

## **Studies of hydrogen interaction with fusion relevant material**

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### **Introduction**

Processes occurring in the edge plasma and on the wall of fusion reactor are important for stable behaviour of fusion core plasma and also for determining power load to the wall. Wall material of fusion reactor has to sustain high temperature and particle flux, needs to have good mechanical properties, low erosion and low hydrogen retention. It is also necessary to understand long-term and short-term hydrogen isotope retention characteristics especially when dealing with tritium. Hydrogen atoms and molecules are present in the edge plasma together with ions and electrons. Interaction of neutral hydrogen with wall material and ions coming from the core plasma has to be considered. A lot of studies and different kind of experiments have been made but there are still a lot of discrepancies between measurements and numerical parameters, determining hydrogen interaction with material [1-2]. There is still lack of experimental results when dealing with atoms and molecules, especially concerning molecules formed by recombination of atoms on the surface.

We have constructed a special cell in order to study interaction of neutral hydrogen with fusion relevant material and retention of hydrogen in it. These studies are performed by *in-situ* measurement of H and D concentration by means of ion beam analytical method ERDA (Elastic Recoil Detection Analysis) [3]. We have investigated processes on tungsten in more details as it is being largely used in fusion devices.

Our next objective is to connect exposure cell with our analyzing tool for vibrational spectroscopy of hydrogen molecules [4]. By combining these two set-ups one would get simultaneous information about the atmosphere above the sample and hydrogen concentration in the sample and therefore a new insight into surface processes.

### **Experiment**

Hydrogen exposure cell (HEC) allows real time *in-situ* measurements of hydrogen concentration depth profile. Cell is schematically shown in figure 1. It is mounted in the vacuum chamber where ERDA measurements are performed on 2 MV Tandem accelerator

at the Jožef Stefan Institute (JSI) in Ljubljana. Sample is mounted on the flat side of HEC. It is heated from the backside and its temperature is monitored by a thermocouple, mounted on the sample holder plate.

Temperature of the cylindrical envelope of HEC is controlled by

water cooling. There are two rectangular windows on the cylindrical envelope, one is the entrance window for incoming high energy probing beam and another window is to allow recoiled particles to reach detector. In order to reduce gas leak out of the cell, both windows are closed with thin Al foils. Gas inlet and pressure measurement ports are on the bottom of the cell. Tungsten filament is mounted on the top of the cell. When heated, this filament is used to dissociate hydrogen molecules.

We use ion beam analytical method ERDA for real-time depth profiling of hydrogen concentration in a sample in order to study processes occurring on a surface and in bulk of sample material. As the probing beam  $4.2 \text{ MeV } ^7\text{Li}^{2+}$  ion beam is used. Energy of recoiled H and D ions is detected in order to determine the depth profile of hydrogen in the sample. By successive ERDA spectra recording processes on the surface and in bulk are followed. Time resolution needed for sufficient spectrum statistics is 10-15 minutes in our case, depending on the hydrogen concentration in a sample.

Input gas ( $\text{H}_2$  or  $\text{D}_2$ ), pressure, sample temperature and degree of dissociation can be varied in experiment. For a set of measurements these parameters are kept constant, so that time evolution of processes can be followed. Surface processes such as adsorption of atoms, isotope exchange, recombination of atoms, dissociative adsorption of molecules and desorption of molecules can be followed in time and also diffusion of atoms into the bulk and to the surface.

## Results

ERDA spectra obtained with Ti sample (annealed, purity 99.6%) during its heating

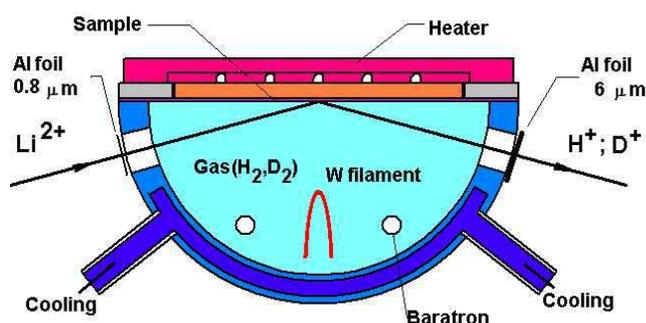


Figure 1: Schematic drawing of HEC

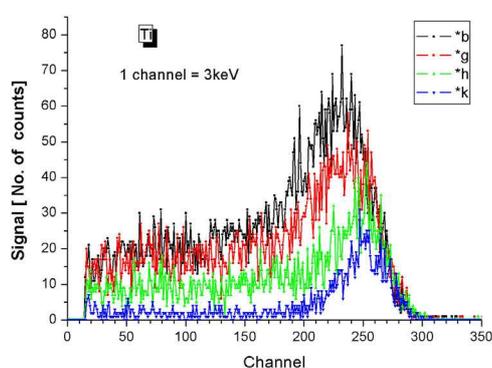


Figure 2: ERDA spectra of H in Ti during heating.

are shown in figure 2. Temperature was gradually increased from room temperature (spectrum “g”) to 130 °C (spectrum “k”). Spectrum “b” was made pre-heating in vacuum. Peak at around channel number 250, 750 keV, is due to recoiled H from the surface while the signal at lower channels (lower energy) is due to H from bulk. One can see that hydrogen concentration decreases with time due to the heating of the sample. Atoms diffuse from the bulk to the surface and desorb from the surface as molecules.

In contrast to Ti, W (rolled, purity 99.95%) has very low bulk concentration and so has only surface H peak as seen in figure 3 (black line). This is characteristic for these two elements due to low and high hydrogen solubility for W and Ti, respectively [6].

Typical ERDA spectrum of hydrogen in tungsten in vacuum, black line, (“a” in figure 4), during sample exposure to deuterium molecules, red line, (“n” in figure 4) and during exposure to partly dissociated deuterium gas, blue line, (“d2” in figure 4) are shown in figure 3. Surface concentrations of H and D for each individual experimental spectrum were

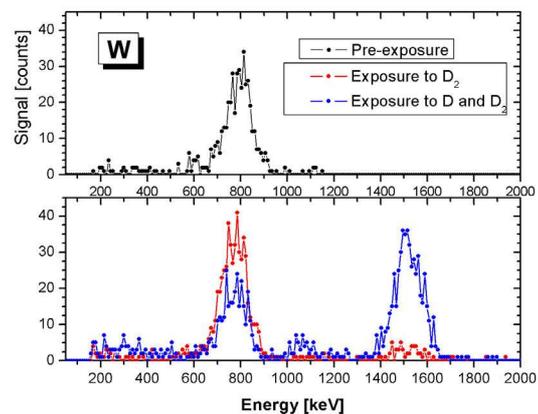


Figure 3: ERDA spectra of H (surface peak  $\approx$  800 keV) and D (surface peak  $\approx$  1500 keV).

obtained by fitting with simulated spectrum from SIMNRA [7]. Results of analysis of successive spectra are shown in figure 4. Initially sample was heated in background vacuum above 100°C. H and D surface concentrations labelled with “g”-“n” present consecutive spectra where W sample was exposed to deuterium molecules. Sample was exposed to D<sub>2</sub> for about three hours. Concentration, i.e. surface signal, of deuterium gradually raises, therefore deuterium molecules have adsorbed on the surface. A model for dissociative adsorption of deuterium molecules on tungsten was developed and fits the measured data rather well.

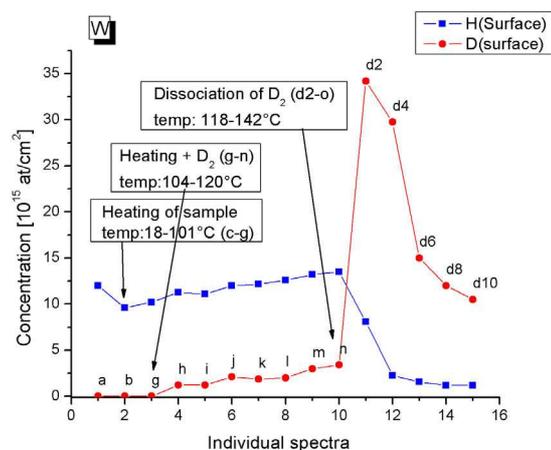


Figure 4: H and D surface concentration of consecutive ERDA spectra at specified

Another phenomenon observed on W is illustrated in figures 4 (“d2”-“d10”) and 5 (“o”-“v”). Sample was in both cases exposed to partially dissociated gas. The D concentration increased and simultaneously H concentration decreased almost instantaneously after turning on the dissociation filament. Presumably deuterium atoms interact with hydrogen atoms, recombine and desorb as HD molecules from the surface. An isotope exchange happens. Sample temperature was not constant during measurement in figure 4, and consecutively D concentration decreased after initial spectrum, due to temperature rise. Temperature was constant, 353 K in another set of measurements, figure 5, and stationary conditions were reached. Moreover exchange of H with D

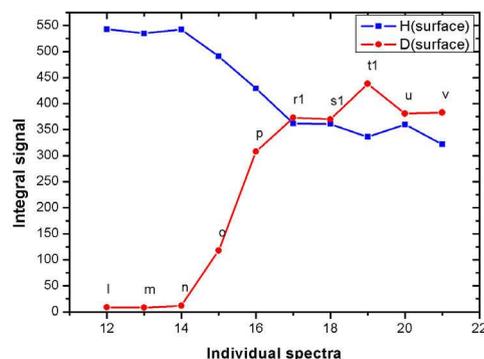


Figure 5: Surface integral (i.e. concentration) of individual spectra of H and D of consecutive spectra.

was faster for the first measurement due to higher pressure and higher content of D atoms. It is not clear yet why concentration of H did not diminished more in second measurement (figure 5) as has in the case in figure 4. We suspect that it is due to impurities on the surface and that one part of hydrogen was more strongly bounded, but further investigation is needed. The same phenomenon was observed when sample was exposed to H atoms, where on the other hand they have exchanged D atoms on the surface.

- [1] R. A. Causey, *J. Nuclear Material*, **300** (2002) 91-117
- [2] E. Serra et al. *J. Nuclear Material*, **255** (1998) 105-115
- [3] J Tirira, Y. Serruyis, P. Trocellier, *Forward Recoil Spectroscopy*, Plenum Press (1996) New York
- [4] S. Markelj et al. and Čadež et al., 27<sup>th</sup> Int. Conf. on Phys. in Ion. Gasses, Eindhoven, the Netherlands, 18-22 July, 2005, Proceedings, 08-237 and 02-236
- [5] E. Fromm, *Kinetics of Metal-Gas Interactions at Low Temperatures*, Springer-Verlag Berlin Heidelberg (1998) Berlin
- [6] Y. Fukai, *The Metal-Hydrogen System*, Springer-Verlag Berlin Heidelberg (2005) Berlin
- [7] <http://www.rzg.mpg.de/~mam/>