

Advanced Smart Future Fusion Power Plant

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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 1000o 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 1.26×10^{26} ion/m². The weight loss of pure tungsten samples after exposure was $\Delta mW = 1000-1150$ μg . The measured weight loss of sputtered smart alloy sample $\Delta mSA = 1240$ 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.

I. Introduction

Tungsten is among prime candidate materials to be used for plasma-facing components in the future fusion power plant. Among the advantages of tungsten for fusion applications are: low sputtering yield by plasma ions, high thermal conductivity at elevated temperature, low retention of radioactive tritium and high melting temperatures. At the same time, the construction of the future power plant will raise significantly the limits and requirements which are imposed on the plasma-facing materials. In addition to already harsh plasma conditions expected in next fusion experiments the accumulated neutron fluence is expected to be orders of magnitude higher bringing several physical and engineering challenges including the high level of the nuclear decay heat at the PFC.

The expected stationary or quasi-stationary operation will extremely increase the plasma particle fluxes towards the plasma-facing components. According to the recent investigations [1], none of existing materials will withstand the combination of particle and heat loads and intensive neutron irradiation. MPT/P5-6 2 In particular, in case of Loss-of-Coolant-Accident (LOCA) with an air ingress, the temperature of non-cooled tungsten components may rise till 1200o C and remain at such a level for several weeks [2]. Activated tungsten forms at these temperatures the radioactive oxide which will be mobilized into atmosphere at a rate of 1 -100 kg/h.

This is an unacceptable value. In order to counteract the undesirable oxidation of tungsten and to keep its advantageous features the new advanced, so-called “smart” alloys were introduced. Smart alloys are supposed to adjust their properties to the environment. During regular plasma operation of power plant, preferential sputtering of alloying elements will leave almost pure tungsten surface turned to plasma. In case of an accident, such a thin tungsten – rich surface of ~ 10 nm will oxidize and will be mobilized into environment. Alloying elements in the bulk of the smart alloy will however, remain. These alloying elements will create their own dense oxide layers protecting tungsten matrix from oxidation and evaporation. Smart alloys provide an essential element of the so-called intrinsic safety of the future power plant. The schematic of operation of smart alloy is provided in Fig.1.

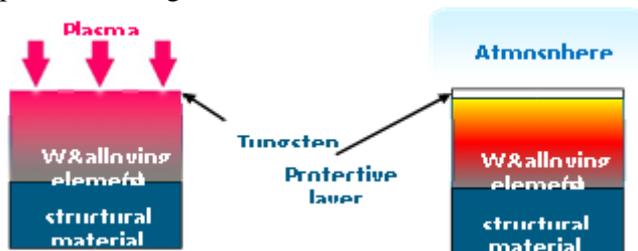


Figure 1. The scheme of operation of smart alloys during the regular plasma operation and under accidental conditions.

There are several essential limitations imposed on alloying elements for the fusion applications. Alloying elements need to possess low activity, ability to form stable oxides and low volume increase during oxidation. Among remaining alloying elements are Cr, Si, Vn, Ti and Y.

Investigations of self-passivating tungsten-based smart alloys

There is an essential research on self-passivating smart alloys [3-4]. Investigations made on binary and ternary systems led to discarding Si-containing systems [5]. The present investigations are focused on ternary chromium-containing smart alloys.

The present paper is focused on newest developments in advanced smart alloys made and studied at the Forschungszentrum Jülich GmbH (FZJ) and provided by the partner institute CEIT, Spain. The research on smart alloys comprise the dedicated studies on the near-to-ideal, homogenous at atomistic scale, well-defined thin (3-7 μm) films produced by magnetron sputtering. Such thin films are being used for the understanding of fundamentals of the oxidation and for initial evaluation of the new smart alloys. At FZJ, a multi-electrode magnetron system from PREVAC was used for production of thin-film alloys. Three targets made from tungsten and alloying elements are depositing material onto a rotating table with samples. The essential features of smart alloys are their resistance to plasma sputtering and the suppression of the oxidation.

Suppressed oxidation most obviously manifests itself via low mass gain in the course of the controlled oxidation. Such measurements are made using the symmetrical double-oven thermogravimetric (TGA) system TAG 16 from Setaram. The system is also equipped with the steam generator allowing performing experiments in humid atmosphere. The TGA system is located in the new just commissioned ThermoLab at the FZJ. Mass change was monitored using the high-resolution microbalance. The surface roughness was monitored using stylus profiler Dektak 6M from Bruker. Visual observations were made using the inverted microscope Carl Zeiss Axio Observer Z1m. All mentioned equipment is located in the MirrorLab [8] at FZJ. In addition, the crystallographic structure, phases of constituents can be investigated using XRay Diffraction (XRD) system from Bruker.

Elemental composition of matrix and alloying elements along the depth of the probe was investigated using Secondary-Ion-Mass-Spectrometry ION-ToF IV system. Elemental composition, morphology, homogeneity was investigated using Carl Zeiss CrossBeam XB 540 a combined scanning electron microscope (SEM) and focused ion beam (FIB)

system. The CrossBeam is equipped with electron back-scattering system (EBSD), energy-dispersive X-ray analysis (EDX), transmission electron module (STEM), plasma cleaner and the vacuum gate system.

Yttrium-containing thin film smart alloys

Good oxidation resistance was observing when yttrium was introduced as a so-called active element in the alloy. Yttrium is primarily known from its successful application in the steel industry. Adding yttrium to alloy system may:

- Introduce pegs at the interface between the alloy and the oxide scale [6, 7]. Pegs are acting as a connection between the oxide and the alloy which may increase the adherence of the oxide scale to the alloy system.
- Alter transport mechanisms through the oxides. Yttrium is usually segregated to the grain boundaries [8-9] where the transport occurs. The oxygen scale grows from the alloy and yttrium acts as a vacancy sink to suppress the formation of voids and pores at the interface [10-11].
- Due to its chemical reactivity, yttrium is actively bonding the impurities preventing them from entering the oxide scale. The presence of impurities in the oxide may otherwise destabilize the oxide layer.

Yttrium is known for its ability to limit the grain size growth [12] and to decrease the growth rate of an oxide scale [13]. This element is shown not to have cation transport moreover, it suppresses the cation transport of the other elements, leading e.g. to a decrease of the growth stresses and improved adherence, when Y is present in small amounts [14].

Yttrium-containing films produced at FZJ featured very good homogeneity. Chromium fraction contributed to apr. 8 to 12 weight %, an amount of Y was about less than 1 wt. % of the alloy. The results of the comparative oxidation study of pure tungsten and smart alloys are provided in Fig. 2. The oxidation rate of W-Cr-Y alloy, characterized by a mass increase is significantly lower than that of any shown by W-Cr

and W-Cr-Ti systems. The microscope and FIB investigations revealed the formation of the thin and dense chromium oxide protecting tungsten from oxidation and subsequent evaporation. The respective FIB scan is provided in Figure 3. Obviously, the formation of such a dense protective layer is responsible for the suppressed oxidation. The series of TEM investigations were made on thin lamella cut from the upper part of the oxidized thin-film smart alloy. The EDX mappings of the constituent elements are shown in Figure 4. The created protective Cr_2O_3 oxide contains no trace of tungsten. What is more interesting however, is the distribution of Y. During investigations two pronounced distributions of Y were detected. Firstly, there are some pre-cursors of the formation of the nucleation sites which is agreement with the expected distribution of Y.

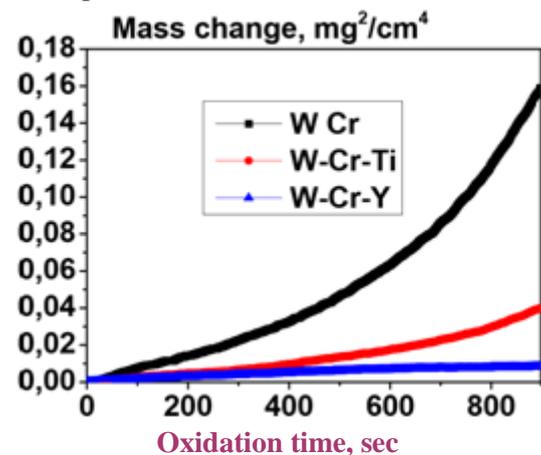


Figure 2. Oxidation of different smart alloys in 80 vol.%Ar + 20% vol.% O₂ at 1 bar and the temperature of 1000°C

At the same time, there is obviously homogenous distribution of yttrium in the bulk of the sample. Whether the nucleation sites are responsible for the good passivation or the homogenous distributions and hence immediate availability of yttrium in the bulk of the sample are playing the decisive role in the passivation - remains an open question. It is supposed that the observed two different distributions of Y may play the decisive role in understanding of the oxidation behavior of the bulk smart alloys.

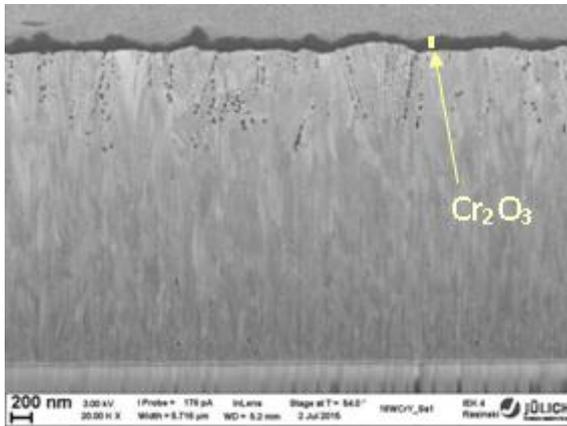


Figure 3. FIB cut of W-Cr-Y thin film smart alloy after 10 minutes of oxidation at 1 bar in 80 vol.% Ar + 20% vol.% O₂ at 1000°C

Knowledge obtained after studies of the model thin-film systems was afterwards applied for production of the bulk smart alloys. Presently, the manufacturing route towards the bulk smart alloy comprises several steps. On the first step, mechanical alloying of initial elemental powders is made using the planetary milling system. The alloyed powder underwent hot isostatic pressing at 1200°C at the pressure of 150 MPa. Manufactured bulk material featured nanocrystalline tungsten grains of about 90-110 nm and contained 10 wt.% of Cr, 2 wt.% of Ti and the rest of W.

Details of the manufacturing process and the initial characterization of manufactured materials are provided in [6].

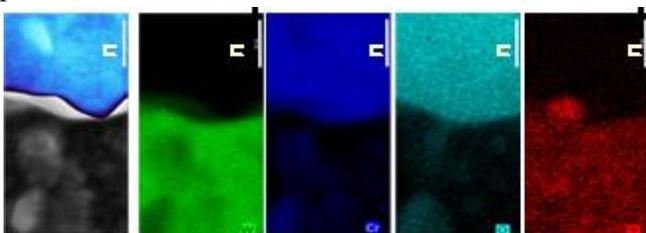


Figure 4. Color maps of the EDX measurements made on the TEM lamella, at the interface between smart alloy and the protective oxide layer.

Plasma exposure of smart alloys and pure tungsten samples

In addition to the suppressed oxidation, new materials need to be qualified under plasma exposure. The first results of such a plasma exposure along with the subsequent oxidation of exposed tungsten and smart alloy samples are reported in this paper. The W-Cr-Ti smart alloy samples were produced at CEIT (Spain) from elemental tungsten, chromium and titanium powders. The alloyed powder underwent hot isostatic pressing. Manufactured bulk material contained 10 wt.% of Cr, 2 wt.% of Ti and the rest of W. Tungsten and smart alloy samples were characterized before the plasma exposure. 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 1µm. The markers were used for direct measurements of the material sputtered during plasma exposure.

The pre-characterized samples were exposed to steady-state deuterium plasma in linear plasma device PSI 2 [15]. During exposure 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/cm³. The measured ion flux was 1×10^{18} D/(cm²×s). Samples 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. The total duration of the exposure was 3.6 hours, the total accumulated fluence was estimated to be 1.3×10^{26} D/cm².

First studies at extreme temperatures 1000°C- 1200°C

Since the temperatures of up to 1200°C are expected to happen during accidental conditions [16], further investigations were focused on studying the oxidation processes at high temperatures. The results of the isothermal oxidation of the thin film W- Cr and W- Cr- Y samples carried out at 1000°C and

oxidation of thin film W- Cr- Y at 1200°C O₂ are presented in Fig. 5. An oxidizing atmosphere contained 80 vol.% of Ar and 20 vol. % of O₂.

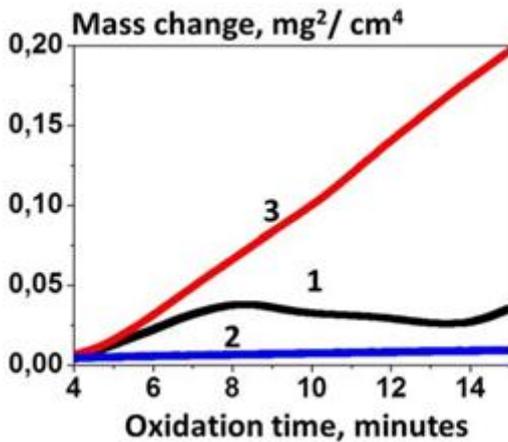


Figure 5. High temperature oxidation of W- Cr and W- Cr- Y alloys. Squared mass change as a function of exposure time for various thin film samples: 1) W- Cr sample exposed at 1000°C, 2) W- Cr- Y sample exposed at 1000°C and 3) W- Cr- Y sample exposed at 1200°C. Exposures were conducted in humid atmosphere containing 20 vol.% O₂ and 80vol.% of N₂.

W- Cr system demonstrated the parabolic dependence on time only for the first 8 minutes followed by the failure of Cr₂O₃ protection and sublimation of WO₃. The parabolic mass change with the time was seen for both W- Cr- Y systems. However, the oxidation constant k_p in case of 1200°C is almost 10²- fold larger than that at 1000°C. Most probable reason is the increased sublimation of the protective chromium oxide at elevated temperatures. These findings also raise questions on applicability of the smart alloy concepts at elevated temperatures around 1200°C.

Oxidation in humid atmosphere

The dedicated study under the most realistic accidental scenarios was conducted. In these studies the controlled thermal oxidation was made in the atmosphere of the synthetic air containing 20 vol. % of O₂ and 80 vol % of nitrogen (N₂) introduced into the reaction chamber of the TGA oven. A humid air

atmosphere was prepared in the vapor chamber of the new Setaram Wetsys gas supply system. For experiments 70% relative humidity at 40°C fed into reaction volume at the pressure of 10⁵ Pa at the temperature of 1000°C. Dedicated preparative experiments were made which prove no effect of N₂ on oxidation behavior. The dependencies of mass change on time are shown in the Fig. 6. There are several peculiarities as can be noticed from the Fig. 6. Several “bumps” on the graph relate to mass loss occurred during the exposure. The corresponding dependencies are outlined in Fig 6. with arrows. The observed loss of mass may be directly connected to the sublimation.

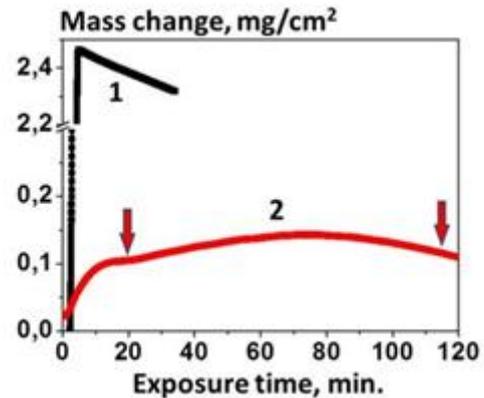


Figure 6. Humid air oxidation and sublimation. Mass change as a function of exposure time for 1) pure tungsten and 2) thin film W- Cr- Y smart alloy.

The dedicated investigations were carried out with pure elements – in order of investigating the main element responsible for strongest reaction in humid air environment. The respective dependencies on the time are shown on Figure 7 as the mass change due to sublimation after the complete oxidation of the studied pure W and Cr samples. It should be noted that the chemical reactivity of tungsten and alloying elements towards the water and humidity can significantly intensify and even dominate an oxidation process.

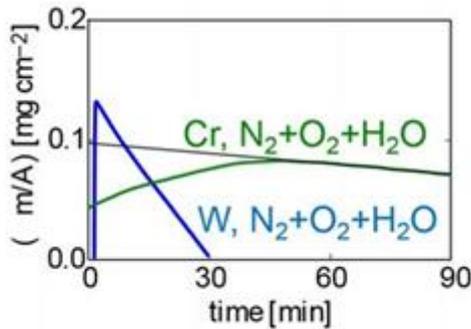


Figure 7. Results of separate oxidation of alloying element (Cr) and tungsten (W) in humid air

From the figure below it can be seen directly, that Cr demonstrate a very moderate reaction with humid air. In contrast, a very strong reaction of WO_3 with humid air was detected. This reaction is obviously dominating the entire mass changes during the exposure of smart alloy described above. The intensified chemical reactivity of tungsten may arise e.g. from the increased penetration of oxygen in the form of OH^- ions through the protective Cr_2O_3 oxide layer from and the following buildup of the partial pressure of oxygen at the interface between the protective layer and the alloy. This would lead to the formation of tungsten oxide and its subsequent volumetric increase, cracking of the protective chromium oxide layer and finally, the sublimation of tungsten oxide through the damaged layer as reported. The stability of the protective Cr_2O_3 layer certainly needs to be increased. However, the further improvement of protection action using the thin film smart alloy samples with their very limited reservoir of Cr needed for protection layer, is difficult.

Summary and outlook

New advanced materials are required for the safe and reliable operation of the future fusion power plant. Among these materials, self-passivating tungsten-based smart alloys are aimed at getting the acceptably low plasma sputtering rate during regular plasma operation and suppressed oxidation of tungsten under accidental conditions.

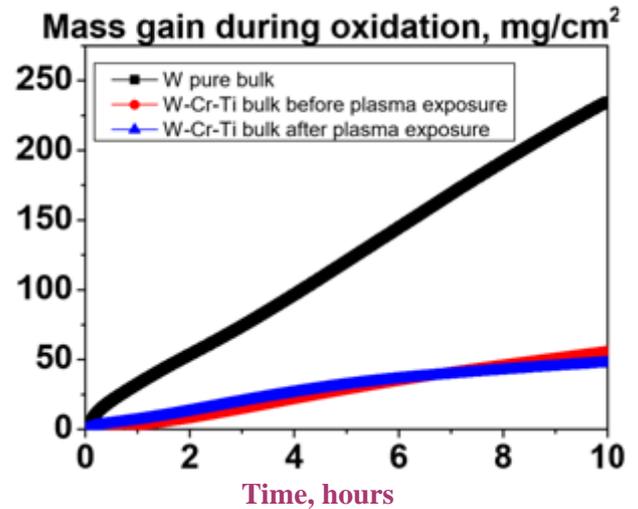


Figure 8. Mass gain caused by the oxidation of the pure tungsten and W-Cr-Ti smart alloys.

Very promising results were reached with the model smart alloy thin films containing yttrium. Adding the small amount of < 1 wt.% of yttrium to the tungsten-chromium alloy was capable to stabilize the protective Cr_2O_3 layer, to make it more compact and dense and decrease the oxygen diffusion into alloy bulk. The remarkable oxidation rates of $3 \times 10^{-6} \text{ mg}^2/\text{cm}^4$ per second were attained, constituting the more than 10^5 -fold difference to that of pure tungsten. Tungsten-chromium-yttrium systems are also featuring the most stable passivation behavior among all studied smart alloy systems.

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. The expected preferential sputtering of alloying elements during the plasma exposure of smart alloys was confirmed experimentally. Controlled oxidation of smart alloys did not reveal any negative effect of plasma exposure on the suppressed oxidation. 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 protective layers. The plasma tests of these innovative new smart alloys are in preparation. 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.

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