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## Experimental studies of the interactions between a hydrogen plasma and a carbon or tungsten wall

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**Abstract.** We present work done at LSPM (Laboratory of Sciences of Processes and Material Sciences), using the CASIMIR ECR plasma reactor device, aimed at answering questions about hydrogen isotope fuel retention and dust production in the context of the plasma-facing components (PFCs) of the International Thermonuclear Experimental Reactor (ITER). The plasma is characterized by means of optical spectroscopy, mass spectrometry and electrostatic probe; furthermore the dust density and size distribution will be measured by a laser diagnostic system. We present some early results obtained from hydrogen plasma exposure of pure tungsten samples, as well as samples of ITER-relevant tungsten-rich powders, produced in-house by the ball-milling technique, which are likely to be a by-product of material erosion and migration during tokamak operation. In particular, we have performed measurements of the specific surface area of these powders as a proxy to their capacity to absorb hydrogen.

### 1. Introduction

At LSPM, we have developed a low pressure / high density ECR plasma reactor named CASIMIR II, along with a smaller prototype reactor, so-called ‘monosource’. In these devices, we consider the behaviour of nanometric ternary powders from the tungsten-rich corner of the W-C-Mg system, as representative of the mixed materials likely to form as a consequence of the plasma-wall interactions and impurity transport phenomena in ITER. We diagnose the plasma phase by means of Optical Emission Spectroscopy (OES), Langmuir Probe, and Mass Spectrometry, as well as with two laser diagnostic systems. The first is an IR (Infra-Red) quantum cascade laser (QCL) devoted to the detection and absolute measurement of light hydrocarbon dust precursors. The second system is a continuous wave (cw) visible laser, in the course of installation, that will be used in extinction and scattering modes to measure the dust density in our plasmas as well as determine the dust size distribution.

### 2. Short presentation of the CASIMIR reactor

Our reactor CASIMIR II (for Chemical Ablation, Sputtering, Ionization, Multi-wall Interaction and Redeposition) relies on the Electron Cyclotron Resonance (ECR) principle. Its aim is to produce low pressure / high density plasmas, using an innovative dipolar plasma source [1, 2], to simulate some plasma/surface processes occurring in parasitic discharges under the divertor dome and in the far Scrape-off Layer (SOL) regions of tokamaks. A full description of the device is given elsewhere [3]. The main idea of this paper is to study the formation under plasma exposure of mixed materials with compositions similar to those foreseen in fusion devices (C/W).

The plasma is generated at the electron cyclotron resonance location, in our case 875 G for a 2.45 GHz excitation frequency. Langmuir probe and optical spectroscopy measurements show the plasma to be homogeneous within the volume enclosed by the 16 ECR sources arranged in a circle. Typical electron densities and temperatures are of the order of  $10^{11} \text{ cm}^{-3}$  and 2-3 eV, respectively, for working pressures between  $10^{-3}$  and  $10^{-2}$  mbar and an injected microwave power of 3 kW. An energy balance of the reactor with a single ECR source



was performed in order to estimate the power absorbed by the plasma to the power supplied to the system ratio  $P_{\text{absorbed}}/P_{\text{injected}}$ . It was shown that around 1/3 of the injected power is in fact coupled to the plasma [4]. These electromagnetic parameters are important to understand the homogeneous growth mechanism of dusts.

To understand the mechanism of dust formation, we installed an Infrared Absorption Spectroscopy system, using a cw Quantum Cascade Laser as light source, emitting around  $1347\text{ cm}^{-1}$  [5, 6]. Its aim is to perform quantitative measurements of carbon dust precursors:  $\text{CH}_4$  and  $\text{C}_2\text{H}_2$ , before going further with mixed materials. This system provides finely-tuned IR laser radiation that is absorbed by specific molecular species within the plasma, before being detected by a very high resolution dedicated spectrometer. The absorption length was increased to 5 m using a custom-made optical cell that makes the laser beam do some 24 passes inside the plasma volume.

In order to detect the apparition of soot in the discharge, we are presently implementing an additional laser setup relying on a cw visible solid-state laser emitting at 532 nm (up to 1 W power). Two sets of measurements will be attempted: (i) Extinction (which corresponds to the decrease of the laser signal after crossing the plasma volume, when the soots start to appear), and (ii) light scattering, in order to gain information on the size distribution of these soots. A photograph of the first step of setup assembly is shown as Figure 1.



Figure 1. The cw visible laser assembly during installation and alignment.

### 3. Carbon dust formation under ECR plasma exposure

Carbon dusts were synthesized at low pressure ( $10^{-3}$  mbar) plasma with feed gas composed of (50:50)  $\text{H}_2:\text{C}_2\text{H}_2$  or  $\text{H}_2:\text{CH}_4$ . SEM (Scanning Electron Microscopy) pictures were performed on Si substrates, showing spherical particles of tens of nm, obtained after 30s. TEM (Transmission Electron Microscopy) measurements exhibit graphitic structures [7]. These observations are quite unusual at such low temperatures, due to the very small residence time of neutral particles. Therefore, ions (positive and negative) were tracked since they are sensitive to the permanent magnetic field of the ECR sources.

Ions detection was performed by means of in-situ mass spectrometry. It is seen that positive ions are by far much more prevalent in the gas phase than negative ions (Figure 2), which corresponds to a different behaviour with respect to higher pressure (1 mbar) RF reactors, in which particle growth is due to trapping of negative ions. In our case, positive ions, confined by the device's magnetic field, seem to be responsible for the dust synthesis.

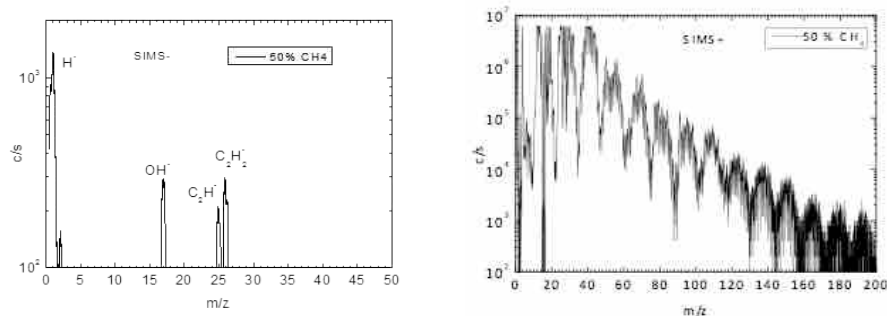


Figure 2. Mass spectra of positive ions (left) and negative ions (right) obtained in the single source ECR reactor. Each broad peak in the left spectrum corresponds to a different number  $x$  of carbon atoms in the  $\text{C}_x\text{H}_y$  hydrocarbons collected.

#### 4. Plasma exposure of prepared mixed materials dust samples

We can also synthesize ternary (W/C/Mg) powders of arbitrary but controlled stoichiometry by the ball milling method [8], also known as mechano-synthesis, as examples of the material mixes that will occur in ITER following erosion of the plasma-facing components [9]. Here we use Mg as a stand-in for Be, which is too dangerous to handle in our facility. The advantage of working with a composite material is to assess the influence of carbon and magnesium on the incorporation of hydrogen by studying the effect of their concentration on the hydrogen amount implanted near the surface and the kinetics of hydrogen diffusion into the bulk of the material. To assess the amounts of diffused and implanted hydrogen, techniques such as thermal desorption are expected to be used.

Measurements of specific surface areas (SSA), done prior to plasma exposure, have shown a value of up to  $2 \text{ m}^2\text{g}^{-1}$  for pure W powder, and increase further to more than  $7 \text{ m}^2\text{g}^{-1}$  when up to 10at% C is added; on the contrary, adding Mg has a clear negative influence on the measured SSA, probably due to an incomplete insertion of Mg into the W lattice, where Mg then has a tendency to bind together the W particles, thereby reducing the SSA. Measurements of the pore size distributions of binary and ternary materials are presented below (Figure 3). The majority of the pores (80%) have a diameter greater than 20 nm. These diameters correspond to mesopores (20-50 nm) and macropores (above 50 nm diameter). Large pore sizes mean that species can be inserted more easily into the material, in particular for hydrogen to get incorporated into the material during plasma implantation. This is beneficial for our future studies on the hydrogen incorporation into the material by plasma implantation.

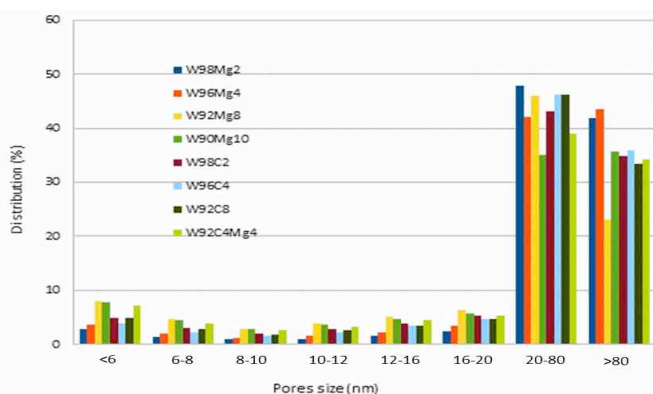


Figure 3: Distribution of pore sizes in binary W-C and W-Mg as well as ternary W-C-Mg materials.

The presence of oxygen is a problem because it changes the material properties and thus distorts the results. To enable the study of hydrogen incorporation in powders, it is necessary to reduce the oxygen concentration. For this we treat our powders with argon plasma. We exposed a sample of milled tungsten powder in ethanol. We performed the experiments in an argon plasma reactor for 1 hour. After treatment, the oxygen concentration decreases to 10%. The treatment is effective for oxygen content reduction on the material surface. It must, however, be renewed after each air exposure event.

The hydrogen plasma exposure experiments were carried out in our reactor with a single dipolar ECR source, which can deliver a power of 180 W and generate high density deuterium plasmas at low pressure. The neutral species flux is  $10^{22} \text{ m}^{-2}\cdot\text{s}^{-1}$  and ion flux is  $10^{20} \text{ m}^{-2}\cdot\text{s}^{-1}$ . The samples were placed on a ceramic support for electric insulation, at floating potential, and situated 1 cm away from the ECR resonance location for 6 hours of plasma exposure.

Three compounds (pure W, binary W/8at%Mg and ternary W/4atC/4atMg) were chosen and exposed to a deuterium plasma. To detect structural changes in the tungsten due to plasma interaction, a series of X ray diffraction patterns were obtained after various exposure times. We can see in Figure 4, the appearance of new peaks after 1 hour which indicates a phase change in the tungsten structure, the exact nature of which is still under study.

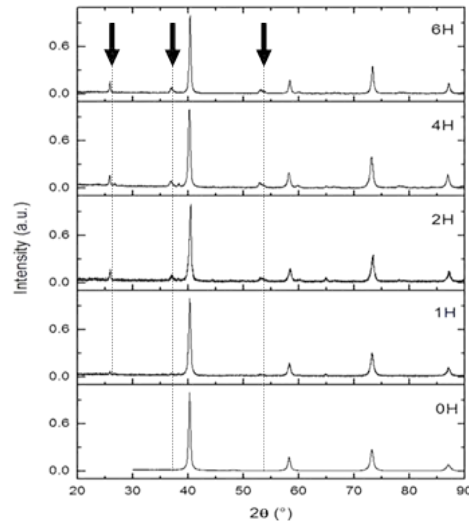


Figure 4 : X-ray diffraction spectra of the pure-W sample as a function of the plasma exposure time. The arrows indicate the positions of new peaks not present before plasma exposure.

The 3 mixed materials samples were imaged by SEM before and after deuterium exposure (Figure 5). The first (a) is the pure tungsten sample; it presents a real surface change, with disappearance of the roughness and a molten aspect that could have a link with phase changes that we can see in Figure 4. The second (b) is the binary compound (W/8%atMg) sample. Before exposure, the surface is flat and has few cracks. Thermal cracking appears after exposure. This is because of deuterium loading leading to large strains and aging of the samples. The black spots appearing are due to SEM chemical contrast between W and Mg, and could be interpreted as the resurgence of the latter to the surface. The third sample (c) is the ternary compound (W/4%atMg/4%atC) sample. Interpretation is the same as for sample (b). The presence of carbon manifests itself through a much larger porosity than for the sample containing only magnesium.

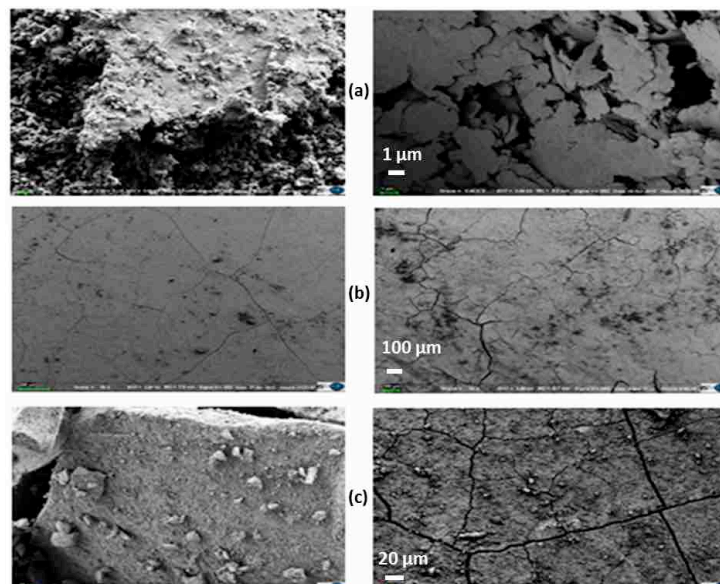


Figure 5: SEM micrographs showing samples before (left) and after plasma exposure (right). Each panel pair is shown at the same magnification. Sample (a): pure W; sample (b): W/Mg8%at; sample (c): W/C4%at/Mg4%at.

## 5. Conclusion

In this paper, we have presented briefly the work performed at LSPM using the CASIMIR II ECR plasma device, and a companion monosource prototype, aimed at plasma-wall interaction studies of fusion-relevant material mixes, concerning dust formation, hydrogen retention, and material changes due to plasma exposure. To date we have synthesized, by mechano-synthesis, a wide variety of tungsten-rich mixtures (with small atomic percentages of C and Mg, as a stand-in for Be) and exposed them to a deuterium plasma in the CASIMIR device and in our single source reactor. Characterization of these samples showed the appearance of new phases of W and the resurgence of the Mg from the W matrix after plasma exposure. In the single source reactor, we also witnessed growth of carbon nanoparticles, in a plasma dominated by positive ions, which is rather unusual and will require further investigation. We have also investigated the changes in specific surface area as function of carbon and magnesium concentrations included in the tungsten matrix. The pore size distribution of the powders showed that the majority of the pores (80%) are meso- and macro-pores, promoting surface adsorption processes.

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