EFDA–JET–CP(08)04/15

C. Ruset, E. Grigore, I. Munteanu, H. Maier, H. Gruner, C. Hopf, V. Phylipps,
G. Matthews and JET EFDA contributors

Industrial Scale 10μm W Coating of CFC Tiles for ITER-like Wall Project at JET

Industrial Scale 10μm W Coating of CFC Tiles for ITER-like Wall Project at JET

C. Ruset[1], E. Grigore[1], I. Munteanu[1], H. Maier[2], H. Gruner[2], C. Hopf[2], V. Phylipps[3], G. Matthews[4] and JET EFDA contributors*

JET-EFDA, Culham Science Centre, OX14 3DB, Abingdon, UK

[1]*National Institute for Laser, Plasma and Radiation Physics, Association Euratom-MEdC, Bucharest, Romania*
[2]*Max-Planck Institut für Plasmaphysik, Euratom Association, 85748 Garching, Germany*
[3]*Forschungszentrum Jülich, Euratom Association, Jülich, Germany*
[4]*EURATOM/UKAEA Fusion Association, Culham Science Centre, Abingdon, UK*
** See annex of M.L. Watkins et al, "Overview of JET Results ",*
(Proc. 21 st IAEA Fusion Energy Conference, Chengdu, China (2006)).

Preprint of Paper to be submitted for publication in Proceedings of the
25th Symposium on Fusion Technology, Rostock, Germany
(15th September 2008 - 19th September 2008)

ABSTRACT
As a result of the R&D phase of the ITER-like wall project, Combined Magnetron Sputtering and Ion Implantation (CMSII) technique was selected for 10μm W coating of approx. 1,000 CFC tiles for the new JET first wall. This technique involves simultaneous magnetron sputtering and high energy ion bombardment. A high voltage pulse discharge is super-posed over the magnetron deposition and by this way positive ions are accelerated, bombarding initially the substrate and then the coating itself during its growth. Based on this method, industrial equipment with a deposition chamber of Φ 800 x 750mm and 24 magnetrons was designed, manufactured and commissioned. The coating productivity is about 1m^2 /week.Tungsten coatings with a thickness of up to 17μm and multilayer structures Mo/W/Mo/W with a thickness of ~25μm were produced and successfully tested at 100 pulses of 16.5MW/m^2 for 1.5s.

1. INTRODUCTION
The identification of an industrial scale manufacturing method for tungsten coatings with a thickness in the micrometer range on carbon substrates was an important subject since W was designed as an armour material for the first wall of magnetic confinement thermonuclear fusion devices. H.Maier, et. al. [1] performed an extended comparative investigation on the properties of W coatings deposited on fine grain graphite by three different methods: electron beam evaporation, magnetron sputtering, and arc deposition. The coatings, with thickness of 0.5μm, 1–3μm and 10μm, were prepared at Max Planck Institut für Plasmaphysik, Garching, Germany, Fraunhofer Institut fur Elektonenstrahl- und Plasmatechnik FEP in Dresden, Germany and Plansee AG, Reutte, Austria respectively. The High Heat Flux (HHF) tests revealed a good behaviour of the coatings deposited by magnetron sputtering, and arc deposition for power density up to 11 MW/m^2 for 2,5s although the coating deposited by magnetron sputtering exhibited a high internal compressive stress. At DIARC Technology Inc., Finland tungsten markers of 3.4μm were deposited on six JET (Joint European Torus) divertor CFC tiles using the DIARC plasma method [2]. The coatings survived to 113 pulses in a neutral beam test facility at power density from 5.8 to 15.7MW/m^2 and duration from 3s to 1s. The maximum surface temperature was 1,553K.

Currently, the primary ITER materials choice is a full beryllium main wall with CFC (carbon fibre composite) at the strike points and tungsten at divertor baffles and dome. Since this combination has never been tested in a tokamak, ITER-like Wall project has been launched at JET (Joint European Torus) with the aim to replace the present CFC first wall with a new one, comprising the same materials choice as it is planned for ITER [3]. In the R&D phase of this project, various PVD and CVD technologies have been developed for 10μm W coating of CFC material cut both parallel and perpendicular to the carbon fibre planes. As a result of HHF tests carried out in the hydrogen beam facility GLADIS at IPP Garching, Combined Magnetron Sputtering and Ion Implantation (CMSII) technology was selected for 10μm W coating of about 1,000 tiles, of different size and dimensions, under industrial conditions [4]. The W coatings deposited on CFC samples by this technology survived without delamination to thermal screening up to $23MW/m^2$ (1.5s) and cycling loading with 200 pulses of $10.5MW/m^2$ (5s). The surface temperature during these tests exceeded in some cases 2,273K. These good performances are mainly due to a number of factors such as: the nano-structure of the W coating, the stress relief induced into the coating by high energy ion bombardment and the introduction of a Mo interlayer between W and CFC substrate. Details about the characteristics of the W coatings deposited on CFC material by CMSII technique are given elsewhere [5]. The transfer of the new CMSII technology from a small experimental unit (Φ 300×420mm) with one magnetron to an industrial unit (Φ 800×750mm) with 24 magnetrons was an important project and some results are presented in this paper.

2. COATING METHOD AND EQUIPMENT

The CMSII technique involves simultaneous magnetron sputtering and high energy ion bombardment. In the deposition process, three low pressure electrical discharges (magnetron discharge, DC bias discharge and high voltage pulse discharge) are superposed. Typical parameters for the high voltage pulse discharge are: U = 30–50kV, τ = 20μs, f = 25Hz. The DC bias up to –900V is applied between pulses.

The plasma ions from the magnetron discharge are accelerated during the high voltage pulses and strike initially the substrate and then the coating itself during its growing with energies of tens of keV. As a result of the periodical ion bombardment the following effects occur:

- An increase of the surface mobility of the deposited atoms which leads to a high densification of the layer.
- An extremely dense, pore free nano-structure is produced. TEM analyses have shown crystallites with a size of less than 10nm [5, 6].
- A stress relief at the interface and within the layer. Due to this effect, coatings with a thickness of 10 – 30μm have been produced.

The deposition rate is in the range of 4 – 8μm/h depending on the coating to be deposited. The deposition temperature is approx. 673K. Schematic representation and the general view of the coating equipment are shown in Fig.1. The deposition chamber, made of stainless steel with double wall for water cooling, has an inner diameter of 800mm and a height of 750mm. Twenty-four magnetrons are positioned inside the chamber in 8 columns of 3 magnetrons each. By this way a usable volume Φ 420×360mm is provided. On the top lid of the chamber there is a ceramic insulator designed to sustain 100kV. On this insulator a double axes rotating load support is installed. Four widows allow the visual inspection of the load during the coating process. Mass Flow Controllers (MFC) are used to introduce argon and reactive gases (if necessary) into the deposition chamber. A TurboMolecular Pump (TMP) of 1,000 l/s ensures the evacuation of the chamber. A 200 HAL gas analyzer with a quadrupole mass spectrometer supplied by Hyden Analytical Ltd., UK is connected to the chamber in order to monitor the composition of the deposition atmosphere. The magnetrons are energized by an 18 channel DC power supply with a total power of 25kW. The current intensity for each channel is stabilized (±1%) and can be adjusted independently in the range of 0.3 – 2.5 A. Arc suppression devices are installed to each channel.

3. CMSII COATING TECHNOLOGY

The difficulty with tungsten coatings on CFC tiles is the anisotropic thermal expansion of the bidirectionally fibre-reinforceced CFC. For the ITER-like Wall Project CFC tiles must be coated both parallel and perpendicular to fibre planes. Direct deposition of W on CFC substrate was not successful because of the poor adhesion, but an intermediate molybdenum layer of 2–3μm has solved the problem.

The beneficial role of Mo seems to be in connection with the adjustment of the thermal expansion mismatch between CFC and W ($\alpha_W = 4-5 \cdot 10^{-6}\ K^{-1}$; $\alpha_{CFC} = 10\text{-}12 \cdot 10^{-6}\ K^{-1}$ perpendicular to fiber and $0\text{-}1 \cdot 10^{-6}\ K^{-1}$ parallel to fiber plane; $\alpha_{Mo} = 7.2 \cdot 10^{-6}\ K^{-1}$). From 24 magnetrons, 12 are loaded with Mo targets and the rest of 12 with W targets. The tiles to be coated are rotated initially in front of the Mo targets and then in front of the W targets. By this way the entire coating is deposited in the same cycle. A batch of CFC tiles positioned on the jigging device just after coating is shown in Fig.2.

4. RESULTS AND DISCUSSION

In accordance with IPP-JET Specification the thickness of the W coating should be in the range of 9–12μm and the impurities within the coating do not exceed 5 at.% for oxygen and 10 at.% for carbon.

Before starting the production the CMSII coating technology was qualified. The qualification process involved the following measurements and tests: a) Determination of the Ar, O and C impurity concentrations b) Determination of the coating thickness c) HHF tests in hydrogen beam GLADIS facility for tiles coated in various zones of the coating area.

4.1. IMPURITY CONCENTRATIONS

In order to measure the O, C and Ar impurity content, tungsten coatings with a thickness of ~200 nm were deposited by CMSII technology on silicon and graphite substrates. The choice of the thickness and substrate was made to meet the requirements of the applied Nuclear Reaction Analysis (NRA), Rutherford-BackScattering (RBS) and Thermal effusion spectroscopy (TES) techniques. NRA with 2.5MeV^{3}He was used to detect C and O. The protons from $^{12}C(^3He, p)^{14}N$ and $16^o(^3He, p)^{18}F$ were detected by a energy-resolved detector. Samples of a-C:D and a-C:H layers with known composition, areal density and thickness and SiO_2 layer of known thickness were used as reference for calibration. RBS spectra were recorded simultaneously with NRA spectra with 165^o scattering angle. For energy calibration, spectra of C, O, Al, Si, Co, Rh and Au were also taken. The spectra were evaluated by simulation with SIMNRA using the areal densities of C and O as determined by NRA. The areal densities of W and Ar were used as free fitting parameters for the simulated SIMNRA RBS spectra. [7]. For TES analysis, the samples were heated up in an UHV system from room temperature to 1,173K with a ramp of 30K/min. The signal of mass 40 (Ar) was recorded by a mass spectrometer. The system was calibrated by feeding a known flow rate of gas just beneath the sample holder.

The highest Ar concentration of up to 8 at.% was found at the Si-W interface within ~50 monolayers of W. In the bulk of W coating, Ar could only be detected, the concentration being of 1 – 2 at.%. As far as concern C and O the maximum depth-averaged concentrations were 6 at.%. The relative error for both C and O concentration was estimated to be about 30%. Calculation of the possible impact of Ar content from W coating on the JET plasma operation lead to conclusion that 2 at. % Ar impurities represent no serious problem on a longer term scale. However at the beginning of operation with neutral beam injection, Ar contamination of plasma will occur. Argon will be gradually depleted in the high power commissioning phase with carefully monitoring of Ar content in the plasma.

4.2. COATING THICKNESS

Glow Discharge Optical Emission Spectrometry (GDOES) is currently used for measurement of the coating thickness and impurities as a quality control technique for industrial production. The equipment used for this purpose is GDA – 750HP machine supplied by SPECTRUMA GmbH, Germany. In each coating run, four witness titanium samples are introduced at different locations including areas where the coating thickness exhibits maximum and minimum values (see Fig.2). This solution has been chosen because CFC is porous and it can not be used directly for GDOES analysis. After coating, Ti witness samples are analyzed. Typical depth profiles of W, Mo, C, O and Ti are shown in Fig.3. As it can be seen the C and O concentrations within the W coating are negligible. The thickness measured by GDOES was compared with the thickness measured on the same sample by optical microscopy with a precision of 0.2µm. The values correspond within an error range of ±1µm.

4.3. HIGH HEAT FLUX (HHF) TESTS

The HHF tests have been carried out in the hydrogen beam facility GLADIS at IPP Garching to examine the thermo-mechanical behaviour of the coatings. In order to check the performances of W coatings deposited by CMSII, layers with thicknesses from 9µm to 17 µm have been deposited both perpendicular and parallel to fibre planes. The first ones survived without delaminations to 100 pulses of 16.5MW/m^2 for 1.5s. The maximum surface temperature was just below 1.873K. The same results has been obtained for 100 cycles at 4 MW/m^2 for 2.5s tests carried out with W coating deposited parallel to fiber planes. During the production process 10% of the coated tiles will be tested on GLADIS at the same heating parameters.

4.4. MO/W MULTILAYER COATINGS

CMSII technology can be extended to thicker coatings. In this respect, the Mo/W structure has been repeated twice and by this way a Mo/W/Mo/W multilayer with a total thickness of ~25µm was produced. The SEM micrographs of this coating are shown in Fig.4. The very good adhesion between W and Mo coatings can be seen at the interfaces shown in Fig.4b. This coating was also tested successfully to 100 pulses of 16.5MW/m^2 for 1.5s.

CONCLUSIONS

Combined Magnetron Sputtering and Ion Implantation technology was developed to industrial scale for 10μm W coating of approx. 1,000 CFC tiles for the JET wall. Tungsten coatings with a thickness of up 17μm and multilayer structures Mo/W/Mo/W with a thickness of ~25μm were deposited by the same technology both perpendicular and parallel to fibre planes of CFC tiles. These coatings were successfully tested to cyclic thermal loading of 100 pulses at 16.5MW/m^2 for 1.5s (coatings perpendicular to fibre planes) and 100 pulses at 4 MW/m^2 for 2.5s (coatings parallel to fibre planes). The maximum surface temperature during these tests was 1,873K.

The impurity content of the W coatings was found to be about 6 at.% as maximum depth-averaged concentrations for C and O and 1–2 at.% of Ar. GDOES method is currently used as a quality control technique for production of W coatings.

ACKNOWLEDGEMENTS

The work was supported by the European Commission and the Romanian Ministry for Education and Research under EURATOM Program EFDA Task Agreement Code: JW6-TA-EP2-ILC-01 (Contract5/2000-NUC-INT(FUS)/TT12) The authors wish to thank to Dr. C. Luculescu for his assistance with SEM analyses. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

REFERENCES

[1]. H. Maier et al., Surf. Coat. Technol., Vol. **142-144**, 2001, 733-737

[2]. S. Lehto et al., Fusion Engineering and Design **66-68**, 2003, 241-245.

[3]. G.F. Matthews et al., Phys. Scr. T128, 2007, 137– 143.

[4]. H. Greuner et al., J. Nucl. Mater. **367-370** (2007) 1444

[5]. C. Ruset et al. ,Phys. Scr. T128, (2007), 171 – 174

[6]. E. Grigore et al., Surf. Coat. Technol., **200**, (2005), pp.744 – 747

[7]. M. Mayer, SIMNRA User´s Guide, Report IPP 9/113, Max-Planck-Institut für Plasmaphysik, Garching, Germany, 1997

Figure 1: CMSII coating equipment: schematic representation (a) and general view (b).

Figure 2: CFC tiles and witness samples coated by CMSII technology

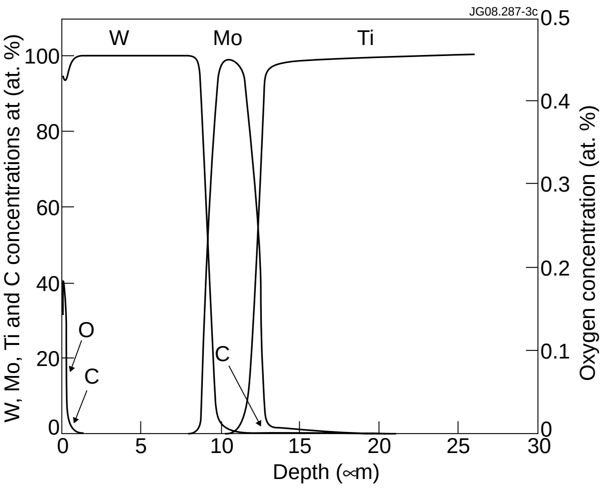
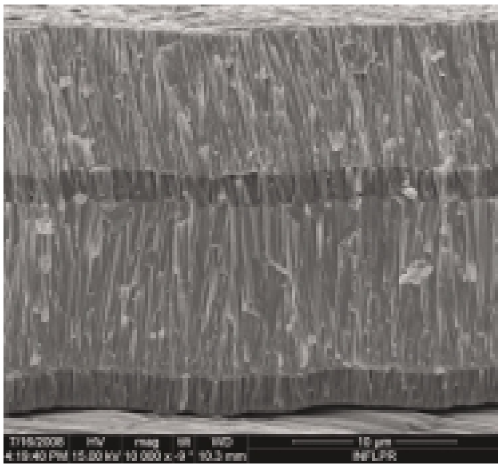


Figure 3: GDOES depth profiles of W, Mo, C and O for a W coating deposited on Ti substrate



(a)

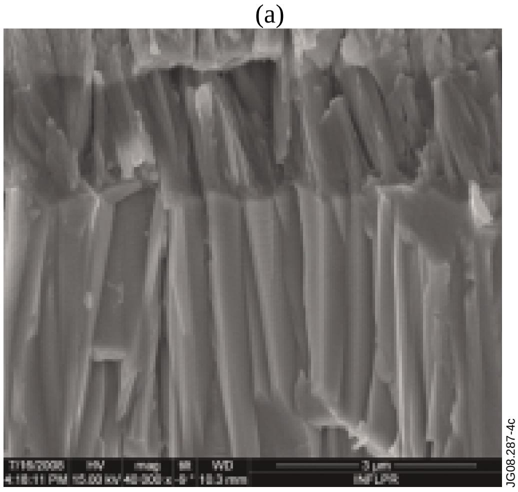


Figure 4:The overall SEM image (a) and the detailed W Mo/W interfaces (b) of the Mo/W/Mo/W multilayer deposited by CMSII technology