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Development of tungsten coated first wall and high heat flux components for application in ASDEX Upgrade

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Abstract

In the tokamak experiment ASDEX Upgrade, the investigation of tungsten as a first wall material is an ongoing research project. In a step-by-step strategy, the tungsten covered surface area is increased from campaign to campaign. For this purpose an industrial-scale method for coating graphite with micrometer tungsten films had to be identified. Test coatings deposited by magnetron sputtering and by plasma-arc deposition were compared. By X-ray analysis it was found that sputter-deposited coatings suffer from high compressive stress (1.7 GPa). This leads to delamination when a film thickness of about 3 μm is exceeded. For arc-deposited coatings, a compressive stress value of 0.5 GPa was determined and no delamination occurred up to the maximum film thicknesses investigated, i.e. 10 μm. Upon thermal loading, none of the arc-deposited coatings failed up to the melting condition, while one sputter-coating delaminated. First results on similar investigations employing CFC substrates are presented.

1. Introduction

In current fusion devices, plasma facing components are typically materials of low atomic number, mainly carbon is employed. Carbon is an ideal material concerning the heat load because of its advantageous thermophysical properties. Concerning its erosion behavior under hydrogen impact, carbon is acceptable because of its relatively low potential for radiative plasma cooling. Chemical elements with low atomic number are fully ionized at the typical plasma temperatures of a nuclear fusion experiment and therefore cannot emit line radiation. The drawback of this material, however, is the high erosion yield and the subsequent strong accumulation of hydrogen in the vessel by co-deposition in the form of hydrocarbon films (see for example [1]): For the International Thermonuclear Experimental Reactor (ITER-FEAT), it has been estimated that amounts of tritium in excess of 2 g per 400 s of plasma discharge are retained within the vessel in the form of hydrocarbon films [2]. Therefore a next-step facility operating with tritium will require an alternative to carbon.

For high-Z materials like molybdenum or tungsten, tritium accumulation by co-deposition is not expected to be a problem. In addition, high-Z materials in general offer the advantage of low sputtering yields as compared to low-Z materials like beryllium or carbon, which would make high-Z the preferential choice from the viewpoint of component lifetime. On the other hand, their potential of radiative plasma cooling is considerably higher, and therefore the allowable concentrations permitted within the plasma are correspondingly low [3].

Replacing low-Z with high-Z first wall materials therefore represents a trade-off between erosion yields and allowable plasma impurity concentration. The choice of materials is not obvious, as both parameters change by orders of magnitude. Therefore such a replacement of first wall materials needs to be tested under realistic conditions.

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Table 1
The tungsten program at ASDEX Upgrade

Divertor	Ref. [4]	1993/1994	W markers and inlays
	Ref. [5]	1994/1995	First coated test tiles
	Ref. [6]	1995/1996	Tungsten divertor experiment
Installation and investigation of Divertor II			
Main chamber	Ref. [9]	07/1998	Siliconization: first step to 'high-*Z*'
	Ref. [7]	1998/1999	First W-coated test tiles
	Ref. [8]	1999/2000	1.2 m^2 of coated surface area
	Ref. [16]	2000/2001	Increase to 5.5 m^2
		From 10/2001	Fully coated inner wall (except CFC)

2. The W program at ASDEX Upgrade

The replacement of graphite with tungsten on the first wall is under investigation in the tokamak experiment ASDEX Upgrade. For the reasons mentioned above, this has been done in a step-by-step approach [4–9]; this process is displayed in Table 1. In the current phase, tungsten coated tiles are installed at the central column of the main chamber. With each campaign, additional tungsten coated tiles were added to the central column. In the last experimental campaign (which ended in August 2001), about 5.5 m^2 of surface area consisted of tungsten coated graphite tiles. The need for coating several hundred tiles with tungsten films of a thickness around 1 μm required the identification of an industrial-scale coating technique. For this purpose, we first investigated sample coatings on fine grain graphite manufactured by two different PVD techniques. The main parameters for evaluating the sample coatings were their adhesion properties – especially under thermal loading conditions, their content of light impurities, and the degree of surface coverage of these thin films on relatively rough fine grain graphite surfaces. In the coming campaign starting in October 2001, the central column will be fully covered with tungsten coated tiles, except for the neutral beam injection shine-through beam dumps, which are made of fiber reinforced carbon (CFC). Therefore, in a second step, after defining the preferred coating technique for fine grain graphite, coatings on CFC materials are to be evaluated.

3. Comparison of PVD techniques

Sample coatings were prepared at two different institutions capable of manufacturing large quantities of coated tiles:

At the Fraunhofer-Institut für Elektronenstrahl- und Plasmatechnik (FEP) in Dresden, Germany, samples were coated by magnetron sputtering. In this case a parameter study was performed, varying the film thickness from 0.5 to 10 μm and the substrate temperature from 150 to 600 °C. The deposition was performed at an Ar pressure of 3×10^{-3} mbar with an average growth rate of 2 nm/s. The substrates were Schunk FU4206 graphite tiles, pre-treated by polishing to an average roughness of 1–1.5 μm, ultrasonic cleaning in water, and subsequent baking of the tiles at 400 °C.

Plansee AG, Reutte, Austria, prepared sample coatings in the range 1–10 μm by an arc-deposition method. Two types of graphite substrates were used (SGL Carbon R6710 and Schunk FU4206), which differed mainly in their average grain size. In the investigations described below, no significantly different results were obtained for the two types of samples. The surface pretreatment was milling, which resulted in about the same surface roughness of the substrates as above. Information about further surface cleaning procedures as well as the actual deposition parameters were not released by the company. Tungsten film thicknesses were determined gravimetrically in all cases, some samples were cross-calibrated by Rutherford backscattering spectroscopy.

3.1. Adhesion and film stress

Fig. 1 shows cross-sectional SEM images from samples deposited by plasma-arc (top) and by sputtering (bottom). The arc-deposited coating shows a dense structure with a relatively smooth surface. The sputter-deposited coating, on the other hand, has a columnar structure which induces an intrinsic surface roughness.

With both coating methods a series of films with a thickness of up to 10 μm was investigated. While the arc-deposited films showed no signs of failure at any thickness and independent of the substrate material, it turned out to be impossible to deposit films with a thickness exceeding 3 μm by the sputtering method: Delamination occurred in form of small flakes with diameters up to the millimeter range, when a certain thickness threshold was exceeded. The threshold value itself depends on the respective substrate surface pre-treatment. The pattern of delamination – small flakes instead of crack formation – hints to compressive stress as the reason for the loss of adhesion. Formation of compressive stress in sputter-deposited metal coatings can be found in the literature:

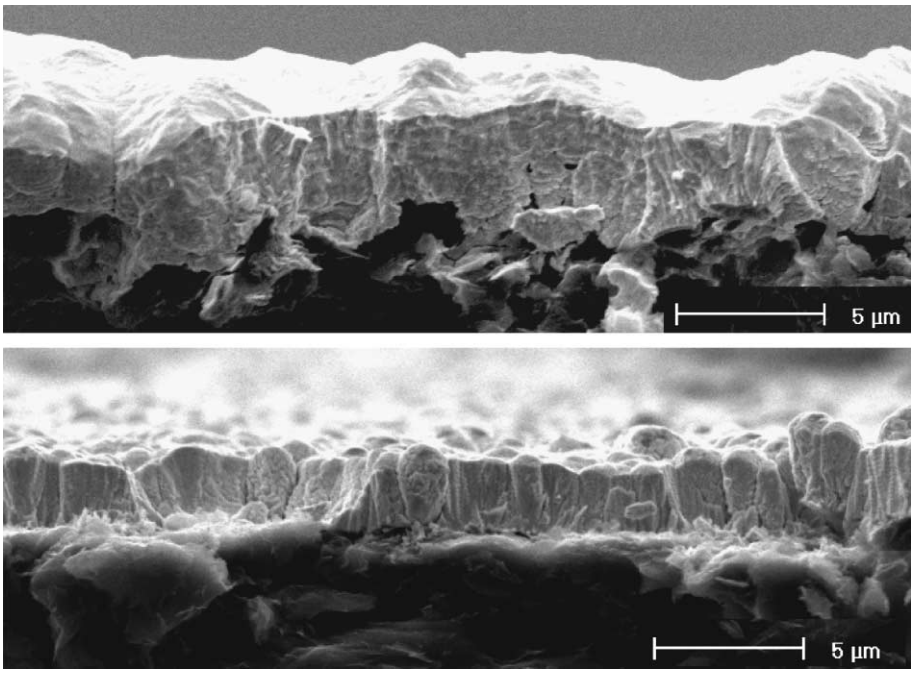

Fig. 1. SEM cross-sectional imaging of the two types of tungsten coatings. A 4 μm plasma-arc coating is shown in the top image, a 3 μm sputter-deposited coating is shown in the bottom image. Note the columnar structure in the lower image.

In a series of papers Thornton and Hoffman reported a very general formation of compressive stress for metal films ranging from aluminum to tungsten, when the working gas pressure went below a mass dependent critical value [10–12]. In the case of tungsten compressive stresses up to 2.3 GPa were reported [12]. Therefore we determined the in-plane stress state of our films by X-ray diffraction using the $\sin^2 \Psi$ method. To obtain stress values from the measured X-ray peak shifts, the Young's modulus and Poisson ratio of bulk tungsten, i.e. $E = 400$ GPa and $\mu = 0.28$ were employed. This yielded compressive stress in both kinds of films with average values of 1.7 GPa for the sputter-deposited coating and 0.5 GPa in the case of arc-deposition. We therefore attribute the loss of adhesion for sputter-deposited coatings to the formation of excessive compressive stress.

3.2. Summary of other fusion-relevant properties

A detailed comparison of the two types of coatings has been published in [13]; this includes the adhesion results from the magnetron sputtering parameter study, other fusion-relevant properties of the two types of coatings, as well as results from thermal screening tests. Therefore these results will only be summarized here.

In the ASDEX Upgrade tungsten divertor experiment sputtering by light plasma impurities originating from the main chamber walls was found to be the dominating mechanism of tungsten erosion [6]. Therefore the degree of surface coverage and the content of light impurities could be important parameters for the erosion of tungsten in a device with a large fraction of tungsten coated first wall surface area, if sputtering by light impurities prevails. The surface was well coated in all cases, even at film thicknesses below the average substrate roughness. The contents of light impurities measured by X-ray photoelectron spectroscopy in combination with sputter depth profiling was around 5–10 at.% for carbon and at or below the detection threshold of 1 at.% for oxygen. The thermal screening test were carried out at the electron beam facility JUDITH [14] at Forschungszentrum Jülich, Germany. Thermal loading caused no failure up to the melting condition of the films except for one case of a coating sputter deposited at a substrate temperature of 150 °C, which delaminated at a thermal load of 11 MW/m^2 and a corresponding surface temperature of about 1600 °C.

4. CFC samples: first results

Two-dimensional (Dunlop DMS 704) and three-dimensional (SEP N11) CFC substrates were coated with tungsten films of 1 and 5 μm thickness, respectively. An investigation of this substrate/coating combination may be necessary for a future further extension of the tungsten first wall surface area in ASDEX Upgrade. Coating was performed by the plasma-arc method by

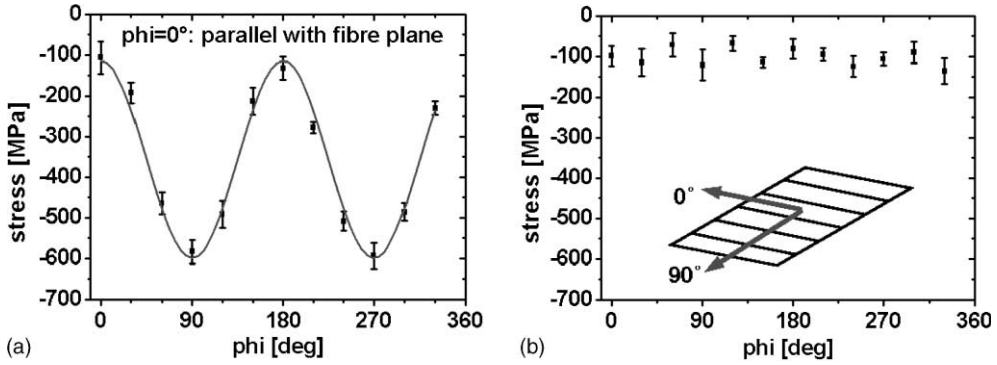

Fig. 2. Angular dependence of the stress state of tungsten films on the 2D material Dunlop DMS 704 (left) and the 3D material SEP N11 (right). The inset in the right hand plot shows the definition of the angle with respect to the fiber planes. The solid line in the left hand plot is a cosine fit.

Plansee AG. The first results we report here concern the degree of surface coverage and the stress states of the respective films. Again the surface coverage is excellent in spite of the even worse surface structure of CFC as compared to fine grain graphite. SEM imaging indicates a complete coverage of the surface by tungsten even within relatively deep pores.

Concerning the stress state of the films X-ray diffraction measurements were performed and evaluated as described above. Since these are anisotropic materials an angular scan was performed, defining $\varphi = 0$ as parallel to the fiber planes. The results are shown in Fig. 2. Again all observed stress values are compressive, i.e. $\sigma < 0$. Fig. 2(a) shows the data obtained from the 2D material Dunlop DMS 704 and Fig. 2(b) shows the data obtained from the 3D material SEP N11 with the inset again showing the definition of angles. As can be seen in Fig. 2(a), there is a distinct variation from −100 MPa in the direction parallel to the fiber planes to about −600 MPa in the direction perpendicular to the planes. Such a variation cannot be observed in the case of the 3D material. At room temperature the coefficient of thermal expansion is 3×10^{-6} K^{-1} and up to 15×10^{-6} K^{-1} for the two respective directions for the 2D material, but varies only from 2.3×10^{-6} to 1.2×10^{-6} K^{-1} for the 3D material. These numbers have to be compared to the room temperature value for tungsten of 4.5×10^{-6} K^{-1}. Therefore we clearly attribute this large stress anisotropy in the case of the 2D material to the mismatch of thermal expansion coefficients due to deposition at elevated temperature. The consequences of this stress anisotropy to the behavior under thermal loading will have to be investigated in the future.

5. Plasma performance in ASDEX Upgrade

Finally we want to focus on the plasma discharges in ASDEX Upgrade. Compared to the value expected from sputtering by charge exchange neutrals only, a high erosion up to the range of 200 nm for the whole campaign was found [15]. In spite of this finding, the central tungsten concentration in the plasma remained in the range of 10^{-6} for standard H-mode discharges as measured by soft X-ray spectroscopy [16]. Consequently the performance was not deteriorated and the energy confinement remained good at an ITER scaling factor of $H_{ITER92Py}$ of approximately 1. These results indicate that the central tungsten concentration is less dependent on the flux of eroded tungsten but more dominated by transport properties in agreement with findings from the tungsten divertor experiment [17].

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