



### Deposition and Qualification of Tungsten Coatings Produced by Plasma Deposition in WF<sub>6</sub> 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.

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



Main chamber: 3-5 µm (PVD) Divertor: 10 µm ( PVD+ CMSII) **JET ILW experiment** 



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## WF<sub>6</sub> as precursor gas



WF<sub>6</sub> is used for chemical vapour deposition (CVD) by thermal decomposition at hot surfaces (400-800°C)

WF<sub>6</sub> 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 WF<sub>6</sub> in TEXTOR

6. Pilot experiment of in situ W coating in TEXTOR



## **Coating Process**



#### RF – assisted DC glow in 95% $H_2$ & 5% $WF_6$

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)









# Promising W coatings have been obtained on small scale samples



steel

silicon

graphite

#### **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

Beam energy : 7.5keV



C-signal (green spots) W-signal (red spots)

 $\rightarrow$  no closed coverage, due to surface porosity



## Heat flux resistance



# Test of coatings (on C, EK98) in e-beam JUDITH facility in ELM-like tests, up to 160 MW/m<sup>2</sup>, 1ms

 $\rightarrow$  no visible damage (no further detailed analysis done)

 $\rightarrow$  further ELM like tests in laser heating



## Laser ELM simulation





#### LASER Penetration: 22 nm

- No visible damage (buckling or cracking) up to 500 MW/m<sup>2</sup>
- > 600 MW/m2 : surface roughing and start of melting on some spots
- Larger melting at 700 MW/m<sup>2</sup>

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

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## **Exposure on TEXTOR testlimiter,** comparison with bulk W

W bulk plate





W coated C • Temperature excursions up to 3000C (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 x  $10^{19}$  WF<sub>6</sub> Each: 2 · x  $10^{20}$  WF<sub>6</sub> molecules



#### WI at 400.8 nm





#### FI at 696.6 nm



Fluor main plasma line (53,521nm)

Deeper penetration of Fluor & larger memory effect







### 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 plasma impurity lines reach background line intensities in about 10 shots





## 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<sub>6</sub> for TEXTOR application

#### Large vacuum Test Facility

Represents one octant of the TEXTOR tokamak





Volume ~ 2.1 m3







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







# TEST coating with WF6 : Sample arrangement





# Test coatings with CH<sub>4</sub> showed only deposition on the orschungszentrum sample holder and no deposition on the walls



Target holder with capacitive RF + DC bias



RF power = 100 W DC= -110 V Sample current 45 mA 23 uA/cm2



Higher oxygen content in first experiments,

Improved by better wall conditioning of system

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### 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 then 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