

EUROFER as wall material: reduced sputtering yields due to W surface enrichment

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Abstract

The performance of EUROFER steel under high flux low energy plasma bombardment is being analyzed. EUROFER contains, other than Fe and Cr, important concentrations of heavy elements with W being the most prominent component with 0.4 at.%. These different components in EUROFER will be eroded differently, leading to changes in surface composition and erosion yield. The plasma exposure was performed in the plasma generator PISCES-A with the parameter ranges of ion energies between 90 and 190 eV, exposure temperatures between 80 and 500°C and fluences between 3×10^{23} and 6×10^{25} m⁻². The erosion yield was mainly determined by weight-loss measurement, and the surface composition after the exposure was analyzed by various ion beam analysis methods.

For temperatures below 180°C the erosion yield of EUROFER was lower than for pure Fe already at the lowest fluence of 3×10^{23} D⁺m⁻², and decreased further to values of less than 1/10 of those for Fe at 6×10^{25} D⁺m⁻². Ion beam analysis showed the enrichment of W at the exposed surface correlating with the yield reduction; however, the experimental depth resolution of about 1 nm did not allow resolving the predicted W enrichment within the first two monolayers. Therefore, it remains unclear whether the W enrichment is the only reason for the yield reduction or if surface oxidation and topography changes contribute to the observed effects. First results for different surface temperatures, however, indicated that at 500°C the erosion yield remains close to the values for pure Fe, indicating that the exposure temperature is also an important factor for the sputtering process of EUROFER.

1. Introduction

The selection of plasma-facing materials in existing tokamak magnetic confinement devices is complicated by the presence of transient power loading due to ELMs and disruptions [1]. However, when one moves to a reactor, or DEMO, the assumption of being able to obtain a stable, quiescent plasma is a given. Facing such plasma the lifetime of components will be determined by their erosion rate. The erosion rate of high-Z elements, such as tungsten, is several orders of magnitude lower than that of low-Z elements, such as beryllium or carbon, at the low ion energies expected near the walls in confined plasma devices [2]. For this reason many DEMO design studies have incorporated the use of thin tungsten armored surfaces for plasma-facing components [3,4].

Due to the large power flux and steady-state environment expected in DEMO, any tungsten coatings, or armor, must be directly bonded to the cooled component. This can be a costly and technologically challenging step in the fabrication process. In remote regions of the first wall, one possible alternative to tungsten armor being applied to the surface of a component is to use a bare steel plasma-facing surface [3]. These areas are mainly subject to charge-exchange neutral fluxes, with the largest fluxes at very low energies of the order of 200 eV and below. Many of the nuclear grade steels (for example, EUROFER, RUSFER and F82H) contain a small concentration of tungsten within the material. At low particle bombarding energy, one theoretically expects the lower mass iron in the steel surface to quickly erode, leaving behind a thin tungsten-rich surface which would be immune to erosion. If preferential sputtering of Fe at the surface occurs, it would lead to changes in surface composition and erosion yield with continuous particle bombardment in contrast to pure Fe, where the erosion yield is constant with fluence after removal of the native surface oxide layer [5].

The goal of this work is to experimentally determine the conditions where erosion-induced surface segregation can result in accumulation of tungsten at the outermost layers of EUROFER samples exposed to energetic deuterium (D) bombardment. D plasma exposure was conducted in the linear plasma device PISCES-A [6] at UCSD where the net erosion yield from EUROFER samples was measured. Post-exposure surface analysis of the samples was performed using ion beam analysis at IPP-Garching.

2. Experimental

2.1. Sample preparation

EUROFER has been developed as a nuclear steel consisting of Fe as the base material, about 10 at.% Cr, and small fractions of low-Z ($C+N+P+S+O \leq 0.2$ at.%) and mid-Z ($Mn+V \leq 0.85$ at.%) elements. In addition, high-Z alloying elements are included, such as W (nominally 1.0-1.2 wt.%, corresponding to approximately 0.4 at.%) and Ta (0.10 - 0.14 wt.%) [7]. EUROFER samples for this study were prepared by cutting from one slab with the dimension of 12 mm \times 15 mm \times 0.6 mm (thickness). The surface to be exposed to plasma was mirror-polished.

For controlled investigation of the influence of high Z elements simple binary Fe-W mixed layers were prepared as model system of EUROFER by magnetron-sputtering deposition with two independent cathodes: one from S235JRG2 steel and another from W. S235JRG2 steel has a composition of about 99 at.% Fe, where Ni is included (~ 0.3 at.%) and the content of high-Z elements is much smaller (Mo 0.08 at.%). Graphite was chosen as the substrate to facilitate the layer characterization and post-mortem analysis. The composition of the layer can be controlled by adjusting the input power for the cathodes. Three depositions with different input power combinations were run, which resulted in Fe-W layers with 3 different W concentrations, i.e. 0.3, 0.5 and 1.0 at.%, respectively. The layer thickness was 250 - 300 nm, which is optimized for the post-mortem analysis and allowed the analysis of the total layer thickness and the loss of Fe and W atoms, independently. It should be noticed that the layers have relatively high oxygen content (8 - 10 at.%) due to a small leakage in the deposition set-up.

In addition, several Fe targets (purity 99.95%) were used as a comparison for measuring the sputtering of pure iron surfaces.

2.2. Exposure conditions

Samples were clamped to the cooled PISCES-A sample manipulator using a special mask to ensure good thermal contact when either water or air cooling was employed. A low-flux D plasma (Γ_i from 2.4×10^{21} to 6.2×10^{21} $m^{-2}s^{-1}$) was used to minimize redeposition of eroded material and allow for the net erosion of the surface to be measured by weight loss. The plasma condition, measured by a reciprocating Langmuir probe to be $n_e \sim 4 \times 10^{17}$ m^{-3} and $T_e \sim 6$ eV, was kept constant for all samples. The deuterium species mix of the plasma was calculated to be ($D^+ : D_2^+ : D_3^+$) = (30:38:32) based on previous measurements in PISCES-A [8]. Although the incident ion flux consisted only to 30% in D^+ ions, only the D^+ fluence was considered in evaluating the erosion yield. The molecular ions result in 1/2 and 1/3 of the incident energy after break up of the molecules and correspondingly much lower erosion yield

close to or below the threshold for sputter erosion. The uncertainty introduced by this assumption could result in an overestimation of the D^+ yield of up to 25%.

Fluence (i.e. exposure time), ion energy and surface temperature (i.e. cooling rate) were varied. The energy of incident plasma ions was varied by applying a negative bias to the target. Bias voltages of 100, 150 and 200 V were chosen, yielding ion energies of 90, 140 and 190 eV, respectively. Temperature was varied between 80 and 500°C by adjusting the sample cooling and measured by a thermocouple pressed to the rear side of the sample.

2.3. Post-exposure analyses

The erosion yield was primarily evaluated by a weight-loss technique. The weight of each sample was measured before and after plasma exposure by a microbalance system, and the erosion rate was then calculated from the weight loss and the total ion fluence. For the calculation, we assumed that the weight loss is dominantly due to the sputtering of Fe and contribution of other elements to the weight change is negligible. The weight-loss measurement was done at UCSD and IPP using different microbalance set-ups for the cross-check, and both results showed almost perfect agreement. Typical weight losses were between 25 and 500 μg ; the uncertainty in the weight loss determination is estimated to be $\pm 10\mu\text{g}$.

The surface composition and elemental depth profiles were measured by Rutherford backscattering spectroscopy (RBS) and X-ray photoelectron spectroscopy (XPS). While the erosion yield of EUROFER could only be analyzed from the weight loss, the erosion of Fe-W layers could be also analyzed by ion beam analysis. RBS was performed first with using 3 MeV $^4\text{He}^+$ as probe beam, which is particularly suitable for the analysis of Fe-W layer samples because the entire thickness of the prepared layer (250-300 nm as mentioned above) can be measured under this beam condition. The total number of sputtered atoms, specified for Fe and W, of the layer can be easily determined by measuring the thickness change between before and after plasma exposure. Modification of surface composition, especially enrichment of W due to preferential sputtering of low-/mid-Z elements, would be an important factor for the erosion process of EUROFER, and is of interest in this study. Numerical calculations had predicted a pronounced surface enrichment of W within fractions of the first nm, which is well below the depth resolution of conventional RBS (scattering angle 165° , normal incidence) of about 10 nm. Therefore, another RBS approach with the Cornell geometry arrangement, i.e. 1 MeV $^4\text{He}^+$ with an scattering angle of 165° and incident angle of 75° to the surface normal, was attempted, which improves the depth resolution down to ~ 3 nm. Some of the EUROFER samples were additionally analyzed by “high-resolution”

RBS at the University of Göttingen [9]. It was performed with an analyzing beam of 450 keV $^4\text{He}^+$ and the backscattering energy distribution was measured using an electrostatic analyzer (ESA) detector, which achieved a depth resolution of ~ 1 nm. The obtained RBS spectra were evaluated with the SIMNRA program [10].

XPS was performed with a PHI 5600 ESCA system using an Al $K\alpha$ source. For the depth profiling, 10 keV Ar^+ was used for the surface etching, and the photoelectron spectra were measured every 20 s of etching time (corresponding to $1.1 \times 10^{15} \text{Ar}^+/\text{cm}^2$). XPS can better separate closely neighbored elements compared to RBS, such as Cr and Fe. The depth resolution is of the order of several monolayers, but needs still to be better quantified by measurements of the sputter crater after extended sputtering.

The surface morphology after plasma exposure was examined by scanning electron microscopy (SEM) equipped with an energy dispersive X-ray spectrometer (EDX).

3. Results and discussion

3.1. Erosion yields

The erosion yields of EUROFER / Fe-W layer were determined for a bias voltage of -150V as function of the incident fluence and sample temperature. As the sample temperature could not be set completely independent from the plasma exposure conditions the fluence dependence was determined in the temperature range of 80 to 180°C, while the temperature dependence was analyzed up to 500°C for medium fluences between 3 and $9 \times 10^{24} \text{m}^{-2}$.

3.1.1. Fluence dependence

Fig. 1 shows the experimental erosion results for 140 eV D^+ as function of fluence obtained for EUROFER and Fe-W layers with different bulk compositions (W $\sim 0.3, 0.5,$ and 1.0 at. %) compared to a numerical simulation calculated by the binary sputtering code TRIDYN [11,12]. All experimental data in Fig. 1 were obtained at low temperatures between 80 and 200°C. The simulation was performed for the sputtering of a Fe-W mixed material with W concentration of 0.5 at.% under 150 eV D^+ bombardment. Due to the much lower erosion yield of W for low energy D ions an enrichment of W in the first few monolayers was predicted. This W enriched layer in turn reduces the Fe sputtering leading to a decrease of the total sputtering yield with increasing fluence as shown in the figure. Because of limited computation time, the reasonably achievable fluence for the TRIDYN simulation was $1 \times 10^{24} \text{m}^{-2}$ at the highest. Therefore, the lowest fluence in PISCES-A of $1.2 \times 10^{24} \text{m}^{-2}$ already exceeds

the highest fluence in the TRIDYN simulation. Nevertheless, the yield values of about 1.2×10^{-2} are in still reasonable agreement at this point. Exposure to higher fluences leads to a further decrease of the experimental values. As the experimental yields represent average values up to the chosen fluence they had to be re-evaluated in order to make them comparable to the simulated values for sequential fluence intervals. The erosion up to the previous fluence had to be subtracted from the next higher fluence and divided by the fluence interval. Actually, most of the erosion occurs in the fluence range up to $5 \times 10^{24} \text{ m}^{-2}$ while at higher fluences steady state erosion with yields around $1 \times 10^{-3} \text{ at./D}^+$ is obtained.

Although the total sputtering yield is dominated by Fe sputtering, the RBS technique allows also the determination of the partial sputtering yield for W for the Fe-W layers. Figure 1b shows the yield for W and reveals similar fluence dependence as for Fe, however about two orders of magnitude lower. Such a yield ratio must be expected as in steady state the W to Fe ratio in the sputtered flux must be equal to the W to Fe ratio in the bulk of the material. It must be noted that a bombarding energy of 140eV is well below the threshold energy for sputtering of pure W of 200 eV, as D ions cannot transfer the energy needed for a W atom to overcome the surface binding energy of 8.6 eV. However, if the energy transfer can go via an intermediate Fe collision in a Fe-W mixed layer, the threshold energy can be reduced to 80 eV and finite yield values for W are measured at even 140 eV.

3.1.2. Energy dependence

Fig. 2 shows the results for the energy dependence of the sputtering yield of Fe and EUROFER in comparison with literature values of pure iron sputtering yields determined experimentally in ion beam studies [2] and by numerical simulation [13]. The values obtained for pure iron at PISCES-A are in close agreement with ion beam data previously obtained from the high current ion source (labeled as “HStQ” in the figure) for iron and steel SS136LN [2], while numerical simulations tend to overestimate iron sputtering [14]. The lower experimental yields may also be due to the influence of oxide surface layers in ion beam experiments [5].

EUROFER data were obtained at medium fluences in the range of $3\text{-}5 \times 10^{24} \text{ m}^{-2}$ and for high fluences close to saturation of the fluence dependence. For temperatures below 150°C the data show a clear reduction, typically by a factor of 3 at medium fluences compared to pure iron and exhibit a threshold at 90 eV. In the highest fluence intervals investigated the experimental values reach at saturation a reduction of a factor of 10 compared to pure iron

(Fig. 2). However, for exposures at 500°C the same yields were obtained for EUROFER as for pure iron, at both 90 and 140 eV.

3.1.3. Temperature dependence

As mentioned above, the temperature of the samples used for obtaining the fluence dependence varied between 80 and 180°C. In order to obtain a clear temperature dependence at 140 eV D⁺ and for medium fluences between 3 and 9×10²⁴ m⁻² the temperature was increased further by reducing the sample cooling during irradiation. Fig. 3 shows data for EUROFER up to 500°C. It can be seen that the reduction of the erosion yield at a fluence of 3×10²⁴ m⁻² and 80°C as compared to the values of pure iron is lost at high temperatures (see Fig. 2). For exposure of EUROFER at 500°C, sputtering yield values similar to those of pure iron are measured. Even for 90 eV D⁺ irradiation of EUROFER, where at low temperature no erosion yield could be determined, the yield increases to 2×10⁻³ at 500°C. Due to the variations in the applied fluences and the limited control of temperature the characteristic temperature for the transition back to pure iron sputtering yield cannot be unambiguously determined, and therefore, further detailed investigations are needed.

Alternatively, using a simplified description of the experimental results inherent parameters can be optimized to make use of all data points for a fit to the fluence and temperature dependence simultaneously. It is assumed that the fluence dependence is due to a reduction of the erosion rate of Fe due to surface W enrichment. The disappearance of this reduction at elevated temperatures may be due to inter-diffusion of Fe and W resulting in the suppression of the W enrichment. Within this description it is reasonable to assume a simple exponential decay of the yield with fluence as

$$Y_F = Y_o \exp\left(-\frac{F}{F_t}\right)$$

where Y_o is the yield for pure iron and F_t is the empirical fitting parameter equivalent to the fluence for a reduction to 1/e. For the temperature dependence an Arrhenius behavior is assumed tentatively describing diffusional effects which reduces the surface enrichment of W. The reduction obtained at a given fluence disappears with increasing temperature according to

$$(Y_o - Y_T) = (Y_o - Y_F) \exp\left(-\frac{T_t}{T}\right)$$

where T_t is the empirical fitting parameter equivalent to the characteristic transition temperature. Using this simple description, optimal values for F_t and T_t are obtained by a

least-mean-square fit of the calculated values to the experimental data. The fitting results are shown in Fig. 4.

The optimal fitting values are $F_t = 2.1 \times 10^{24} \text{ m}^{-2}$ and $T_t = 330^\circ\text{C}$ for the characteristic transition of the yield values as function of fluence and temperature, respectively. Introducing a frequency factor in the Arrhenius dependence of 10^{13} s^{-1} would result in an activation energy of 2 eV for the process reducing the W surface enrichment, but the large error associated with the uncertainties of the data requires more dedicated experiments on the temperature dependence. The overall fit shows a reasonable agreement of calculated values with experimental data, the straggling of the calculated values resulting mainly from uncertainties in the temperature data ($\pm 30^\circ\text{C}$). For the error bars for the experimental data the weight loss ($\pm 10 \mu\text{g}$) is the dominant uncertainty.

3.2. Surface compositional changes:

The predictions of the sputtering yield in TRIDYN and in the simple description are based on the assumption that the much lower erosion yield of W compared to Fe leads to a strong enrichment of W at the surface and in consequence to a reduction of the total sputtering yield. Therefore, the surface composition of the components in EUROFER was investigated by ion beam analysis (RBS) with different surface resolutions.

Fig. 5 shows the raw data from RBS using 1.0 MeV ^4He ions at glancing angle to the surface for plasma exposed samples. Clearly, a pronounced W peak at the surface can be seen after D^+ irradiation. Note that no spontaneous composition / phase change or elemental segregation in EUROFER is expected under the present experimental temperature and background pressure range [15], meaning the surface W enrichment is caused by the D^+ irradiation. Actually, on binary Fe-W not always the entire surface area was irradiated, leaving unirradiated areas without any change in the W surface concentration. The evaluation of such spectra using SIMNRA results in typical surface resolution of $30 \times 10^{19} \text{ at/m}^2$ with W concentrations up to 10 at.%, i.e. enrichment of W by about a factor of 20. Such enrichment can only explain a yield reduction of about 10%, which is far less than the observed values of a factor of 10. This discrepancy may be due to a W enrichment much shallower than the surface resolution with correspondingly higher W concentrations, such as predicted by TRIDYN for similar experimental conditions (Fig. 6). Fig. 6 shows the development of the depth distribution of the W surface concentration with fluence. For low fluences, the large W enrichment extends over a distance of less than two monolayers which is smaller than the best achievable the resolution of the measurement ($\sim 1 \text{ nm}$). In contrast, the broadening of the W

enrichment peak with increasing fluence is well resolved within the resolution limit of the RBS technique. The broadening could be an indication for ion induced W diffusion, but also could also be explained by the development of a pronounced surface topography as seen in the SEM after extensive surface erosion [16].

4. Complications and uncertainties:

At present, it appears reasonable to assume that W enrichment within a very shallow surface layer due to low energy D bombardment can indeed drastically reduce the erosion of EUROFER at temperatures below 200°C. However, there are further effects which could be of importance in the detailed explanation of all effects:

- EUROFER is a multi-elemental alloy, where other components could be of importance. X-ray photoelectron spectroscopy (XPS) shows strong changes in the surface concentrations of different components which cannot be distinguished by RBS. Surface enrichment of Cr is seen in the XPS data already for the unexposed samples, being reduced with increasing fluence. However, even for XPS the calculated thickness of W surface enrichment cannot be resolved. XPS sputter depth profiling using 5 keV Ar ions shows W enrichment up to a depth of to more than 3 nm.
- Oxygen is a major impurity both in the unexposed and in the exposed samples. Within the depth of Cr modification and W enrichment also O uptake is seen in XPS analysis. This can result from oxidation on air after exposure, but also be due to oxygen impurity ions in the incident plasma, amounting in PISCES A to about 0.5%. Further experiments in PISCES B with much lower oxygen content could clarify the influence of oxygen.
- The EUROFER samples are by no means a homogeneous material. Precipitations of WC are found on the virgin samples and prolonged plasma exposure enhances the development of pronounced surface topography [16].
- D implantation into EUROFER leads to high near surface concentrations up to about 10 at.%, similar as in pure W [17]. This may be accompanied by subsurface lattice damage influencing the atomic mobility in the near surface.

5. Conclusion

First preliminary results show that for low energy D irradiation (90 to 190 eV) of EUROFER sputtering yields are strongly reduced with increasing ion fluence up to $6 \times 10^{25} \text{ m}^{-2}$ and saturate at values between 5×10^{-4} and 10×10^{-4} . These findings can be interpreted by preferential erosion of Fe and Cr and surface enrichment of W. At temperatures above 200°C

this effect is reduced and at 500°C erosion values are close to those of pure iron. A simple empirical description used to separate the effects of fluence and temperature results in an exponential yield reduction with fluence with a characteristic fluence of $2.7 \times 10^{24} \text{ m}^{-2}$ and a recovery with a characteristic temperature of 280°C.

Acknowledgments

Thanks are due to many helpful colleagues in the course of this work. We thank Chiara Falsini and Freimut Koch for the excellent preparation of the Fe-W layers and the EUROFER samples. The accelerator group of Prof. H. Hofsäss and Dr. K. Zhang at University Göttingen made their high resolution RBS set-up available and provided data prior to publication. Dr. Klaus Schmid helped in the set up of the empirical fitting procedure of the model to the experimental data. Dr. Wolfgang Eckstein extended TRIDYN simulations to high fluences. One of us (JR) thanks the PISCES group at UCSD for placing PISCES A at his disposal and coping with his daily requests during a 5 weeks visit.

Figure captions

Figure 1:

Fluence dependence of the sputtering yield of EUROFER (solid points) and binary Fe-W layers with different W concentrations (open points) under exposure of D plasma with a bias of -150 V. For comparison, computer simulation of the sputtering yield for a Fe-0.5% W mixture is shown up to a fluence of $1 \times 10^{24} \text{ D}^+ \text{ m}^{-2}$. a) Fe sputtering yields; b) W sputtering yields.

Figure 2:

Energy dependence of the sputtering yield of pure iron and EUROFER under exposure to 90, 140 and 190 eV D^+ from the PISCES A plasma in the temperatures range of 80 to 180°C and at 500°C. The data are presented for medium fluences around $5 \times 10^{24} \text{ m}^{-2}$ and high fluence, steady state conditions close to saturation of the sputtering yield. For comparison, computer simulation of the sputtering yield for pure iron is shown as well as experimental ion beam data for iron and steel SS316 LN.

Figure 3:

Temperature dependence of the sputtering yield of EUROFER under exposure of a D plasma with a bias of -100 and -150 V at medium fluences around $5 \times 10^{24} \text{ m}^{-2}$.

Figure 4:

Fit of sputtering yield values obtained from the simple description (see text) to experimental sputtering yield data for EUROFER at different ion fluences and surface temperatures taking both, the fluence and temperature dependence into account. Error bars are estimated from the weight loss uncertainties and the temperature variations during the plasma exposures.

Figure 5:

Rutherford backscattering spectra (RBS) for 1 MeV 4He scattering for 75 degree angle of incidence for EUROFER samples exposed to a 90, 140 and 190 eV D^+ from the plasma at low fluences. The W peak indicates the surface enrichment of W while the backscattering level at lower energies corresponds to the 0.5 at.% W bulk concentration.

Figure 6:

Depth distribution of the W surface enrichment obtained with high resolution RBS (ESA [9]) for -150 V bias and different fluences. For comparison, results from numerical simulations (TRIDYN) are shown. The depth scale was expressed in nm using the atomic density of pure Fe.

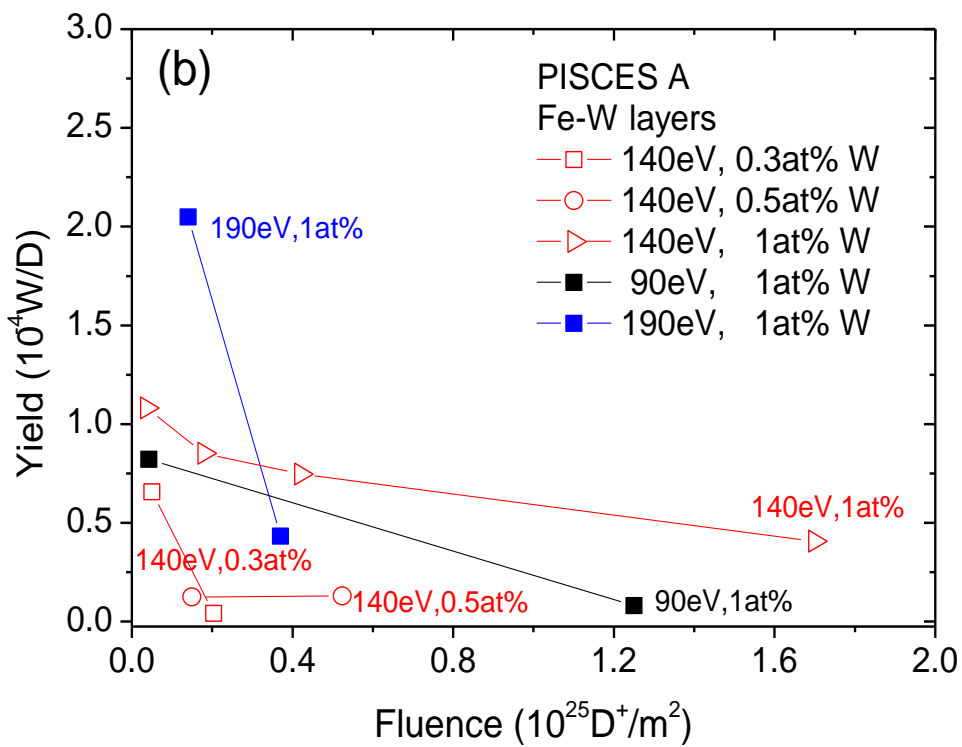
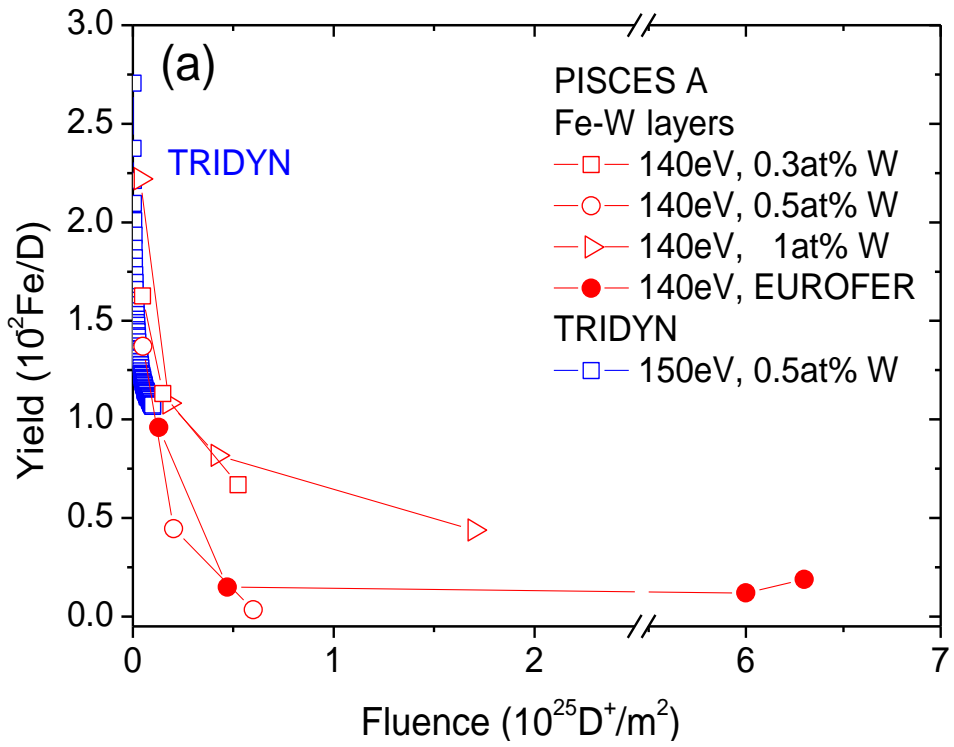


Figure 1

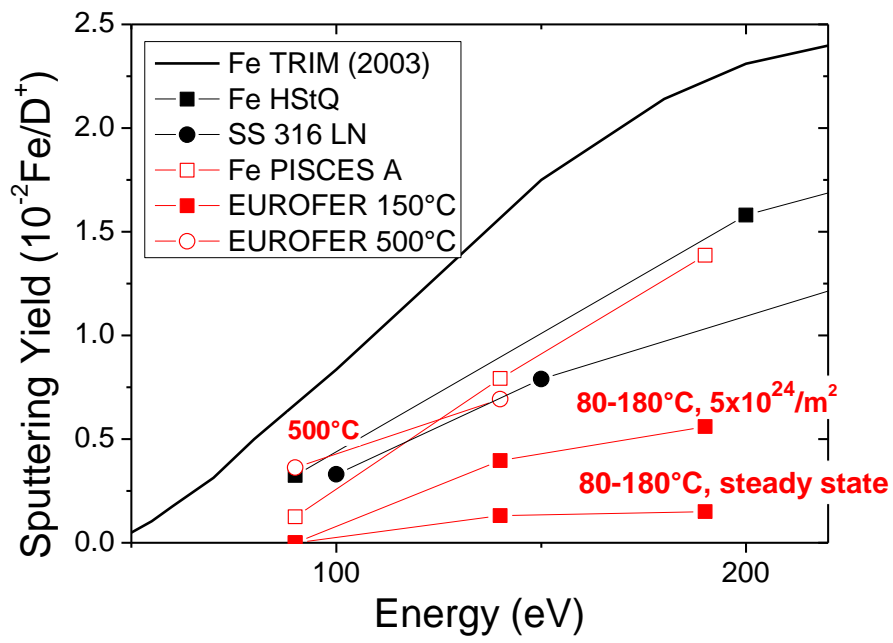


Figure 2

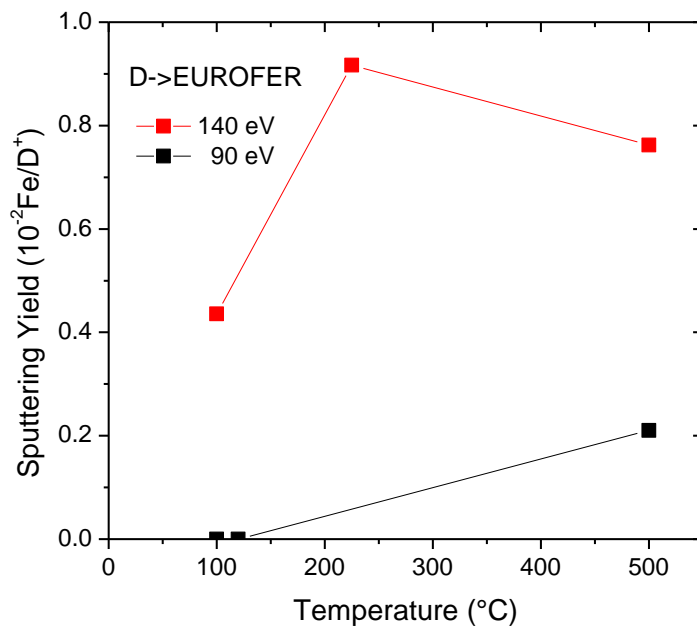


Figure 3

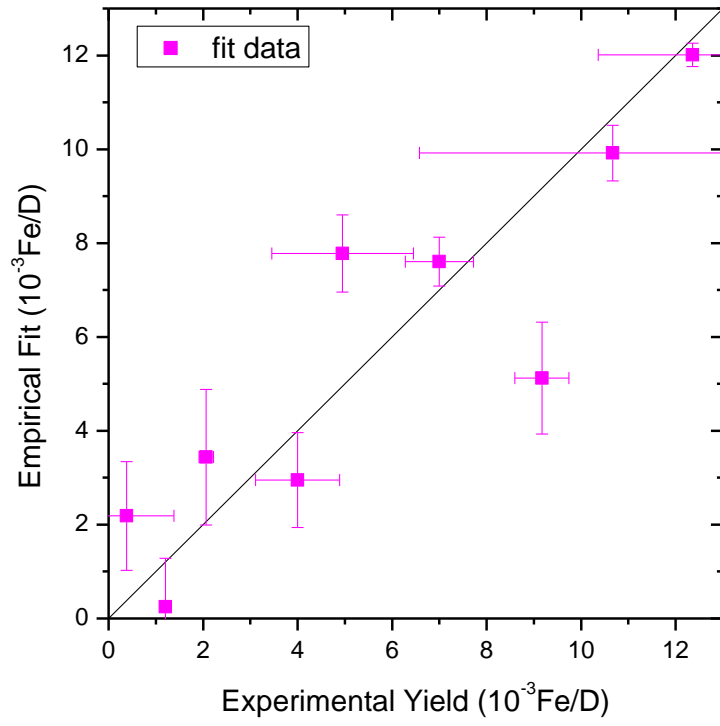


Figure 4

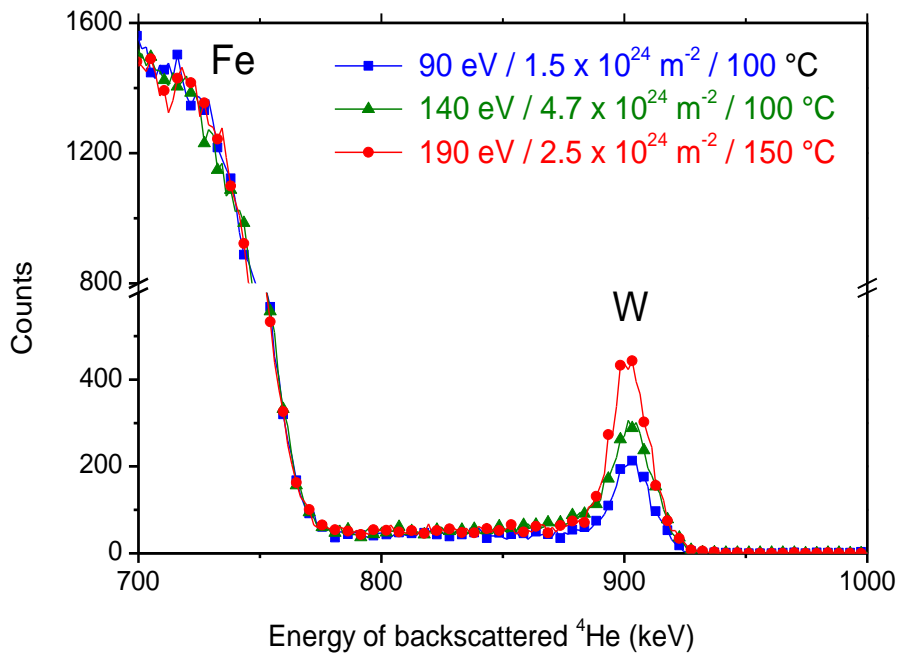


Figure 5

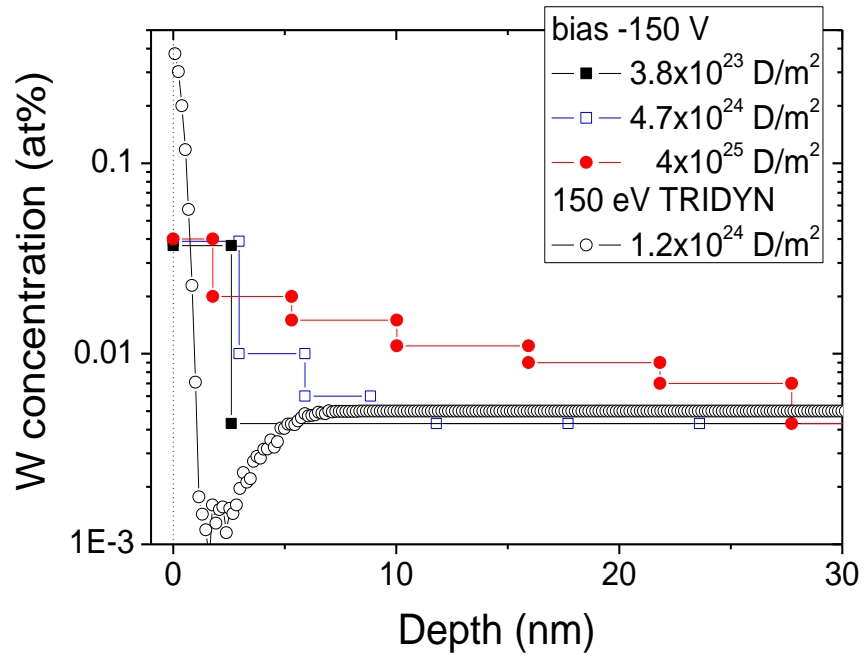


Figure 6

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