

**National Research Nuclear University MEPhI**  
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# **Deuterium retention, desorption and migration in plasma facing materials for fusion devices**

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## **Introduction**

Plasma-wall interaction is recognized as one of the key issues on the way to fusion power devices. Material, design and operation conditions of plasma facing components (PFC) are strongly discussed. In fusion devices, PFC should survive under extreme heat loads, have a reasonable erosion rate, be compatible with the plasma, and comply with safety requirements.

Investigation of hydrogen isotopes retention, desorption and migration in PFC is very important for the safety issues, since the total tritium inventory in the machine and permeation into the coolant system is restricted by safety regulations. On the other hand, the wall of fusion devices can accumulate much more hydrogen than the amount involved in plasma discharges. This can cause serious problems in the case of uncontrollable gas release from the wall during transient events. Therefore, characteristics of hydrogen trapping and release from materials are also very important for hydrogen recycling control and plasma operation in general. IAEA launched recently several coordinated research projects devoted to tritium retention and plasma-wall interaction with candidate PFC materials.

Various materials are considered for applications in fusion devices. Serious progress in fusion devices has been achieved using carbon materials due to a low  $Z$ , sustainability to high temperatures and absence of melting. The international experimental reactor ITER is planned to operate with Be first wall and W divertor. Beryllium has also a low  $Z$  and high oxygen getter characteristics. Tungsten has a very high melting temperature, and a high sputtering threshold.

One of the innovative concepts is the use of liquid metals as PFC that give an opportunity to create a renewable surface. This is especially important for steady state operation of fusion devices. The most perspective is, probably, lithium due to a low  $Z$  and good getter properties. The high resistance under high heat loads and many positive effects of lithium on plasma operation in tokamaks have been demonstrated already.

This work is devoted to investigation of deuterium retention, desorption and migration in main candidate PFC materials. Main regularities in deuterium bulk retention and accumulation in co-deposits and the capability of thermal desorption spectroscopy, as the main tool to investigate these topics in this work, are discussed.

## **Experimental methods**

### **a. TDS**

Most of the experiments in this thesis work have been done using thermal desorption spectroscopy (TDS). There exist many other methods, but TDS gives information not only about deuterium in the limited surface layer, but also about D accumulated deeply in the bulk. In general, it is rather easy method. The sample should be heated up gradually, and the dynamics of the deuterium (or any other gases) release is analyzed. However, one can get a lot of information from TDS in some cases.

First of all, TDS gives the total D retention and characteristic temperatures of D release from the material. In addition, various parameters of hydrogen interaction with materials can be also derived.

Deuterium desorption from endothermic metals is often limited by D detrapping from various kinds of lattice defects. In this case TDS spectra usually consist of many peaks and one can get information about detrapping energies. For this purpose, one has to analyze a set of

experiments. Varying the heating rate for several identical samples, one can get the detrapping energy analytically. In other cases, one should use computer codes and various assumptions.

In the case of exothermic metals, TDS spectra often have one major peak that is usually attributed to surface processes. Characteristics of the surface can be derived from these experiments.

The probe method can be used for analysis of evolution of radiation defects in materials. In this case, the preliminary damaged sample is filled by low energy ions with a small fluence. TDS spectra are used as a characteristic of defect structure.

Four different installations equipped with TDS analysis has been constructed and used in experiments: 1 – TDS stand for analysis of samples irradiated outside the device (MEPhI), 2 – Ion beam experiment with in-situ TDS “MEDION” (MEPhI), 3 – Multifunctional installation “MD-2” for experiments on deuterium co-deposition with materials (MEPhI), 4 – TDS stand for Be samples analysis (VNIINM). All installations are different, but they have also several general features. Serious attention was paid to minimize the background and provide UHV conditions. Most of installations are equipped with a transfer chamber for sample loading. In the case of MEDION, the low background was achieved due to the differential pumping system and the low D pressure during irradiation. All installations were calibrated using a precise baratron and a system of valves.

#### **b. Ion-driven permeation**

All experiments on ion-driven permeation have been done at the PERMEX facility in the Max Planck Institute for Plasma Physics (Garching, Germany). The experiment was optimized to perform long time irradiation, because the experiments took a few days in some cases. From the analysis of the dynamics of deuterium permeation through materials one can get characteristics of deuterium transport in the material, trapping efficiency of lattice defects and characteristics of surface.

Additionally, the effect of surface layer modification (impurity implantation, coatings, etc.) on deuterium bulk retention can be investigated, since the deuterium flux into the bulk determines the retention in many materials at high fluences.

#### **c. Ion beam analysis**

Rutherford Backscattering Spectroscopy (RBS) and Nuclear Reaction Analysis (NRA) have been used for the surface composition analysis and deuterium retention investigations. All measurements have been done in Max Planck Institute for Plasma Physics (Garching, Germany) at the TANDEM accelerator. The deuterium retention in the top layer (6  $\mu\text{m}$  in depth for W) of materials was measured using the  $\text{D}(^3\text{He},\text{p})^4\text{He}$  reaction. Varying the energy of incident  $^3\text{He}^+$  ions in the range of 0.7 – 4.0 MeV, the deuterium depth profile was also derived. NRA was used also for the determination of the carbon amount using the  $^{12}\text{C}(^3\text{He},\text{p})^{14}\text{N}$  reaction.

Lithium amount determination was done by two methods: using the nuclear reaction  $^6\text{Li}(^3\text{He},\text{p})^8\text{Be}$  for the second Li isotope (natural amount – 7.5 %) and by RBS with 2.7 MeV protons using the enhanced Non-Rutherford cross section.

## **Bulk retention**

The highest deuterium concentration in plasma irradiated material is usually in the implantation range close to the surface. However, the integral deuterium amount in the bulk even with a relatively low concentration can be much higher, especially at high fluences. If one assumes that the concentration of traps  $n_t = 0.05$  at.% in 1 cm thick material and 100% population of traps by deuterium and tritium (D:T= 1:1), the areal concentration of tritium will be  $0.75 \text{ g/m}^2$ . This is equivalent to the inventory of 750 g in a machine with a PFC surface area of  $1000 \text{ m}^2$ . ITER has approximately  $850 \text{ m}^2$  and the administrative limit for ITER is 1kg.

Most of PFC candidate materials are endothermic (with the positive heat of hydrogen solution  $Q_s > 0$ ) for hydrogen and the equilibrium solute concentration is rather low. However, the hydrogen isotopes retention can be high in the presence of radiation damage or natural defects. The deuterium concentration in radiation damaged W can reach several at.%. Neutrons produced in fusion reactions will produce damage in the whole volume of PFC, therefore, the bulk retention can be a problem for future fusion devices.

The population and the filling rate of radiation defects depends on many factors, the PFC temperature, binding energies and concentrations of traps, diffusivity, surface conditions, and the incident ions flux and energy. Therefore, investigation of all these parameters is important to understand hydrogen isotope retention.

### **a. Tungsten**

Tungsten has a very low solubility for hydrogen. Therefore, deuterium retention in W is determined mainly by the defect structure of the material. The recombination rate at the surface is usually assumed to be extremely high. Deuterium can quickly release from tungsten just after termination of ion/plasma irradiation. However, surface influence can not always be neglected. The surface effect is especially high in the presence of impurities in the plasma. Seeding of deuterium plasma by several percent of nitrogen led to increase in D retention by 2-3 times at the fluence of  $2 \times 10^{25} \text{ D/m}^2$ . This difference increased with the fluence, because the formation the nitrogen-enriched increased the solute concentration at the implantation depth and the diffusion flux into the bulk. Nitrogen release from the surface started only at 1100–1200 K, therefore, in long term operation of fusion devices one can easily imagine the formation of such layer in some areas. On the other hand, He irradiation decreases the D retention in the bulk of W, likely, due to formation of a kind of diffusion barrier behind the implantation region.

The radiation damage drastically changes deuterium retention in tungsten. The radiation damage produced by deuterium ions in a wide range of incident energies and by MeV  $W^{6+}$  ions has been investigated. Experiments demonstrated that a remarkable deuterium amount was accumulated in damaged W even after exposure at high temperatures (up to 800 K) that is an evidence of production of defects with a high binding energy. To derive quantitative characteristics of defects experimental TDS spectra have been simulated using computer codes (TMAP7, DIFTRAP). A good agreement with the experiment was achieved assuming the traps with the detrapping energy of 1.7-2.0 eV. Similar values have been derived from ion-driven permeation experiments. These traps are assumed to be the sites at the surface of inner voids or vacancy clusters. The detrapping energy for the vacancies in W is also very high. According to our last experiments, it is about 1.55 eV.

The annealing of the radiation defects was investigated by the probe method. It was shown that at the temperatures 600-700 K vacancies are actively agglomerate to complex defects. Further increase of the temperature leads to increase of the clusters. At high

temperatures above 1400 K, annealing of these vacancy clusters is possible. In the case of the high concentration of defects, large (tens of nanometers) pores can be formed which are stable, at least up to 1800 K.

### **b. Steels**

The Reduced-activation ferritic–martensitic steels (RAFMS) with high resistance to neutron irradiation are considered as a structural material for future fusion devices. However, there are some concepts considering the use of steels as PFC. Steels have a positive heat of solution, but much lower than tungsten. The binding energies for defects are also much lower.

In general, experiments with steels demonstrated rather small deuterium retention in various experiments. Deuterium retention decreases strongly with the increase of temperature up to 450 – 500 K. TDS spectra of deuterium from the steels irradiated by plasma at low temperatures (below 450 K) have one major peak that is typical for exothermic metals. It was observed also that the position of this peak moves to higher temperatures after air exposure. Therefore, this peak was associated with the surface barrier that should be chromium oxide.

### **c. Carbon materials**

D retention in the bulk of carbon PFC materials is very specific, since most of them (fine grain graphites and carbon fiber composites) are very porous. Hydrogen diffusivity in the bulk of carbon is very small; however, hydrogen can migrate deeply through the open porosity of the material, where it can be trapped on dangling bonds. These bonds do not depend on the material structure. Therefore, TDS spectra for various carbon materials demonstrate the same peak positions. The continuous growth of deuterium retention up to highest fluences has been observed in experiments and is due to diffusion deep into material. At the surface of C materials, an amorphous layer with a high concentration of deuterium is formed. A significant part of deuterium stays in C materials up to very high temperature, above 1000 K.

## **Retention in co-deposits and erosion products**

Erosion of PFC and further co-deposition with hydrogen isotopes is a seriously contribute to the hydrogen inventory in tokamaks. For example, after operation with the full C wall (2005-2009), co-deposits with a thickness of ~50  $\mu\text{m}$  have been observed in some areas. The deuterium concentration in co-deposits is usually very high. It can be at the level of tens of atomic percent for most materials at the room temperature deposition.

The erosion rate depends strongly on the material and on the energy and flux of incident particles. For some materials, the total erosion rate can be increased strongly by chemical sputtering. During transient heat loads (ELMs) and abnormal events (major disruptions and vertical displacement events (VDE)) macroscopic particles can be ejected from the surface. This mechanism can be the main source of Be erosion and dust production. Simulations for the unmitigated major disruption in ITER predicted up to several kg of Be per single event. On the other hand, hydrogen isotope retention in these particles should be small due to the high surface temperature during these events.

### **a. C-D and C-W-D films**

Deuterium co-deposition with C and W was investigated in two very different conditions: in a stationary magnetron discharge with a relatively low incident flux and in the high quasi-stationary plasma accelerator QSPA-T (TRINITI) with very high pulsed loads (~2.5 MJ/m<sup>2</sup>,

0.5 ms). In both cases, sputtering of the pure W target led to formation of mixed C-W-D films. In spite of very different conditions and deposition rates, the D concentration in films deposited at room temperature was approximately the same, in the range of 0.1-0.4 for the wide range of parameters (C/W ratio, deposition rate).

In the case of low energy incident particles coming to the substrate, TDS spectra for C-D and C-W-D films were rather similar with the major peak at 700-800 K. For carbon, biasing of the substrate led to increase of the high temperature part (1100-1300 K) that is typical for “hard” C-D films.

The amount of deposited material at one pulse in QSPA experiments was a factor of 20 higher for C films than for W films. Therefore, the integral D retention in the film was also about 20 times higher (at room temperature).

The dependence on substrate temperature was also very different for C and W. In QSPA experiments, the D concentration in C films was nearly constant up to 450 K, and then decreased slowly with the temperature. The D concentration in W films decreased exponentially with the deposition temperature.

### **b. Beryllium**

Deuterium retention in erosion products of beryllium formed during high heat loads has been investigated in the QSPA-Be facility (VNIINM). Beryllium tiles were irradiated by ten pulses of deuterium plasma with the energy of 1 MJ/m<sup>2</sup> and the pulse duration of 0.5 ms. The base temperature before the shot was 250 or 500 C in various experiments. The upper layer of the tile was melted, and surface layer was transferred from the center to the outer regions of the target forming re-solidification streams and droplets. These droplets were collected by scraping from the surface TDS analysis.

The main part of deuterium released from the droplets in the temperature range of 900–1350 K. D release at these temperatures was not observed in ion beam experiments, however, the peaks are close to experiments on tritium release from n-irradiated Be. The total deuterium amount in various samples was varied from  $N_D/N_{Be}=3\times 10^{-6}-10^{-4}$ . Deuterium retention was found to be dependent on beryllium grade, it was higher in S-65C than in TGP-56FW by three times in average.

### **c. JET experiments**

Investigation of erosion and deposition patterns at PFCs in JET tokamak (UK) was done using ion beam analysis (RBS and NRA) in Max Planck Institute for Plasmaphysics (Garching, Germany). Two campaigns, full C machine (2005-2009) and with ITER-like wall (2011-2012) have been compared. The amount of co-deposits and deuterium inventory in them was drastically decreased in the case of ITER-like wall (first wall – Be, divertor -W). Chemical erosion of C by low energy particles is the most probable explanation for the comparatively lower Be erosion rate. This led to significant reduction in total deuterium inventory in the machine in the campaign with ITER-like wall.

### **d. Lithium**

Lithium was successfully used in many tokamaks for wall conditioning as well as a material for plasma-facing components. Large amounts of lithium can be deposited on walls of the vacuum chamber in both cases, and large amounts of hydrogen isotopes can be accumulated in deposited lithium. Co-deposition of Li and D on the Mo substrate was investigated in

conditions of deuterium magnetron discharge with liquid lithium cathode. The major part of deuterium accumulated in the film desorbed in a very narrow peak with maximum at 650-700 K. Some TDS spectra had an additional peak with maximum at 850 K, however it was not reproduced in later experiments. The deuterium amount in the film was calculated to be 10-20 at.%.

Li-D films kept in atmospheric air or in water vapor loose deuterium intensively even at room temperature. After three days of air exposure, deuterium was not found in the sample. This makes experiments with lithium samples difficult. On the other hand, exposure in water vapor can be recommended for tritium removal from lithium.

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