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Hydrogen isotope depth profiling in carbon samples from the erosion dominated inner vessel walls of JET

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Abstract

In order to investigate the hydrogen isotope accumulation at the erosion dominated inner walls of fusion experiments, several small carbon samples have been mounted in the inner wall tiles of the torus of the JET Joint Undertaking. After about 2.5 years of JET operation, including the extended D/T phase and the D/D cleaning phase, the samples have been removed and analysed by RBS, ERDA and AMS. In the near surface region only small T concentrations are measured. A much larger T concentration is found at depths between about 1 and 2 μm. This may originate from energetic (~1 MeV) T which is produced during the D/D discharges and implanted to large depth. The lower energy (keV) T which is implanted during the D/T discharges became effectively removed from the inner wall, by isotope exchange with implanted D or H and/or by erosion of the wall. The total amount of T within a depth of about 2 μm was found to be about $1–3 \times 10^{13}$ T/cm^2, while in a surface layer of about 0.5 μm about $1–3 \times 10^{12}$ T/cm^2, about $2–4 \times 10^{16}$ D/cm^2 and about $4–9 \times 10^{16}$ H/cm^2 were measured.

Keywords: Hydrogen retention; Deuterium retention; Tritium retention; JET; Erosion

1. Introduction

In fusion experiments with magnetically confined hot plasmas, the surrounding vessel walls are bombarded by energetic ions and neutral atoms of the hydrogen isotopes [1–3], and high concentrations of the hydrogen isotopes build-up in surface layers of the plasma facing vessel walls. Extra plasma fuelling is necessary because of this accumulation of hydrogen in the wall materials. Furthermore, the accumulation of tritium in the vessel walls represents a critical radiological problem. A major accumulation of hydrogen isotopes was found to occur at deposition dominated colder more remote wall areas, such as, at the sides of limiters [4,5] and at areas in the shadow of adjacent wall tiles and the divertor corners [5–9]. This occurred mostly by 'co-deposition' [7,8,10,11], i.e. the simultaneous implantation of hydrogen isotopes from the plasma into the deposited layers (which consist predominantly of carbon). We report on investigations about the build-up of hydrogen isotopes at areas that are not deposition dominated but erosion dominated, such as the inner wall areas. 'Long-term samples' (LTSs) have been installed in the carbon tiles of the inner walls of the European tokamak experiment JET during the last discharge period, ending in 1999, including the period when D and T have been used routinely as the fuelling gases [12–15].

2. Experimental

The JET Joint Undertaking is a divertor tokamak experiment with a major radius of about 3 m, a minor

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radius of about 1.25 m and a vertical elongation of about 1.7. The maximum toroidal magnetic field on the axis is about 4 T, the plasma current is generally ~3 MA and the total discharges last for about 20 s, out of which the divertor is active for about 5–10 s. In 1997/99 while the T trapping at the inner walls was investigated, about 5700 discharges were performed in D and about 1400 discharges in D and T, of which 220 discharges had a T concentration above 40% (Fig. 1).

For measuring the hydrogen isotope trapping in the vessel walls, small 'Long-term samples' (LTSs) with dimensions of about $0.9 \times 0.9 \times 0.4$ cm^3 have been installed in holes of the inner wall tiles in octant 4 at several poloidal positions, ranging from sample 'iw 402' close to the top to sample 'iw 411' close to the bottom. The LTS have been made out of CFC material, the same material that is used for the inner wall tiles. The plasma-exposed surfaces were polished and had been implanted with Au markers at depths of about 2 and 4 μm. Furthermore about 1/3 of the plasma exposed sides were covered with a layer of about 0.5 μm Al. The LTS could be removed remotely during the shutdown of JET in 1999. Due to the small dimensions, the radioactivity was sufficiently low so that the LTS could be sent to IPP Garching for a detailed analysis.

Several analysis methods were applied for measuring both the surface layer composition as well as the hydrogen isotope concentrations. These are RBS, ERDA and AMS. However, due to the structure of this CFC material, even the polished surfaces are always non-uniform and had pores typically in the μm range. Some techniques, such as RBS and AMS, allow one to measure a depth profile also at rough surfaces with the depth scale relative to the actual local surface area as well as the total amount of trapped hydrogen isotopes [16]. For the ERDA measurements relatively flat surface areas have been taken. However, the depth scale has an uncertainty corresponding to the surface roughness.

3. Rutherford backscattering (RBS)

All samples have been analysed with RBS using 2.6 MeV ^{4}He-ions at a scattering angle of 165°. Two spectra from an Al deposited area of a wall sample, before and after the plasma exposure, are shown in Fig. 2. After the plasma exposure the Al layer has been mostly eroded, confirming that the inner walls are erosion dominated due to sputtering by energetic charge exchange neutrals from the plasma [9,17]. The total erosion of the Al layer can be estimated from previous erosion measurements [9] to be about 1.5 μm, which is in agreement with the observation that the 0.5 μm thick Al-layer has mostly disappeared. Some Al may be still present at the bottom of pores, where it is less eroded. The estimated carbon erosion is at least 3.6 μm, again in agreement with the observation that the Au markers which had been

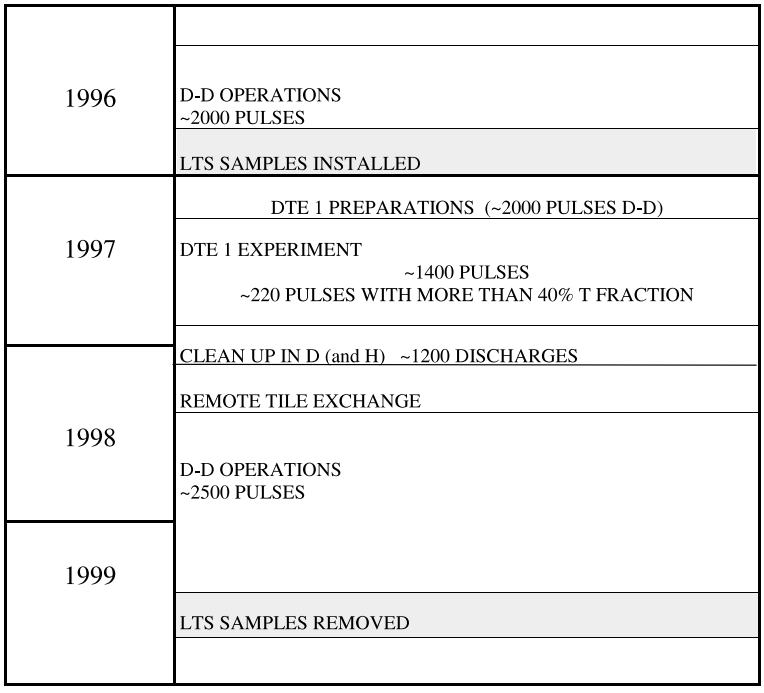

Fig. 1. JET in-vessel inventions between 1996 and 1999.

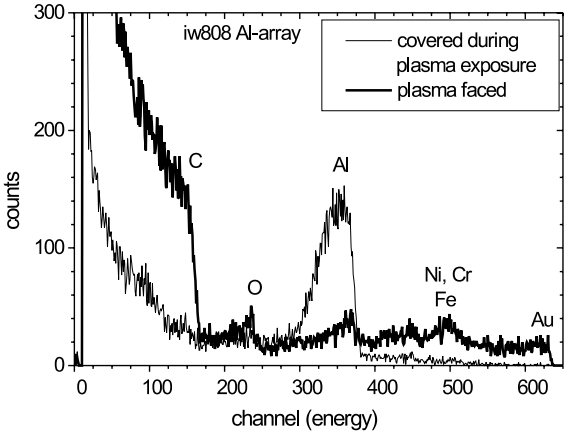

Fig. 2. RBS depth profile at JET inner wall tile 'iw 808' (octant 8 lower middle part), before (covered during plasma exposure) and after plasma exposure.

implanted to depths of about 2 and 4 μm have been mostly eroded. There is also a small deposition of Ni, Fe, Cr and Ti, which originates from the other areas of the vessel walls.

4. Elastic recoil detection analysis (ERDA)

The hydrogen isotopes have been measured by ERDA using both 2.6 MeV ^{4}He-ions at IPP Garching [18] and 35 MeV Cl-ions at Forschungszentrum Rossendorf [19]. The depth profiles calculated from the measured recoil energy spectra are shown in Fig. 3(a) and (b). Due to some surface roughness also at the relatively flat areas selected for the analysis, they have some uncertainties. Hydrogen and deuterium are trapped mostly in the calculated surface layer of about 0.5 μm at concentrations up to about 2 and 5.5 at.%. There is up to about a factor of 2 more hydrogen than deuterium, but they are distributed with a similar depth distribution. The total amounts of hydrogen and of deuterium in the analysed surface layer of about 0.5 μm, are about 4–9 10^{16} H/cm^2 and 2–4 10^{16} D/cm^2. In the ERDA measurement with 35 MeV Cl ions at samples 'iw 405' from octant 4 and 'iw 608' from octant 6, i.e. at a similar position as sample 'iw 408' from octant 4, a few pulses at the energies corresponding to T in a surface layer of ≈0.5 μm allowed a rough estimate of the T content to be made, resulting in about 6 ($\pm$5) $\times$ 10^{13} and about 2 ($\pm$2) $\times$ 10^{13} T/cm^2, respectively. The values are introduced in Table 1. The numbers are larger than those measured with AMS for the depth $\leqslant$0.5 μm. Taking into account the poor statistics and the uncertainties in depth due to the surface roughness the ERDA and the AMS results are more or less consistent.

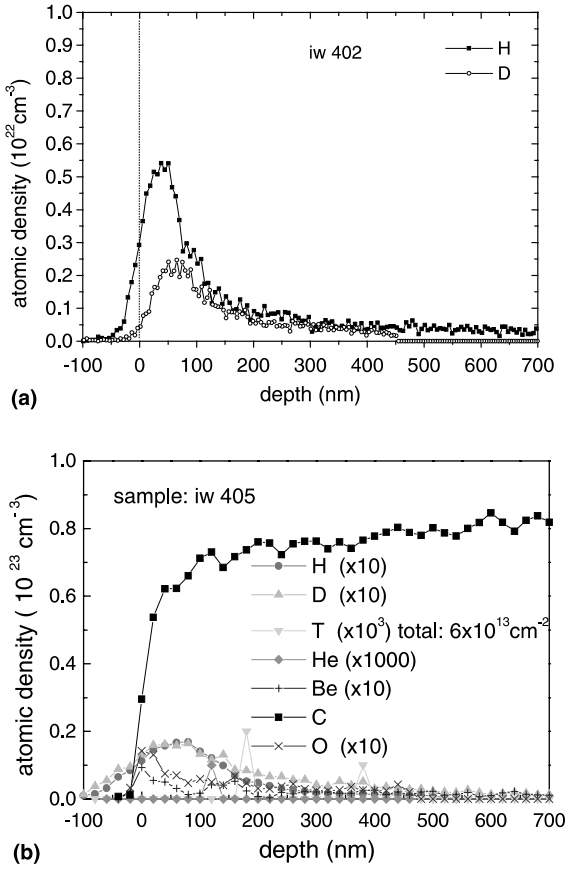

Fig. 3. (a) ERDA depth profile at JET inner wall tile 'iw 402' (octant 4, upper part) as measured with 2.6 MeV ^{4}He-ions, (b) ERDA depth profile of several low Z elements, including T, at JET inner wall tile 'iw405' (octant 4, upper middle part) as measured with 35 MeV Cl-ions.

5. Accelerator mass spectrometry (AMS)

AMS is the most sensitive method for measuring very small amounts of isotopes such as the T trapped and accumulated in tiles of the vessel walls of fusion experiments [20,21]. In the present work, tritium in the LTS was measured using the Ultra Clean Injector (UCI) of the 15 MV MP-tandem accelerator at the accelerator laboratory of Ludwigs Maximilian Universität (LMU) and Technische Universität München (TUM) in Garching, with an external magnetically analysed Caesium (Cs) ion beam for sputter erosion of the target to be analysed.

In order to obtain a good depth resolution and to avoid crater effects the UCI was upgraded [22]. The samples to be analysed were mounted onto a computer controlled, UHV compatible *x*-*y* scanning unit, which moves the target holder in front of the Cs-sputter beam. The surface is scanned in *x* and *y* in steps of 3 mm on an area of about 36 mm^2. The sputtered negative ions are

Table 1
The amounts of hydrogen isotopes in different LTS from the inner walls of JET, as measured by AMS in depths up to about 0.5 and 2 μm and by ERDA in depths up to about 0.5 μm

JET sample	Total amount (T/cm^2) (AMS and ERDA)	Total amount (H/cm^2) (ERDA)	Total amount (D/cm^2) (ERDA)
iw 402	$2.6(\pm1)\times10^{13}$ (depth $\approx$2 μm) $\approx3\times(\pm2)10^{12}$ (depth <0.5 μm)	9×10^{16}	4×10^{16}
iw 405	$\approx6(\pm5)\times10^{13}$ (depth < 0.5 μm) (ERDA measurement)	3.4×10^{16}	4×10^{16}
iw 408	$1.5(\pm1)\times10^{13}$ (depth $\approx$2 μm) $\approx10^{12}$ (depth <0.5 μm)	6.4×10^{16}	2.5×10^{16}
iw 411	$1.2(\pm0.8)\times10^{13}$ (depth $\approx$2 μm) $\approx10^{12}$ (depth <0.5 μm)	4.4×10^{16}	2×10^{16}
1w 608	$\approx2(\pm2)\times10^{13}$ (depth < 0.5 μm) (ERDA measurement)	3.8×10^{16}	3.2×10^{16}

mass analysed and the mass 3 ions are injected into the tandem accelerator. In the stripper, at high voltage the molecules, such as HD, are split up and the mass 3 tritium ions are finally detected by a system of five sequential PIN diodes. The area scanned by the sputtering Cs ion beam gives a matrix of 100 data registers. In order to avoid effects of the rim at the sides of the sputtered crater, only the results of the measurements in the centre of the analysed spot are taken in account. Using the measured sputter beam intensity and the total depth of the crater as measured with a profilometer at the end of the experiment the cycle number is converted to depth. The calibration of the measured tritium concentration is performed by using two tritium standards (concentrations T/C: 10^{-9} and 10^{-10}). [23]. Two depth profiles measured at samples 'iw 402' from the upper part of the JET inner wall and at 'iw 411' from the lower part of the JET inner wall in octant 4 are shown in Fig. 4. Generally the T concentration near the surface is low. It increases by more than a factor of 10 at a depth larger than about 1 μm. At depth larger than about 2 μm, the T concentration decreases to zero, however small concentrations of T have been measured up to depth of several

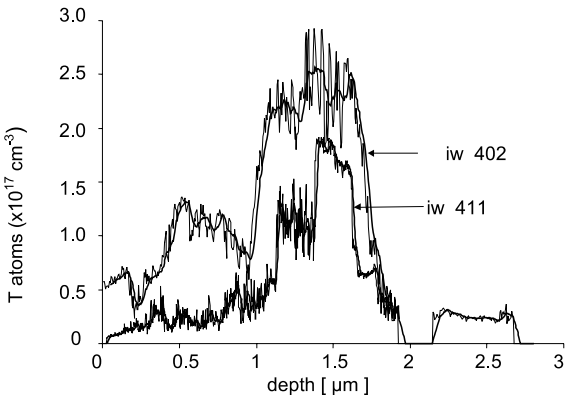

Fig. 4. T depth profiles for the JET inner wall tiles 'iw 402' (octant 4, upper part) and 'iw411' (octant 4, very low part) as measured by AMS with the 15 MV tandem accelerator at Technical University München, Garching. The solid lines represent more smooth interpolations to the dashed curves connecting the measured data points.

10 μm, not shown in the figure. The T found at these large depth may be the result of the surface roughness and inhomogeneities in the material and has not been evaluated further.

The total amount of tritium in the different samples was calculated from these measured depth distributions, the results are also shown in Table 1. AMS allows one to analyse to much larger depths (several μm), compared to the analysis with MeV ion beam where only layers of about 0.5 μm are measured. For a comparison of the amount of T measured with AMS and with ERDA only the amount of T within a surface layer of 0.5 μm is calculated from the AMS results. The numbers are also shown in Table 1.

6. Discussion and conclusions

The total amounts of T at the erosion and implantation dominated inner CFC wall tiles of the JET vessel walls after the 1997/99 discharge period, as measured by AMS and ERDA, are found to be up to about 2.5×10^{13} T/cm^2 in a layer of up to 2 μm with a concentration of up to 2×10^{-4} at.%. For the surface layer of about 0.5 μm, such as measured typically in MeV ion beam analysis, the T concentration is about one order of magnitude lower with total amounts of up to 10^{12} T/cm^2. The results of the different T analysis techniques differ by up to an order of magnitude which is within the uncertainties of the different measuring techniques, mainly due to the surface roughness and poor counting statistics. The large concentrations of 2 to 5 at.% of H and D measured in a surface layer of about 0.5 μm are due to implantation of neutrals with keV energies during the plasma discharges. Generally the H is larger or about the same as the D, though the H/D ratio in the JET plasma is only a few %. Large amounts of H in tokamak wall samples were also observed in other experiments [24], and are most likely due to uptake or isotopic exchange of H from water vapour during exposure to air.

For understanding the T amounts and depth distributions several effects have to be taken into account. During the experimental period when the T was built up

in the inner vessel walls there was an erosion of more than 3.5 μm of carbon, i.e., of the same order of magnitude as the measured depths of T in the vessel walls. During the DTE 1 campaign tritium was implanted into the vessel walls with a mean range of about 100–200 nm. Due to the much larger total wall erosion, however, the majority of this T was released again during the clean up and D–D operations phases following the DTE 1 campaign. Another source of T are D/D nuclear reactions. The T produced in these reactions has a starting energy of about 1 MeV. If it is implanted in carbon at normal incidence it has a mean range of about 8.5 μm [25]. The 1 MeV T ions are partly slowed down in the plasma and will finally reach the vessel wall surface with an energy distribution ranging to lower energies and partly at oblique angles of incidence. This results in a broad implantation distribution for the T ranging up to 8.5 μm. During the cleaning discharges in D (and H) relatively large concentrations of D (and H) are implanted in the vessel walls with keV energies up to mean depth of about 0.2 μm [25]. These implanted D and H atoms are detected in our measurements. The T concentration measured at depth up to >2 μm and deeper likely originate from the 1 MeV T implanted up to even larger depth. The total amount of T agrees reasonably with the measured amount of D/D neutrons which is equal to the amount of produced T, if this is uniformly distributed over the first wall. The final depth distributions are modified by the isotope exchange in the surface layers, the erosion of up to 3.5 μm and in addition by some diffusion in the CFC carbon material.

The area of the flat inner wall tiles at JET (i.e. not including the inner bumper limiter tiles) is approximately 7 m^2. At an average T concentration of 2×10^{13} T atoms cm^2, this represents a retention of 1.4×10^{18} T atoms, or 7 μg T.

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