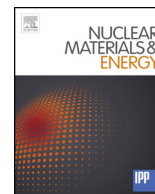




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WCrY smart alloys as advanced plasma-facing materials – Exposure to steady-state pure deuterium plasmas in PSI-2

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ABSTRACT

In this paper the impact of steady state pure D plasma on WCrY smart alloys at ion energies of 120 and 220 eV is reported. For this purpose a comparison with simultaneously exposed pure W samples is drawn. Different analysis techniques employed for pre- and post-plasma sample analysis hint at a significant depletion of Cr and enrichment of W for lower ion energies. Preferential sputtering leads to enhanced volumetric loss at 220 eV. Analysis of redeposited material indicated local redeposition of Cr. Modelling the ion irradiation with SDTrimSP is used to further interpret experimental results. Depending on the sample temperature during plasma exposure and the magnitude of the ion flux, diffusion of Cr towards the surface is a determining factor for erosion of smart alloys for higher ion energies.

1. Introduction

An essential part of future fusion reactors is the so-called first wall, the wall directly facing the plasma and therefore protecting structural parts of the vessel. Only a few materials are suitable to be used in fusion environment. Among other requirements described for instance in [1], the armour material has to withstand high heat loads, feature low tritium retention and moreover show only minor activation during neutron irradiation. Tungsten (W), currently the preferred first wall material for the next step fusion devices such as DEMO, possesses many advantages. These include a very high melting point and low erosion yields during ion irradiation. However, for the development of W-based wall components some of the material's drawbacks have to be improved. Besides its inherent brittleness, a further drawback of pure W consists in fast oxidation when coming into contact with oxygen (O). During accidental reactor conditions (Loss-Of-Coolant-Accident: LOCA) the cooling system fails. Additionally air ingress may occur. W oxidises and forms radioactive WO_3 . Due to nuclear decay heat wall temperatures rise to above 1000°C for several months [2] leading to the mobilisation of radioactive material into the environment. In order to establish intrinsic reactor safety, W-based smart alloys are currently being

developed aiming at preserving W-like behaviour during plasma operation while suppressing oxidation in case of LOCA. The most promising systems in terms of passivation behaviour feature chromium (Cr) as passivating element as well as yttrium (Y) [3]. Y serves as active element facilitating Cr transport towards the alloy's surface during oxidation and adding to the stability of the oxide scale. So that according to [4] the addition of Y to the WCr-system reduces the mass gain significantly by supporting the formation of a continuously growing, well adhering and dense Cr_2O_3 layer. These alloying elements possess a lighter mass compared to W and are therefore more easily sputtered during plasma operation. Preferential sputtering leads to depletion of Cr and Y, and enrichment of W, leaving a pure W surface facing the plasma.

Newly developed WCrY-systems demonstrated a significant oxidation suppression of more than five orders of magnitude in comparison to pure W [5]. In 2017 these systems have been exposed to plasma for the first time [5]. This paper reports in detail on the first plasma exposure of WCrY smart alloys. Moreover experimental findings of a recently conducted second plasma experiment at lower ion energies are covered and a comparison to simulation results obtained with SDTrimSP [6] are drawn.

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2. Experimental setup and analytic techniques

2.1. Sample preparation

Samples used for the plasma experiments reported in this paper were obtained by using Field-Assisted Sintering Technology (FAST) [7] at Forschungszentrum Jülich (FZJ). The composition of all exposed smart alloy samples is W-11.4wt%Cr-0.6wt%Y. WCrY samples exposed simultaneously were produced from the same powder batch. The name convention is as follows: samples SA11 and SA12 were exposed during the first plasma experiment (PE1) at an ion energy of 220 eV, while samples SA21 to 23 were exposed during the second experiment (PE2) at lower ion energies (120 eV). For the sintering process of samples SA11 and SA12 a uniaxial ramp of 200 °C/min, a maximum pressure of 50 MPa and a holding time of 1 min at a maximum temperature of 1550 °C were used. FAST parameters for samples SA21 to SA23 differ from SA11 and SA12 in a maximum temperature of 1460 °C after which no holding time was applied.

The WCrY samples investigated in the two plasma exposures were produced using slightly different FAST parameters. For samples in the second plasma experiment, SA21 to SA23, optimised FAST parameters were used. As a result for these samples a fine sub-micrograin structure and improved oxidation resistance as referred in [5] compared to samples produced for the first plasma experiment were obtained. However, plasma performance is not expected to significantly differ. There may be a small difference due to enhanced Cr-mobility by providing smaller grains and thus more grain boundaries in the material. Consequently, bulk WCrY samples with small WCr-grains and finely dispersed yttrium oxide (Y_2O_3) particles were obtained. Because of the small size of the yttrium oxide particles it is difficult to investigate the exact composition of the yttrium oxide, but as Y_2O_3 is the most stable yttrium oxide we assume most of the yttrium oxide to have this stoichiometry. Details of the sample production and the resulting microstructure can be found in [5].

Samples are cut from the smart alloy ingots by means of wire erosion to fit the PSI-2 mask geometry. To be able to hang the samples in the furnace for oxidation studies after exposure, a hole is added to the sample geometry (see Fig. 2c). W samples exposed alongside with the afore-mentioned WCrY samples for direct comparison were all cut from the same pure W piece by wire erosion. The plasma-facing surface of W and WCrY samples spans over 1 cm × 1 cm. In order to remove residues from the wire erosion process and ensure the same surface properties, all samples were manually ground. For this purpose different silicon carbide grinding papers (SiC P) were used following a defined sequence. The last paper used within this sequence defines the surface roughness of the ground samples. Generally SiC P1200, with a SiC particle size of around 15 μm , is used as the last paper in this sequence. For one W sample and one WCrY sample of PE2, P180 with a SiC particle size of about 82 μm was used to obtain a rougher surface and

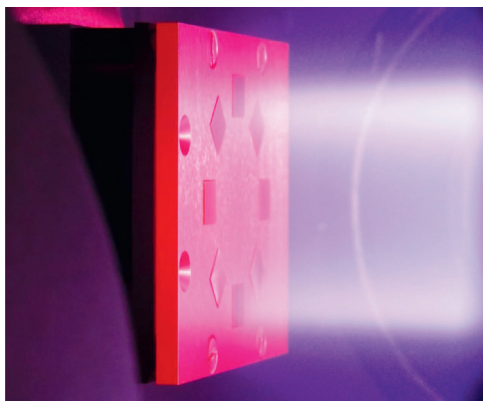


Fig. 1. Plasma exposure of WCrY and W samples in PSI-2.

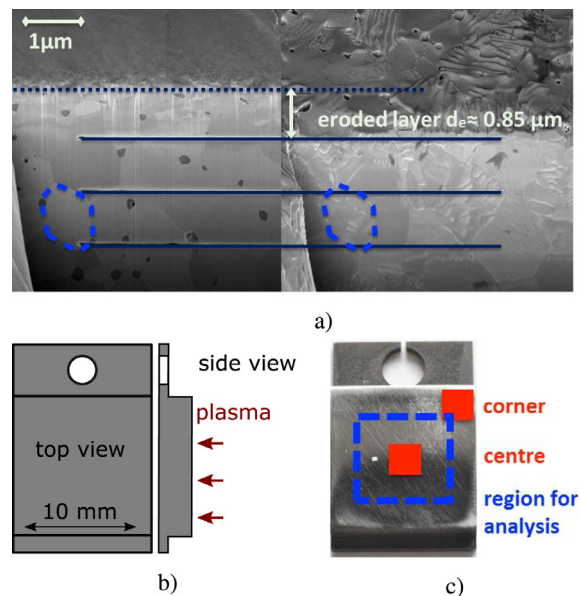


Fig. 2. a) comparison of FIB marker positions relative to the surface before and after plasma for sample SA21, b) sketch of sample geometry for smart alloys, top and side view where the plasma-facing surface is indicated c) photograph of smart alloy (top view), centre and corner positions for FIB craters and analysis region are indicated.

compare the performance of these samples to the smoother ones.

2.2. Analysis methods

A variety of methods were employed for pre- and post-plasma analysis: For assuring a clean sample surface and investigating changes in the smart alloy's depth-resolved composition, Time-of-Flight Secondary Ion Mass Spectroscopy (ToF-SIMS) was used, here the specific setup is IonToF IV. The SIMS technique uses two alternating ion beams for destructive surface analysis. The primary or sputtering ion beam consisted of O^{2+} ions, while Bi^{3+} was used for the analysis presented in this paper. The sputtered area amounts to $300 \times 300 \mu\text{m}^2$, while the size of the analysed area is $47 \times 47 \mu\text{m}^2$.

To infer the local erosion during plasma exposure, a crater featuring an orthogonal cut relative to the sample's surface was created by aid of the Focussed-Ion-Beam (FIB) technique prior to plasma exposure. Equidistant markers are generated onto that orthogonal cut (see Fig. 2) so that after plasma exposure the local erosion can be inferred directly by comparing the distance from the surface to the uppermost FIB marker. Within the same analysis device, a combined SEM-FIB system Carl Zeiss CrossBeam XB540, images displaying the sample's surface and microstructure are taken using Scanning Electron Microscopy (SEM). Global erosion or weight loss measurements consist in weighing the samples before and after plasma exposure with a Sartorius MSA225P microbalance with a resolution of 10 μg . With a stylus profiler Dektak 6M several line scans across the sample's surface were conducted, aiming at assessing the surface roughness before and after exposure. During each line scan a tip with a radius of 2.5 μm is drawn across the surface. Linear fitting and discrete fourier transformation are applied for separating defects from cutting or the nonplanarity of the surface from the surface profile before the arithmetic average height R_a (see [8]) is calculated. X-ray Photoelectron Spectroscopy (XPS) is employed to obtain a higher resolution of the elemental composition of the smart alloy's surface composition. For the current XPS-setup, which uses a $Al K_{\alpha}$ X-ray source, the depth from which radiation is still emitted amounts to approximately 5 nm in W according to the Lambert-Beer law.

At FZJ Nuclear Reaction Analysis (NRA) is available. A μ -NRA setup

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