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M Mayer et al.

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Tungsten surface enrichment in EUROFER and Fe-W model systems studied by high-resolution time-of-flight Rutherford backscattering spectroscopy

M. Mayer^a, T.F. Silva^{a,b}, R. Arredondo^a, M. Balden^a, I. Bogdanović-Radović^c, T. Höschen^a,
H. Maier^a, M. Oberkofler^a, L. Ru^a, Z. Siketić^c

^a *Max-Planck-Institut für Plasmaphysik, Boltzmannstr. 2, 85748 Garching, Germany*

^b *Instituto de Física da Universidade de São Paulo, Rua do Matão, trav. R 187, 05508-090 São Paulo, Brazil*

^c *Laboratory for Ion Beam Interactions, Ruđer Bošković Institute, Bijenička 54, HR-10000 Zagreb, Croatia*

Time-of-flight Rutherford backscattering spectroscopy (ToF-RBS) with incident heavy ions offers a substantially improved depth resolution compared to conventional RBS with incident He ions. The new Garching ToF-RBS detector is located at a scattering angle of 150° with a free flight path of 1.313 m and a time resolution of 600 ps. The experimentally achieved depth resolution at the surface of W-containing Fe samples is 2–3 nm using incident Si ions. Model systems consisting of Fe layers with 0.7, 1.5 and 4.2 at% W and EUROFER steel (containing 0.34 at% W + Ta) were eroded by 200 eV D ions to a fluence of 10²³ D/m² at 310 K and 900 K. W depth profiles were measured using ToF-RBS, light impurities at the surface were detected using time-of-flight elastic recoil detection analysis (ToF-ERDA). The data sets from the two techniques were analyzed self-consistently. In all cases W enrichment at the surface was observed. The enrichment at 310 K is in good agreement with SDTrimSP simulations if the experimentally achieved depth resolution is taken into account. Annealing of the model systems with 0.7 and 1.5 at% W to 900 K for 10 hours in vacuum resulted also in a W-enriched surface layer.

1. Introduction

In future fusion devices such as DEMO a crucial factor for the lifetime of plasma-facing wall components is their erosion rate by bombardment with energetic ions or neutrals from the fusion plasma [1]. At sufficiently low particle energies the erosion rates of high-Z elements, especially of tungsten, are several orders of magnitude lower than those of low- or medium-Z elements [2]. Due to this low erosion rate of tungsten a number of DEMO design studies have foreseen a layer of thin tungsten armor for plasma-facing wall components [3,4,5]. Due to the large power fluxes and steady-state environment expected in DEMO the tungsten armor must be directly bonded to the cooled component. This can be a costly and technologically challenging step in the fabrication process.

At remote regions of the first wall with smaller power and particle fluxes, a possible alternative might be the use of a bare steel plasma-facing surface [3]. These areas are mainly subject to charge-exchange neutral particles, with the largest fluxes at low energies of the order of 200 eV and below. Different types of reduced activation ferritic-martensitic (RAFM) steels have been developed for fusion applications: EUROFER [6] in the European Union, RUSFER [7] in the Russian Federation, F82H [8] in Japan and CLAM steel [9] in China. While many types of steel are not suitable as first-wall materials due to their relatively high erosion yields [2], low-activation steels contain small concentrations of heavy elements, especially tungsten and tantalum. It has been predicted by computer simulations using the SDTrimSP code that during erosion by energetic hydrogen atoms or ions a W-enriched surface layer can be formed by preferential sputtering, which could lower the over-all erosion rate to acceptable levels [1,10]. This renders low-activation steels as potential candidates as first-wall materials for recessed areas with low particle and power fluxes of the plasma-facing wall in a fusion reactor.

The existence of W-enriched surface layers has been demonstrated experimentally in Fe-W model systems and RAFM steels [1,10,11,12,13,14,15], however, due to the limited depth resolution of the used methods the comparison to simulation results proved to be difficult. Time-of-flight Rutherford backscattering spectroscopy (ToF-RBS) with incident heavy ions (for example Si or Cu ions) offers a 10-20 times better depth resolution than conventional RBS with incident He ions. This work describes measurements of W enrichment at the surface of Fe-W model systems and EUROFER at the new Garching ToF-RBS detector setup.

2. Experimental

The ToF-RBS setup is shown schematically in Fig. 1. The energy of particles backscattered from the sample is derived from the flight time between the Start foil and a copper plate which gives the Stop signal. The free flight path was determined by fitting the path length to time-of-flight backscattering spectra from different elements and is 1.313(2) m. The Start pulse is triggered by secondary electrons emitted when an ion passes through a $3 \mu\text{g}/\text{cm}^2$ thin carbon foil; the Stop trigger is produced by secondary electrons emitted when the ion hits a (thick) copper plate. The time detectors were built according to Busch et al. [16] and are both oriented for the collection of backward emitted secondary electrons. The Stop detector is approximately 20% larger than the Start detector in order to compensate the angular spread introduced by the carbon foil. The emitted electrons are accelerated by electrical fields produced by a grid system, deflected by 90° , and finally accelerated onto a multi-channel plate where they are amplified. The scattering angle of the system is 150° ; measurements were performed at two different incident angles: Normal to the sample surface and at 60° with respect to the surface normal. The typical time resolution obtained was around 600 ps, limited by the impedance matching at the Stop detector.

ToF-RBS spectra were measured with 2.0, 5.0 and 11.5 MeV Si ions. The recorded time spectra were converted to energy spectra using the “Read spectrum data: Energy/Counts” feature of the SIMNRA software [17] which allows correct re-binning of non-equidistant channels without destroying Gaussian counting statistics.

As ToF-RBS gives only information about heavier elements, in addition time-of-flight elastic recoil detection analysis (ToF-ERDA) spectra were measured at the Ruđer Bošković Institute in Zagreb [18] in order to obtain information about light elements. The measurements were performed using 20 MeV I ions at a recoil angle of 37.5° . The ToF-RBS and ToF-ERDA spectra were simultaneously fitted by MultiSIMNRA [19] using SIMNRA [17] as simulation kernel with SRIM stopping powers [20].

Fe-W model systems were produced by simultaneous sputter-deposition of Fe and W in a Denton Discovery 18 device using two individual cathodes on Fe substrates. Initial layer thicknesses

were about 350 nm. Samples with concentrations of 0.7, 1.5, and 4.2 at.% W in Fe were produced. These samples were treated in three different ways:

- Heated to 900 K in vacuum for 10 hours in order to study temperature-induced effects, in particular segregation.
- Eroded at 310 K by 200 eV D ions to a fluence of nominally 10^{23} D/m².
- Eroded at 900 K by 200 eV D ions to a fluence of nominally 10^{23} D/m² in order to study the effects of erosion at elevated temperatures.

The D irradiations were performed at the SIESTA device [21] using a magnetically analyzed mono-energetic beam of 600 eV D₃⁺ ions. The beam current was 25-30 μA; by wobbling the beam a spot size of 72 ± 2 mm² was obtained. At the nominal fluence of 10^{23} D/m² the real fluences were in the range 9.5 - 1.1×10^{23} D/m². The samples eroded at 310 K were kept at ambient temperature but their temperature rose to about 310 K due to beam heating.

EUROFER is a reduced activation ferritic-martensitic steel with a composition of 8.8 wt.% Cr, 1 wt.% W, 0.1 wt.% Ta, and 0.1 wt.% C; the rest is mainly Fe [22]. Mirror-polished EUROFER samples were eroded at 310 K by 200 eV D ions to a fluence of nominally 10^{23} D/m² at the SIESTA device [21].

Depth profiling by sputter-XPS was performed on some samples at a PHI 5600 ESCA system using incident 10 keV Ar⁺ ions at an incident angle of 20° with respect to the surface normal.

3. Results and discussion

ToF-RBS energy spectra of Fe-W model systems with 1.5 at.% W in Fe are shown in Fig. 2. The spectrum of the pristine sample is indicated by the blue line and the blue area. The high-energy edge around 6500 keV is relatively broad, indicating the presence of a thicker oxide layer. The layer eroded at 310 K (dash-dotted green line) shows some erosion of the layer (characterized by the increased energy of the low-energy edge of the W signal at around 3000 keV as compared to the pristine sample) and a small increase of the W concentration at the surface, characterized by the small peak in the W signal at around 6500 keV. The layer annealed at 900 K (dotted red line) shows a stronger enrichment of W at the layer surface (the peak in the spectrum at around

6500 keV, see Fig. 2), but also at the interface between the Fe substrate and the Fe-W layer (the peak in the spectrum at around 3000 keV, see Fig. 2). These peaks are associated with a decrease of the W signal close to the surface (the dip at around 6000 keV) and close to the interface (the dip at around 3500 keV), i.e. the additional W at the surface and at the interface originates from depths slightly below the surface and close to the interface from where the W diffused to the surface or interface. This shows clearly that segregation of W plays an important role at 900 K. The appearance of the peak between the Fe substrate and the Fe-W layer might indicate that oxygen plays a role in this segregation process. The sample eroded at 900 K (dashed orange line) shows the strongest surface enrichment of W. This enrichment is considerably higher than the superposition of the effects of erosion and heating alone and may indicate that ion-induced mobility plays a role. The W-enrichment at the interface to the Fe substrate is smaller compared to the annealing-only case: This can be due to the shorter time the sample was kept at high temperature (about 3 h for reaching the fluence of 10^{23} D/m²), but can be also due to a stronger smearing of the low energy edge due to surface roughening.

It has been predicted by combined sputter-diffusion modeling [23], that at elevated temperatures the W surface enrichment should decrease due to diffusion. From the present experimental results this prediction is not confirmed and an even higher surface enrichment is observed at 900 K than at 310 K. This is due to segregation of W at the surface, which is not included in sputter-diffusion modelling [24].

A typical ToF-ERDA spectrum is shown in Fig. 3. In addition to recoils of the heavier elements Fe and W together with forward scattered incident I ions also recoiling light elements, especially H, C and O, are visible in the spectrum. The signals from other elements are very low.

W depth profiles in the Fe-W model systems, as derived by simultaneous fitting of the ToF-RBS and the ToF-ERDA spectra using MultiSIMNRA [19], are shown in Fig. 4. The depth resolution for W in these samples is 2-3 nm at the surface but deteriorates with increasing depth due to electronic energy loss straggling and multiple scattering [25]. The W concentration in the pristine samples decreases towards the surface due to the presence of oxygen within a range of 20-30 nm from the surface, i.e., the sample surfaces were oxidized within this range. A constant W concentration was reached in a depth of 20-30 nm. The eroded samples had a layer of hydrogen, carbon and oxygen at the surface, i.e., the surface was oxidized and additionally covered by a

hydrocarbon and/or water layer. The thickness of this layer varied from sample to sample and was between 5 and 40 nm thick. The W enrichment at the surface can be clearly seen in the W depth profiles. The depth profiles shown in Fig. 4 were qualitatively confirmed by sputter-XPS measurements.

W depth profiles in EUROFER, as derived by simultaneous fitting of the ToF-RBS and the ToF-ERDA spectra using MultiSIMNRA [19], are shown in Fig. 5. The measured W concentration in the EUROFER bulk is about 0.35 at.% (corresponding to 1.2 wt.%), which is in good agreement with the nominal concentrations of W + Ta of 1.1 wt.% - W and Ta cannot be distinguished by the present measurements. In the top 6 nm of the pristine sample the W concentration decreased to 0.15 at.%, again mainly due to the presence of oxygen. In the eroded EUROFER sample the W concentration is enriched to about 0.7 at.% in the top 4-5 nm.

The surface concentrations of W after erosion by 200 eV D ions to a fluence of 10^{23} D/m² at 310 K are shown in Fig. 6 as function of the initial W concentration for the model systems with initially 0.7, 1.5 and 4.2 at.% W in Fe and for EUROFER with initially 0.35 at.% W + Ta. For all samples a clear enrichment of W is observed. The experimental data are compared to SDTrimSP simulations (lines in Fig. 6.) SDTrimSP predicts W enrichment within an extremely thin surface layer with a thickness below 0.1 nm [10]: This stretches the physics model implemented in SDTrimSP beyond its validity range [10] and cannot be resolved experimentally. In order to be able to compare the SDTrimSP simulations to the experimental data the simulations were averaged over the experimentally achieved depth resolution of 2-3 nm. The two lines in Fig. 6 mark these two maximum and minimum values. The experimental values should lie between these two lines: Taking the experimentally achieved depth resolution into account the experimental data agree well with the simulations, see Fig. 6.

4. Conclusions

Time-of-flight Rutherford backscattering spectroscopy (ToF-RBS) with incident heavy ions offers a considerably better depth resolution than conventional RBS with incident He ions. The new Garching ToF-RBS detector is located at a scattering angle of 150° with a free flight path of 1.313 m and a time resolution of 600 ps. The experimentally achieved depth resolution for W at

the surface of W-containing Fe samples is 2–3 nm, which is about 10-20 times better than conventional RBS with incident He ions.

Model systems consisting of Fe layers with 0.7, 1.5 and 4.2 at% W and EUROFER steel (which contains 0.35 at% W) were eroded by 200 eV D ions to a fluence of 10^{23} D/m² at 310 K and 900 K. W depth profiles were measured using ToF-RBS with 2.0, 5.0, and 11.5 MeV Si ions, light impurities at the surface (especially C and O) were detected using time-of-flight elastic recoil detection with 20.0 MeV I ions at a recoil angle of 37.5°. The data sets from the two techniques were analyzed self-consistently using MultiSIMNRA [19].

Erosion of the model systems and of EUROFER resulted in surface enrichment of W at 310 K and at 900 K. The observed surface enrichment at 310 K is in good agreement with SDTrimSP simulations if the experimentally achieved depth resolution is taken into account. Annealing of the model systems with 0.7 and 1.5 at% W to 900 K for 10 hours in vacuum resulted in a W-enriched surface layer with a W concentration of 2-3 at% and a thickness of about 15 nm. The additional W at the very surface originated from a near-surface layer with a thickness of about 100 nm where the W was depleted. A similar effect was observed after erosion of the model systems by 200 eV D ions to a fluence of 10^{23} D/m² at 900 K. These results are in contradiction to combined erosion/diffusion simulations [23], where a decrease of the surface enrichment effect was predicted at elevated temperatures. The reason for this discrepancy is segregation of W at the surface, which is not included in the erosion/diffusion simulations. There are hints that oxygen may play a role in this segregation process.

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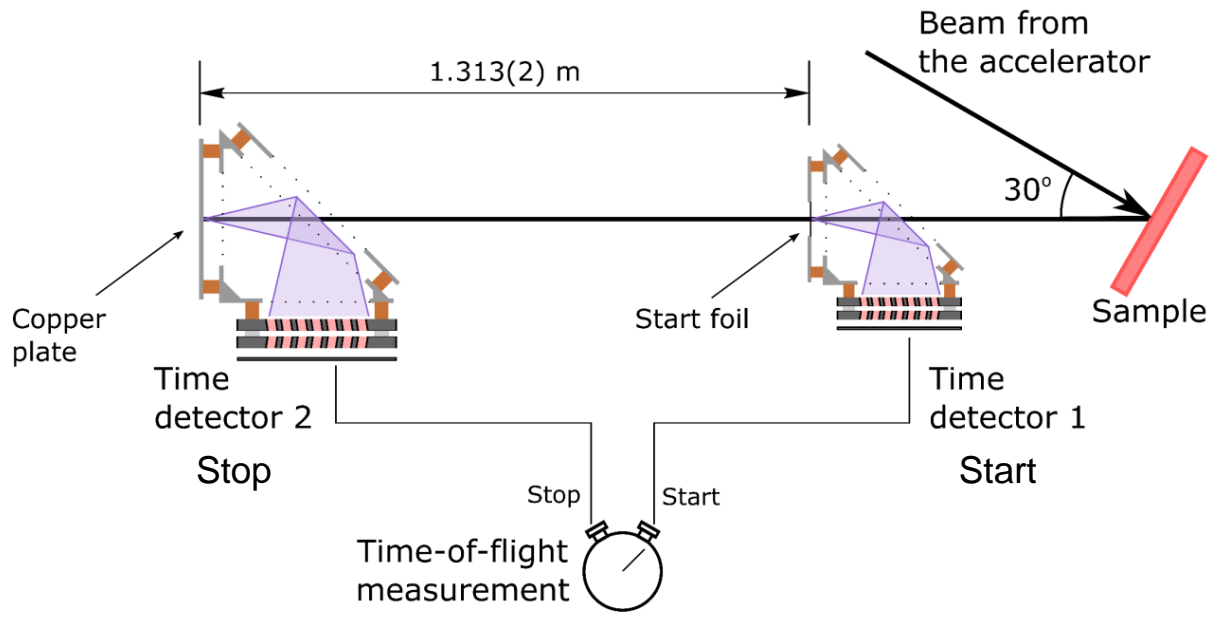


Fig. 1: Schematic representation of the ToF-RBS setup.

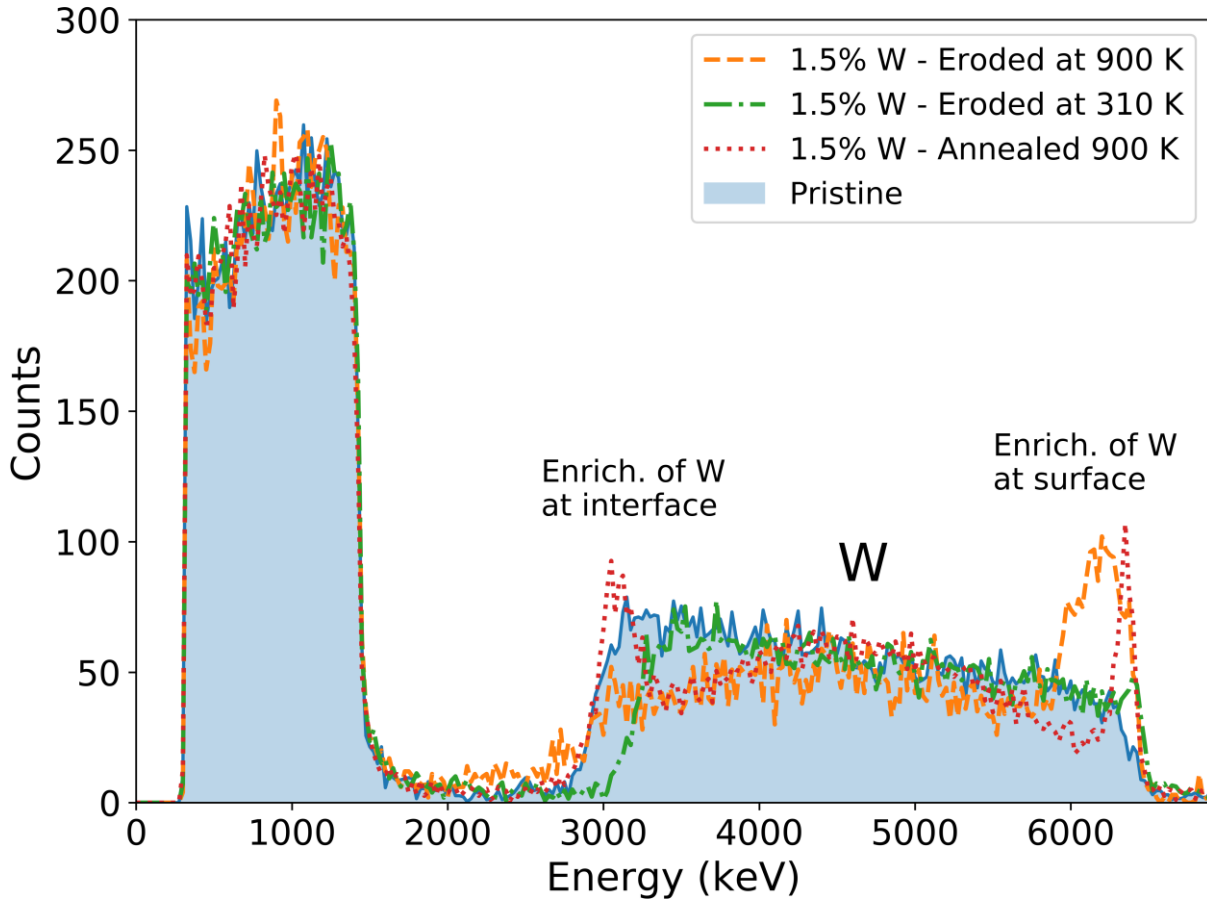


Fig. 2: ToF-RBS energy spectra of Fe-W model systems with 1.5 at.% W, measured with 11.5 MeV Si ions at a scattering angle of 150° . The signal at energies below 1500 keV is backscattering from Fe from the Fe-W model system; the signal between 2500 and 6500 keV is backscattering from W. Blue line and blue area: Pristine sample; Dashed orange line: Eroded with 200 eV D ions at 900 K; Dash-dotted green line: Eroded with 200 eV D ions at 310 K; Dotted red line: Annealed at 900 K for 10 h.

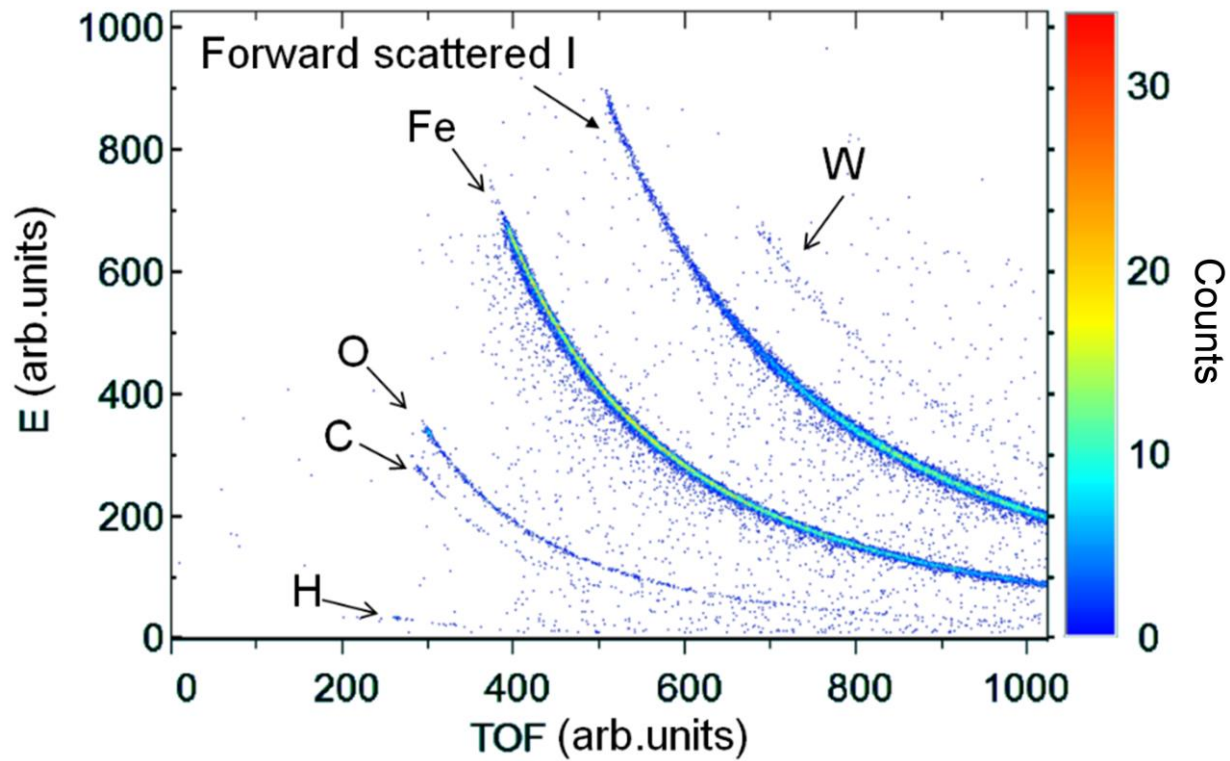


Fig. 3: ToF-ERDA spectrum of a Fe-W model system with 1.5 at.% W, measured with 20 MeV I ions at a recoil angle of 37.5° . The x-axis is the time-of-flight in channels; the y-axis is the ion energy in channels.

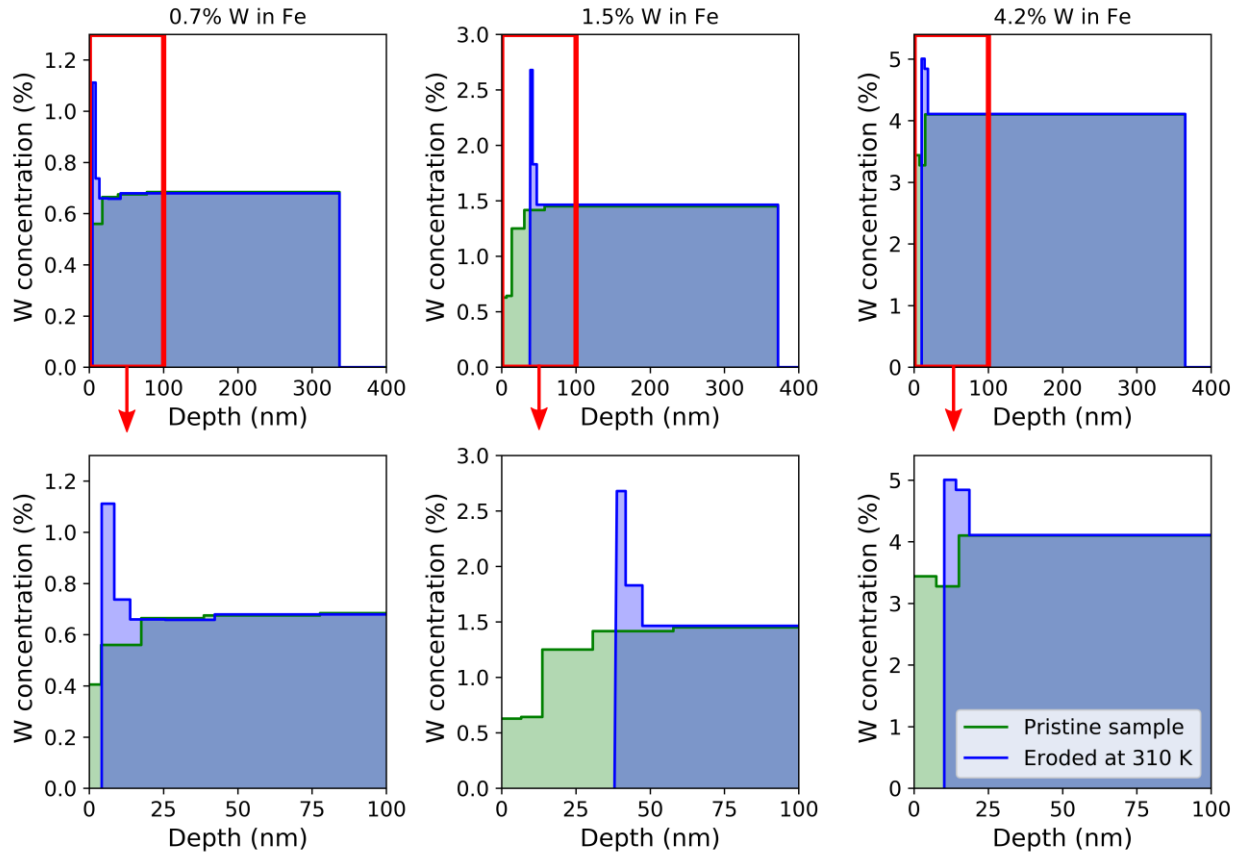


Fig. 4: W depth profiles in the Fe-W model systems, as derived by simultaneous fitting of the ToF-RBS and the ToF-ERDA spectra using MultiSIMNRA. Green lines: Pristine samples; blue lines: After erosion by 200 eV D ions to a fluence of 10^{23} D/m² at 310 K. Top row: Depth profile throughout the whole layer; bottom row: Enlargement of the top 100 nm. The W concentration is given in at.%.

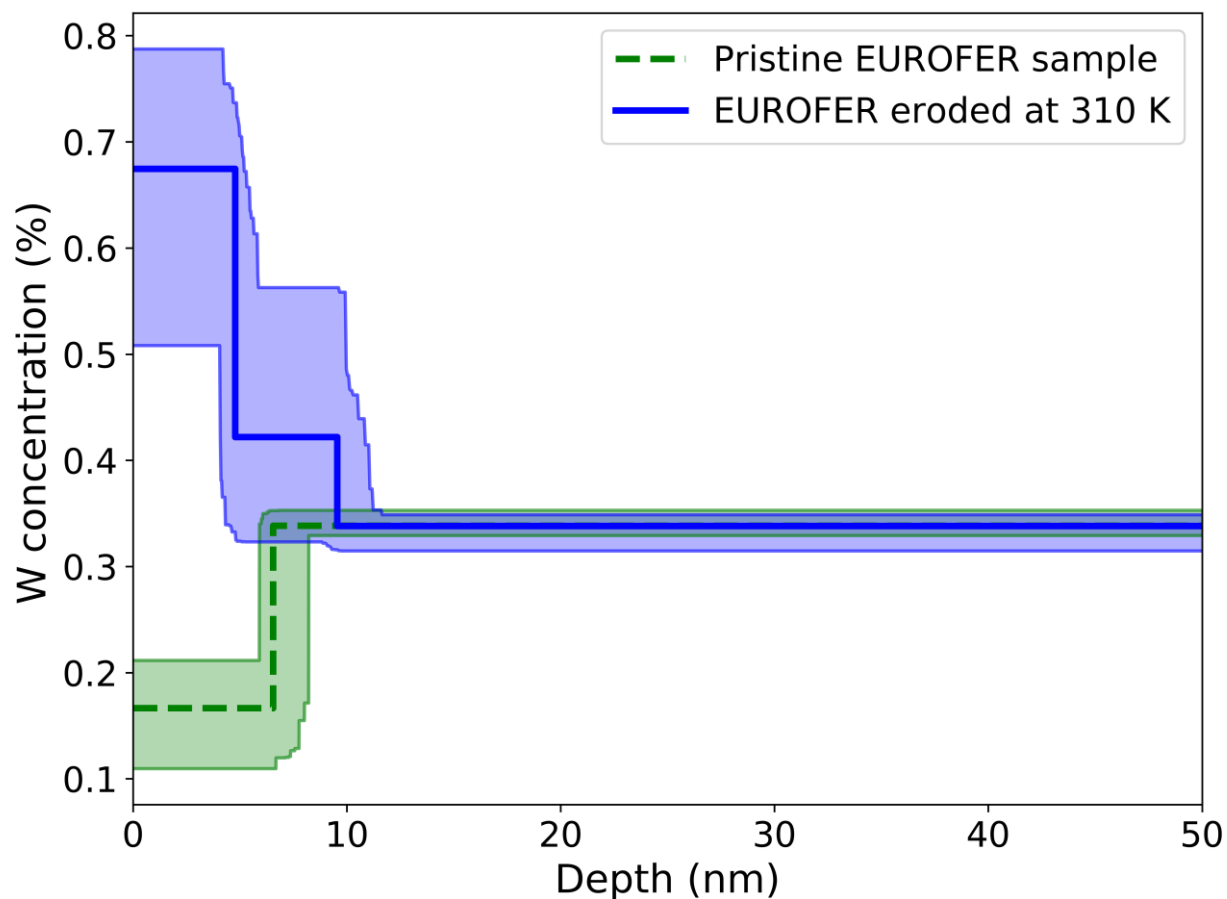


Fig. 5: W depth profile in EUROFER as derived by simultaneous fitting of the ToF-RBS and the ToF-ERDA spectra using MultiSIMNRA. Dashed green line: Pristine sample; Thin solid green lines and green area: Uncertainty band of the W concentration of the pristine sample; solid blue line: After erosion by 200 eV D ions to a fluence of 10^{23} D/m² at 310 K; thin blue lines and blue area: Uncertainty band of the W concentration of the eroded sample. The W concentration is given in at.%.

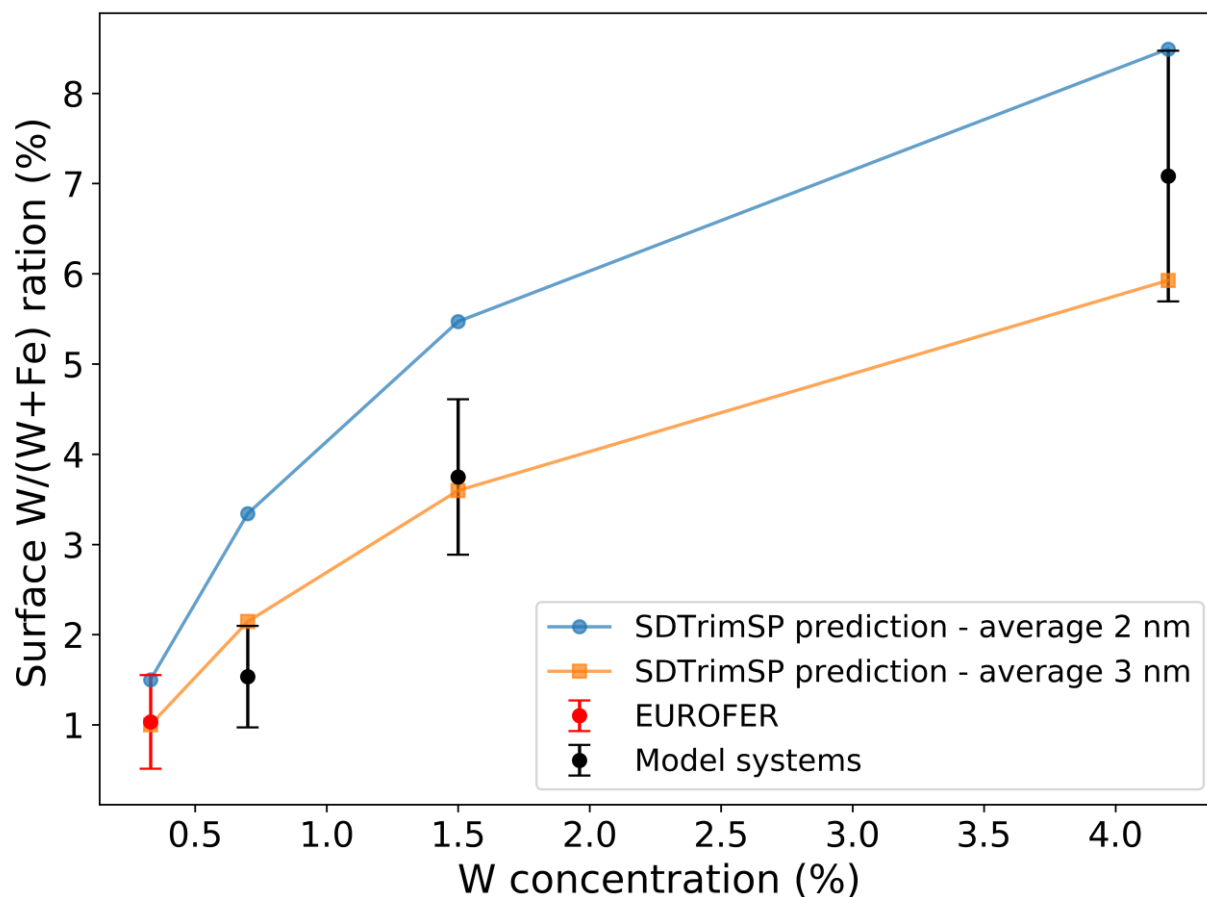


Fig. 6: Surface concentration of W after erosion by 200 eV D ions to a fluence of 10^{23} D/m² at 310 K as function of the initial W concentration in at.%. The surface concentration is given as the ratio W/(W + Fe). Black dots: Model systems with initially 0.7, 1.5 and 4.2 at.% W in Fe; red dot: EUROFER with initially 0.34 at.% W + Ta. The lines are results of SDTrimSP simulation calculations averaged with the depth resolution. Blue line with circles: Depth resolution 2 nm; Orange line with squares: Depth resolution 3 nm.

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