>N and <sup>16</sup>O ions. Respective nuclear reactions are in Table 2.
- Particle Induced X-ray Emission (PIXE) and/or Gamma Emission (PIGE) using a primary 1.5 4 MeV beams of H<sup>+</sup>, <sup>3</sup>He<sup>+</sup>, <sup>4</sup>He<sup>+</sup>.
- Time-of-Flight Elastic Recoil Detection Analysis (ToF-ERDA) with <sup>4</sup>He+ or the high ion version (ToF-HIERDA) using for instance multiply charged ion beams of C<sup>n+</sup>, Si<sup>n+</sup>, Br<sup>n+</sup> or I<sup>n+</sup> beams. Depth profiling down to 700 nm.
- Accelerator Mass Spectrometry (AMS) in trace analysis of T, <sup>10</sup>B, 14C.
- Medium Energy Ion Scattering (MEIS) using a 100 400 keV <sup>4</sup>He<sup>+</sup> beam.
- Secondary Ion Mass Spectrometry (SIMS) with primary beams of Ar<sup>+</sup>, Cs<sup>+</sup> or O<sup>-</sup> of a few keV. The method is sensitive but the quantification in complex mixed-material co-deposits is difficult.

Species	Origin and role in a reactor	IBA Method	Reaction (of practical use)*	Remarks and references
Н	Wall cleaning - conditioning gas	NRA, ERDA	<sup>1</sup> H( <sup>15</sup> N, <sup>4</sup> He) <sup>12</sup> C	H is always present in vacuum systems; information depth below 1 μm; strong detrapping by the <sup>15</sup> N beam.
D	Fuel	NRA, ERDA, EPS	<sup>2</sup> D( <sup>3</sup> He, <sup>1</sup> H) <sup>4</sup> He	NRA is the main technique in D analysis [70-80]. Depth profiling in C-H layers to over 30 μm at 6 MeV [73]
Т	Fuel	NRA, ERDA, AMS	<sup>3</sup> T( <sup>12</sup> C, <sup>4</sup> He) <sup>11</sup> B <sup>3</sup> T ( <sup>12</sup> C, <sup>1</sup> H) <sup>14</sup> C <sup>3</sup> T (D, <sup>4</sup> He)n	Use of IBA is limited. Low sensitivity of <sup>12</sup> C- <sup>3</sup> T reactions <sup>12</sup> C- <sup>3</sup> T reactions tried on JET materials [81], while the <sup>2</sup> D - <sup>3</sup> T was used on TFTR tiles [82]. AMS in trace analysis [83].
<sup>3</sup> He	Minority gas for ICRF heating	ERDA		[84]
<sup>4</sup> He	Ash of D-T reaction; Wall cleaning - conditioning gas	ERDA		[84]
<sup>6</sup> Li <sup>, 7</sup> Li	Li-beam diagnostic, wall coatings	NRA, ERDA, PIGE	<sup>7</sup> Li( <sup>1</sup> H,nγ) <sup>8</sup> Be	
9Be	Wall material	NRA, ERDA	<sup>9</sup> Be( <sup>3</sup> He, <sup>1</sup> H) <sup>11</sup> B <sup>9</sup> Be( <sup>2</sup> D, <sup>1</sup> H) <sup>10</sup> Be <sup>9</sup> Be( <sup>2</sup> D, <sup>4</sup> He) <sup>7</sup> Li	[20,71,72,75,77,85]
<sup>10</sup> Be	Be migration marker	AMS		Marker n-activated <sup>9</sup> Be tile [86]
<sup>10</sup> B and <sup>11</sup> B	Wall conditioning by low plasma in $B_2H_6$ , $B(CH_3)_3$ or evaporation from $B_{10}H_{14}$	NRA, ERDA	<sup>11</sup> B( <sup>1</sup> H, <sup>4</sup> He) <sup>8</sup> Be <sup>11</sup> B( <sup>3</sup> He, <sup>1</sup> H) <sup>13</sup> C	Analysis of PFMC from boronised machines [87-91]
<sup>12</sup> C	Wall material	NRA, EPS, ERDA	<sup>12</sup> C( <sup>3</sup> He, <sup>1</sup> H) <sup>14</sup> N <sup>12</sup> C( <sup>2</sup> D, <sup>1</sup> H) <sup>13</sup> C <sup>12</sup> C( <sup>1</sup> H, <sup>1</sup> H) <sup>12</sup> C	[18,70-72,77,78,92] <sup>12</sup> C( <sup>2</sup> D, <sup>1</sup> H) <sup>13</sup> C for C analysis on Be targets [85]
<sup>13</sup> C	Tracer in C migration studies	NRA, EPS, ERDA	<sup>13</sup> C( <sup>3</sup> He, <sup>1</sup> H) <sup>15</sup> N <sup>13</sup> C( <sup>1</sup> H, <sup>1</sup> H) <sup>13</sup> C	[93-96]
<sup>14</sup> N	Edge cooling	NRA, ERDA	<sup>14</sup> N( <sup>2</sup> D, <sup>1</sup> H) <sup>15</sup> N <sup>14</sup> N( <sup>2</sup> D, <sup>4</sup> He) <sup>12</sup> C	[85,93,97,98]
<sup>15</sup> N	Tracer	NRA, ERDA	$^{15}N(^{1}H,^{4}He\gamma)^{12}C$	[96,98-100]
<sup>16</sup> O	Major impurity	RBS, EPS, NRA, ERDA	16O( <sup>2</sup> D, <sup>1</sup> H) <sup>17</sup> O <sup>16</sup> O( <sup>1</sup> H, <sup>1</sup> H) <sup>16</sup> O	[93]
<sup>18</sup> O	Tracer for in- vessel oxidation studies	NRA, ERDA	<sup>18</sup> O( <sup>1</sup> H, <sup>4</sup> He) <sup>15</sup> N	[65,78]

TABLE 2. Species to be analysed/determined and their role in a reactor.

\*Only reactions of practical use are listed, i.e. reactions with the detection limit of minimum  $5x10^{14}$  cm<sup>-2</sup>.

Species	Origin and role in a reactor	IBA Method	Reaction (of practical use)*	Remarks and references
<sup>20</sup> Ne	Edge cooling agent	ERDA, RBS		[84,97]
<sup>21</sup> Ne, <sup>22</sup> Ne	Considered as tracers	ERDA, RBS		RBS only on light substrates
Al	Impurity from structural material of RH systems	RBS, PIXE, ERDA		
Si	Component of in- vessel diagnostics	RBS, PIXE, ERDA		SiH <sub>4</sub> (SiD <sub>4</sub> ) used for wall conditioning (siliconisation) [101]
Ar	Edge cooling agent	RBS, PIXE, ERDA		[ <mark>94</mark> ]
Fe, Cr, Ni	Vacuum vessel and antennae materials: Steel, Inconel	RBS, PIXE, ERDA		Separation with RBS is difficult. ERDA and MEIS allow for separation of Cr and Ni
Cu	Impurity from NBI system	RBS, PIXE, ERDA		PIXE in presence of Fe, Cr, Ni
Kr	Edge cooling agent	RBS, PIXE, ERDA		[94]
Мо	Vacuum vessel and antennae materials: Steel, Inconel; First mirrors	RBS, PIXE, ERDA		
W	Wall material	RBS, PIXE, ERDA		
Re	Proposed addition to W	RBS, PIXE, ERDA		Only PIXE in the presence of W
Au	In-vessel diagnostics: bolometers, coated mirrors	RBS, PIXE, ERDA		

TABLE 2. (cont'd).

\*Only reactions of practical use are listed, i.e. reactions with the detection limit of minimum  $5 \times 10^{14}$  cm<sup>-2</sup>.

For most techniques, besides ERDA, the standard lateral resolution determined by beam diameter is in the range 0.6-1.2 mm. Detailed mapping of species with a resolution of 1-30  $\mu$ m is carried out (if needed) with  $\mu$ -RBS,  $\mu$ -NRA,  $\mu$ -EPS and  $\mu$ -PIXE, i.e., using micro-beams formed in a quadrupole-equipped beamline. In ERDA or HIERDA which are based on the target irradiation at a shallow angle (usually 22.50) the beam spot is elongated: 1x4 mm.

Taking into account a range of ion beams, beam spot size, broad energy spectrum, tens of nuclear reactions and data processing software, the "toolbox" offers a huge number of analytical options. It is also clear that there is no single technique to address all needs taking into account the differences in the information depth and sensitivity for detecting respective species because these parameters are decided by energy-dependent stopping powers in ion-target systems, and by cross-sections of individual processes.

The IBA methods are complementary to each other and, they are complementary to other techniques for characterisation of PFMC and fuel inventory. In the case of light isotopes, particularly in fuel retention studies, <sup>3</sup>He-based NRA plays a prominent role. It is the only method to determine quantitatively the areal distribution and depth profiles of deuterium down to tens of micrometres in light substrates [73]. Micro-NRA facilities deuterium

mapping in regions of highly not uniform content of that isotope [102,103] and even in single grains of dust [104,105]. NRA complements results of the gas balance assessment in fusion devices [50,79,106,107] and thermal desorption spectroscopy (TDS) data [108] to obtain an overview of the global fuel retention. Determination of the fuel content in PFMC is crucial to obtain reference targets in the development of in-situ techniques: laser-induced desorption (LIDS), breakdown (LIBS) or ablation (LIAS) spectroscopy techniques [109-114].

In the third column of Table 2 ERDA is listed in every set of useful/recommended techniques. ToF- HIERDA is an extremely powerful tool in the determination of low-Z isotopes on surfaces, especially when using a gas ionization chamber (GIC) detector [115], as it has been shown in studies of PFMC and wall probes from the TEXTOR [89,90], JET [77], COMPASS [91] tokamaks, and from laboratory experiments on mirror testing [116]. A great advantage is a simultaneous analysis of H, D, <sup>3</sup>He and <sup>4</sup>He [84]. High mass resolution facilitates conclusive results in material migration studies which involve the injection of tracer gases labelled with rare isotopes such as <sup>13</sup>CH<sub>4</sub> [90], <sup>15</sup>N<sub>2</sub> [90], <sup>18</sup>O<sub>2</sub> [65,84] when it is essential to discriminate between the main and minor isotopes, e.g., <sup>12</sup>C eroded from PFMC and the injected <sup>13</sup>C tracer. The GIC detector opens possibilities for applying other tracers: <sup>10</sup>B<sub>2</sub>H<sub>6</sub>, <sup>21</sup>Ne, <sup>22</sup>Ne. Figure 2 shows a spectrum of species detected with a 42 MeV <sup>127</sup>I<sup>8+</sup> beam on the PFC surface retrieved from the TEXTOR tokamak after experiments with <sup>13</sup>CH<sub>4</sub> and <sup>15</sup>N<sub>2</sub> tracers.



FIG. 2. ToF HIERDA spectrum recorded after tracer experiments for the limiter tile of TEXTOR.

### 3.2 Ion-induced damage in materials

The other role of accelerators in fusion research is in the ion-induced simulation of neutron damage in materials [10,116-118]. The damaged surface structure has a major impact on fuel retention in PFC and, also on optical performance of crucial diagnostic components like so-called first mirrors, i.e., metal mirrors acting as plasma-facing components in all optical plasma diagnosis systems (spectroscopy and imaging) in ITER, i.e., the reactor-class machine under construction. The impact of irradiation with H, <sup>4</sup>He (transmutation simulation) and Mo, Zr, Nb (simulation of n-induced damage) on the optically active layer of Mo mirrors has been presented [116-117]. There are three key points in such study: (i) the selection of irradiation conditions to by H, <sup>4</sup>He and high-Z species to influence changes in the optically active layer (OAL) of the mirror, i.e., maximum 30 nm of the outermost surface; (ii) the irradiation and determination of reflectivity changes; (iii) ToF HIERDA measurements of H and <sup>4</sup>He depth profiles, their changes in time and the dependence on the irradiation sequence. Plots in Figure 3(a) and (b) show, respectively, the depth profiles of H and He following the irradiation of polycrystalline Mo mirrors only with a 2 keV H<sup>+</sup> beam 14x10<sup>16</sup> cm<sup>-2</sup> and, first with 5x10<sup>16</sup> cm<sup>-2</sup> of 2 keV <sup>4</sup>He<sup>+</sup> and then with 14x10<sup>16</sup> cm<sup>-2</sup> of 2 keV H<sup>+</sup>. The results indicate that the damage produced by helium has a strong impact on the amount and depth distribution of hydrogen: the H profile is deeper when combined with H<sup>+</sup> only: from 2% to 4% atomic.



FIG. 3. ToF HIERDA depth profiles of H and He following two types of irradiation:(a) irradiation only with H; (b) irradiation with He followed by H.

### 3.3 Instrumentation

A pre-requisite for the advanced accelerator-based material research, either analysis or modification, is a laboratory (or a network of laboratories) with equipment providing a broad range of capabilities regarding the beam composition, energy, current, particle detection and, the control of experimental parameters: gas feed, temperature etc. A review of twelve accelerator laboratories with a detailed account on the facilities relevant to studies of fusion-related materials has been given in [72]. Among them, there are six laboratories capable of handling and analysing Be- and T- contaminated materials from JET: from full not sectioned Be tiles (12cm x 20cm) to smaller sectioned samples and dust. Work procedures with such materials (handling, transport etc.) have been addressed in [71,72], while very details are in [119].



FIG. 4. Tandem Laboratory at the Uppsala University: (a) the accelerator; (b) analysing magnet; (c) six beamlines with the description of their main purpose.

New developments of the instrumentation are crucial to enhance and to broaden research capabilities. Images in Figure 4 show the 5 MV Pelletron Tandem (National Electrostatic Corporation, NEC) and the beamline arrangement at the Tandem Laboratory, Uppsala University, Sweden. Two gas and two sputter ion sources allow for the formation of beams in all mass ranges, from low-Z (H - Li), medium-Z (Be – Si) and, with some exceptions, high-Z up to Au. There are six beamlines for standard IBA (PIXE, RBS, NRA and ToF ERDA with GIC detector [115]) and very specific tasks like <sup>15</sup>N NRA with a gamma detector, AMS used mainly in the <sup>10</sup>Be [86] and <sup>14</sup>C analyses, µ-beam with PIXE, RBS, NRA. A separate line is dedicated to material modification by ion irradiation while the newly developed system on the sixth beamline is for in-situ and in-operando research: Set-up for *In- situ* Growth, Material modification and Analysis (SIGMA) [120,121].



FIG. 5. SIGMA chamber – 1; viewing ports on both sides of the chamber – 2 and 2'; triple evaporator – 3; residual gas analyzer – 4; sample manipulator – 5; ion gun – 6; load-lock chamber – 7.

As already mentioned, all processes involved in PWI are dynamic. Direct in-situ material studies inside fusion devices are technically either very challenging or not possible at all. Some fundamental processes can therefore be investigated under controlled laboratory conditions. The SIGMA system, presented in Figure 5(a) and (b), has been designed to facilitate material modification with in-situ IBA employing both light and heavy beams for RBS, NRA, PIXE, PIGE, ToF-ERDA at the 2- 50 MeV energy range. Due to large viewing ports optical characterization is also carried out. Several gas feeds, three evaporation cells, a sputter gun (1-5 MeV) enable diverse material modification scenarios. Sample annealing to 1100 °C combined with gas phase analysis offers a wide range of experimental possibilities in studies of fuel retention in fusion-relevant targets.

Two other accelerating systems at Uppsala University extend research on material modification [121]. With a 350 kV implanter (Danfysik) equipped with three changeable ion sources (gas, oven-based, sputter) the simulation of neutron-induced damage by means of light and heavy ion irradiation is carried on mirrors tested for diagnostic and heating systems in future fusion devices [116-118]. Two other beamlines are for: (i) ToF-MEIS and (ii) low energy RBS and NRA. The application of MEIS [77] ensured sensitive high-resolution determination of surface composition and has led to new topics in material migration.

A low energy ion gun (up to 10 kV) in another system equipped with two chambers is the base for ToF Low Energy Ion Scattering (ToF LEIS), Auger Electron Spectroscopy (AES) and Low Energy Electron Diffraction (LEED) [122]. Material modification capabilities annealing, sputtering and in-situ growth of thin layers. In all materials analyses, the quantification of composition is essential. In the case of IBA, it relies on the energy dependent cross-sections in the interactions of fast particles with matter.

#### 4. STOPPING AND REACTION CROSS-SECTIONS

The energy deposition by energetic charged particles in matter is conveniently described by the energy deposition per unit path length, commonly referred to as stopping power (S). Dependent on the nature of the interaction, i.e. whether energy is deposited by elastic interaction between ion and target nuclei or by excitation of the electronic system of the target, one refers respectively to electronic (Se) or nuclear (Sn) stopping power [123]. By a convenient transformation one obtains the stopping cross section by normalization by the atomic density N, which yields a quantity independent of the mass density of the target material. In any representation, accurate knowledge on the specific energy deposition of charged particles forms a key ingredient for quantification in ion beam analytical methods, by providing depth scales, in ion implantation by allowing for a prediction of particle range and in modelling of, e.g., sputtering processes and defect formation [69,124]. At high energies, the interaction with the target electronic system has been already early modelled successfully by Bethe [125] with subsequent further improvements [126-128]. Towards lower energies, interaction becomes more complex even for the lightest ions, as details of the electronic structure of the material were predicted to affect the energy deposition [129]. These effects of the density of states of the irradiated material were later been confirmed in several experiments for metals with excitation thresholds for specific electronic states [130-131] as well as for insulators featuring a band gap [132,133]. For light ions different from protons, also projectile excitation becomes increasingly relevant [134,135] still challenging predictions up to date [136-137]. In a similar fashion as

calculations feature an increasing complexity towards low ion energies, the same applies to experiments: stopping powers are experimentally most straightforwardly obtained in transmission experiments [138], for which, at lower energies, however the deteriorating influence of surface contaminations increases. In backscattering geometry, effects of surface contamination are drastically reduced, however, at lower energies, effects of plural and multiple scattering affect the spectra, complicating the analysis, requiring accurate simulations [139,140]. An additional option, available when sufficiently thin films of the target material cannot reliably be obtained is evaluation from the height of a spectrum recorded for a thicker film or bulk of the material of interest [141. In all cases, however, material purity is of utmost importance, which is challenging to guarantee for thin layers near a surface [142]. For all the reasons above, the database of electronic stopping powers hosted by IAEA [143,144] features only a limited number of datasets at low ion energies. Also, the materials, for which stopping powers have been measured or calculated is found limited [145]. The most commonly employed source for tabulated stopping powers, the semi empirical SRIM-code [146], is thus challenged in its predictive capacity. For many aspects of research on plasmawall interaction is it, however, these low energies, which are most relevant. Low ion energies are not only relevant to model sputtering, fuel retention or defect formation, but are similarly necessary for quantification in analytical approaches such as Low- and Medium Energy Ion Scattering (LEIS & MEIS) [122,147]. Table 3, summarizes the status quo for a number of elemental target materials highly relevant for next generation fusion devices, indicating the almost complete absence of data at low energies, as well as the presence of an ambiguity of available data.

Element	H ions	He ions
Be	No data below 10 keV – no reliable data below 1 MeV.	No data below 200 keV.
С	High number of datasets	High number of datasets; limitations at low energies.
Мо	No data below 50 keV – data spread in the stopping maximum.	Only one low-energy dataset – spread in the stopping maximum.
W	Only one dataset below 100 keV.	Only one dataset below 300 keV; two datasets differing by 10% at classical IBA energies.

TABLE 3. Account on availability of the stopping powers data for selected elements.

Plots in Figures 6 and 7, show respectively detailed data for H in Be and He in W, thus illustrating the limited availability and reliability of reference data at intermediate energies or their complete absence at low energies respectively. Recently, the development of new computational approaches such as time-dependent density functional theory [148,149] or abandoning the modelling of a homogeneous electron gas [150] provides successively better predictions for specific systems, but commonly with high computational expenses. Dedicated experiments providing a better insight into the dependence of stopping powers on  $Z_2$  [151] or specifically targeting materials for PFMC [152] enhance simultaneously the predictive power of semi-empirical approaches. Nevertheless, due to the large number of relevant ion-target combinations, energies and experimental approaches, a concerted action - as proposed by a number of research groups in the CRP-F11023 coordinated by IAEA - will be necessary to build up comprehensive knowledge for the relevant materials in the relevant energy range on the stopping of H and He in Be, Fe, Mo and W. A very similar lack of data and thus necessity for acquisition of high-quality reference data is found for reaction cross-sections of especially <sup>3</sup>He with isotopes of Li, Be, B, C, also N and O in the 1-6 MeV energy range.

### 5. CONCLUDING REMARKS

The accelerator-based analysis and modification of materials is not an isolated or a passive strand of fusion research. The results directly contribute to decisions regarding the wall composition and diagnostic planning in the current and future devices, e.g., ITER and DEMO. It is a driving force for improvements and development of analytical capabilities (nuclear data sets, detectors, chambers) to ensure cutting edge research. To keep this status, continual development of the methods in accordance with what was outlined above is required. Especially the role of in-situ and in-operando systems for the material modification and analyses will be crucial for a deep insight into the dynamics of fuel retention and segregation of metals in PFMC relevant materials such as EUROFER.



FIG. 6. Data availability of experimental reference electronic stopping cross-sections for hydrogen on beryllium.



FIG. 7. Data availability on experimental reference electronic stopping cross-sections for helium-4 on tungsten.

# ACKNOWLEDGEMENTS

The work has been supported by the Swedish Research Council (VR), Grant 2016–05380. Financial support of the Tandem Accelerator Infrastructure by VR-RFI (contract #2017–00646\_9) as well as the Swedish Foundation for Strategic Research (SSF) under contract RIF14–0053 is gratefully acknowledged

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Acronym	Meaning
AMS	Accelerator Mass Spectroscopy
ERDA	Elastic Recoil Detection Analysis
GDOES	Glow Discharge Optical Emission Spectroscopy
GIC	Gas Ionization Chamber (detector)
HIERDA	Heavy Ion ERDA
IBA	Ion Beam Analysis
ILW	ITER-Like Wall
JET	Joint European Torus
JET-C	JET with Carbon wall
JET-ILW	JET with ITER-Like Wall
LEIS	Low Energy Ion Source
LIAS	Laser-Induced Ablation Spectroscopy
LIBS	Laser-Induced Breakdown Spectroscopy
LID	Laser-Induced Desorption
NRA	Nuclear Reaction Analysis
PFC	Plasma-Facing Components
PFM	Plasma-Facing Materials
RBS	Rutherford Backscattering Spectrometry
RF	Radio Frequency
RGA	Residual Gas Analyser
RH	Remote Handling
SIGMA	Set-up for In-situ Growth, Material modification and Analysis
SIMS	Secondary Ion Mass Spectroscopy
TDS	Thermal Desorption Spectroscopy
ToF-ERDA	Time-of-Flight ERDA/HIERDA

#### List of Acronyms