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EUROFUSION WPMAT-PR(16) 14885

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Preprint of Paper to be submitted for publication in  
22nd International Conference on Plasma Surface Interactions  
in Controlled Fusion Devices (22nd PSI)



This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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# Smart alloys for a future fusion power plant: first studies under stationary plasma load

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## Abstract

Due to its high melting point, low sputtering yield and high thermal conductivity tungsten is presently deemed as a prime candidate for the first wall cladding of the future power plants like DEMO. However, in the event of a so-called loss of coolant accident with simultaneous air ingress the temperature of tungsten will rise above 1000°C and will last for weeks because of neutron decay heat. Neutron-irradiated radioactive tungsten at such a temperature forms volatile oxide which then can be mobilized into the environment. New advanced materials - so-called “smart” alloys are being developed to address this issue. During regular operation the selective sputtering of alloying elements by plasma should leave almost pure tungsten surface facing the plasma. In the accidental conditions the alloying elements in the bulk of smart alloy will form an oxide layer protecting tungsten from oxidation.

The first direct comparative test of pure tungsten and smart alloys under identical plasma conditions was performed. Tungsten-chromium-titanium alloys were exposed simultaneously with tungsten samples to stationary deuterium plasma in PSI 2 linear facility under DEMO relevant conditions. The accumulated fluence was  $1.26 \times 10^{26}$  ion/m<sup>2</sup>. The weight loss of pure tungsten samples after exposure was  $\Delta m_w = 1000-1150$  µg. The measured weight loss of sputtered smart alloy sample  $\Delta m_{SA} = 1240$  µg corresponds very well to that of pure tungsten providing experimental evidence of good resistance of smart alloys to plasma sputtering.

## Keywords:

DEMO, Advanced plasma-facing materials, smart tungsten alloys, suppressed oxidation, plasma sputtering, accidental conditions

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## **Smart alloys for a future fusion power plant: first studies under stationary plasma load**

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### **Highlights**

- Smart tungsten-based advanced alloys are proposed for future fusion power plant
- Smart alloys feature advantages of pure tungsten during plasma operation and reduced oxidation in case of an accident
- First comparative test of smart alloy and pure tungsten was made under DEMO-relevant plasma conditions
- Tungsten and smart alloy samples demonstrated similar erosion yields
- Expected and favorable selective sputtering of alloying elements was detected experimentally

## **Introduction and motivation**

In future fusion power plant like DEMO, the in-vessel components will be subjected to the unprecedented steady-state particle and neutron loads. Currently envisaged materials for fusion reactor experiments will face the challenge of the rapidly degrading performance in the power plant. Presently, tungsten is deemed as the best-suited plasma facing material for the first wall of DEMO. Tungsten features low fusion fuel retention, low sputtering by plasma ions, perfect thermal conductivity at elevated temperatures. However in the case of an accident in fusion power plant, the application of tungsten could be questionable. During the so-called loss-of-coolant accident (LOCA) combined with an air ingress, the tungsten plasma-facing components (PFCs) will be heated up to 1000°C-1200°C due to nuclear decay heat [1]. Such an elevated temperature will remain for months at the absence of an active cooling. At such a temperature the radioactive tungsten and its isotopes will form volatile oxides, which can be then mobilized into atmosphere with the rates 10-100 kg per hour. Therefore, the oxidation of tungsten must be suppressed at the maximum possible extent.

New advanced tungsten-based so-called “smart alloys” represent an attractive option for providing the intrinsic safety to the fusion power plant. These materials possess the capability of adjusting their properties according to environment. During the routine plasma operation in the power plant, the first few nanometers of the surface will be sputtered by the plasma ions. The lighter alloying elements will be sputtered earlier, leaving almost a pure tungsten surface facing the plasma. In the case of an accident however, the alloying elements in the bulk of the smart alloy will react with oxygen and create their own stable oxides to protect tungsten from mobilization.

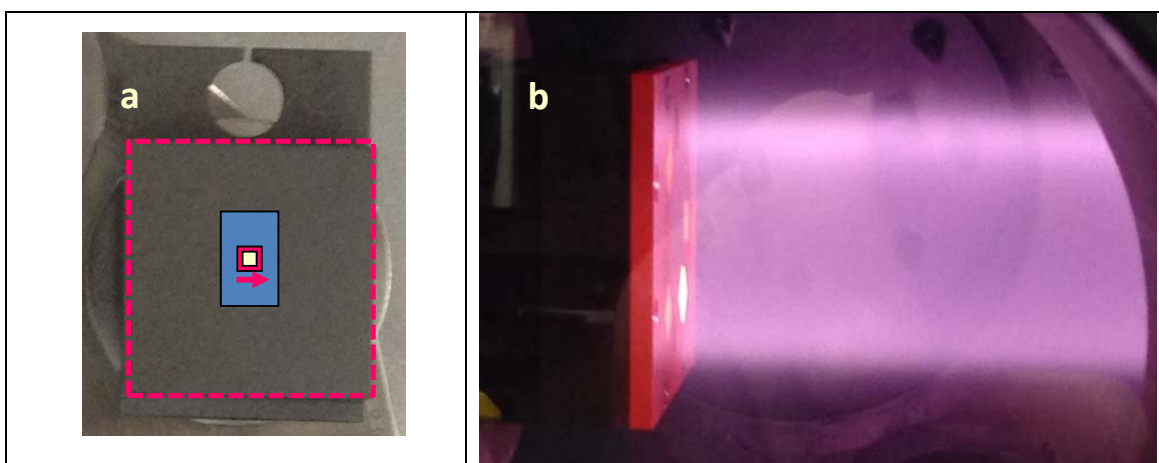
The development of self-passivating tungsten alloys and oxidation tests of their performance under accidental conditions was reported in [2-6]. However, besides the suppressed oxidation, new materials need to be qualified under plasma exposure. The first results of such a test are reported in this paper.

## **Manufacture of smart alloy, pre-characterization and plasma exposure**

The W-Cr-Ti smart alloy samples were produced at the CEIT (Spain) from tungsten, chromium and titanium powders mechanically alloyed using the planetary milling with tungsten carbide milling balls. The alloyed powder underwent hot isostatic pressing at 1200°C at the pressure of 150 MPa. Manufactured bulk material featured small-size grains of tungsten of about 90-110 nm. Details of the manufacturing process and the initial characterization of manufactured

materials are provided in [7]. The ingot of the W-Cr-Ti smart alloy was procured to Forschungszentrum Jülich (FZJ) where samples were produced out of this material using the spark erosion following the standard methods for tungsten processing. The samples had dimensions of 10×10 mm and 10×15 mm with the thickness of 3 and 3.5 mm respectively. Samples were mechanically grinded to avoid the rest deposits from spark erosion cutting. Both tungsten and smart alloy samples were characterized before the plasma exposure. The total weight of the sample was measured using Sartorius MSA225P microbalance with an accuracy of 10 µg in the MirrorLab [8]. Surface roughness  $R_a$  was evaluated using the stylus profiler Dektak 6M from Bruker. Scan locations are shown in Fig.1a. Each scan of surface roughness consisted from five to seven measurements, the final result was averaged.

Time-of-flight Secondary Ion Mass-Spectrometry (ToF SIMS) investigations were made in the middle of each sample using ION ToF IV facility on locations shown in Fig 1a. SIMS measurements provided a depth profiling of the elemental composition of all samples. The investigations were made with both 2 keV  $\text{Cs}^+$  sputter ions for better resolution of non-metallic elements and oxides and with 2 keV  $\text{O}_2^+$  ions for better resolution of metals.



**Figure 1.** Smart alloys and tungsten samples: a) A view of a typical sample with the measurement locations: arrow show the location of surface roughness scan with stylus profiler, large area is the location of SEM surveys, smaller square in the middle is the location of SIMS depth profiles and the smallest square shows the location of FIB cut and b) Exposure of smart alloys and pure tungsten samples in steady-state deuterium plasma of PSI 2 linear device. A side view to the heated tungsten and smart alloy samples mounted into the holder and glowing during plasma exposure is presented.

Scanning electron microscopy (SEM) surface surveys were made on each sample on the area shown in Fig. 1a using the Carl Zeiss CrossBeam XB 540 microscope equipped with the focused ion beam (FIB). Cross-section viewing by FIB was performed in the central area of each sample shown with the smallest square in Fig.1a. Special markers were made with an ion beam on the side surface of each FIB crater. The distance between the two neighboring markers

corresponded to 1  $\mu\text{m}$ . The markers were used to directly measure the material sputtered during plasma exposure.

The pre-characterized samples were installed to the designed sample holder and exposed to steady-state deuterium plasma. The view of the samples during the plasma exposure is presented in Fig.1b. During exposure the plasma parameters were monitored using the moveable Langmuir probe. The measured electron temperature was 30-35 eV, the plasma density was  $N_e \sim 7 \times 10^{11}$  ion/cm<sup>3</sup>. The measured ion flux was  $1 \times 10^{18}$  D/(cm<sup>2</sup>×s). Sample were biased at -250 V. The temperature of the samples was controlled via thermocouple mounted behind the sample and using the infrared FLIR camera. During the exposure the temperature of samples was ranging from 576°C to 715°C as provided in Table 1. Apart from the fact, that such temperatures are not affecting the sputtering coefficients, temperatures for the pairs of samples W1-SA1 and W2-SA2 correlate fairly well, providing an evidence of identical plasma conditions for these pairs of samples. The total duration of the exposure was 3.6 hours, the total accumulated fluence was estimated to be  $1,33 \times 10^{22}$  D/cm<sup>2</sup>.

### Results of exposure and analysis

After the exposure the samples were weighted. For exposed tungsten sample W1 a mass loss of 1000  $\mu\text{g}$  was measured, tungsten sample W2 exhibited the mass loss of 1150  $\mu\text{g}$  as provided in the table 1. The decrease of the mass loss of exposed smart alloy SA1 sample was measured to be 1240  $\mu\text{g}$ . Measured mass loss of tungsten was then used to estimate the experimental sputtering rate of this material. The attained sputtering rate of  $2.4 \times 10^{-4}$  at/ion corresponds well to the experimental data provided in [9] however, it is a bit higher than that expected from theory [10, 11]. The reason for more effective erosion in plasma of PSI 2 linear device may be attributed to the small 0.1% fraction of oxygen in plasma. The calculated removal of material by sputtering from the mass loss measurements was estimated to be: 500 nm for tungsten and about 1000 nm for a smart alloy.

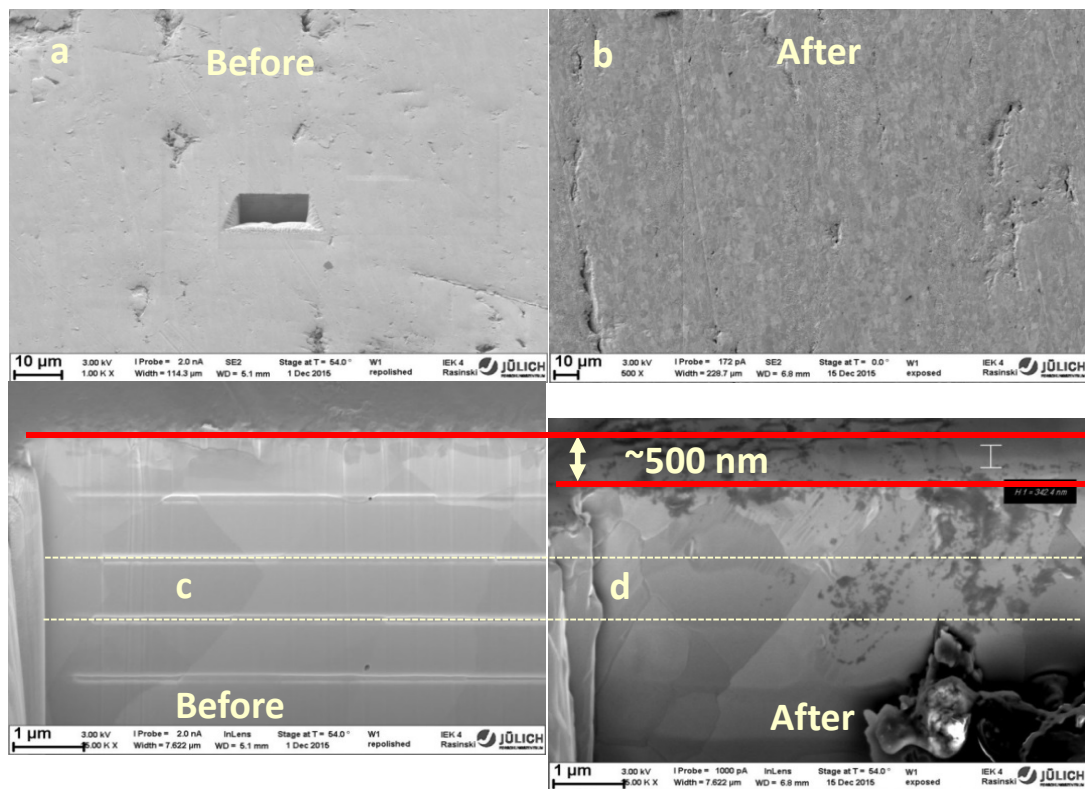
**Table 1.** Characteristics of tungsten and smart alloy samples during and after plasma exposure

Sample	Temperature during exposure, °C	Weight loss during exposure, $\mu\text{g}$	Roughness $R_a$ before exposure, nm	Roughness $R_a$ after exposure, nm
W1	702	1000	215	255
W2	665	1150	232	324
SA1	715	1240	690	377
SA2	576	n.a.	500	n.a.

The sputtered depth was measured directly using the craters pre-marked with the focused ion beam as described earlier. The comparison of the cross-section viewing before and after

exposure is provided on Fig. 2 for pure tungsten sample W1 and on Fig.3 for smart alloy SA1. The removed amount of material due to sputtering from the weight loss measurements was compared for tungsten with the value measured using FIB crater. The expected loss of tungsten was 500 nm, whereas the direct measurements showed 560 nm outlining the perfect correlation between weight loss and direct measurements of sputtered material. The measured material removal from the smart alloy sample was about 800 nm which is in a very good correlation with the weight loss measurements.

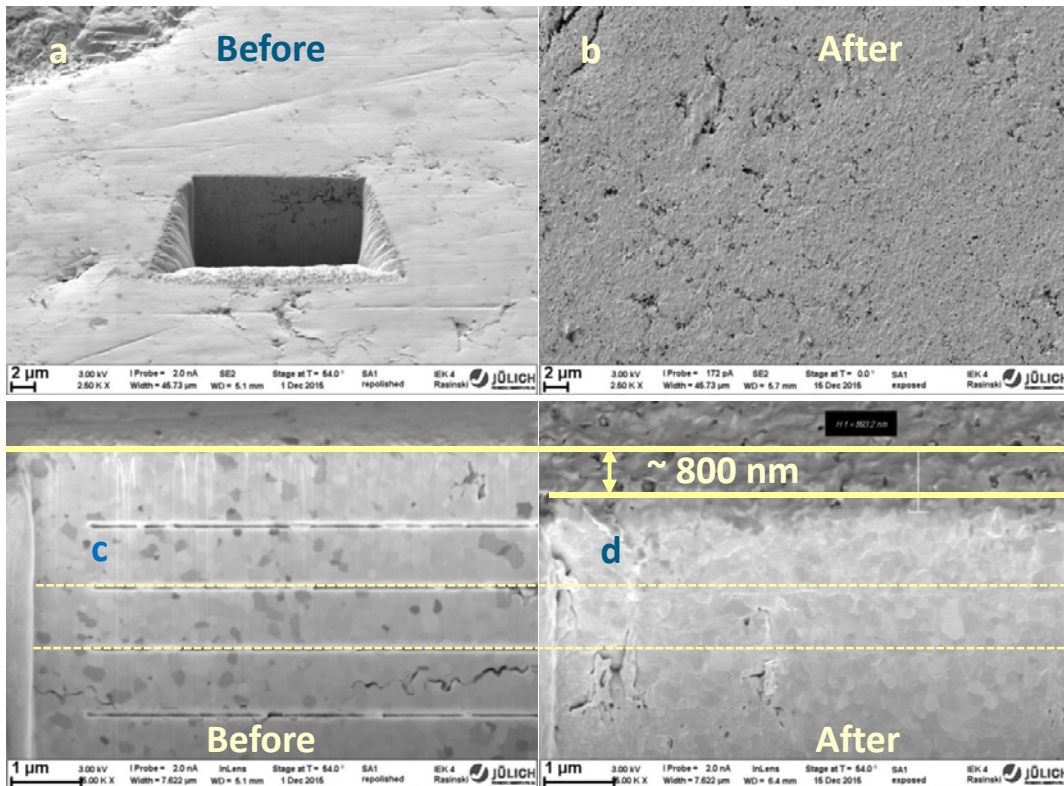
Surface morphology of all the samples was investigated with SEM microscope before and after exposure. The respective SEM photos can be found in Fig.2 for tungsten sample W1 and on Fig.3 for the smart alloy sample SA1. Observations show the slight morphology changes under the ion bombardment. On the surface of smart alloy some hole-like structures with the size less than 500 nm have been detected. Most probably, these structures were formed on locations of alloying elements sputtered by plasma ions.



**Figure 2.** Surface morphology and cross-section viewing of pure tungsten sample before and after exposure to steady –state deuterium plasma: a) top surface before exposure, b) top surface after exposure, c) cross-section viewing before exposure and d) cross section viewing after exposure. The FIB labels are marked on Fig 2c with the dashed lines. The thickness of removed layer is shown on Fig. 2d.



Surface roughness was measured in the middle of all samples after exposure in PSI 2 on the locations shown in Fig 1a. The results of surface roughness measurements are presented in Table 1. A slight increase of surface roughness from 215 to 255 nm for sample W1 and from 232 to 324 nm for tungsten sample W2 was detected. Such an increase of roughness can be due sputtering of polycrystalline tungsten by plasma ions. Different grains of a polycrystalline material are sputtered with the different efficiency leading to the increase of surface roughness. The impact of plasma exposure on surface roughness of smart alloy is more surprising at a first glance. The initially high roughness of the sample SA1 of 690 nm decreased down to 377 nm – a value similar to that measured on pure tungsten samples. The observed effect can be described by the initial fast sputtering of the lighter alloying elements having higher efficiency of sputtering by plasma ions. The depleted surface contains almost pure tungsten and its evolution becomes similar to that of pure tungsten sample. The measured material removed from the smart alloy sample during plasma exposure is 800 nm which already exceeds the initial surface roughness value. This finding implies that the final surface morphology after plasma exposure is dominated mostly by the plasma sputtering and not by initial surface roughness.



**Figure 3.** Surface morphology and cross-section viewing of smart alloy sample before and after exposure to steady –state deuterium plasma: a) top surface before exposure, b) top surface after exposure, c) cross-section viewing before exposure and d) cross section viewing after exposure. The FIB labels are marked on Fig 3c with dashed lines. The thickness of removed layer is shown on Fig. 3d.

The evidence of such a selective sputtering of alloying elements was provided by the dedicated SIMS measurements made on all samples on locations shown on Fig 1a. Prior to exposure, all smart alloy samples featured the homogeneous distribution of tungsten and alloying elements along the bulk of the sample. The example of the SIMS depth profile measured on the sample SA1 after the plasma exposure is shown in Fig. 4. Concentrations of both main (W) as well as alloying materials (Cr and Ti) are plotted along the depth of the sample. As it can be inferred from the depth distributions, tungsten material matrix remains almost unchanged during the exposure whereas Cr and Ti fractions are depleted at the surface till the depth of ~ 50 nm and reach the stationary values only at the depth >75 nm. These results support our initial expectation of preferential sputtering of alloying elements and can explain the observed increasing similarity of the surface roughness of smart alloys and pure tungsten samples reported above.

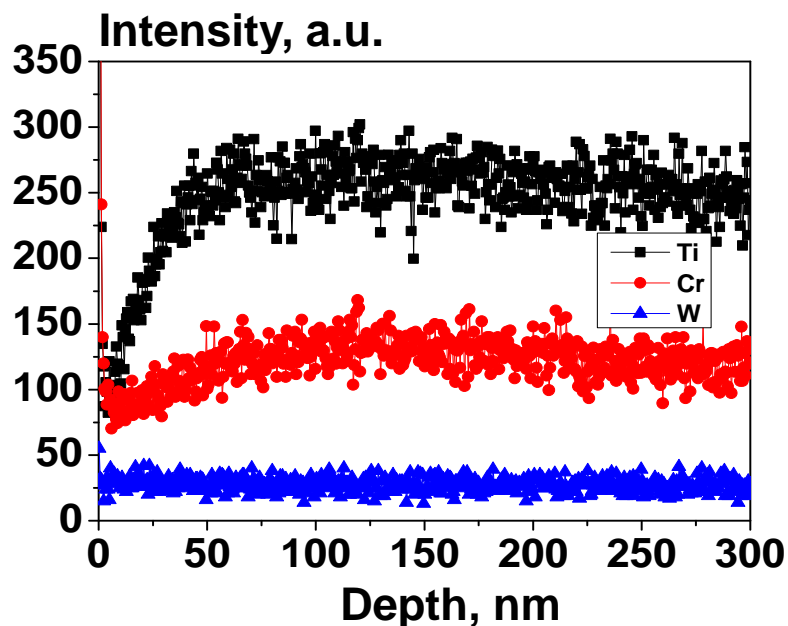


Figure 4. The distribution of tungsten and alloying elements in the depth of the smart alloy sample after plasma exposure

### Summary and outlook

The bulk advanced W-Cr-Ti smart alloys with the suppressed oxidation became manufactured using mechanical alloying followed by HIPing procedure. Smart alloy samples were machined with conventional tools used for tungsten machining without any modifications of tooling or machining procedures.

First direct comparative plasma test of advanced smart tungsten-based alloys and pure tungsten samples was made under conditions expected at the first wall in DEMO. Exposed under identical plasma conditions, tungsten and smart alloy samples demonstrated similar sputtering under plasma ion bombardment in the PSI 2 linear device. Only a moderate change of surface morphology was detected after exposure despite of 500 nm of removed material for tungsten samples and about 800 nm of material removed from smart alloys. At the same time, the expected preferential sputtering of alloying elements during the plasma exposure of smart alloys was confirmed experimentally. The final surface roughness of smart alloys after plasma exposure was similar to that of pure tungsten samples. These findings outline promising features of new advanced tungsten – based materials for the use in future fusion power plants and call for continuation of this promising study.

New yttrium-containing tungsten smart alloys became available recently. The new W-Cr-Y systems feature more effective long-term suppression of tungsten oxidation coupled with better long-term stability of the self-passivating protective layers. The plasma tests of these innovative new smart alloys are in the focus of future research. The sputtering of new smart alloys by plasma, corresponding surface changes and the deuterium retention in the exposed samples is in focus of future research. The combined full working cycle test including plasma and oxidation testing of new systems is of prime importance. Future studies will be carried out to optimize the overall performance of smart alloys including thermo-mechanical properties of these materials such as: thermal conductivity, hardness and ductile-to-brittle transition temperature.

### **Acknowledgments**

The author would like to express the sincere gratitude to our colleagues from the Central Institute of Engineering, Electronics and Analytics of the Forschungszentrum Jülich: to Mr. A. Schwaitzer for help and advices on sample holders and to Mr. K.-H. Junglas for organizing the machining of smart alloy and tungsten samples. We are grateful to Mr. J. Faupel for a fast and efficient commissioning and support of our thermogravimetric facility. A part of these studies has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement N° 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

## References:

- [1] D. Maisonnier et al., A Conceptual Study of Commercial Fusion Power Plants, Final Report, 2005, EFDA-RP-RE-5.0.
- [2] F. Koch and H. Bolt Phys. Scr. T128 (2007)100
- [3] F. Koch, S. Köppl and H. Bolt, J. Nucl. Mater. 386–388 (2009) 572-574.
- [4] F. Koch, J. Brinkmann, S. Lindig, T.P. Mishra and Ch. Linsmeier, Phys. Scr. T145 (2011) 014019
- [5] P. López-Ruiz, N. Ordás N, S. Lindig et al Phys. Scr. T145 (2011) 014018
- [6] C. García-Rosales, P. López-Ruiz, S. Alvarez-Martín, A. Calvo, N. Ordás, F. Koch and J. Brinkmann, Fusion Eng. Des. 89 (2014) 1611-1616
- [7] A. Calvo, N. Ordás, I. Iturriza et al., «Manufacturing and testing of self-passivating tungsten alloys of different composition », Proceedings of the International Conference on Fusion Reactor Materials (ICFRM-17), Po 3-29, Aachen, Germany, October 11-16, 2015
- [8] MirrorLab Website (<https://tec.ipp.kfa-juelich.de/mirrorlab/>), Access details: mirrorlab@fz-juelich.de
- [9] W. Eckstein, IPP Garching Report 9/82 (1993) 116
- [10] W. Eckstein, IPP Garching Report 9/132 (2002) 211
- [11] “Sputtering by particle bombardment: Experiments and Computer Calculations from Threshold to MeV Energies” R. Behrisch and W. Eckstein (Eds), Springer, Berlin, 2007.