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Tracer techniques for the assessment of material migration and surface modification of plasma-facing components

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HIGHLIGHTS

- Tracer techniques were used in the TEXTOR tokamak to determine high-Z metal migration and the retention of species used for plasma edge cooling or wall cleaning under different operation conditions.
- Volatile molybdenum hexa-fluoride, nitrogen-15 and oxygen-18 were used as markers in tokamak or ion cyclotron wall conditioning discharges (ICWC).
- The objective was to obtain qualitative and quantitative of a global and local deposition pattern and material mixing effects.
- The deposition and retention was studied on plasma-facing components, collector probes and test limiters.
- Optical spectroscopy and ex-situ analysis techniques were used to determine the plasma response to tracer injection and surface composition modification.

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ABSTRACT

Tracer techniques were used in the TEXTOR tokamak to determine high-Z metal migration and the deposition of species used for plasma edge cooling or wall conditioning under different types of operation conditions. Volatile molybdenum hexa-fluoride, nitrogen-15 and oxygen-18 were used as markers in tokamak or ion cyclotron wall conditioning discharges (ICWC). The objective was to obtain qualitative and quantitative of a global and local deposition pattern and material mixing effects. The deposition and retention was studied on plasma-facing components, collector probes and test limiters. Optical spectroscopy and ex-situ analysis techniques were used to determine the plasma response to tracer injection and the modification of surface composition. Molybdenum and light isotopes were detected on all types of limiters and short-term probes retrieved from the vessel showing that both helium and nitrogen are trapped following wall conditioning and edge cooling. Only small amounts below 1×10^{19} m^{-2} of ^{18}O were detected on surfaces treated by oxygen-assisted ICWC.

1. Introduction

Tracer techniques are widely used in science and industry to determine for instance flows, reaction rates and mechanisms, i.e. to reveal decisive steps in studied processes. In the field of plasma-wall interactions (PWI) such techniques have been used to study material migration which is decisive for lifetime of plasma-facing components (PFC) and fuel inventory. The term "tracer" denotes species introduced on purpose to plasma edge, either by puffing exotic (e.g. WF_6) or rare isotope (e.g. $^{15}N_2$, $^{18}O_2$) gases, by ablating tracer material using lasers or, by exposing marker tiles coated with well defined sandwich-type layers of heavy and light elements. Experimental approach to the determination of erosion phenomena involves the combined application of spectroscopy, mass spectrometry, surface probes for ex-situ studies and tracer materials. The selection and application of tracers requires availability of a tracer, its affordable cost, proper surface probes placed in several locations and relevant analytical methods in order to obtain a deposition pattern.

The amount and distribution of eroded, transported and then re-deposited species is determined by means of several surface-sensitive methods with a leading role of special variants of ion beam analysis using either a micro-beam or heavy ions for elastic

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recoil detection. For many years a programme dedicated to testing of PFC and studies of erosion processes by tracer techniques have been carried out at the TEXTOR tokamak, especially using methane labeled with carbon-13 [1–5]. This was followed by ^{13}C migration experiments in JET [6–12] and other machines [13–15]. Isotope-based tracers are impractical in the case of heavy metals (e.g. W) both because of a tremendous cost of isotopic enrichment and the lack of quantitative surface analysis techniques capable of reliable distinguishing heavy isotopes of similar masses (resolution problem). Instead, another refractory metal being a proxy to tungsten can be deposited as a coating on a W component or a volatile compound may be used.

The intention of this work is to provide a brief overview of results regarding: (a) oxygen impact on PFC and retention after oxidative fuel removal methods with an ^{18}O marker; (b) detection of nitrogen co-deposition after plasma edge cooling with a ^{15}N marker; (c) mobility of heavy metal following the injection of volatile hexa-fluorides. MoF_6 was injected to test whether such compound can be considered as a tracer for W migration studies in a device with a tungsten wall.

2. Experiments and methods

The experimental programme was carried out at the TEXTOR tokamak during: (a) standard plasma operation with neutral beam injection (NBI) heating and (b) ion cyclotron wall conditioning (ICWC). TEXTOR, in its operation till December 2013, was a mission-oriented machine with a focus on PWI processes and material testing. It was equipped with a system of three complex manipulators for the exposure of dedicated targets: two test limiter locks at the bottom and top of the torus [16] and a rotated collector probe in the equatorial plane [17].

Oxygen-18 (natural abundance 0.2%) was used in ICWC discharges in a $He/^{18}O_2$ mixture. The pulses lasting in total for 136 s were generated using either one or two radio frequency (29 MHz) antennae (50 or 100 kW) in the presence of magnetic field of 0.23 T or 1.9 T; operation details have been described in a paper on ICWC development [18]. 3.6×10^{22} atoms of ^{18}O were injected into the ICWC discharges. The major point was to assess oxygen retention. Using a tracer one can discriminate between the oxygen retained in materials under the discharge, from air oxygen (O-16) chemisorbed on surfaces during the sample transfer to a surface analysis station. A number of different substrates was exposed, including a set of silicon targets pre-coated with deuterated carbon films.

Intense tungsten testing done over the years in TEXTOR has led to a remarkable W background level on graphite PFC. As a consequence, molybdenum was selected as a proxy of tungsten to study high-Z migration. Molybdenum hexafluoride (MoF_6) was puffed locally from the bottom of the machine through an inlet in a polished graphite plate installed on a test limiter. The entire procedure was similar to that used for the WF_6 injection experiments [19]. The puffing was done during 31 neutral beam injection (NBI) heated discharges: 14.2×10^{20} Mo atoms in total, injection at 0.8–1.8 s into the discharge. The heating phase lasted 4.5 s in the period 0.8–5.3 s into the discharge. Nitrogen-15 was simultaneously (timing: 1–5 s) puffed into the plasma edge: 5.3×10^{21} ^{15}N atoms in 22 pulses. Graphite and titanium plates were installed as flux collectors on test limiters at the top and the bottom of TEXTOR, while a rotated collector probe was at the equatorial plane. The injection was done on the last operation day of TEXTOR and followed by the dismantling of all in-vessel components making them available for ex-situ studies. Samples of dust and debris were also collected.

Local and core spectroscopy measurements during the experiment with MoF_6 were performed using systems described in [20,21]. The local studies with cameras and imaging spectrometer (Acton Research Corporation, model SpectraPro 500) with a holographic grating in Czerny–Turner arrangement were used at a central wavelength of 395 nm to cover both FII (402 nm) and MoI (390 nm) lines visible during the injection. In order to study lines related to the core plasma and its impurities a VUV spectrometer (SPRED) [21] was utilized for recording spectra in the range 10–200 nm. This allowed observation of higher ionization states of the most abundant plasma impurities including fluorine (53.521 nm) and nitrogen (26.629 nm). The injection system typically causes a delay of ~200 ms between opening the valve and spectroscopic visibility of the injection.

X-ray spectroscopy, both wavelength (WDS) and energy dispersive (EDS), and ion beam methods were used for analysis of the main and test limiter plates and collector probes. Time-of-flight heavy ion elastic recoil detection analysis (ToF-HIERDA) with a 32 MeV $^{127}I^{9+}$ beam was used to determine species deposited on the test limiter after the MoF_6 injection, Rutherford backscattering spectroscopy for the determination of Mo on the toroidal limiter, while ^{18}O and deuterium were quantified using nuclear reactions $^{18}O(p,\alpha)^{15}N$ [22] and $^{3}He(d,p)\alpha$, respectively. The sensitivity level for the ^{18}O detection is about $2–3 \times 10^{18}\ m^{-2}$. EDS and scanning electron microscopy (SEM) were used for studies of layers on limiter tiles and dust collected from the machine after the last experiment in TEXTOR.

3. Results and discussion

3.1. Oxygen-assisted ICWC operation: ^{18}O tracer

Over twenty silicon discs coated with approximately 150–240 nm thick a: C–D layers were used as probes exposed to ICWC discharges in the $He/^{18}O_2$ mixture. A complex holder, as shown in [23], was installed on the test limiter lock. Post exposure measurements have shown that ^{16}O is a dominant oxygen species with a content $1.5–4.0 \times 10^{21}\ m^{-2}$. It has partly originated from the silicon oxidation (SiO_2) before the coating process and also from atmospheric oxygen ad-/absorption in the carbon film after the exposure in TEXTOR. Therefore, to make a correct assessment of the ^{18}O retention from the ICWC plasma, the natural abundance of that isotope was also taken into account: range $3–8 \times 10^{18}\ m^{-2}$ for the above mentioned ^{16}O content. The total amounts of ^{18}O found on surfaces of the Si discs after the exposure were $6–10 \times 10^{18}\ m^{-2}$ showing that only around $2–4 \times 10^{18}\ m^{-2}$ tracer atoms were deposited from the ICWC plasma and retained. This content is very low: over 15 times smaller than that detected on the pre-characterised graphite probes (cut-outs from limiters) exposed to the same discharges [23]. This difference may be attributed to the surface roughness of the limiters, i.e. greater specific area in comparison to smooth silicon substrates with carbon films. Isotope exchange upon exposure to air cannot be excluded, but no quantitative conclusions can be drawn here. In summary, this tracer experiment has shown a minimal impact of oxygen-assisted ICWC on the retention of that species on surfaces other than carbon or with only a small quantity of carbon.

3.2. Molybdenum and nitrogen migration

Fig. 1 shows a typical evolution of the local spectroscopy signals of MoI and FII. The timing of injection marked in the graph corresponds to the opening and shutting of the valve located more than one meter from the vacuum vessel. As a result, the heavy gas enters the torus with a delay of approx. 0.45 s. This explains the shift of the corresponding Mo and F signals in comparison to the start/end of the puff. The shape of both traces is the same and