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First results of laser-induced desorption - quadrupole mass spectrometry (LID-QMS) at JET

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Abstract

The paper reports the first demonstration of *in situ* laser-induced desorption — quadrupole mass spectrometry (LID-QMS) application on a large scale fusion device performed in summer 2023. LID-QMS allows direct measurements of the fuel inventory of plasma facing components without retrieving them from the fusion device. The diagnostic desorbs the retained gases by heating a 3 mm diameter spot on the wall using a 1 ms long laser pulse and detects them by QMS. Thus, it can measure the gas content at any wall position accessible to the laser. The successful LID-QMS application in laboratory scale and on medium size fusion devices has now been demonstrated on the larger scale and it is already foreseen as tritium monitor diagnostic in ITER. This *in situ* diagnostic gives direct access to retention physics on a short timescale instead of campaign-integrated measurements and can assess the space-resolved

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efficacy of detritation methods. LID-QMS can be applied on many materials: on Be deposits like in JET, B deposits like in TEXTOR, C based materials or on bulk-W.

Keywords: fuel retention, laser, tritium, deuterium, desorption, JET, beryllium

(Some figures may appear in colour only in the online journal)

1. Introduction

Fusion fuel retention especially retention of tritium at the chamber wall is problematic because of several reasons of which the most important are the following:

- the risk of T release in case of a large leak of the vessel. Thus, in ITER the amount of tritium in the vacuum vessel has been limited to 1 kg to keep the contamination of the surrounding below evacuation limits.
- T losses in the fuel cycle and thus problems to keep up a self-sustained T supply by breeding;
- adverse effects on the properties of the wall material due to hydrogen isotope retention, e.g. embrittlement.

Even after removal of carbon as surface material for plasma facing components (PFCs) from JET (Joint European Torus) [1], the issue of co-deposited fuel retention at the wall is still significant although diminished. The experience from JET after the installation of the ITER-Like Wall (JET-ILW) [2] in 2011 shows that the use of Be as main chamber PFC material and W as divertor PFC material leads to erosion of Be, its transport by scrape-off layer flows mainly towards the central solenoid and deposition of Be:D layers predominantly on the inner upper divertor [3]. Sample tiles have been retrieved from the chamber and their *ex situ* analyses have shown the highest Be and D concentration on the uppermost inner divertor tile (tile 0) and on top of tile 1 [4]. So far a direct measurement of the fuel inventory of PFCs was only possible by *ex situ* methods after retrieving the components from a fusion device. This is a complex and time-consuming process that includes venting the vacuum chamber (in case no air lock is available) and exposure of these components to air, which already influences the results. In order to measure retention directly in the fusion device several laser-based methods have been developed that allow *in situ* application, e.g. laser-induced ablation and laser-induced breakdown spectroscopy which create craters in the wall and analyse the eroded material. Unlike these methods laser-induced desorption (LID) [5–8] only heats the surface and does not remove material in such large extent. However, still typical surface modifications due to heating can occur like roughening of the surface, melting of the co-deposited layer in case of high laser power, minor evaporation at the surface or layer delamination due to thermo-mechanical stresses. Typically a laser spot diameter of few mm is used to be large enough that the spot edge effects due to the lower edge temperatures are negligible, but not too large so that the laser power density is high enough to reach high enough temperatures. At JET we decided to use a spot of 3 mm diameter, which is a

good trade-off. The laser pulse duration is usually in the ms range as this determines the depth of the heating, which should be larger than the fuel retention depth. As the plasma deposited layers contain most of the fuel rather than the bulk material carbon-fibre composite (covered with a 3 μm Mo interlayer and 12 μm W top layer) a 1 ms short pulse duration is sufficient to heat the complete deposit depth of about 10–20 μm in JET. Thicker layers are expected in ITER where the pulse duration thus will have to be increased to probably 3 ms. Previous laboratory applications of LID have shown on Be:D layers that already for the thickness of JET layers melting of the Be layer is necessary, i.e. more than 1550 K, to release the D completely [9]. Since almost all the fuel is retained in the deposited layer, the fuel retention depth is virtually identical to the layer thickness in this case. This also corresponds to the fuel desorption depth as the complete layer is molten by the laser heating and the gas release is complete. The fuel desorbs predominantly as HD, D₂ and in case of JET also as DT and T₂ molecules, which are quantified by residual gas analysers (RGAs). Mainly a quadrupole mass spectrometer (QMS) is used to quantify these rapid partial pressure jumps of these masses ($m/q = 3, 4, 5$ and 6). Thus, the diagnostic method is called LID-QMS. Beside the *ex situ* application in laboratories where it has successfully been applied to carbon-based materials like C layers and bulk W, it has already been applied *in situ* at medium-size fusion devices like TEXTOR on B deposits [10]. Applied as *in situ* diagnostic it

- gives direct access to retention physics on a short timescale instead of campaign-integrated measurements;
- can provide space-resolved fuel retention maps of the wall and
- can assess the space-resolved efficacy of detritation methods.

2. Ex situ results

Before the design, installation and application of *in situ* LID-QMS on JET, the method was tested on a JET divertor tile that was exposed to JET plasmas during 2014–2016 at the uppermost inner divertor position (tile 0). The deposited layer can already be located visually by the colourful appearance due to light interference in the poloidal position above 50 mm (cf figure 1 where the scale of the graph corresponds to the image of the tile). Several cores of 20 mm outer diameter, which yield circular samples of 17 mm inner diameter were cored (cores 1 c–7 c) few cm toroidally from the edge of the tile and analysed by thermal desorption spectroscopy (TDS). The upper halves of core 1, 3 and 5 were desorbed earlier by

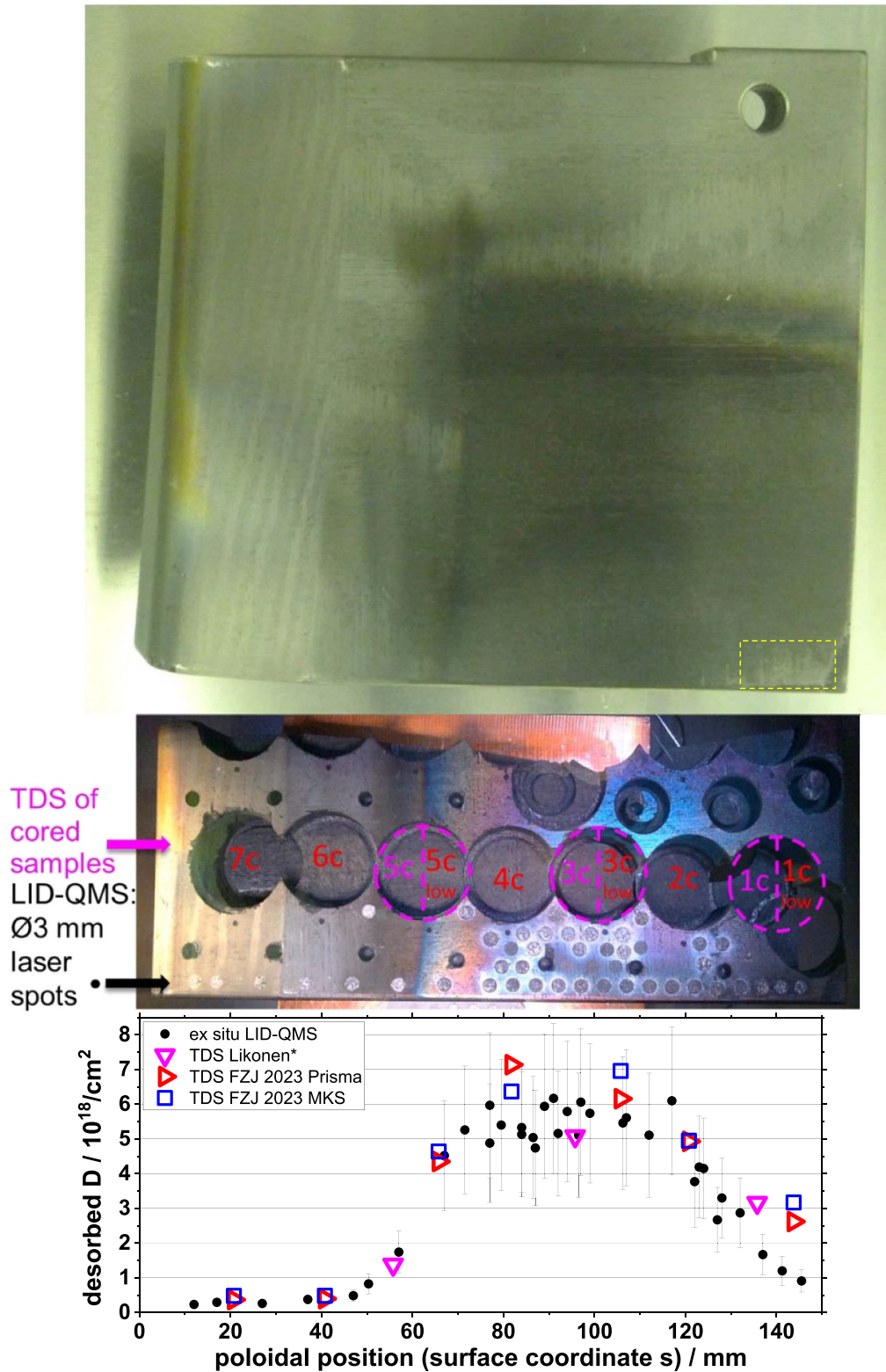


Figure 1. *Ex situ* LID-QMS compared to *ex situ* TDS on a sample of a JET upper inner divertor tile 0 (LH14N) exposed to plasma during 2014–2016. * data from [11].

Likonen *et al* [11] while the lower halves of these samples and the complete cores 2, 4, 6 and 7 were recently analysed in the FREDIS device [12] in FZJ. The desorbed D content is in very good agreement with the *ex situ* LID-QMS performed in various positions between the cores and the edge of the tile.

Most TDS values are very close to the corresponding LID-QMS value at the same poloidal position or positions nearby. In the plateau region between 80 and 110 mm some deviations occur, which are still well within the error bars. The LID-QMS error bar is $\pm 35\%$ according to the Gaussian error propagation.

It is summed up quadratically from two main sources of error. Firstly, the error estimated in quantification of the desorbed gases by QMS ($\pm 33\%$) comprising calibration leak rate uncertainty, calibration fitting errors and QMS sensitivity variations between time of calibration and time of LID-QMS. Secondly, the uncertainty of the desorbed surface area ($\pm 10\%$).

Only at the right hand side, which is closest to the plasma, TDS shows more than LID-QMS. This discrepancy can most probably be explained by a partial removal of the deposited layer in the yellow box shown in figure 1.

After this proof that LID-QMS yields correct results on real deposits from JET the *in situ* application was started in a cooperation of EUROfusion, FZJ and UKAEA in order to demonstrate the feasibility of this method in a large vacuum volume about three orders of magnitude larger than the FREDIS device.

3. *In situ* diagnostic setup of LID-QMS

In the roof lab above the JET torus hall, a laser pulse of ms duration with up to 25.5 kW power is generated by a diode-pumped ytterbium fibre laser (IPG YLS-25000, 1071 nm) and guided via 12 mirrors through a penetration tube in the ceiling and through a vacuum window onto the divertor (cf figure 2). The first mirror (M1), which is just in front of the vacuum window is rotatable in two axes and thus can move the laser spot to any location within the laser accessible area in octant 5 of the torus (red area in figure 2). This area contains the tile 0 and top of tile 1, where the majority of fuel retention is located [4]. On the optics table in the roof lab three cylindrical mirrors (M8–M10) change the circular beam into an elliptic beam such that the projected laser spot on the target tile is circular despite the oblique angle of incidence.

Most of the mirrors are gold-coated Cu mirrors in order to have high reflectivity in the range 600–2200 nm. This not only allows to pass for the IR laser and the guide lasers (at 661 and 685 nm) but also for visible light and IR light in reverse direction for a coaxial camera view of the target and for IR pyrometry inside the laser spot. Those coaxial viewing systems are combined with the laser beam path via beam splitters (BS1, BS2 in figure 2) and hence always move together with the laser beam. A plan-convex field lens with 9561 mm focal length is used to obtain a larger field-of-view for the coaxial camera observation of ca. 2 cm. The optics setup projects an image of the laser fibre end onto the target tile using mirror 12 at the fibre to generate an almost parallel laser beam and the curved mirrors M7 and M4 in conjunction with the field lens to produce the image. Except the three cylindrical mirrors discussed above, all other mirrors in the laser beam path are flat mirrors only for deflection of the laser to the appropriate locations.

The laser part of the diagnostic setup is outside the vacuum, hence there was no need of breaking the vacuum for installation. The total transmission of the laser beam path has been measured in a test setup in FZJ. The total optical transmission of the beam path at the laser wavelength was 53%. The transmission of the JET vacuum window has been measured

in reflection with a spectrometer to be ca. 85%. Further power losses occur due to laser reflection on the surface, which has been measured in a spectrophotometer on the JET tile presented in figure 1 to be ca. 25% at the laser wavelength. Thus, overall only about 1/3 of the initial laser power is used for heating of the tile.

A water cooled beam dump can be used to operate the laser pulses only on the optics table by inserting the movable mirror M15 into the beam path that deflects the laser into the beam dump. A very small fraction (below 1%) of the laser beam is not reflected by the beam splitter 1 and thus passes through it. This fraction of the laser power is absorbed by a passively cooled beam dump.

Beam splitter 1 reflects almost all of the laser light but transmits most of the visible and IR range. The visible range is reflected by beam splitter 2 to the coaxial camera to observe the laser spot and its surrounding. BS2 transmits the IR range for the pyrometry. This light is focussed by the curved mirror 14 into the pyrometer fibre, which guides the light to the pyrometer box. Here the curved mirror creates a parallel light beam, which is then split by two beam splitters and a mirror in the wavelength range of 1.4–2.2 μm into the three point pyrometers according to their individual detection wavelength range.

The detection of the desorbed gas makes use of the incumbent residual gas analysis (RGA) system of JET mainly utilising its QMS devices (figure 3). Most sensitive especially to tritium containing molecules are the QMS located closest to the desorption position. In particular this is QMS5 (=‘MS5’) in octant 6 and QMS1 and QMS2 (=‘MS1’, ‘MS2’) located in octant 1. The RGAs in the sub-divertor below octant 8 are further away from the torus and thus have especially low sensitivity to the T containing molecules DT and T₂. The reason is probably that the ducts and pipes on the way to these remote RGAs are not saturated with T after years of deuterium plasma operation and only few weeks of DT plasma operation. Thus, those molecules are adsorbed by the walls, while the D₂ partial pressure jump during the laser pulse is high as the walls are saturated with deuterium.

4. *In situ* results

One of the first LID-QMS results on JET demonstrating deuterium release are shown in figure 5. To confirm the laser parameters for full fuel desorption known from *ex situ* measurements, firstly a toroidal and poloidal scan of the tile was performed with these *ex situ* parameters. Hence, an area of constant D retention was identified (green area in figure 4). Within this area laser parameter variations of laser pulse duration (0.5–10 ms) and laser power (cf figure 5) were performed. A laser pulse of 1 ms and 19 kW laser output power (dark blue line in figure 5) was found to be sufficient for full fuel desorption from the laser spot as the partial pressure jump of the masses did not increase at higher laser power of 21 kW (cyan line in figure 5) although the total pressure signal still increased due to the release of other gases. The laser power was varied in 4 steps as shown in the top graph. Accordingly,

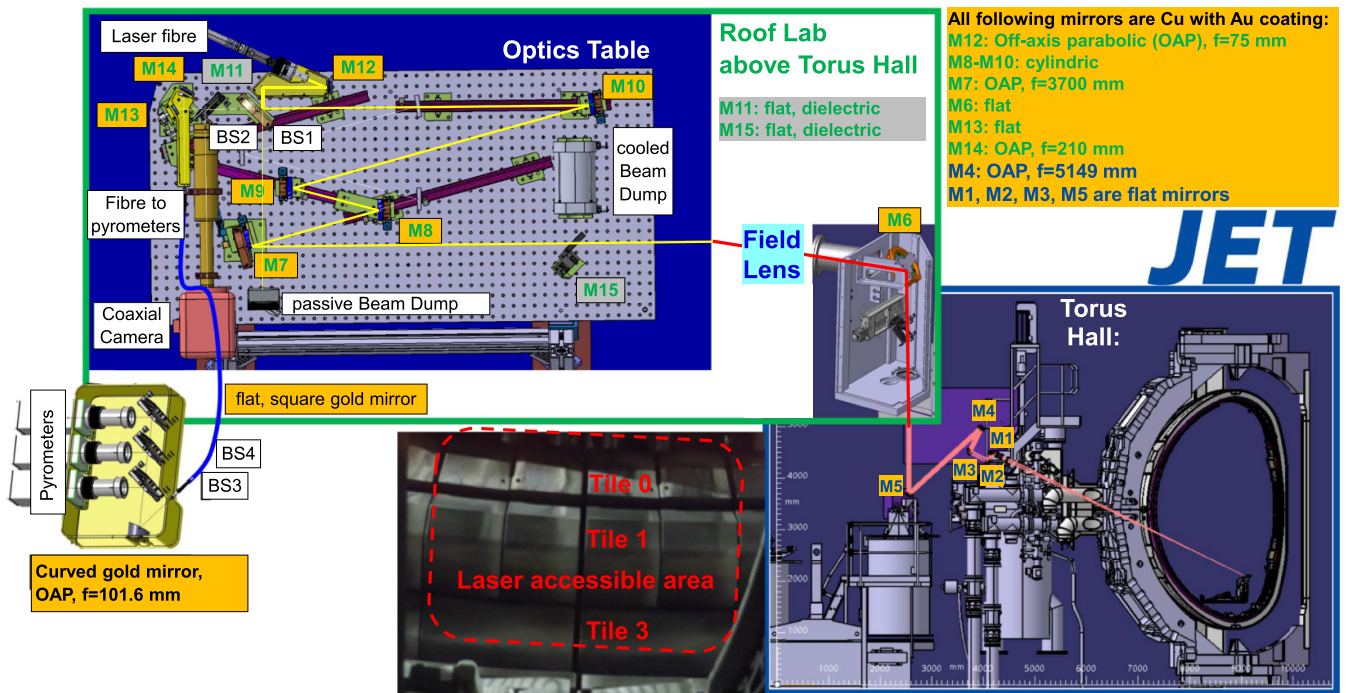


Figure 2. Laser part of the LID-QMS setup at JET. The detection part of the setup utilises the incumbent RGA system. The laser injection part shown here consists of a mirror-based beam path that mainly uses gold-coated copper mirrors (yellow labels), two dielectric mirrors (grey labels), beam splitters (BS) and a field lens. It guides the laser beam that emerges from the laser fibre from the roof lab above the torus hall through the bioshield penetration (between M6 and M5) into the torus hall and onto the laser accessible area on the inner divertor PFCs (red marked area). In reverse direction the beam path is used to observe the laser spot with a coaxial camera and measure the surface temperature inside the laser spot with three point pyrometers. More details in the text.

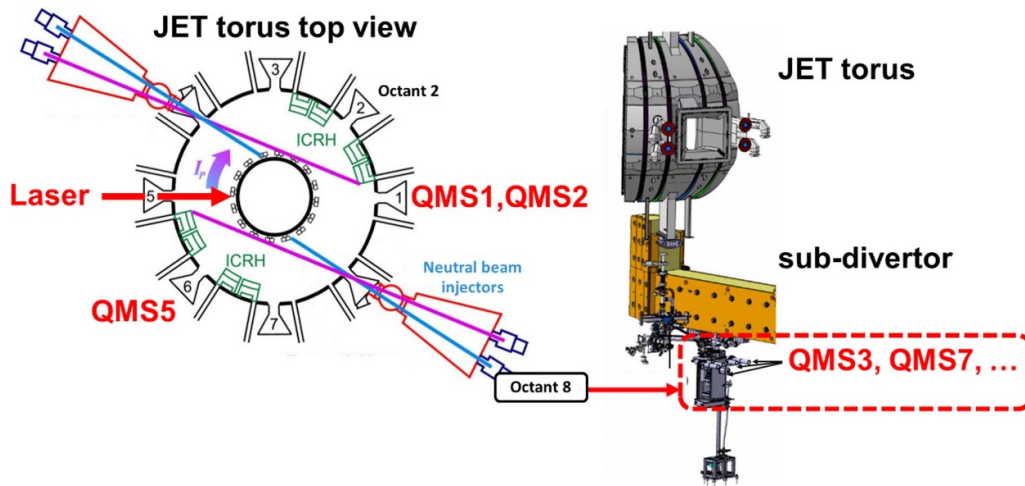


Figure 3. The incumbent residual gas analysis (RGA) system of JET with the positions of the QMS used for LID-QMS.

the surface temperature at the end of the 1 ms long laser pulse increased as shown in the graph below. The temperatures shown here are not the real surface temperatures, which are much higher above the Be melting point of 1550 K. For simplicity, the graph shows only the signal of one of the three pyrometers in the temperature scale shown on the pyrometer device, for which the temperature calibration using a black body radiator oven has to be applied and then combined with the other two pyrometer signals to obtain the real surface temperature. The next graph shows the gas release from the laser spot using a total pressure gauge in the main torus chamber.

The release of deuterium seen as jump in the partial pressure of mass 3 (HD) and 4 (D₂) was clearly detected for each laser spot on the target tile even in the areas of low retention at the top of the tile. Especially the mass 4 signal of QMS3 shown in the bottom left graph clearly shows the increasing partial pressure jump with increasing laser power and the same jump for the two highest laser powers. Thus, 19 kW were in principle sufficient for full fuel desorption. However, this value might just be at the edge of the full desorption range. Therefore, in the DT campaign that followed after this study, we used the 21 kW laser power in order to be a bit above this edge and

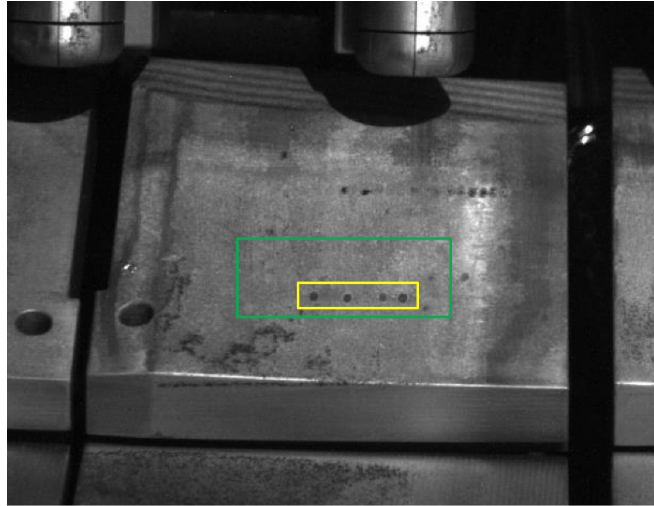


Figure 4. Target tile 0 (RH14W); green: area with constant D retention; yellow: the 4 laser spots discussed below.

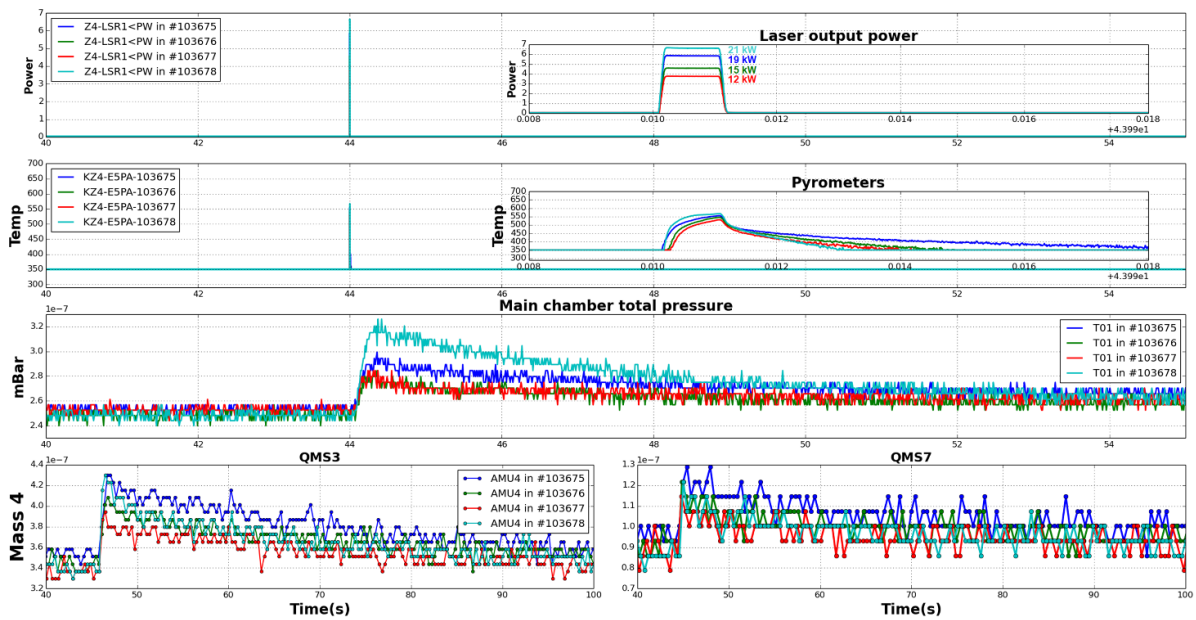


Figure 5. LID-QMS measurements with a single laser spot always on a new position containing identical D content. From top to bottom: varied laser power with a zoom window at 44 s (the time of the laser pulse), pyrometer signals with the same zoom window, main torus total pressure, mass 4 signal of different mass spectrometers located in the sub-divertor showing the D_2 release from the laser spot, which increases with laser power but saturates for the two highest laser powers of 19 and 21 kW.

compensate for any optics transmission degradation or target surface effects like higher reflectivity at some positions and still be in the full desorption range.

Furthermore, to increase the amount of desorbed gas, a quick raster mode was successfully applied with several laser spots fired next to each other or overlapping in one or several rows within few seconds. Up to 128 laser spots were fired within 2.5 s which allows to raise the signal above the background pressure e.g. if LID-QMS is required shortly after a plasma pulse. The limit are 512 laser spots fired in 10.2 s with 3 mm step size. With this raster mode we were able to detect the very low tritium retention during the 2023 DT campaign after each week of plasma operation. In particular six rectangular areas of 40.5 mm \times 33 mm were cleaned with overlapping

laser spots with a step of 1.5 mm before the DT campaign to remove any residual T and D from these areas (figure 6). After each week of DT operation the next rectangle was measured and in addition in each week rectangle 1 was measured and cleaned again after the measurement.

It was observed that the most constant LID-QMS signal and calibration signal during puffing of a known amount of gas was obtained with low pumping, i.e. cryo pump at liquid N_2 temperature and only 1 of 4 turbo molecular pumps pumping (figure 7). This increases the pump-out time from few seconds (with cryo pump at liquid He temperature and all 4 turbo molecular pumps) to minutes, which gives enough time to measure the QMS spectra. Their measurement repetition time depends on the device and scan setting, but the minimum

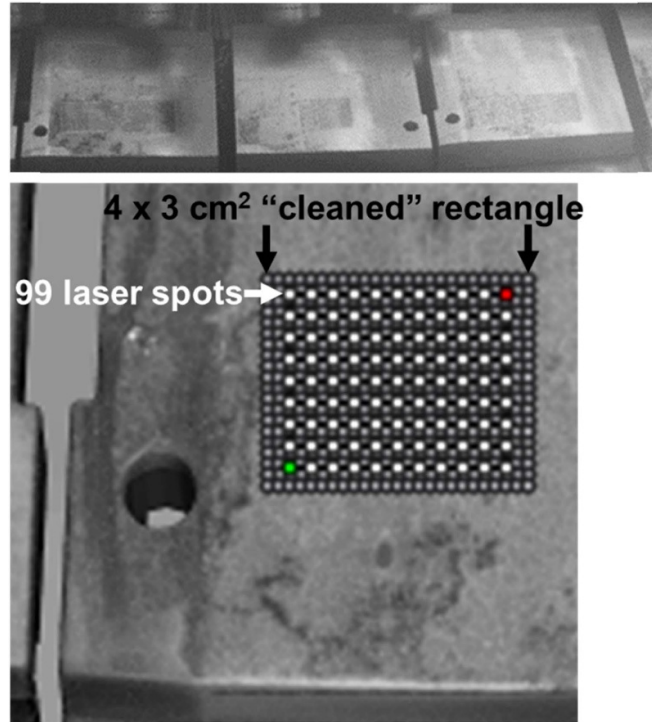


Figure 6. Tiles 0 of JET with ‘cleaned’ rectangles desorbed by the laser with overlapping laser spots (step 1.5 mm, grey spots) and the 99 laser spots (with step 3 mm) for the LID-QMS measurement a week later inside the cleaned area.

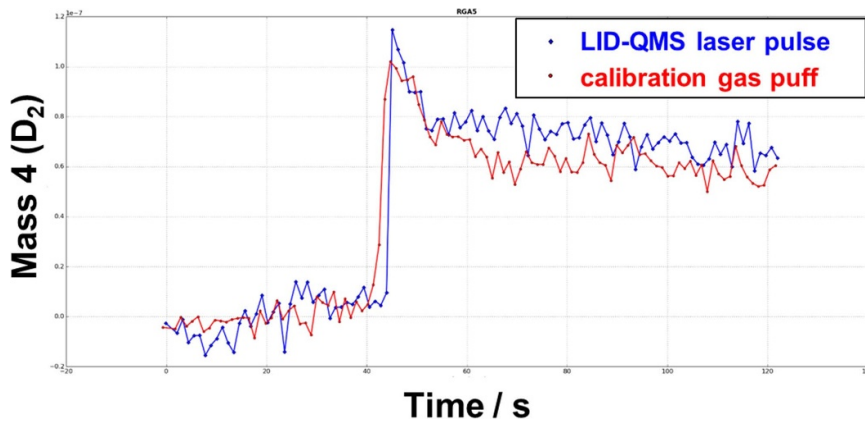


Figure 7. QMS5 signal during a calibration gas puff with D_2 and a LID-QMS measurement pulse for comparison. Both signals after subtraction of the initial value before gas injection.

required scan is a mass scan of the first 6 masses which takes about 5 s for the QMS5 close to the laser spots and thus can resolve the partial pressure jumps even if the measurement raster takes 2 s. This was not possible in the strong pumping conditions as the pump-out time was in the same range as the gas release time duration.

After one week of DT plasma operation the D and T retention could clearly be measured (figure 8) as partial pressure jumps on masses 3 (HD), 4 (D_2) and 5 (DT). These masses are unambiguous as the potential TH molecule on mass 4 and HD_2 on mass 5 only have negligible contribution as there is little H (mass 1) and H_2 (mass 2) present in the residual gas. By accumulation of the weekly signal on the same rectangle, the build-up of fuel retention during the DT campaign in September/October was monitored.

5. Outlook

After the DT campaign a T cleaning campaign was performed at JET with different methods to reduce the T amount:

- Backing of the torus to 320 °C,
- Ion cyclotron wall cleaning plasmas,
- Raised inner strike point plasmas (RISP plasmas)
- Glow discharge plasmas.

After each cleaning method a poloidal LID-QMS scan was performed on a toroidally shifted position only few mm next to the previous scan on tile 0 and 1. In this way the decrease of tritium retention after the campaign has been monitored by the LID-QMS diagnostic as a test run for ITER. The data will be

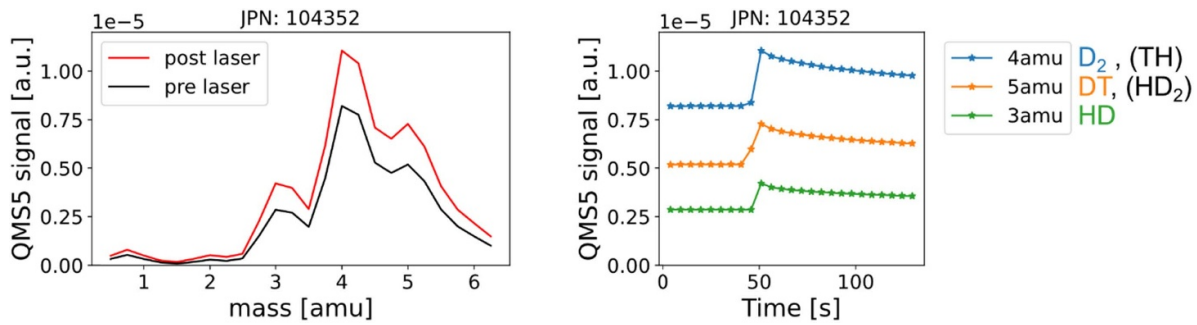


Figure 8. LID-QMS spectra before and after the laser pulses and time traces of the main masses desorbed with 99 laser spots in an area with accumulated fuel retention of one week of JET operation.

evaluated and published in the future. But already in the raw data a clear decrease of the tritium signal during LID-QMS has been observed. The decrease was especially pronounced after the RISP plasmas.

In the last two days of JET operation in 2023, after the last plasmas operation, LID-QMS measured the fuel retention in many positions of the accessible PFCs. It is planned to retrieve these tiles from the torus and measure the retention *ex situ* close to these positions in order to compare the *in situ* and *ex situ* results.

6. Conclusion










The LID-QMS method measures the fuel retention by thermally induced desorption of D and T containing gases from the PFC by heating a 3 mm spot with a 1 ms IR laser pulse and their detection by QMS. In the past it was successfully applied to carbon based materials and bulk-W and in recent years also qualified on laboratory-originated homogeneous Be layers and Be deposits from the JET-ILW in *ex situ* setups. Comparisons with other *ex situ* methods like TDS have proven accurate quantification of the gases on JET tiles and other samples by LID-QMS. Thus, the method was implemented as *in situ* fuel retention diagnostic at JET using a mirror-based beam path through the bioshield with a movable targeting mirror and coaxial observation and pyrometry of the laser spot in reversed optical direction. Laser parameter scans on areas with constant D retention on the upper inner divertor of JET have confirmed that the *ex situ* laser parameters are sufficient to desorb the fuel completely also in a large fusion device. In this setup LID-QMS measured the deuterium and tritium retention during the 2023 DT campaign at JET and monitored the fuel increase on a weekly basis by re-visiting rectangular areas that have been depleted from fuel by overlapping laser spots before the DT campaign. The successful fuel detection after short plasma exposures by quick scans of larger areas with multiple laser pulses and the detection of long-term retention using a single laser spot even in remote areas have qualified LID-QMS on large-scale fusion devices. Thus, the method is already being designed as tritium monitor diagnostic for ITER.

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