Deposition and Qualification of Tungsten Coatings
Produced by Plasma Deposition in WF₆ Precursor Gas

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Tungsten is the plasma facing candidate for future fusion devices

Additional R&D needed to

• Qualify tungsten under high power loads (cracking, fatigue, melting)

• Develop W materials with improved ductility and reduced grain grow

• Study further the compatibility with all plasma scenarios and heating schemes
Aim of the work

Develop an in situ method to deposit W-coatings on the first wall of fusion devices

Provide an environment to study W-PFCs with all plasma scenarios and heating schemes.

→ Discussion ongoing on the need for a full W wall in JET and ITER, also for EAST and JT60-SA → to qualify W for DEMO

Main chamber: 3-5 µm (PVD)
Divertor: 10 µm (PVD+ CMSII)

AUG full W experiment

JET ILW experiment

10-25 µm W on CFC by CMSII

W-bulk

Louver s
WF₆ as precursor gas

WF₆ is used for chemical vapour deposition (CVD) by thermal decomposition at hot surfaces (400-800°C)

WF₆ has also been used in lab experiments for plasma assisted W deposition (e.g. A. Cambe, E. Gauthier, J. Nuc. Mat (2001) 331)

Plasma deposition method like boronisation/siliconisation used in present tokamaks

Approach
1. Define the deposition process parameters
2. Coating properties: density, purity, adhesion, heat flux and thermal shock capability
3. Identify and minimise the (negative) role of Fluor
4. Large scale deposition experiment
5. Injection of (smaller) amounts of WF₆ in TEXTOR
6. Pilot experiment of in situ W coating in TEXTOR
Coating Process

RF – assisted DC glow in 95% H₂ & 5 % WF₆

RF Power: 60 W, Bias Voltage: 200 – 300V, Substrate Temp: 200 C, Pressure: 0.06 mbar, Plasma Exposure: up to 5 hours

Samples: Silicon, Stainless steel, Graphite(EK98)

small scale lab experiment

WF₆ + 3 H₂ → W + 6HF

W⁺, WFₓ⁺, H₂⁺, HF
Promising W coatings have been obtained on small scale samples

Deposition rate : 95 nm / hr.

Coatings up to 0.5 μm achieved

≈ 5 h operation, no physics limit identified
Coating properties

Optical microscopy

SEM

Element analysis

SEM on cross section
Coating properties (2)

Layer analysis by Electron induced X-ray emission

Beam energy: 7 keV (range ≈ 100 nm), 25 keV (2µm)

W layer with few impurities, some oxygen, free of Fluor

→ no closed coverage, due to surface porosity

Beam energy: 7.5 keV

C-signal (green spots) W-signal (red spots)
Heat flux resistance

Test of coatings (on C, EK98) in e-beam JUDITH facility in ELM-like tests, up to 160 MW/m², 1ms

→ no visible damage (no further detailed analysis done)

→ further ELM like tests in laser heating
• No visible damage (buckling or cracking) up to 500 MW/m²

• > 600 MW/m²: surface roughing and start of melting on some spots

• Larger melting at 700 MW/m²

Calculated temperature using bulk W data: 1500 K → heat conductivity of layer and/or heat transfer to graphite reduced

JET W coatings (20μm) analysed under same conditions: failure at 200-300 MW/m²
Exposure on TEXTOR testlimiter, comparison with bulk W

- Temperature excursions up to 3000°C (poor contact of plate to graphite holder)
- No visible damage of W layer
- Slightly reduced W erosion

Larger surface roughness of W coating lead to increased redeposition on rough structures
Injection of WF6 in TEXTOR

Study W migration

Study WI line emission

Study the impact of Fluor on plasma behaviour and operation

7 WF6 injections with $3 \times 10^{19}$ WF$_6$
Each: $2 \times 10^{20}$ WF$_6$ molecules
WL at 400.8 nm

Fluor main plasma line (53,521 nm)

Deeper penetration of Fluor & larger memory effect
Fluor plasma impurity lines reach background line intensities in about 10 shots.

Post mortem analysis of deposited W layer
(RBS, SIMS, EPMA)

Local deposition of a “pure” W layer with low amount of Fluor
Small W local deposition efficiency
(about 1% of W found on plate, 30% found on main TEXTOR limiter after immediate TEXTOR)
Fluor impurity behaviour in TEXTOR

5 shots days in TEXTOR with strong WF6 injection

No particular effect on long term behaviour of line integrated Fluor VI line emission
Large scale W coatings from Wf$_6$ for TEXTOR application

Large vacuum Test Facility
Represents one octant of the TEXTOR tokamak

Diameter: 1.3 m
Length: 2m
Volume ~ 2.1 m$^3$
standard TEXTOR configuration
Antenna with 13.56 MHz RF generator with inductive coupling (Anode) + DC voltage to wall (cathode)

New arrangement:
Direct capacitive coupling of RF to large sample holder + bias of -100...-500V DC potential

attracts the ions to the holder and prevents coating of walls
A-c:H layer deposition by CH$_4$ showed only deposition on the sample holder and no deposition on the walls.
Test coatings with CH$_4$ showed only deposition on the sample holder and no deposition on the walls.

- RF power = 100 W
- DC = -110 V
- Sample current 45 mA
- 23 μA/cm$^2$

Target holder with capacitive RF + DC bias.

Higher oxygen content in first experiments, improved by better wall conditioning of system.
Summary

W layers have been deposited on graphite by plasma deposition in WF$_6$ and H$_2$

Layers with sufficient purity and very low amount of Fluor have been deposited with good adhesion on graphite and promising thermal shock behaviour

Injection of smaller amount of WF6 in running Tetxor shots has resulted in local deposition of pure W layers

Increased Fluor plasma contamination disappeared in less than 20 shots

In a new RF deposition arrangement, local deposition of an C film was achieved with no deposition on the rest of the wall

RF plasma deposition of W layers with DC ion acceleration on graphite appears a promising technique for in situ local W coating of wall tiles

Further optimisation ongoing

Preparation for TEXTOR W coating ongoing