


# PRESENTATION OF THE RESULTS FOR DEUTERIUM RETENTION AND THERMAL RELEASE IN A NEW TYPE OF LOW ACTIVATION FERRITIC-MARTENSITIC STEEL EUROFER

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## *Abstract:*

*This work presents the results of the investigation into deuterium retention and thermal release in low activation ferritic-martensitic steel EUROFER and its main components, Fe and Cr, using ion implantation, nuclear reaction depth profiling and the thermal desorption spectrometry technique. The samples were exposed to deuterium ECR plasma of  $6.5 \times 10^{24} \text{ Dm}^{-2}$  fluence. Our NRA results show that, at 300 K, most of deuterium is trapped in the near-surface region. A part of the implanted deuterium diffuses into the bulk and is trapped by defects beyond the implantation range. The results of the TDS measurement of the Cr sample confirm the formation of an ordered Cr-hydride phase.*

*Key words: nuclear reactor materials, radiation effects, diffusion, nuclear resonances.*

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## Introduction

Reduced activation ferritic-martensitic (RAFM) steels are presently the leading candidate materials for the first wall and breeding blanket structural application for ITER and future power plants. This class of steels has been selected mainly because of their improved radiation resistance and the potential of reduced activation. These materials have been developed by replacing alloying elements like Mo, Nb and Ni in the commercial martensitic steels by other elements which exhibit faster decay of induced radioactivity such as Ta, W and V (Kohyama, 1996, pp.138-147). They also show excellent mechanical and thermo-physical properties (Lindau, 2005, pp.989-996) and swelling resistance (Baluc, 2007, pp.33-41).

EUROFER'97 is the RAFM steel developed for the requirements of the European fusion technology program (Gasparoto, 2003, pp.129-137). Its chemical composition has been designed to achieve good activation properties and irradiation resistance (Van der Schaaf, 2003, pp.197-203), (Tavassoli, 2004, pp.257-262). It is expected that Fe and Cr, as its main components, will have the most impact on its overall properties. As a structural material for the future fusion application, EUROFER will be exposed to intense fluxes of energetic hydrogen isotope ions and neutrals as products of nuclear reactions (Yao, 2005, pp.1285-1291). Retention and further transport of the hydrogen isotopes will have implications on material stability, environmental safety and recycling to the plasma. Hydrogen transport and trapping in EUROFER have been recently studied by means of the gas evolution permeation technique (Esteban, 2007, pp.473-477) assuming that martensitic steel is close to a bcc structure where H occupies only tetrahedral interstitial positions (Seeger, 1976, pp.137-138).

An experiment has shown that the H transport is limited to diffusive phenomena rather than surface effects, though a complete lack of surface effects cannot be proven. Activation energy ( $E_a$ ), found to be close to 59 kJ/mol, corresponds to high angle grain boundaries (Esteban, 2000, pp.34-41). This indicates that the governing cause of the H trapping in EUROFER may be grain boundaries and/or fine martensitic laths, characteristic for this type of steel.

This paper presents a study on the retention and thermal release of deuterium in Fe, Cr and EUROFER irradiated with D ions by means of ion beam analysis techniques and thermal desorption spectroscopy (TDS).

## *Experimental details*

Polycrystalline Fe and Cr foils of quoted purity 99.99%, 0.5 mm in thickness delivered by Goodfellow and EUROFER steel foil, 1 mm in thickness, were used in this work. Note that the main alloying elements of the EUROFER are Fe and 9.2 at.% Cr. Rectangular-shape samples 12×15mm in size were cut from the foils and then mechanically polished prior the measurements.

The samples were exposed to deuterium plasma in an electron-cyclotron-resonance source PlaQ in IPP, Garching (Manhard, 2011, pp.015-010). Thus source is equipped with a biased sample holder and is well suited for the implantation of deuterium ions. A PlaQ experiment consists of a stainless steel chamber where plasma is confined in a cylindrical steel mesh cage with a height of 15 cm and a diameter of 14 cm. A plasma beam consisting mainly of  $D_3^+$  ions reaches a sample holder through a circular aperture of the steel plate placed at the bottom of the cage. The whole sample is homogeneously irradiated. A typical achievable deuteron flux is in the range  $10^{19}$  to  $10^{20}$  D m<sup>-2</sup>s<sup>-1</sup>. Achievable energies range from several keV up to 200keV per deuteron.

The surface morphology of the samples subjected to the D plasma exposures was examined using scanning electron microscopy.

Deuterium depth profiles were determined using the nuclear reaction analysis technique (NRA) at a Tandem Accelerator in IPP, Garching. The analysis was carried out by directing a  $^3\text{He}$  beam normally on the samples and measuring the proton yield from the induced nuclear reaction  $D(^3\text{He},p)^4\text{He}$  as a function of an incident  $^3\text{He}$  energy beam. Series of seven different  $^3\text{He}$  energies were used in the range from 0.5 to 4.5 MeV, following the reaction cross section which was obtained from the literature (Alimov, 2005, pp.169-175) and which exhibits a maximum at around 0.65 MeV. The computer program NRADC was used for the analysis of the data (Schmid, 2012, pp.64-71). Here, a deuterium concentration profile was extracted from the proton yield using an iterative deconvolution procedure.

Protons from the nuclear reaction  $D(^3\text{He},p)^4\text{He}$  were counted with a Si surface barrier detector which subtended a solid angle of 0.106 Sr at 150° from the incident-beam direction. This detector was covered with 0.4 mm of Al foil to range out unwanted charged particles. The detector acceptance angle was calibrated by Rutherford backscattering from a C film on a Be substrate, with the intervening Al foil removed.

Thermal desorption spectroscopy (TDS) measurements were performed with a custom built set-up in INN Vinča, Belgrade, consisting of a quartz tube placed inside the furnace. The samples were outgassed at 293 K under  $1.9 \times 10^{-8}$  Pa and subsequently subjected to a linear

heating rate of 20 K/min to 1273 K under high vacuum. The partial pressures of H<sub>2</sub>, H<sub>2</sub>O and D<sub>2</sub> released were monitored with a quadrupole mass spectrometer (Extorr 3000).

## Results and Discussion

### *D retention in Fe*

In the first set of experiments, two Fe samples were used: the first Fe sample as prepared, i.e., mechanically polished, and the second one additionally annealed at 1120 K for 1 h in vacuum under  $\sim 10^{-5}$  Pa to enlarge grains and remove the cold-work damage. The SEM micrographs of the samples show that in the case of the sample without previous heat treatment, no pronounced surface structures were observed. On the other hand, the microstructure of the annealed sample consists of anisotropically distributed grains of white borders. The mean grain size observed by SEM was 50-100  $\mu\text{m}$ .

After that, both samples were simultaneously exposed to deuterium plasma in PLAQ at room temperature. The energy of deuterium ions was  $< 5$  keV/D and the irradiation flux was  $5.6 \times 10^{19} \text{ Dm}^2\text{s}^{-1}$ . The total ion fluence was  $4 \times 10^{24} \text{ Dm}^{-2}$ .

The calculations performed by the SRIM code (Ziegler, 2004, pp.1027-1036) indicate that this energy corresponds to the approximate deuterium penetration depth (projected range) of 400 nm. A displacement damage (damage distribution profile) peak is located at 50 nm from the surface. The projected range of D is increasing monotonically with increasing ion energy. Along its trajectory, ion is producing damage which can serve as trapping sites for deuterium. In most ion irradiations in Fe, the damage region is usually within a few micrometers of the irradiated surface (Horton, 1981, pp.1343-1347). The defect structures significantly depend on the distance from the irradiated surface. However, a comparison to binding energies of deuterium to various traps in iron and ferritic-martensitic steels indicate that probably in both cases vacancies are the major traps (Forcey, 1990, pp. 357-363), (Pressouyre, 1979, pp.1571-1573).

Deuterium is highly mobile in Fe with a diffusion activation energy of  $\sim 0.05$  eV and a room-temperature diffusivity of  $\sim 10^{-9} \text{ m}^2\text{s}^{-1}$ .

Hydrogen diffusivity in iron and martensitic MANET II shows a pronounced dependence on gas pressure and a deviation from Arrhenius type temperature dependence at temperatures below 300°C (Wedig, 1997, pp.138-146). For the case of hydrogen transport and trapping in austenitic stainless steels, Langley (Langley, 1984, pp.622-628)

proposed a two-region diffusion model where the solid is considered to consist of two components, a surface layer and a bulk material. In this model, the surface layer is characterized by a smaller diffusion coefficient than the bulk. This was confirmed by the measurements of Wilson and Baskes (Wilson, 1978, pp.291-297) on deuterium trapping in 316 stainless steels. Their desorption data showed that there are two dominant mechanisms: bulk migration of mobile deuterium atoms with  $\sim 0.6$  eV migration energy and release from near surface traps with a net detrapping energy of  $\sim 0.9$  eV. Here, the diffusivity was determined to be  $D_0 = 0.12 \text{ cm}^2/\text{s}$  and  $E_{\text{activ}} = 0.61$  eV.

After the plasma exposure, the concentration of D in each sample was determined by the nuclear reaction analysis (NRA). The NRA results are shown in Figure 1.

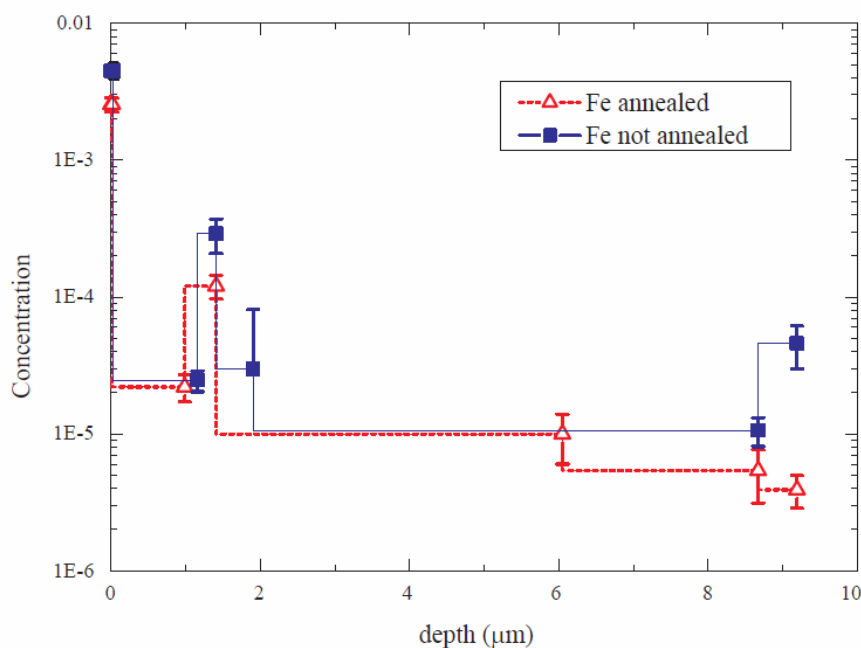


Figure 1 – Deuterium depth profile for the Fe samples exposed to deuterium plasma at 300 K to a fluence of  $F = 4 \times 10^{24} \text{ Dm}^{-2}$  (-) not annealed and (----) annealed in vacuum at 1120 K for 1 h

Slika 1 – Dubinski profil deuterijuma za uzorke Fe ozračene plazmom deuterijuma na 300 K i pri fluksu od  $F = 4 \times 10^{24} \text{ Dm}^{-2}$  (-) nezagrevani i (----) zagrevani u vakuumu na 1120 K u trajanju od 1 h

Рис. 1 – глубинный профиль дейтерия образца Fe, облучённый дейтериевой плазмой на 300K и при флюенсе  $F = 4 \times 10^{24} \text{ Dm}^{-2}$  (-) не разогретый и (----) разогретый в вакууме на 1120 K в течение 1 часа

The depth profiles in the both samples are characterized by a very high concentration of deuterium in the near surface region, probably due to the trapping at the plasma irradiation induced defects. At depths above 1  $\mu\text{m}$ , the concentration exponentially decreases due to the D diffusion into the bulk. The only unusual feature is the increase of the concentration in the bulk which appears for both samples at around 1  $\mu\text{m}$  and will be discussed later in the text.

### *D retention in Fe, Cr and EUROFER*

In the second set of measurements, five different samples were used:

- two Fe samples (one unprocessed and another additionally coated with a 130 nm thick Au layer on one side)
- two EUROFER samples (one unprocessed and another additionally coated with a 130 nm thick Au layer on one side)
- one Cr sample, as prepared

The reason for using Au coating is that a hydrogen barrier is planned to be placed between the plasma facing material and EUROFER which is planned to be used as the first wall in future fusion reactors. This barrier will prevent tritium transport and retention through the first wall. The study on alumina (Levchuk, 2004, pp.103-106) has shown that this material is a promising candidate for this purpose. It showed a significant decrease of the permeation flux compared to the bare material ( $\sim 10^3$  factor) where surface effects seem to play an important role. With this experiment, we wanted to test Au as a potential diffusion barrier for D in a combination with EUROFER as a substrate. Similarly to the mentioned study, a very thin Au coating was used to prevent the neutron induced activation from Au.

All samples were annealed at 873 K for 3 h in vacuum under  $\sim 10^{-5}$  Pa to enlarge grains and remove the cold-work damage. SEM micrographs of the EUROFER and Cr samples show that Fe samples look basically the same as in the first measurement (the formation of a grain structure is observed after the annealing procedure). In the case of the EUROFER sample, the structure of the alloy did not change significantly after the heating procedure. In the case of the Cr sample, a network crack structure was observed before the annealing procedure. The microstructure of the annealed sample consists of additional anisotropically distributed grains. The mean grain size observed by SEM was 1  $\mu\text{m}$ .

After that, all samples were exposed simultaneously to deuterium plasma in PLAQ at room temperature. The energy of deuterium ions was 38 keV/D and the irradiation flux was  $9 \times 10^{19} \text{ Dm}^{-2}\text{s}^{-1}$ . The total ion fluence was  $6 \times 10^{24} \text{ Dm}^{-2}$ .

The calculations performed by the SRIM code indicate that this energy corresponds to the approximate deuterium penetration depth of 500 nm in Fe. A displacement damage peak is located at 300 nm from the surface. Deuterium trapping in stainless steel occurs in radiation induced damage sites (e.g., vacancies, interstitial loops, etc.), in voids, grain boundaries and impurities (Wilson, 1981, pp.453-463). The previous measurements indicate that two dominant radiation damage traps exist with binding energies of  $\sim 0.1$  and  $0.3$  eV (Bohdansky, 1980, pp.594-600).

After the plasma exposure in our experiment, the D concentration in each sample was determined by means of the nuclear reaction analysis (NRA). Figure 2 gives the D depth profile in the Fe samples. The depth profile is similar as in the first experiment except that an unusual step-structure appears at a greater depth of  $\sim 4\mu\text{m}$ . This indicates the structure is not constant and is related to the energy of the injected ions.

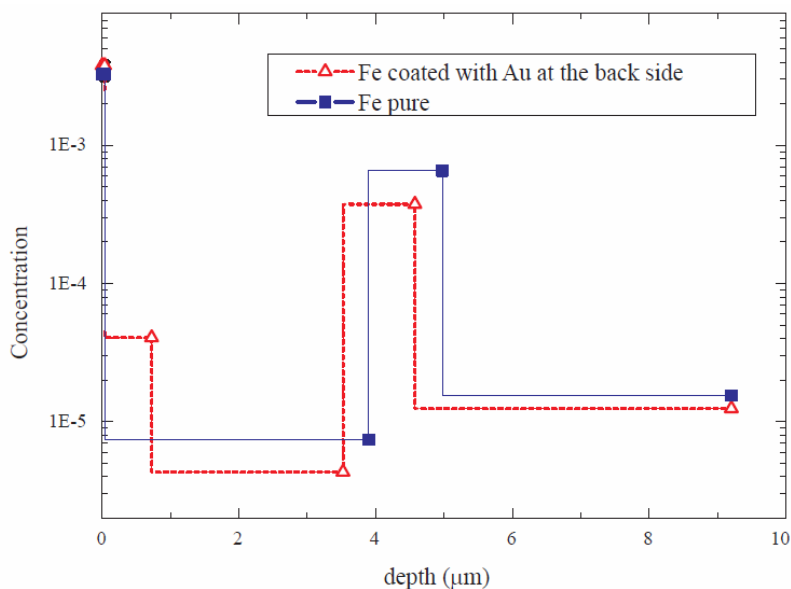


Figure 2 – Deuterium depth profile for the Fe samples annealed in vacuum at 873 K for 1h and then exposed to deuterium plasma at 300 K to a fluence of  $F = 6 \times 10^{24} \text{ Dm}^{-2}$

Slika 2 – Dubinski profil deuterijuma za uzorke Fe zagrevane u vakuumu na 873K u trajanju od 1h, a zatim ozračene plazmom deuterijuma na 300 K pri fluksu  $F = 6 \times 10^{24} \text{ Dm}^{-2}$

Рис. 2 – глубинный профиль дейтерия образца Fe, разогретый в вакууме на 873 K в течение 1 часа, а затем облученный дейтериевой плазмой на 300K при флюенсе  $F = 6 \times 10^{24} \text{ Dm}^{-2}$

Figure 3 and Figure 4 give the D depth profile in the EUROFER and Cr samples. The depth profile of EUROFER shows a similar structure as in the case of the pure Fe samples except that the concentration is about

two times higher than in Fe. The peculiar peak-like structure in the concentration profile of EUROFER probably comes from Fe as the main constituent of this steel. Our measurements are supported by the results of (Spitsyn, 2014, pp.561-567) who have performed a similar kind of measurements on the RUSFER (a Russian version of EUROFER). They have observed similar structures in the depth profiles measuring them for different temperature and energy series. They also showed that, by increasing the temperature, the concentration decreases and already at 200° is below the sensitivity of the detector.

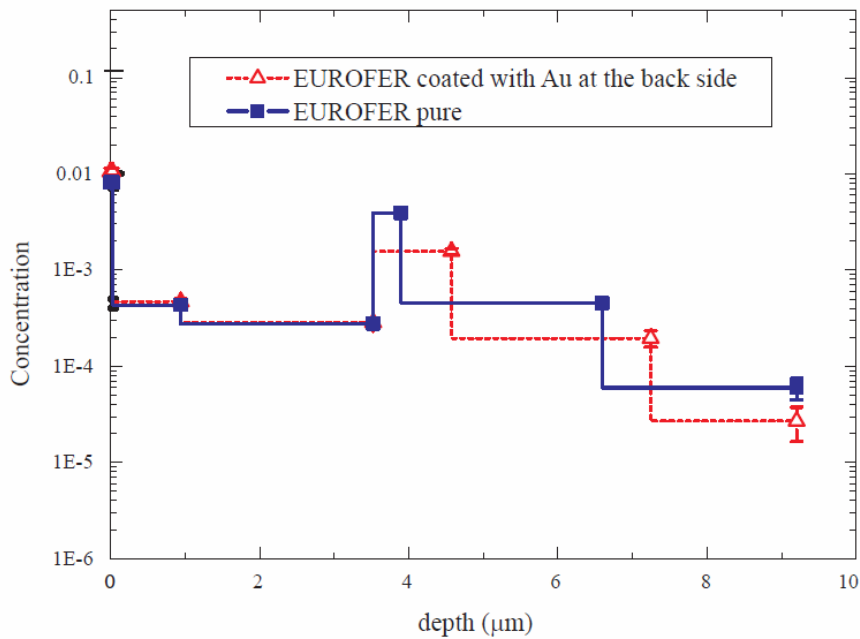


Figure 3 – Deuterium depth profile for the EUROFER samples annealed in vacuum at 873 K for 1h and then exposed to deuterium plasma at RT to a fluence of  $F = 6 \times 10^{24} \text{ Dm}^{-2}$  (-) not coated with Au and (---) coated with a 130 nm thick Au layer

Slika 3 – Dubinski profil deuterijuma za uzorke EUROFER-a zagrevane u vakuumu na 873K u trajanju od 1h, a zatim ozračene plazmom deuterijuma na sobnoj temperaturi i fluksu  $F = 6 \times 10^{24} \text{ Dm}^{-2}$  (-) neobloženi slojem Au i (---)obloženi slojem Au debljine 130 nm  
 Puc. 3 – глyбинный профиль дейтерия образцов EUROFER, разогретых в вакууме на 873 K в течение 1 часа, а затем облученных дейтериевой плазмой на комнатной температуре при флюенсе  $F = 6 \times 10^{24} \text{ Dm}^{-2}$  (-) необлицованные Au i (---)облицованные Au coated толщиной 130 nm

The D concentration profile in the Cr sample is characterized by a very high concentration peak in the near surface region (almost 10 magnitudes of order higher than in the pure Fe samples!) and the further



exponential decay of the concentration in the bulk. This suggests that in the case of Cr there is probably the formation of the chromium hydride on the surface of the sample.

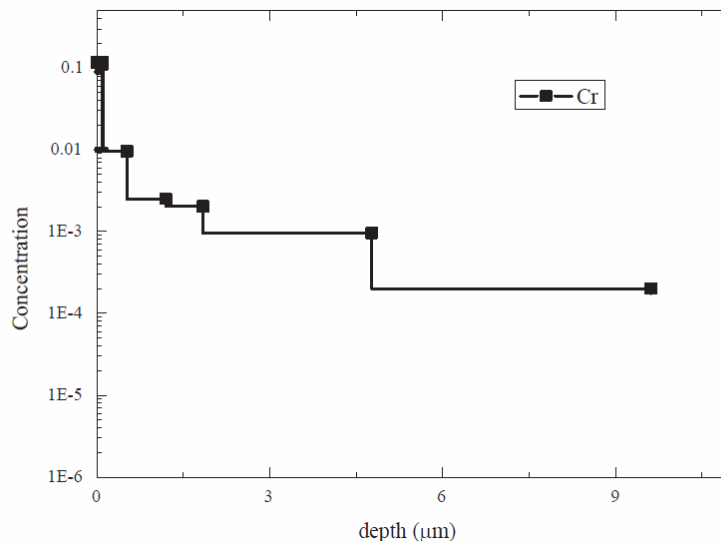


Figure 4 – Deuterium depth profile for the Cr samples annealed in vacuum at 873 K for 1h and then exposed to deuterium plasma at 300 K to a fluence of  $F = 6 \times 10^{24} \text{ Dm}^{-2}$

Slika 4 – Dubinski profil deuterijuma za uzorke Cr zagrevane u vakuumu na 873K u trajanju od 1h, a zatim ozračene plazmom deuterijuma na 300 K pri flukusu  $F = 6 \times 10^{24} \text{ Dm}^{-2}$

Рис. 4 – Глубинный профиль дейтерия образца Cr, разогретый в вакууме на 873 K в течение 1 часа, а затем облученный дейтериевой плазмой на 300K при флюенсе  $F = 6 \times 10^{24} \text{ Dm}^{-2}$

### Formation of Cr-hydride

Metallic chromium metal normally has a body-centered cubic crystal structure. In the interaction with hydrogen, it forms chromium-hydride which appears in two phases: face centred cubic (fcc)  $\text{CrH}_2$ , and hexagonal closed packed (hcp)  $\text{CrH}$ , both having a high hydrogen concentration (Baranowski, 1978, pp.157-200). Both these hydrides were originally formed by the cathodic electrodeposition of Cr (Snively, 1947, pp.537-577), but one of them was later synthesized by a direct reaction with high pressure  $\text{H}_2$  gas as well (Baranowski, 1972, pp.525-527). The fcc hydride prepared this way was reported to be unstable whereas the hcp hydride could be stored indefinitely under ambient conditions. In addition, recent X-ray diffraction measurements with the use of

synchrotron radiation (Fukai, 2002, pp.1079-1084) gave evidence of superabundant vacancy (SAV) formation, as observed in several other metal-hydrogen alloys.

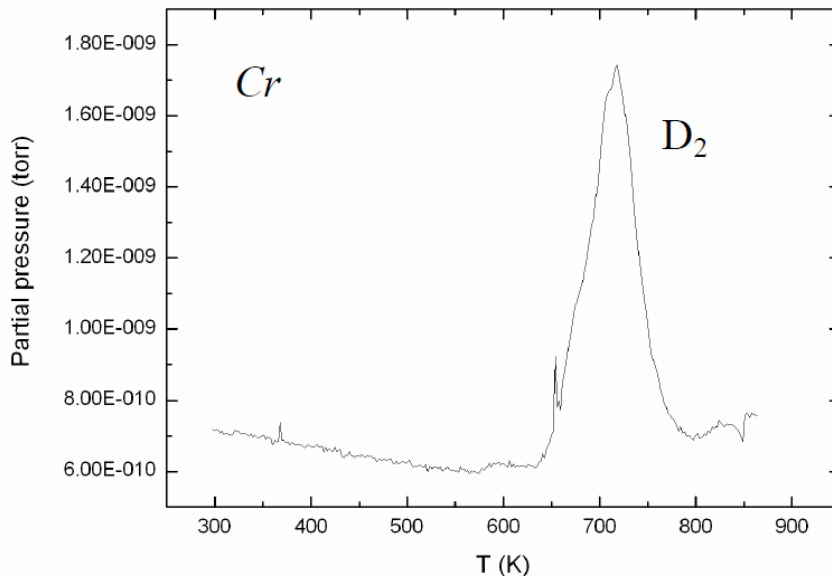


Figure 5 – Thermal desorption spectra of deuterium from Cr exposed to deuterium plasma at 300 K with a fluence of  $F = 6 \times 10^{24} \text{ Dm}^{-2}$ . The temperature ramp was 0.3 K/s.

Slika 5 – Spektar termalne desorpcije deuterijuma iz uzorka Cr izloženog plazmi deuterijuma na 300 K i pri fluksu  $F = 6 \times 10^{24} \text{ Dm}^{-2}$ . Temperaturski koraci bili su 0.3 K/s.

Рис. 5 – Спектр термальной десорбции дейтерия образца Cr, облученного дейтериевой плазмой на 300 K при флюенсе  $F = 6 \times 10^{24} \text{ Dm}^{-2}$ . Температурный рост составлял 0,3 K/c.

We have performed TDS measurements of the Cr sample in order to reveal the presence of the hydride phase on the surface of the sample. The result of this measurement is shown in Figure 5. Deuterium desorption as HD, HDO and D<sub>2</sub>O was not found to be significant to the total deuterium desorption. As it can be seen, deuterium release peak appears at 720 K. Using the Redhead formula (Redhead, 1962, pp.203-211) for first order kinetics, which assumes a linear change of sample temperature with time ( $T=T_0+\beta t$ ):

$$E_{act} = RT_{max} \left[ \ln \left( \frac{10^{13} T_{max}}{\beta} \right) - 3.64 \right] \quad (1)$$

one can estimate the activation energy of the trapping site. In equation (1),  $R$  is the universal gas constant,  $T_{\max}$  temperature which corresponds to the desorption peak and  $\beta$  is the heating rate, which in our case is 20K/min. For our data, the formula gives the value of 2.3 eV. The 2.3 eV trap corresponds to D at the near-octahedral interstitial site, where it is believed to be associated with a vacancy. In addition, a less pronounced peak from  $H_2$  appears at 610 K.

### Proton spectra of the coated samples

In order to study the influence of the Au coated layer on the diffusion of D in the samples, we rotated the samples for  $180^\circ$  and recorded the proton spectra for the  $^3\text{He}$  energy of 3.2 MeV.

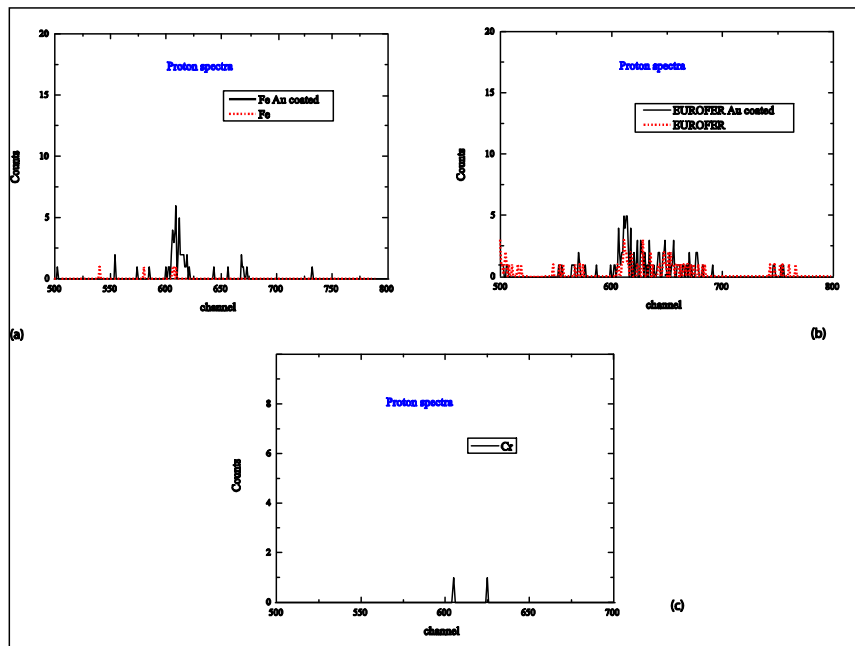


Figure 6 – Proton spectra from (a) the back side of the Fe samples, coated with a 130 nm thick Au layer (-) and pure (---), (b) the back side of the EUROFER samples, coated with a 130 nm thick Au layer (-) and pure (---), (c) back side of the Cr sample (-)

Slika 6 – Protonski spektar sa (a) zadnje strane uzorka Fe, obloženog slojem Au debljine 130 nm (-) i čistog Fe uzorka (---), (b) zadnje strane uzorka EUROFER-a, obloženog slojem Au debljine 130 nm i čistog EUROFER uzorka(---), (c) sa zadnje strane uzorka Cr (-)

Рис. 6 – Спектр протонов с (а) задней стороны образца Fe, облицованного слоем Au толщиной 130nm (-) и чистого Fe образца (---), (b) с задней стороны образца EUROFER, облицованного слоем Au толщиной 130nm и чистого EUROFER образца (---), (c) с задней стороны образца Cr (-)

Figures 6(a) and 6(b) show the proton spectra for the pure and Au coated Fe and EUROFER samples. As it can be seen, the signal from the coated samples was considerably higher than in the case of the pure samples which indicated the diffusion and higher retention and also the reduction of losses on the back side. On the other hand, in the case of Cr (Figure 6(c)) there was almost no signal on the back side indicating the absence of diffusion.

## Conclusions

This work deals with deuterium retention in EUROFER and its main components, Fe and Cr, to be used as structural materials for future fusion reactors. Two sets of measurements performed at different implantation energies of D showed the following results:

- \* Retention in Cr is much higher than in Fe (due to the strong formation of hydride on the surface of the sample)
- \* Retention in EUROFER is higher than in Fe for a factor of 2
- \* A peculiar structure is observed in Fe and EUROFER at a depth of  $\sim 4 \mu\text{m}$  (similar observation by Golubeva *et al.* for RUSFER)
- \* Comparison of the Au coated samples and the pure samples showed a higher degree of diffusivity and the retention of D indicating the role of Au as a diffusion barrier.

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РЕЗУЛЬТАТЫ ИССЛЕДОВАНИЯ ПО УДЕРЖАНИЮ ДЕЙТЕРИЯ И  
ТЕРМИЧЕСКОЙ ДЕСОРБЦИИ В УСЛОВИЯХ НИЗКОЙ  
АКТИВАЦИИ ФЕРРИТНО-МАРТЕНСИТНОЙ СТАЛИ EUROFER

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ОБЛАСТЬ: материалы  
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*Резюме:*

*В данной статье приведены результаты исследований по удержанию и термической десорбции дейтерия в железе, хrome и сплаве EUROFER – представляющем новейший материал для фузионного (термоядерного) реактора. Исследования показали следующие результаты:*

*\*Удержание дейтерия в хrome намного выше, чем в железе (из-за образования гибридов хрома)*

*\*Удержание дейтерия в сплаве EUROFER выше на фактор 2, чем в железе*

*\*Выявлена специфическая структура в концентрационном профиле железа и сплава EUROFER на глубине ~ 4 мкм*

*\*Высокий уровень диффузии и удержания дейтерия свидетельствует о потенциальном применении Au в качестве диффузионного барьера в фузионном (термоядерном) реакторе.*

*Ключевые слова: материалы для ядерного реактора, радиационные эффекты, диффузия, ядерный резонанс.*

REZULTATI ZADRŽAVANJA I TERMALNE DESORPCIJE  
DEUTERIJUMA U EUROFER-U, NOVOJ VRSTI FERITNO-  
MARTENZITNOG ČELIKA NISKE AKTIVACIJE

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OBLAST: materijali  
VRSTA ČLANKA: originalni naučni članak  
JEZIK ČLANKA: engleski

**Sažetak:**

*U ovom radu ispitivano je zadržavanje i termalna desorpcija deuterijuma u gvožđu, hromu i EUROFER-u, leguri koja se razmatra kao najnoviji materijal za buduće fuzione reaktore. Studija je pokazala sledeće rezultate:*

- zadržavanje deuterijuma u hromu je mnogo veće nego u gvožđu (usled formiranja hidrida hroma),*
- zadržavanje deuterijuma u EUROFER-u je za faktor 2 veće nego u gvožđu,*
- primećena je specifična struktura u koncentracionom profilu gvožđa i EUROFER-a na dubini ~ 4 μm,*
- veliki stepen difuznosti i zadržavanja deuterijuma govore o potencijalnoj upotrebi Au kao difuzione barijere u fuzionom reaktoru.*

**Ključne reči:** *materijali za nuklearne reaktore, radijacioni efekti, difuzija, nuklearne rezonance.*

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