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Tritium retention and outgassing from JET first wall materials due to tritiated atmosphere exposure: the Tritium Soaking Facility

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Abstract
Tritium inventory inside the vacuum vessel of a tokamak is restricted for safety reasons. Therefore it is, of great importance to assess tritium retention and outgassing in first wall materials (Be and W) in order to establish its inventory. With this aim, one of the activities foreseen in the "Technical exploitation of JET DT operations" (WPJET3) project is devoted to the studies of tritium retention and outgassing in Plasma Facing Components (PFCs) in laboratory-scaled experiments under controlled conditions to support the understanding of tritium retention in the tokamak. Two facilities have therefore been designed, and are currently under construction, by CCFE and ENEA: the Tritium Loading Facility (TLF) and the Tritium Soaking Facility (TSF). Particularly, the TLF will study the tritium retention caused by energetic tritium ions implantation in PFCs, and the TSF will investigate the tritium retention due to the exposure to a tritiated atmosphere. This work describes particularly the samples to be investigated, the foreseen experimental plan and the design of the TSF. Concerning the experimental plan, this has been arranged to reproduce JET conditions under cryopump divertor regeneration during which a significant amount of tritium is temporarily released in the torus. The samples to be investigated are bulk Be and W, in order to establish the effect of radiation damage on the hydrogen transport properties inside such materials both unexposed and exposed samples will be tested. During the experimental campaign of the TSF, two different phases are foreseen: 1) the loading (or soaking) phase, in which the tritium soaking chamber is filled with a tritiated atmosphere and 2) the outgassing phase, in which the tritium is released by the sample under different inactive atmosphere, including vacuum. Here it will be interesting to measure the tritium release as a function of the temperature, of the time and also of the gas venting composition (i.e. effect of doping gas like H_2, O_2 and H_2O). For the smallest samples the outgassing rate will be measured via thermal desorption spectroscopy up to 1273 K. Finally the layout and the technical description of the TSF are presented.

Key words: tritium retention, first wall materials, JET3 program.

1. Introduction

One of the main concerns of fusion technology is represented by the use of tritium as fuel. Tritium is an unstable isotope of hydrogen and is naturally present only in trace amounts. Therefore, in order to guarantee the self-sufficiency in future fusion reactors, it must be produced by interaction between the neutrons and the lithium present inside the so-called breeding blanket region [1]. Due to its radioactivity and scarcity, any kind of tritium loss must be minimised and the inventory inside and outside the tokamak region has to be maintained below the limits imposed by safety concerns. With regards to the tritium inside the vacuum chamber, main issues are related to erosion, dust generation and retention in first wall materials [2]; in this view, the next deuterium/tritium campaign in JET with the ITER-like Wall provides a unique opportunity for studying tritium

plasma interactions with the tokamak wall in ITER, and other future fusion devices, conditions relevant. Moreover, in the frame of the WPJET3 "Technical exploitation of JET DT operations", tritium retention and outgassing from Be and W samples will be also studied in laboratory-scale experiments under controlled conditions to support the understanding of tritium retention in the tokamak; in addition the results obtained inside the laboratory facilities will be used to validate simulation tools also developed within the WPJET3 project.
In this view two facilities are currently under construction, they are: the Tritium Loading Facility (TLF) and the Tritium Soaking facility (TSF) [3]. The main aim of the TLF is to investigate the tritium retention in Be caused by ion implantation and its possible release; in fact, during plasma operation, high flux of low energy H isotopes are implanted on first wall materials. Referring to the TSF, described in further detail in Section 4, its main objective is to assess the tritium adsorption due to gas soaking and tritium outgassing behaviour in order to reproduce the tokamak conditions during and after cryopumps regeneration. Usually the pressure in the vacuum chamber is low, close to vacuum conditions, ranging from 10^{-7} mbar before a plasma to 10^{-5} mbar during a plasma but, during cryopumps regeneration (in JET the helium panel temperature is increased up to 90 K for more than half an hour and then is cooled back to 20 K), the pressure inside the vacuum vessel reaches values up to 10^{-2} mbar, since the amount of hydrogen isotopes cumulated in the pump charcoal is released inside the chamber [4]. Then, once the regeneration has ended, the tokamak is pumped by the cryopumps of the Active Gas Handling System. The specific conditions inside the vacuum chamber during the cryopumps regeneration phase may cause adsorption, diffusion and thus permeation of tritium through the wall too. A graphical observation of this event is clearly illustrated in Figure 1 [5]: the pressure inside the vacuum vessel increases when injection starts, the plasma itself may stay a few tens of seconds, then the pressure starts to decrease first rapidly and then on a time scale of one hour then, during the cryopumps regeneration, the chamber pressure exhibits a sharp increase (up to a value three orders of magnitude higher than during the plasma phase) lasting for all the regeneration time.

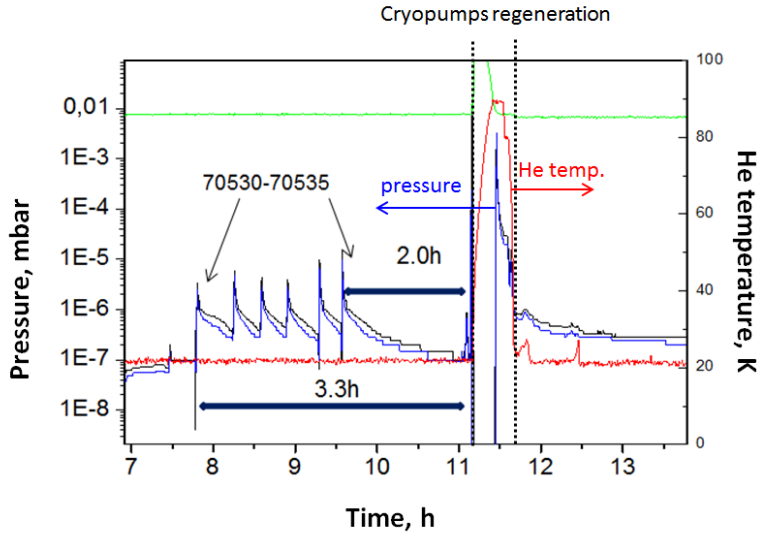


Figure 1. Pressure in the main chamber as a function of time (during a series of plasma shots and during the cryopumps regeneration phase). Graph extracted from [5].

In this view, to investigate the tritium retention and outgassing from JET first wall, this work describes the activities carried out so far in the design and modelling of the TSF. Firstly a brief recall of the basic theory and the main literature results on tritium transport parameters in Be and W is provided, then the samples and the experimental conditions that will be investigated inside the

TSF are described and, finally, the preliminary layout and the technical design of the TSF are illustrated.

2. Basic hydrogen isotopes transport parameters in Be and W

The aim of the TLF is to measure the tritium adsorption, retention and outgassing in Be and W when they are exposed to a tritiated atmosphere (to reproduce the vacuum vessel conditions during pump regeneration) under several operating conditions. In this view it is useful to consider that the hydrogen concentration of species *i*, c_i, in a wall of thickness *d* at depth *x* and time *t* is composed of a solute concentration c_{si} and a trapping concentration c_{ti} [6]:

$$c_i(x,t) = c_{si}(x,t) + c_{ti}(x,t) \qquad (1)$$

where *i* indicates protium, deuterium or tritium. For one of the hydrogen isotopes species, c_s is given by the diffusion equation while trapping is a process that delays the flow of hydrogen in a solid via the capture and release of hydrogen atoms by sites other than the ordinary solution ones. The parameters that characterise the trapping phenomenon are the number of trap sites N_t and their average energies E_t. Under the steady state conditions and not considering temperature gradients, the diffusive flux (J_s) can be expressed by the Fick's first law:

$$J_s(x) = -D\frac{\partial c_s}{\partial x} \qquad (2)$$

where *D* is the diffusivity (m^2 s^{-1}). Then, according to the Sievert's law, the hydrogen atoms concentration into a metal can be expressed as:

$$c_s = K_s p^{0.5} \qquad (3)$$

where K_s is the solubility (or Sieverts') constant (mol m^{-3} $Pa^{-1/2}$) and p is the hydrogen gas pressure [7]. Therefore by combining Eqs. 2 and 3 and considering the boundary conditions at the inner and outer surface of the wall at x=0 and x=d, the diffusion flux can be expressed as:

$$J_s = \frac{DK_s}{d}\left(\sqrt{p_d} - \sqrt{p_0}\right) \qquad (4)$$

Therefore, in order to assess the hydrogen transport inside Be and W, the main fundamental parameters to know are: *D*, K_s, N_t and E_t. A more precise assessment should also foresee the adsorption and release surface constants (usually called k_1 and k_2). Finally, it is useful also to recall that the diffusivity and solubility are thermal-activated processes and thus they can be expressed using an Arrhenius' law:

$$D = D_0 \exp\left(-E_m / RT\right) \qquad (6)$$

$$K_s = K_0 \exp\left(-Q_s / RT\right) \qquad (7)$$

Literature provides some works reporting hydrogen isotopes (mainly H and D) solubility and diffusivity values in Be and W. A comprehensive review has been done by Skinner et al. [8] which main outcomes are illustrated in Figure 2 for beryllium and in Figure 3 for tungsten. Two critical aspects can be noted: 1) the data measured in the different experiments show large discrepancies

(usually more than two orders of magnitude) and 2) the temperature range of the measurements is quite limited and usually far from the JET wall temperature (523 K).

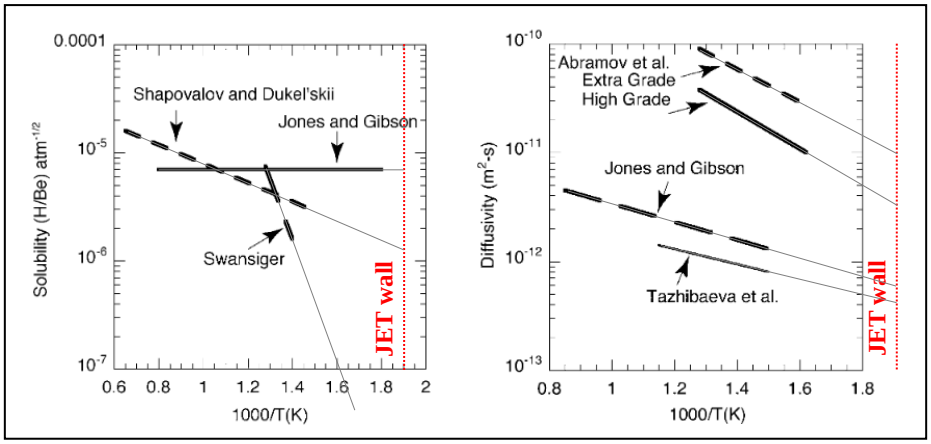


Figure 2. Hydrogen solubility (left) and diffusivity (right) in beryllium [8].

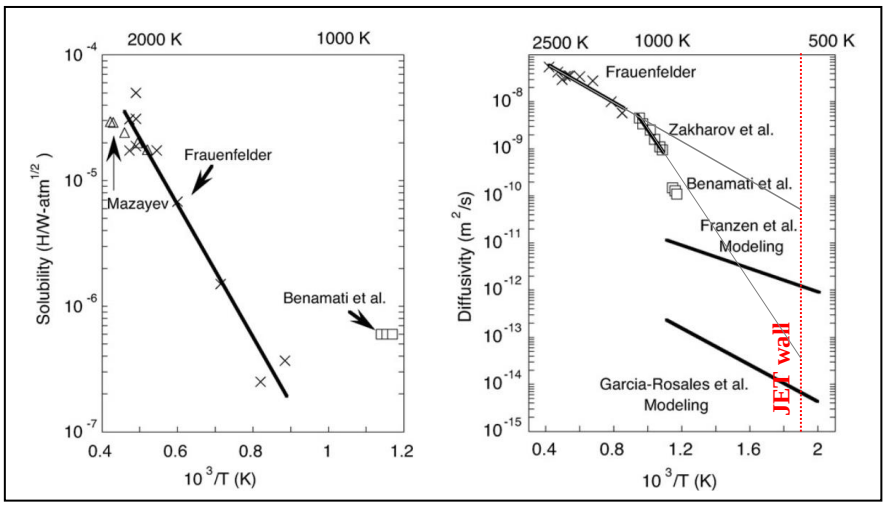
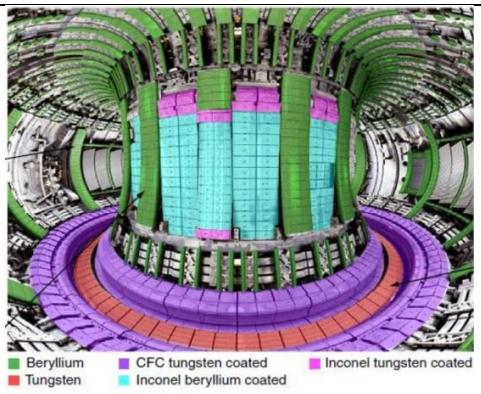


Figure 3. Hydrogen solubility (left) and diffusivity (right) in tungsten [8].

3. Sample definition and experimental plan

The TSF aims to investigate tritium adsorption and outgassing in Be and W due to tritiated atmosphere exposure. Thus the samples studied within this scope are bulk beryllium and bulk tungsten as found in the torus. Particularly, beryllium samples are extracted from the centre part of the Inner Wall Guard Limiter (IWGL) and tungsten comes from the divertor lamellae. In order to avoid conflicts of the retention mechanisms on multi-layered materials or coatings, it has been chosen to focus on bulk materials only. Moreover, due to the importance of the material microstructure on the tritium behaviour, both new (not exposed) and used (exposed) samples will be studied. In fact, during the next D-T campaigns of JET, fusion reactions in the plasma are expected to produce high energy neutrons flux which will be partially absorbed by the wall. Neutrons are known to create damage in materials causing the formation of vacancies, dislocation loops, clusters or nanovoids that are efficient traps of gas atoms as helium, hydrogen and hydrogen isotopes; this will surely affects the hydrogen transport properties. In this view, one piece of unexposed and

exposed Be tile and W lamellae have been reserved for this project which have been cut and characterised. Figure 4 illustrates the different materials in the JET torus and reports the dimension of the samples after the cutting procedure performed in Romania at the MEdC institute. Such procedure has been already defined for Be samples [9], while for W is still under discussion.



Sample dimensions

Material	Size, mm
Be	75 x 60 x 50
	12 x 12 x 12
	12 x 12 x 2.5
W	10 x 5.5 x 3

Figure 4. The different materials in the JET torus and the Be and W samples dimensions after cutting.

During the experimental campaign planned from the end of 2016 until the end of 2018, the TSF will measure the tritium adsorption and outgassing in JET relevant operating conditions. Starting from a reference experiment, the variable parameters will be: source term, soaking time, carrier gas type and carrier gas pressure. In practice, during a typical experiment, the Be (or W) sample is heated up to 523 K and exposed (soaked) in a D-T atmosphere for a given time. Then the tritiated atmosphere is evacuated and the tritium outgassing rate, either under vacuum and in presence of a carrier gas, is monitored for one week. For the smallest samples, after the soaking phase, the tritium outgassing is measured also by Thermal Desorption Spectrometer up to 1273 K [10]. Table 1 illustrates the values of the operating conditions foreseen in the experimental plan.

Table 1. Experimental plan of the TSF.

Experiment	**Soaking temperature, K**	**Soaking time, h**	**Source term, TBq m^{-3} (mbar)**	**Carrier gas**	**Carrier gas pressure, mbar**
Reference experiment	523	1	10 (0.2)	None (vacuum)	10^{-5}
Influence of source term		1	5-20 (0.1-0.4)	None (vacuum)	10^{-5}
Influence of soaking time		2	10 (0.2)	None (vacuum)	10^{-5}
Influence of carrier gas type		1	10 (0.2)	N_2, He and N_2+Air+humidity	1000
Influence of carrier gas pressure		1	10 (0.2)	N_2	200

Also important is the study of the influence of the repetition exposure. Indeed, the Plasma Facing Components (PFCs) will not be exposed to the plasma and cryopump regeneration, once but several times; it is likely to obtain an outgassing signal after several exposures and a retention build-up as a function of the number of plasmas carried out. In this view, the above-listed experiment will be repeated several times.

4. Layout and technical design of the TSF

The TSF will be housed inside a dedicated (tritium and beryllium compatible) glovebox of the Active Gas Handling System (AGHS). Due to budget constraints, the TSF has to share some components with the TLF (not described in this work);

Figure **5** illustrates the process flow diagram of both TLF and TSF. In practice, the two facilities share the pumping system, the residual gas analyser (RGA) for the outgassing measurements and part of the tritium lines. The pump set from Leybold consists of an air cooled turbo pump (115 L s^{-1} in H_2, 145 L s^{-1} in N_2) and a dry scroll pump (5.4 m^3 h^{-1} in N_2). The turbo pump speed is variable, allowing control of the base pressure during the tritium loading phase, while the ultimate pressure is 10^{-10} mbar.

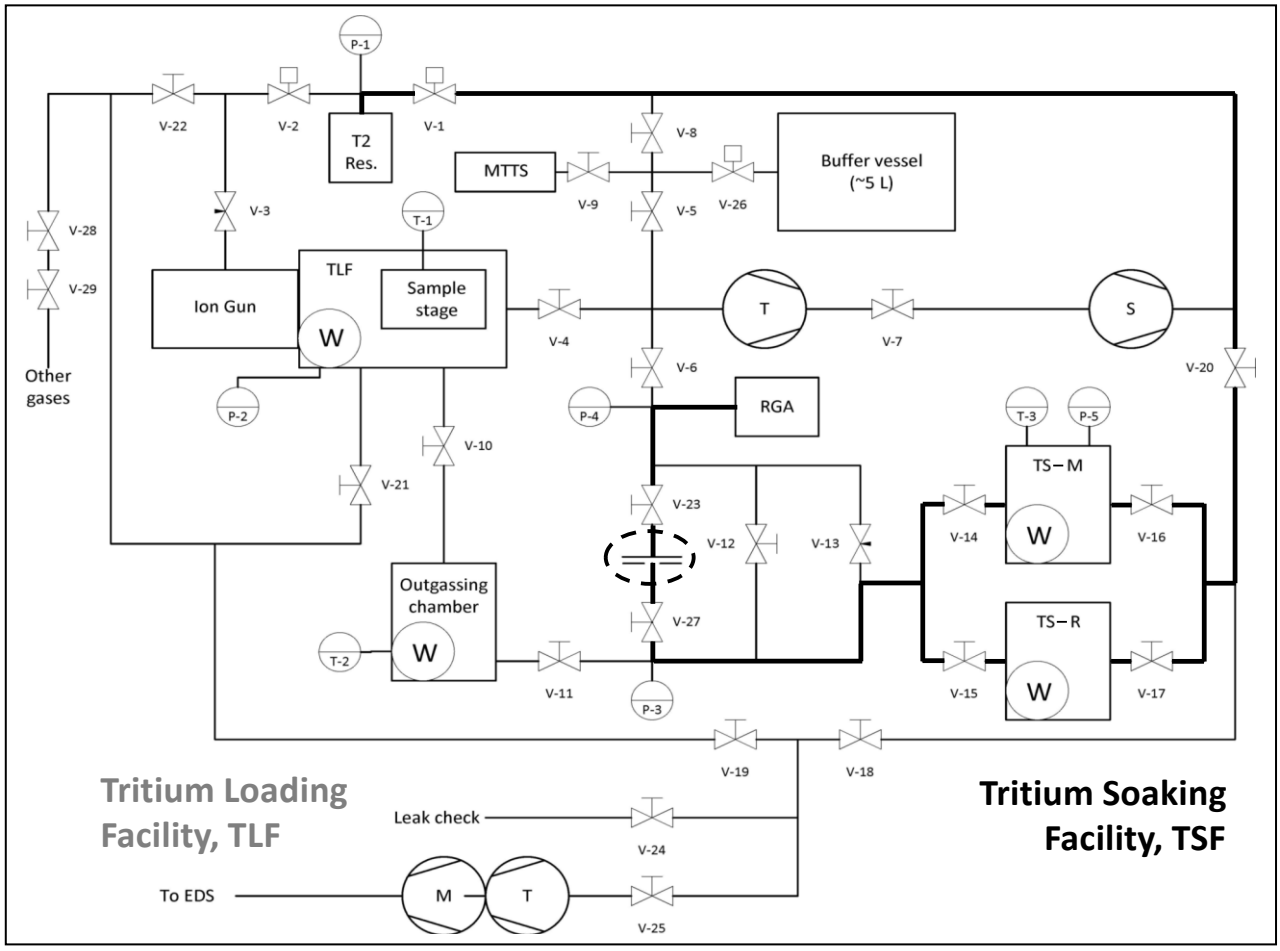


Figure 5. Process flow diagram of the TLF (left side) and TSF (right side).

Referring to the TSF (bold lines in
Figure **5**), the tritium-deuterium gas mixture is sent from a dedicated reservoir (T_2 res.) into the tritium soaking-measuring (TS-M) chamber, where the sample is located. The TS-M is equipped with temperature and pressure sensors; when the deuterium-tritium atmosphere reaches the desired pressure value, the valve in the inlet line (V-16) is closed. The Be (or W) sample inside the TS-M

chamber is soaked in the tritiated atmosphere for the duration fixed in the experimental plan (see Table I). Then the TS-M is briefly evacuated by the vacuum pump and the tritium outgassing from the sample is monitored through a known conductance restriction (see the dashed line in Figure **5**) and measured by RGA. An experiment having the same operating conditions will run concurrently in the tritium soaking-reference chamber (TS-R) – a chamber identical to the TS-M one but without a sample,– in order to estimate the background contribution due to the D-T interaction with the chamber material itself. As previously explained, the outgassing rate up to 523 K from medium and large samples will be monitored inside the facility itself while, for the smallest samples, the outgassing rate up to 1273 K will be measured by Thermal Desorption Spectrometry (TDS). Each TS chamber has a cylindrical shape with a volume of 801 cm^3 (internal diameter of 108.3 mm and height of 87 mm). It is equipped with two ½" VCR connections for the inlet and outlet gas lines and with three thermocouples for temperature measurement in both chamber and sample. The heating system is external to the chamber. The base of the chamber is realised with a 6-3/4" CF flange that allows its opening to replace the sample. The commissioning of the chamber (presently under construction) foresees a leak test ensuring a leak rate lower than 10^{-9} mbar L s^{-1}.

5. Conclusion

The aim of the TRI sub-project (within the WPJET3 project) is to investigate the tritium retention and outgassing in/from first wall materials due to two mechanisms: tritium ions implantation and tritiated atmosphere exposure. These mechanisms will be studied through two dedicated laboratory facilities: the Tritium Loading Facility (TLF) and the Tritium Soaking Facility (TSF), respectively.
This work has presented the status of the activity related to the TSF; firstly a review of main hydrogen transport properties has been presented: results in literature show very large discrepancies and the temperature ranges at which the measurements were performed are far from JET first wall temperature. Thus a more JET-relevant experimental plan has been foreseen in which the soaking and outgassing measurements are performed at the JET first wall temperature (523 K) and the tritiated atmosphere in the TSF chamber reproduces the atmosphere inside the VV during cryopump regeneration. Due to the fact that high energetic neutrons create damages in FW materials, both unexposed and exposed samples will be tested. Particularly, in the smallest samples, the outgassing rate will be monitored up to 1273 K via TDS. In addition, this work has showed the layout of the experimental facilities currently under construction, which commissioning is foreseen within the end of 2016.
The uniqueness of these two facilities is related to the possibility they have of handling both Be and tritium, in this sense they represent a very useful tool for material characterization in the fusion community.

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