

Outgassing measurements for the ITER EC H&CD Upper Launcher

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In most of applications involving both vacuum and high temperatures, outgassing of structural materials is a critical issue. For instance, this is the case of fusion test devices, where the gas released from the vessel walls contaminates the plasma. Four upper ports in the ITER vacuum vessel are reserved for Electron Cyclotron Heating and Current Drive (EC H&CD) Upper Launchers (UL), which have to provide plasma MHD stabilization by localized deposition of high power microwave beams. The structural material foreseen for the UL is the 316L(N)-IG stainless steel. It has to withstand temperatures in the range 120-150°C during normal operation and 240°C during the baking process. One of the preferred manufacturing routes for the UL is the Hot Isostatic Pressing (HIP) which is a very sophisticated method to manufacture structural components of complex geometry with good mechanical properties. The materials for use in the ITER vacuum systems have to comply with the outgassing limits given in the ITER vacuum handbook, but no outgassing data for HIPed stainless steel are available in literature, thus they must be obtained by experimental measurements. In this paper, measurements of partial outgassing rates are shown and discussed for stainless steel prototype samples AISI 316LN (on which the 316L(N)-IG is based) and AISI 317LMN, obtained by powder and solid HIPing method. A variant of the gas throughput method in vacuum systems was used for the measurements which were carried out over periods larger than 8 hours and at different temperatures.

Keywords: ECRH; Upper Launcher; Outgassing measurements; HIPing method; Stainless steel samples.

1. Introduction

The EC H&CD UL has to counteract the neoclassical tearing modes and the sawteeth instabilities of the plasma. During normal ITER conditions the vacuum inside the UL is in the order of $10^{-3} - 10^{-2}$ Pa. The lower the pressure and the higher the temperature, the more important outgassing of the structural material becomes. Since the type of material and its history have a big influence on the outgassing rates, it is not practical to make estimations on the basis of analytical calculations. Experimental tests are usually done on material samples under controlled conditions.

The outgassing rates of materials used in the ITER vacuum systems must be consistent with the limits given in the ITER vacuum handbook [1]. The structural material for the UL and the other in-vessel components is the 316L(N)-IG stainless steel. Manufacturing routes for the UL are under development at KIT and the most elaborated one is the HIPing method [2]. Outgassing data for HIPed stainless steels are not yet available in literature, so an experimental setup has been developed for investigating the partial outgassing rates of HIPed samples and preliminary results have been obtained.

2. Measurement technique

Vacuum measurements of gas release can be either static or dynamic. The dynamic measurement is more suitable for low outgassing rates, thus a variant of such method was used in this work [3]. Consider a vacuum system formed by a vessel which is connected to a

pumping station through a known conductance. Suppose this system has reached equilibrium and no leaks exist. The mass flow rate of gas through the system coincides with the total outgassing rate Q of the system and so the latter is expressed in $\text{Pa}\cdot\text{m}^3\cdot\text{s}^{-1}$ as

$$Q = C(P_1 - P_2) \quad (1)$$

where C in $\text{m}^3\cdot\text{s}^{-1}$ is the conductance, P_1 and P_2 in Pa are the inlet and outlet total pressures of C . Considering molecular flow conditions and S_p the speed of the pumping station in $\text{m}^3\cdot\text{s}^{-1}$, it can be easily demonstrated using the fundamental equation of the vacuum systems that, if $C \ll S_p$, P_2 is very much less than P_1 and so can be ignored [4]. The total outgassing rate of the system therefore becomes $Q = CP$ with $P = P_1$ the total pressure inside the vacuum vessel.

The experimental setup and its schematic representation are shown in fig. 1. A quartz tube of internal diameter $D \sim 5 \times 10^{-2}$ m and length $L \sim 8 \times 10^{-1}$ m is used as vacuum chamber. It is installed in an oven associated to a proportional-integrative-derivative (PID) controller which allows setting the desired temperature. The left side of the chamber is connected to a pumping station through a tube of internal diameter $d \sim 1.6 \times 10^{-2}$ m and length $l \sim 3.6 \times 10^{-1}$ m. Vacuum is obtained by a membrane pump (not shown here) and a turbomolecular pump (HY.CONE 200) connected in series. Through an equal 4-way cross the chamber right side is attached to a Pirani-cold cathode combination gauge, a quadrupole mass spectrometer,

while the last aperture is used for inserting the samples into the vacuum vessel.

During the measurements the pressure is in the order of 10^{-4} Pa and thus molecular flow conditions prevail. Since a long ($l/d \approx 23$) cylindrical tube is used to connect the vacuum chamber to the pumps, the molecular conductance of the tube is given by

$$C = \frac{1}{6} \sqrt{\frac{2\pi RT}{M}} \frac{d^3}{l} \quad (2)$$

where R is the universal gas constant, T is the absolute temperature and M is the molar mass of the gas in SI units [5]. For N_2 , the formula (2) at 20°C gives $C = 1.40 \times 10^{-3} \text{ m}^3 \cdot \text{s}^{-1}$ while S_p is $2 \times 10^{-1} \text{ m}^3 \cdot \text{s}^{-1}$. With $S_p/C \approx 143$ the hypothesis $C \ll S_p$ is therefore well fulfilled.

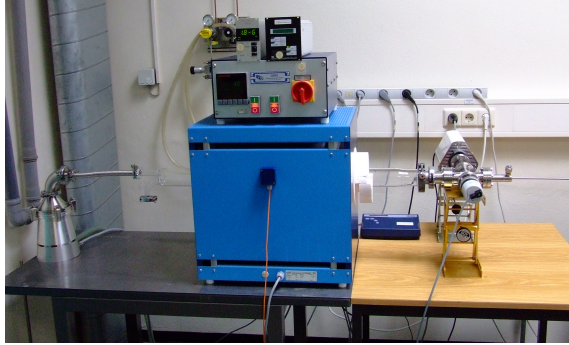
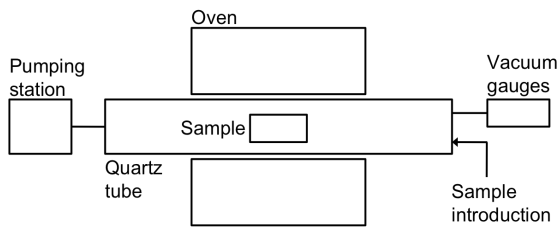


Fig. 1. Experimental setup and its schematic representation.

In order to calculate the sample outgassing rate, blank and sample runs were done in the same experimental conditions for the pressure measurement. In literature outgassing data are reported per unit of area, so the specific total outgassing rate q of the sample is expressed in $\text{Pa} \cdot \text{m} \cdot \text{s}^{-1}$ as

$$q = \frac{C(P_{sR} - P_{bR})}{A} \quad (3)$$

where P_{sR} and P_{bR} are the total pressures during the sample and blank runs and A is the sample surface area in m^2 . Specific partial (i.e. species dependent) outgassing rates of the samples were calculated using

Table 1: Summary of the samples investigated in the present work.

	Material	Fabrication technique	Surface finish	f_s
Sample A	AISI 316LN	Powder HIPing	Polished	1.112
Sample B	AISI 316LN	Powder HIPing	Rills	1.655
Sample C	AISI 317LMN	Rolling	Polished	1.207

(3), where the conductance for the particular gas species is given by (2), and the partial pressures were determined using the mass spectrometer and the combination gauge.

3. Samples

A total of six stainless steel samples were investigated considering the powder and solid HIPing methods. The samples have cylindrical shape with diameter 10^{-2} m and length 3×10^{-2} m which define their geometric surface area. Table 1 reports the features of the specimens. Since the 316L(N)-IG stainless steel is not a standard steel in the international markets, it is very expensive and so it was not used in the present work. However, it is based on the AISI 316LN stainless steel specifications with a narrower composition range of the main alloying elements and a controlled addition of nitrogen. The AISI 317LMN steel with respect to AISI 316LN has essentially a higher molybdenum content.

Many factors may affect the outgassing rates, for example the surface finish of the material and the cleaning method of the surface [3]. The first factor was investigated by considering samples with two different surface finishes shown in fig. 2. Roughness measurements were performed to characterize the surface of the samples and for each one the surface factor $f_s = A_r/A_g$, with A_r and A_g respectively the real and geometric area, was determined. The real area of the samples was used in the formula (3) for the outgassing calculations. On one hand in the appendix 17 of the ITER vacuum handbook [3] no specifications are given for the area of the desorbing surface. On the other hand the American Vacuum Society [6] recommends using the geometric surface area. In the present work the real area of the samples was used, as it allows observing the effect of the surface finish. No special cleaning method was applied to the surface of the investigated specimens.

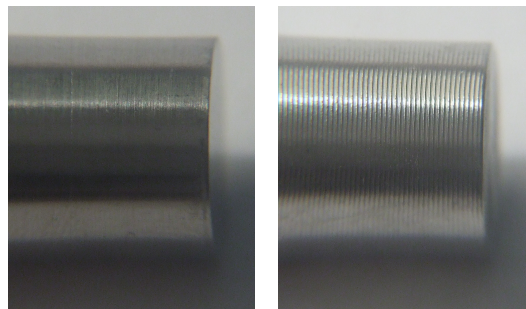


Fig. 2. Samples with different surface finish: polished surface on the left and surface with rills on the right.

Sample	Material	Process	Surface	Value
Sample D	AISI 317LMN	Rolling	Rills	1.770
Sample E	AISI 317LMN	Rolling and solid HIPing	Polished	1.397
Sample F	AISI 317LMN	Rolling and solid HIPing	Rills	1.943

4. Calibration

4.1 Temperature

The outgassing rate of a surface is temperature dependent. The samples to be tested are put inside the quartz tube with the central cross section in the midpoint of the oven. Thus, the temperature at the midpoint has to be known, but the PID controller does not give the real temperature at the location of the samples. Since an equipment that measures the real temperature during the outgassing tests may influence the sample outgassing, a temperature calibration before starting the tests was preferred.

The calibration was carried out at pressures of $\sim 10^{-5}$ Pa between the controller and a K-thermocouple put in the midpoint, covering the temperature range of interest. A linear calibration curve was found and the thermocouple temperature T_{TC} was used as real temperature which the outgassing tests are referred to. In fact, the temperature profile corresponding to the length of the samples along the axis of the quartz tube was also measured for some temperatures. The profiles are flat, so a good thermal equilibrium is present along the samples.

4.2 Mass spectrometer and partial pressures

The quadrupole mass spectrometer BALZERS QMS 200 (PRISMA) with the application program Quadstar 32-bit was used in the present work. Before the outgassing measurements, tests with blank and sample runs were carried out in order to optimize the spectrometer parameters and recognize all the gas species involved in the measurements. Fig. 3 reports the typical mass spectrum. By observing the fragment ions in the spectrum all the probable parent gas species were identified. QMS 200 does not directly provide the partial pressures of the gas species, rather their concentrations related to a reference quantity. The ion currents are measured in the mass numbers of interest, so they become input to a solution matrix and the gas concentrations are determined via calibration factors. These factors indicate the relative mass spectrometer sensitivities for each fragment ion of a given gas species and they are usually determined by calibration gas whose components do not overlap in the mass spectrum.

However, in this work, as a first approach, the calibration factors were gathered from the physics considering the ionization probability of each gas species and the relative intensities of the fragment ions in the mass spectrum of the same gas species. Once the gas concentrations are obtained, they must be normalized. Since all the gas species involved in the measurements are known, the sum of the concentrations is set equal to 100%. The partial pressures of the gas species are therefore calculated multiplying the corresponding

percentage concentrations and the total pressure given by the Pirani-cold cathode combination gauge.

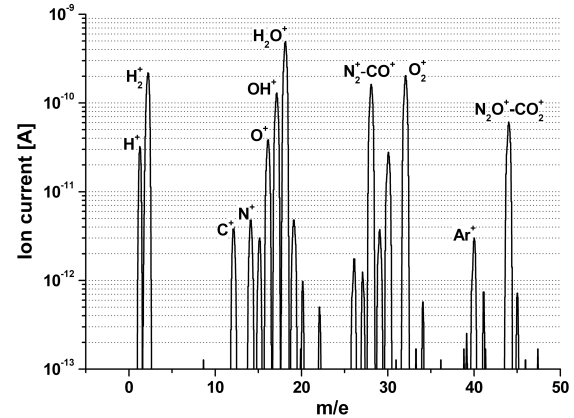


Fig. 3. Typical mass spectrum obtained during tests with blank and sample runs. The fragment ions are shown for some peaks.

5. Measurements

Outgassing rate of a surface is a decreasing function of exposure time of the surface to vacuum. Specific partial outgassing rates were obtained as a function of the pump-down time at $T_{TC} = 100^\circ\text{C}$ for all the samples reported in table 1 and at $T_{TC} = 240^\circ\text{C}$ only for the polished ones. Blank and sample runs were performed under same experimental conditions. Between two consecutive runs the vacuum conditions were broken down and created again for allowing the introduction of the samples into the quartz tube or their removal.

Time zero for data acquisition was fixed when the recorded total pressure was $\sim 5 \times 10^{-3}$ Pa because the mass spectrometer stands a pressure limit of 1×10^{-2} Pa. The selected value of total pressure was usually reached ~ 20 minutes after the start of the pump-down. The acquisition time was larger than 8 hours in order to obtain outgassing rates in quasi-steady state. The calculated specific partial outgassing rates of the samples after 9 hours were taken to compare the results and at this time the recorded total pressure was $\sim 1 \times 10^{-4}$ Pa.

6. Results and discussion

The gas species generally released from the samples were H_2 , H_2O , CO/N_2 , O_2 , Ar, $\text{CO}_2/\text{N}_2\text{O}$. For the UL, the outgassing limits are given at 100°C and only two values are specified in the vacuum handbook: one for hydrogen and the other for impurities.

Fig. 4 reports the specific outgassing rate of hydrogen q_{H_2} as a function of the pump-down time at

$T_{TC} = 100^\circ\text{C}$ for the samples C and E which differ only in the solid HIPing applied to the sample E. It can be observed that during the first 3.5 hours the curves are very close. Later on, the solid HIPing leads to outgassing values which are slightly greater: $q_{H_2} = 5.18 \times 10^{-5}$ Pa.m.s⁻¹ for the sample C and $q_{H_2} = 7.08 \times 10^{-5}$ Pa.m.s⁻¹ for the sample E. When solid HIPing is applied to a material, the outgassing rates can be lower or higher than those of the initial material, according to the combination of temperature and pressure (1000°C and 100 MPa in the present work) used during the HIPing process. However comparing the partial outgassing rates between the samples A, C and E or the samples B, D and F, no significant difference due to the HIPing method has been found. A possible reason could be that the samples are too small in order to appreciate the effect of the different fabrication technique. Moreover, because of such small dimensions, the amount of some gases released from the samples has not been appreciated sometimes; so higher sample/chamber volume ratio must be taken into account.

Fig. 5 reports the specific outgassing rate of hydrogen q_{H_2} as a function of the pump-down time for the sample E at $T_{TC} = 100^\circ\text{C}$ and $T_{TC} = 240^\circ\text{C}$. Since the history of a material affects the outgassing rate, the use of the same specimen first at low and then at higher temperature should be noticed. As expected, for increasing temperatures the outgassing rate grows; in the case of the sample E, q_{H_2} becomes 1.31×10^{-4} Pa.m.s⁻¹. Interestingly, it can be observed that at 240°C , as the measurement time grows, the outgassing rate reaches a well defined maximum before decreasing.

Finally, comparing for instance the specific partial outgassing rates at $T_{TC} = 100^\circ\text{C}$ between the samples A and B which differ only because of the surface finish, it has been observed that in the case of the rills the outgassing value is about 4 times greater than that of the polished sample. It should be remembered that the outgassing values are calculated per unit of *real* area of the samples and not geometric, so this increase cannot be explained with a bigger sample area exposed to the vacuum. The explanation for this increase would be the structural change of the surface that the material undergoes when the rills are generated.

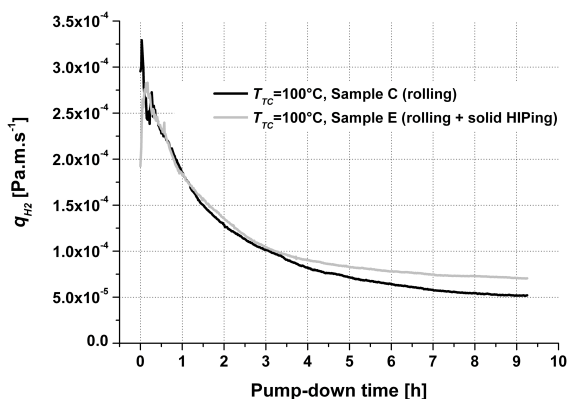


Fig. 4. Effect of the solid HIPing method on the specific outgassing rate of hydrogen.

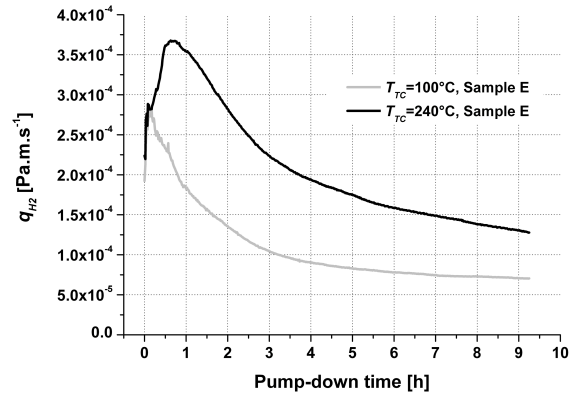


Fig. 5. Effect of the temperature on the specific outgassing rate of hydrogen.

7. Conclusions and outlook

An experimental setup was shown for measuring the partial outgassing rates of stainless steel samples obtained by HIPing method with chemical composition close to that of the structural material for the ITER in-vessel components. The obtained results are only preliminary and as a consequence cannot be compared to the outgassing limits yet. Future work aims to make further improvements of the experimental setup considering in particular higher dimensions of the samples, different cleaning methods of their surface and calibration factors in the mass spectrometer determined by calibration gas.

Acknowledgments

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