PAPER

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Gas analyses of the first complete JET cryopump regeneration with ITER-like wall

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Abstract
Analytical results of a complete JET cryopump regeneration, including the nitrogen panel, following the first ITER-like wall campaign are presented along with the *in situ* analyses of residual gas. H/D mixtures and impurities such as nitrogen and neon were injected during plasma operation in the vessel to study radiation cooling in the scrape-off-layer and divertor region. The global gas inventory over the campaign is incomplete, suggesting residual volatile impurities are remaining on the cryogenic panel. This paper presents results on (i) residual deuterium on the panel which is very loosely related to the campaign, (ii) impurities like nitrogen which stick on the panel, and (iii) the ammonia production which can be observed by mass spectrometry.

Keywords: JET cryogenic pump, JET exhaust, ITER-like wall, gas chromatograph, RGA, regeneration

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

1. Introduction

During the 2010 shutdown all JET plasma-facing components (PFCs) made of carbon-fibre composite (CFC) were replaced with bulk beryllium or Be-coated tiles and tungsten (W) tiles in the divertor to mimic the configuration envisaged for ITER, ITER-like wall (ILW). Following the installation an 11-month experimental campaign was executed to investigate key issues of plasma–wall interaction such as plasma compatibility of the W divertor and fuel retention in PFCs. In order to minimize the W sputtering and allow long operation on the inertially cooled PFCs, impurity seeding is used to mitigate the divertor heat load by radiation cooling. The radiation cooling can be induced by a combination of high recycling and seeding of impurities such as noble gases or nitrogen which has a radiation potential optimized for the temperatures in the divertor. In JET dedicated experiments have been executed to study the impact of nitrogen on the PFCs and in particular on the production of chemically reactive species such as ammonia (NQ_3, Q = H and D) in the gas exhaust [1, 2].

A critical issue with the usage of nitrogen in a hydrogen (H, D and T) environment is the formation of nitrides and ammonia or other derivatives as they are caustic and hazardous. This can cause damage to systems and poisoning of the adsorption material and catalyst material. Condensed ammonia for example is corrosive, and in anhydrous state it can cause stress-corrosion cracking of stressed carbon steels or high-strength, low-alloy steels. For direct contact of ammonia, the best material considered is

[8] See the appendix of Romanelli F *et al* 2012 *Proc. 24th IAEA Fusion Energy Conf. (San Diego, CA, 2012)*, OV/1-3.

316L type stainless steel which is assumed to have the lowest corrosion risk and is used in ammonia production plants [3]. Nitrogen and ammonia have a poisoning effect on tritium uranium-getter-beds and will reduce the adsorption capacity; for this reason only hydrogen isotopes (H, D and T) and noble gases are allowed to pass over a uranium-getter-bed at the tritium plant, the Active Gas Handling System (AGHS) [4] at JET. The effects of ammonia on palladium-catalyst used for detritiation purposes in the AGHS are under investigation at the moment. Also the effect, for example, on bellows used in pumps and valves, pressure gauge membranes, valve seat materials, diagnostic windows and other materials, other than 316L type stainless steel, has to be assessed and taken into account.

2. JET pumped divertor cryopump regeneration

The JET tokamak is equipped with a pumped divertor with integrated cryogenic pump. Hydrogen isotopes and impurities injected or generated during plasma operations are primarily pumped out and trapped by the cryogenic pump. The pump consists of two panels cooled by (i) liquid helium (LHe) and (ii) liquid nitrogen (LN_2). The LHe panel which trapped mostly hydrogen isotopes was frequently regenerated (78 times) at 77 K for either dedicated gas balance experiments [5] or when the D_2 inventory limit of 30 000 Pa m^3 was reached during the campaign, whereas the LN_2 panel only was regenerated once at the end of the experimental campaign, in order not to compromise the good vessel conditions by releasing pumped water. Two tons of W and two tons of Be new plasma-facing material were installed and no LN_2 regeneration after the initial backing cycle and glow discharge operation has been executed. The first and so far only complete regeneration under JET-ILW conditions representing 11 months of operation can be compared with a complete regeneration at the end of the JET-C operation, but collecting only 1 month of JET operation with carbon-based PFCs. A first comparison between the exhaust from the two wall configurations can be made.

2.1. Liquid helium panel regeneration

At the end of the last day of plasma operation and following an out-gassing time of 15 h for the PFCs the LHe supply to the JET cryopump was isolated. This completed an experimental period of 151 identical H-plasmas including two light cryogenic regenerations of the LHe panel [5]. The vacuum valves, isolating the neutral beam injection (NBI) cryopump to the torus vessel, were closed and the NBI cryopump had been already warmed up to 77 K and the released gas had been discharged.

The LHe panel, which had had an accumulation time of 1 day of operation (ten discharges), gradually warmed up to 77 K and the released gas passed through the torus vessel and was mechanically turbo pumped for 4 h via the horizontal pumping duct over a pipe bridge to the AGHS. The AGHS from its side employed in addition a mechanical pump to collect the released gas in a reservoir and performed pressure–volume–temperature–concentration analyses. In total 2210 Pa m^3 normalized to 293.15 K was collected and analysed with two gas chromatographs (GC) and a residual gas analyser (RGA). Results are in vol%: 0.2% He, 0.3% H_2, 2.6% HD, 95.7% D_2, 0.9% Ar, 0.08% N_2, 0.14% CO_2, 0.09% methane, <0.03% C2–C6. A small constant air leak on the vacuum vessel of $< 8 \times 10^{-5}$ Pa m^3 s^{-1} would accumulate in 1 day to a concentration of 0.24% N_2, 0.003% Ar and 0.0001% CO_2.

2.2. Liquid nitrogen panel regeneration

After the complete regeneration of the LHe panel and pumping of the remaining gas from the transfer lines, the liquid nitrogen coolant was stopped. ND radicals during N_2 seeding experiments were observed by divertor spectrometer system as described in [6] which could indicate ammonia formation via plasma/surface reactions [1]. No such ammonia in plasma formation but CN production by surface interaction was observed in JET-C experiments due to surface interaction with the graphite CFC [6]. Ammonia would be trapped on the LN_2 panel together with water which originated mostly through D_2 glow discharge operation (100 h) but also from the initial pump down after the ILW installation and a small constant air leak. In order to avoid mixing up NQ_3 and water and possibly creating corrosive ammonium hydroxide which would immediately be adsorbed onto surfaces in the stainless steel pipework and reservoirs, the released gas was collected in separate batches. NQ_3 which has a lower boiling point than water was expected to be released first from the LN_2 panel. The first batch of released gas at a LN_2 panel-temperature from 77 to 200 K was collected at AGHS on a cold trap cooled to 77 K and further connected to a 25 l reservoir. The cold trap was than warmed up and the gas analysed. The second batch from a LN_2 panel-temperature from 200 K until ambient temperature, containing long-chain hydrocarbons (HC), was collected via a mechanical roots pump leading to the same 25 l reservoir as the first gas collection. The reservoir contained both the non-condensate components of the first batch and the total of the second collection. Finally this reservoir was sampled and analysed with both the GC and RGA technique. Both gas analyses, batch 1 and batch 2, were used to calculate the total gas composition for the LN_2 panel. The LN_2 panel reached 200 K after 2 h but took another 16 h to flatten out at around 322 K. The released gas was pumped for 33 h and a total of 830 Pa m^3 at 293.15 K was collected.

3. Gas chromatograph and RGA analyses

3.1. Methane and higher hydrocarbons

Hydrogen isotope species, molecular nitrogen, air components, carbon oxides, methane and long-chain HC were analysed by standard GC technology. The sample showed a reduction and different profile of long-chain HC as compared with the JET-C (carbon-wall) data (1 month-operation) [7]. A fast decay of C in the residual plasma and of methane during the first year of ILW plasma operation was observed, which indicates that HC mostly originated from the initial operation phase [8] and levelled at about 0.1% which is the detection limit for methane; see figure 1(A). This indicated that a large fraction of the initially observed methane resulted from the installation and plasma clean-up of the ILW which included

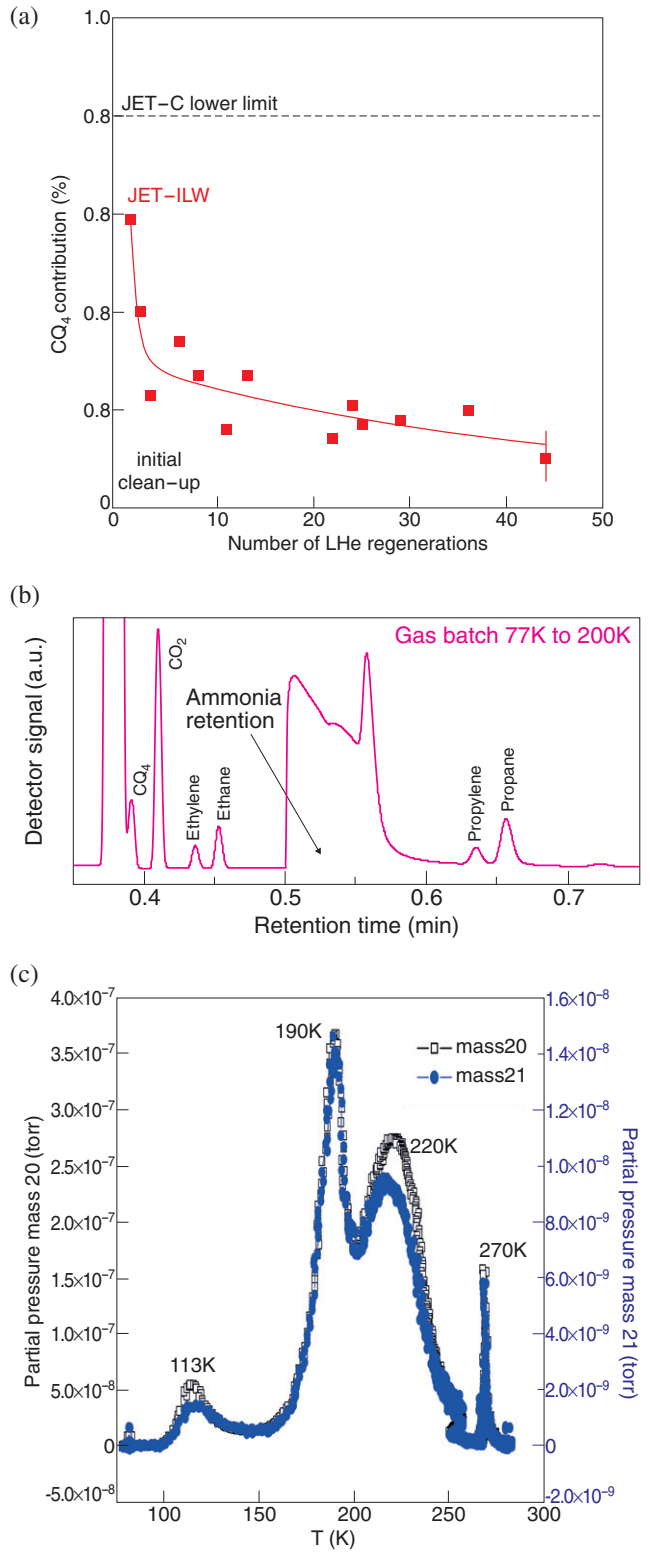

Figure 1. (A). Methane decrease with ILW. (B). GC chromatogram of first gas batch. (C). RGA1, $m/z = 20/21$ amu.

new material out-gassing. The JET-C data were recorded long after a major installation of new wall components with a well-conditioned carbon wall (>10 years). Overall the HC concentration is much lower in comparison with the JET-C wall. But the HC distribution showed a different pattern to the carbon wall, e.g. C2–C4 is 16 times less whereas the C8–C10 group show an increase of four times. The longest chains might be related to cleaning substrates such as oil. The quantified components of the LN_2 panel in vol% are: 30% Q_2, 13% N_2, 2% CQ_4 and 11% long-chain HC. The missing remaining part concludes that the sample must contain some other components which could not be quantified or detected by GC or RGA.

3.2. Ammonia and water analyses

The use of the existing AGHS analytical systems, GC and internal RGA, for the analysis of ammonia and water has been assessed. GC is not the preferred analytical technique to detect ammonia and water due to their high polarity and corrosive attribute in the case of NQ_3. Only qualitative analyses can be performed, but an attempt has been made to calibrate the GC system with a 500 ppm ammonia standard, which is the highest recommended concentration allowed to enter the GC without damaging the system by etching. However, no definite signal for NQ_3 could be obtained on the GC separation columns, PoraPLOT Q and CP-Sil 5CB, despite literature giving elution factors for ammonia for these columns. Also the internal RGA connected to the same sample manifold as the GCs has shown no signal at mass-to-charge-ratio $(m/z) = 17$ atomic mass unit (amu). One explanation of the above observation could be that the 500 ppm NH_3 concentration is too low and the ammonia gets entirely adsorbed onto the surfaces. Ammonia is prone to adsorb onto surfaces especially in combination with water. The ammonia standard gas cylinder was connected to the analytical system via a ~5 m long and $\frac{1}{4}$ inch in diameter stainless steel pipework. The analysis of the first gas batch collected from the LN_2 panel has shown a multiple, overlaid asymmetric peak where the literature gives ammonia retention; see figure 1(B). Water was detected by GC but could not been quantified. Another piece of evidence for the presence of ammonia and water is that the humidity sensor installed in the sampling line gave positive indication in both gas collections for a polar molecule. Therefore ammonia and water can contribute to the unquantifiable components of the released gas of the LN_2 panel.

3.3. RGA analyses

Several quadrupole mass spectrometers for RGAs installed at the JET vacuum vessel were used to measure the partial pressures of gases released from the LN_2 panel. One analyser, RGA1 (HIDEN) [9], is installed in the sub-divertor region, in a vertical duct (Octant 8) close to the divertor cryopump—closest to the released gas from the panels. Two further analysers, RGA2 (Pfeiffer) and RGA3 (HIDEN), are positioned in a bypass section in the main horizontal pump duct of the JET vessel in front of the torus exhaust turbo pump leading to the AGHS. A fourth analyser, internal RGA or RGA4 was employed in the AGHS installed in the same sample manifold leading to the GC systems. All four RGAs were operated in a range of $m/z = 1$–100 amu and have shown slightly different signals which is plausible considering the different instrument type and installation position. Additionally adsorption and re-adsorption from surfaces (torus vessel ~180 m^3 and pipework ~11 m^3) during the pumping process transferring the gas to AGHS might

have led to a slight variation of the RGA signals. The most dominant signal was observed in the 16–20 amu mass range on the RGA1 which is the closest to the LN_2 panel and might indicate the presence of deuterated water and deuterated ammonia. Methane can be neglected due to low concentration (GC$<2\%$). The signals of all employed RGAs (RGA1–4) were widely populated up to $m/z = 100$ amu which indicates the presence of long-chain HC and was confirmed by the GC analyses. For evaluation cracking pattern and cryogenic pump temperature has to be taken into account. A low signal on $m/z = 21$ amu was observed on RGA1–3 and RGA4 (first gas collection), the strongest candidate being $^{15}ND_3$ following a $^{15}N_2$ seeding trace experiment, where pure $^{15}N_2$ was injected into the divertor at the beginning of the 2-week operation in identical H-mode plasmas before the cryopump regeneration. No other $^{15}N_2$ has been injected in the JET-ILW. A comparison between the partial pressures of $m/z = 21$ and 20 is considered to be an effective way to distinguish ammonia ($^{15}ND_3$) from the other species populating $m/z = 20$. Figure 1(C) shows two major peaks, one peak at 190 K, one peak at 220 K and one small peak at 270 K. This most likely indicates the presence of ammonia (first peak), overlaid by a second ammonia peak. Water sublimates at low pressure (below 1.33 Pa) at around 200 K, so peak 2 and peak 3 could be trapped ammonia in water. Positive ammonia detection via RGA was reported from ASDEX Upgrade during N_2 seeding plasma operation [10].

RGA1–4 have shown high signals on $m/z = 2$–4 amu (Q_2) and $m/z = 28$ amu (N_2, CO and C_2), confirmed by GC analyses. Q_2 and N_2 contribute almost half to the quantified amount of the collected LN_2 panel sample. This indicates that Q_2 and N_2 which are released during frequent LHe panel regenerations might get re-trapped on the LN_2 surfaces, possibly via cryosorption on frost formation. Therefore the Q_2 and N_2 content on the LN_2 panel has to be included in future gas balance calculations. The total amount of retained D_2 on the LN_2 panel is 200 Pa m^3 or 0.05% in comparison to the total amount of D_2 pumped over the campaign and light regeneration by the LHe panel (100 h of D_2 glow discharge not included). The panel released 110 Pa m^3 N_2 or 5.1% of the total injected nitrogen during the campaign. In total 130 Pa m^3 of $^{15}N_2$ or 6.1% of total nitrogen ($^{14}N_2$ +$^{15}N_2$) was injected.

4. Summary

Gas analyses of the first complete JET cryopump regeneration with ILW were performed. The released gas of the JET cryopump was collected and analysed by the GC and RGA techniques. A reduction and different profile of long-chain HC was detected. The methane profile during the first year of ILW operation indicates that HC mostly originated from the initial operation phase and installation of the ILW. Water was detected by GC which originated mostly through D_2 glow discharge operation (100 h) but also from the initial pump down after the ILW installation and a small constant air leak ($< 8 \times 10^{-5}$ Pa m^3 s^{-1}). The GC revealed an overlaid asymmetric peak, where literature gives ammonia retention. All employed RGAs have seen a low signal on $m/z = 21$ amu which indicates the presence of $^{15}ND_3$. The comparison between the partial pressures of $m/z = 21$ and 20 is an effective way to distinguish ammonia ($^{15}ND_3$) from the other species populating $m/z = 20$.

The LN_2 panel sample contained 0.05% of the total injected D_2 during the ILW campaign. The panel released 110 Pa m^3 N_2 or 5.1% of the total injected nitrogen during the campaign. A clear identification of ammonia has to be confirmed by ammonia calibration such as (i) ammonia calibration of GC and RGA at AGHS with higher NH_3 concentration, (ii) ammonia calibration of JET vessel RGAs, (iii) improved set-up and calibration of pressure gauges close to the JET cryopump and (iv) slower warm-up of the cryopump to take advantage of the different boiling points

$$T(\text{methane}) < T(\text{ammonia}) < T(\text{water}).$$

An upgrade/calibration of the present JET analytical system is foreseen for the next experimental campaign.

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