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Hydrogen retention studies on lithiated tungsten exposed to glow discharge plasmas under varying lithiation environments using Thermal Desorption Spectroscopy and mass spectrometry

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ABSTRACT

For the design of a Fusion Reactor based on a liquid lithium divertor target and a tungsten first wall at high temperature, the interaction of the wall material with plasmas of significant lithium content must be assessed, as issues like fuel retention, tungsten embrittlement and enhanced sputtering may represent a showstopper for the selection of the first wall material compatible with the presence of liquid metal divertor.

In this work we address this topic for the first time at the laboratory level, hot W samples (100 °C) have been exposed to Glow Discharges of H₂ or Li-seeded H₂ followed by in situ thermal desorption studies (TDS) of the uptake of H₂ on the samples. Pure and pre-lithiated tungsten was investigated in order to evaluate the differential effect of Li ion implantation on H retention. Global particle balance was also used for the determination of trapped H into the full W wall of the plasma chamber. A factor of 3–4 lower retention was deduced for samples and main W wall exposed to H/Li plasma than that measured on pre-lithiated W.

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1. Introduction

Tungsten has been selected as the Plasma Facing Material (PFM) for the ITER divertor [1] and is the basic material taken into account for the PFM selection in the design of a future fusion reactor as DEMO [2]. Its selection is motivated by the good thermal properties of tungsten that has the highest melting point among metals, its low sputtering yield under particle/ion bombardment and the low hydrogenic isotope retention of tungsten compared with other studied materials as carbon. Despite these advantages, the nominal heat loads on the divertor region (5–20 MW/m²) during plasma operation and even more the extreme conditions expected during transient events such as ELMs or disruptions can induce permanent damage and irreversible degradation on this solid material that would seriously limit its lifetime.

On the other hand the use of liquid lithium as PFM offers unique advantages in terms of heat handling capabilities and plasma per-

formance improvement [3,4]. Furthermore its use as a liquid metal would avoid the problems related with induced permanent damage on solid materials exposed to burning plasma and the renewable surface of a liquid material could suppress its disintegration and the dust formation during plasma operation, increasing the lifetime of the Plasma Facing Components (PFC). Hence, lithium is considered as the favorite liquid metal PFM for future fusion devices, but its high H retention [5] makes the associated Tritium inventory a possible showstopper.

The lithium research as an alternative PFM has been addressed in a wide variety of experiments involving hot plasma devices [3–9], linear plasma facilities [10,11], laboratory configurations [12,13] and innovative concepts for future fusion devices [14,15,16], showing promising results in respect of improved plasma performance, power exhaust handling and the possibility of control and decrease the lithium hydrogen uptake. In addition some studies about the interaction of W-Li hybrid layers with linear plasma have been performed. Neff et al. [10] studied the D retention in W-Li coatings exposed to a high fluence linear deuterium plasmas in Magnum PSI device and reported the inhibition of Li-D-O complex formation responsible of the enhanced fuel retention on the coatings when the

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deuterium linear plasma is seeded with helium. Other experiments involving lithiated tungsten samples exposed to Magnum PSI linear plasma [17] have shown the possible saturation in the deuterium retention and the inhibition of blistering on the W surface due to the protective effect of the Li coating. However, the possible effect that a simultaneous irradiation with hydrogenic species and lithium on W-Li layers (conditions that can be expected in a fusion reactor environment with W-Li PFM), can have in multiple Plasma Wall Interaction (PWI) phenomena as T global retention, re-deposition, material erosion, co-deposition and mixing is unavoidable. Under this scenario a specific investigation involving PWI and Material Science topics in terms of fuel retention, surface erosion and material compatibility are clearly necessary. In this work dedicated experiments and analysis oriented to quantify the H retention on several W-Li samples exposed to H₂ Direct Current (DC) Glow Discharge (GD) plasmas under different environments have been performed by using Thermal Desorption Spectroscopy (TDS) in order to induce the in situ hydrogen desorption from the W-Li layers and Mass Spectrometry to quantify it.

2. Experimental setup and procedure

The experiments were carried out in a full W wall device designed at CIEMAT and similar to the one previously used and described [17]. An overview of the setup can be visualized in Fig. 1. The primary vessel consists on a cylindrical stainless steel vacuum chamber that acts as the grounded cathode where the irradiation with the H₂ GD plasmas is carried out. The plasma chamber (V=5.4L) has its inner wall totally covered with high purity cold rolled tungsten (A=0.14 m²) and is pumped out by using a unit that consist on a set of turbomolecular-rotary pump that decreases the base pressure until 10⁻⁵ Pa approximately. The pressure during the plasma operation and the lithiation process is measured with a capacitance manometer (0.1–100 Pa). Inside this chamber a cylindrical, stainless steel oven (3 cm of diameter and 6 cm of height) filled with high purity (>99%) lithium is placed to produce the evaporation of lithium for the in situ lithiation of the samples and the main W wall, by the effusion of the evaporated Li atoms through a circular hole (~1 cm of diameter) placed on the top of the oven. An AC power supply with varying voltage is used to heat up the oven. The temperature of the lithium is measured during the evaporation by a thermocouple type K that is placed inside the oven, in direct contact with the lithium. Before the lithiation process the lithium placed in the oven is outgassed by heating it until 300 °C of temperature during 30 minutes approximately inducing the hydrogen, water and impurities desorption from the lithium bulk.

On the top of the plasma chamber and open to it, a small chamber intended to harbor the in situ TDS analysis on the W-Li samples is located. To measure the H retention during the desorption process, a Residual Gas Analyzer (RGA) Mass Spectrometer (SRS 100) is connected to this chamber. A manipulator that contains a holder for the W-Li samples and a pure resistive filament (fed with a direct current and 60 W of total heating power) in order to heat up the samples by radiation and induce the H desorption, is placed on the top of this TDS chamber. The temperature evolution on the sample during the TDS analysis is measured by using a type K thermocouple welded on the holder and that is in direct contact with the sample. The manipulator can move the samples from the plasma chamber to the TDS chamber without breaking the vacuum conditions after the H₂ GD plasma irradiation. In this way the possibility of contamination and hydrogen release from the samples before the TDS analysis can be excluded.

The samples consist on a cold rolled, high purity tungsten pieces with dimensions of 12.6 ± 0.2 mm of width, 21.7 ± 0.2 mm of height and 0.1 mm of thickness with a surface area exposed to the plasma

of 273 ± 7 mm². These samples are introduced in the holder and are outgassed by heating up to 550 °C during 15 minutes previously to the plasma exposure. Before the H₂ irradiation one of the samples was exposed to a previous in situ lithiation by Li evaporation at 500 °C–550 °C during 30 minutes, (Li evaporation rate of 3·10¹⁸ atoms/cm² s and total evaporation of 0.1 g approximately) under a H₂ flow (~4 Pa of total pressure) atmosphere. Another sample was exposed directly to a H₂ GD plasma in a lithium evaporation environment (Li at 500 °C–550 °C during 30 minutes as in the pre-lithiated case), producing a Li-seeded H₂ GD plasma in order to study the hydrogen retention under simultaneous irradiation of Li and H on the W sample. Finally a pure W sample was also irradiated with pure H₂ GD plasma in order to compare the hydrogen retention with the two cases previously described.

Between the plasma and the TDS chamber a stainless steel grid with boron nitride insulation pieces are placed to limit the plasma in this region and assure that the plasma does not touch any part of the TDS chamber, excluding in this way the possibility of contamination of the TDS analysis by the desorption of hydrogen from other parts non exposed in principle to the plasma. Additionally and also connected to the plasma chamber a differentially pumped analysis chamber is placed to monitor, with a Residual Gas Analyzer (RGA) Mass Spectrometer (Pfeiffer Vacuum Prisma QMS 200), the plasma operation. This analysis chamber is pumped down with another turbomolecular-rotary pump set until a base pressure around 10⁻⁷ Pa, being this pressure measured with an Ionization Gauge. All the experiments were carried out after a wall conditioning on the main W wall and samples that assures the reproducibility of the experiments and a fresh/clean surface prior to the lithiation/irradiation processes. This wall conditioning consists on pure DC-GD Helium plasma during 20 minutes in order to desorb and clean by sputtering the impurities present on the sample and main wall surface.

To produce the discharge, the high purity (>99.999%) gases (H₂ and Helium for the wall conditioning) are introduced into the chamber by an electronic mass flow controller. The plasma characteristics of the H₂ irradiation are: ~4 Pa of total pressure, a discharge voltage between 100 and 250 V, plasma current (300–350 mA) and total ionic fluence up to 7.5·10²² m⁻² (being the dominant plasma ions at a pressure of 4 Pa a mixture of H₂⁺ and H₃⁺ [18]), with a total duration of 30 minutes. During the irradiation the main W wall and the samples were not heated up externally, but due to the plasma bombardment the surface temperature was increased until 90–100 °C approximately. To complete the measurements during the plasma operation an Optical Emission Spectroscopy (OES) diagnostic was also used to measure the intensity of the lines related with Li I (670.7 nm) and H α (656.3 nm) transitions. The OES diagnostic consists of a focusing lens that directs the plasma emitted light to a monochromator (Oriel 77250) in order to select the registered wavelength during the plasma operation (performing a 650–675 nm wavelength scan) and a photomultiplier, (Hamamatsu R3896) with high efficiency spectral response for the 400–700 nm wavelength range, that amplifies the signal measured from the plasma emission collected through an optical window situated in the main plasma chamber.

After the H₂ GD plasma irradiation the treated sample is moved to the TDS chamber by using the manipulator and the hydrogen uptake on the samples is measured by TDS assisted mass spectrometry. The filament that induces the heating of the sample and the hydrogen desorption is fed with a DC power supply with a nominal voltage of 12 V and a maximum current of 5 A. Finally, an absolute calibration work that relates the single RGA peak at 2 amu/e signal with its associated H₂ partial pressure was performed in order to quantify the hydrogen retention on the samples and on the full W wall.

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