Thin coatings using combined magnetron sputtering and ion implantation technique

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Outline

- ☑ Introduction
- ☑ Tungsten coatings on carbon based materials for fusion applications
- ☑ Combined treatment of laser alloying and plasma nitriding; application to forging dies
- ☑ Conclusions

Material Candidates for Plasma Facing Components in Fusion Devices

- Plasma temperature in the core of the tokamak: 50-100 millions K

- Carbon Fiber Composite (*CFC*) – a very good temperature resistance, a reasonable mechanical resistance, but high tritium retention and high chemical sputtering coefficient ⇒ plasma contamination

- Be – low Z, low tritium retention, but low melting temperature (1283 °C)

- W – low tritium retention, high melting temperature (3400 °C), low sputtering coefficient, but high Z

The first option for *ITER* (International Thermonuclear Experimental Reactor) wall is beryllium for the main chamber and tungsten for divertor, but this configuration was never tested on a relevant tokamak.

To do this, the "*ITER-like* wall project" was initiated at *JET* (Joint European Torus), UK, the biggest operating tokamak in the world at the moment.

The objective of this project was to replace the actual *CFC* wall with a new one using Be for the main chamber, bulk W for particular tiles of divertor and W coated *CFC* for the remaining divertor and specific tiles from the main chamber.

ITER-like Wall Project – R&D on W Coating of *CFC* Tiles

- Ten *PVD* and *CVD* techniques at various processing parameters have been applied by European companies to coat with W layers of 4 μm and 10 μm ten sets of *CFC* samples (80 x 70 x 40 mm).

- The high heat flux *HHF* testing program of these samples included a thermal screening up 23 MW/m^2 for 1.5 s and a cyclic loading of 200 pulses at 10.5 MW/m^2 for 5 s. The surface temperature exceeded in some cases 2,000 °C.

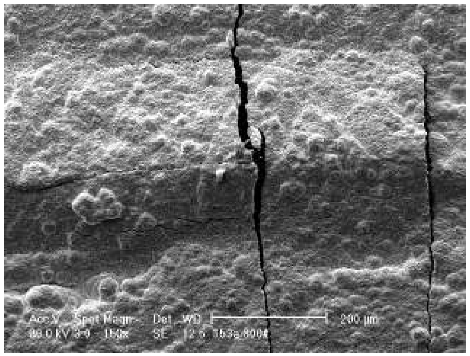
- Only W coatings deposited by Combined Magnetron Sputtering and Ion Implantation (*CMSII*), a technique developed at MEdC (our Euratom association) survived to these tests without exfoliations. The difficulty with tungsten coatings on *CFC* tiles is the anisotropic thermal expansion of the bidirectionally fibre-reinforced *CFC*.

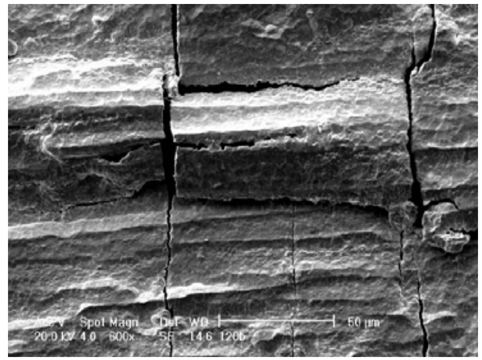
- As a result of the R&D phase of the *ITER-like* wall project:

  \- *CMSII* technique was selected for 10 μm W coating of approx. 1,000 *CFC* tiles for the main chamber of the new *JET* wall.

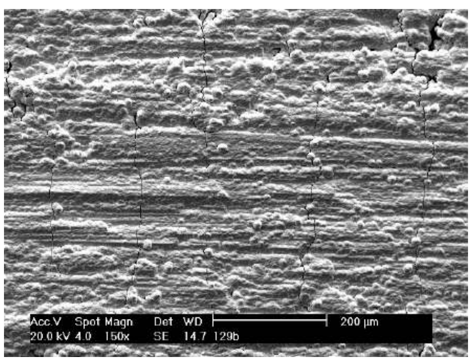
The work has been carried out in co-operation with Max-Plank Institute for Plasma Physics, Garching, Germany and *CCFE*, UK under the *EURATOM* Program

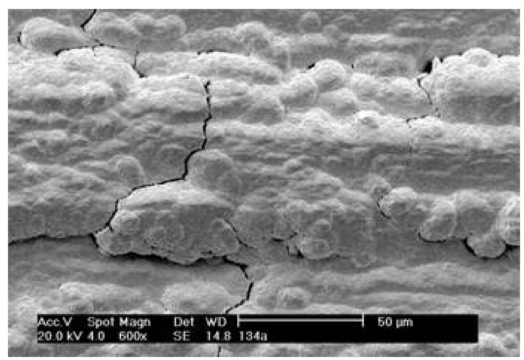
After *HHF* tests in *GLADIS*





Coatings deposited by conventional *PVD* or *CVD* techniques

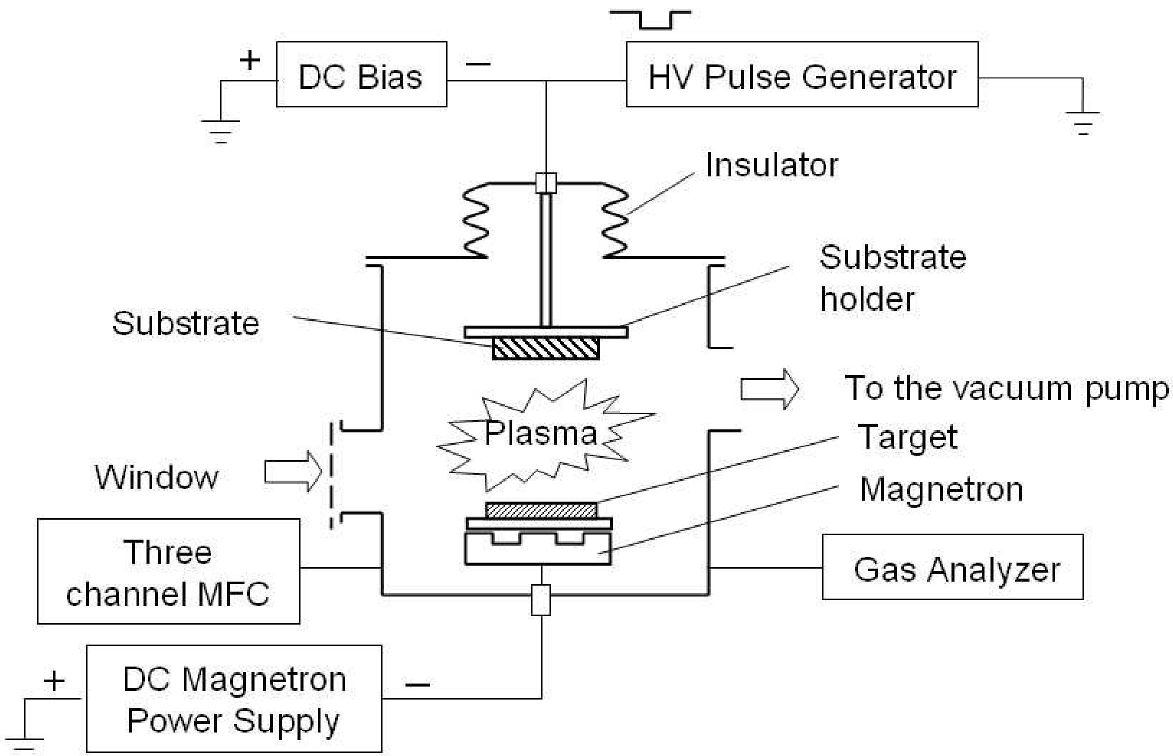




Coatings deposited by *CMSII* technique

Combined Magnetron Sputtering & Ion Implantation

Magnetron sputtering + Ion Implantation = CMSII



Schematic representation of *CMSII* setup

Specific characteristics of *CMSII* coatings

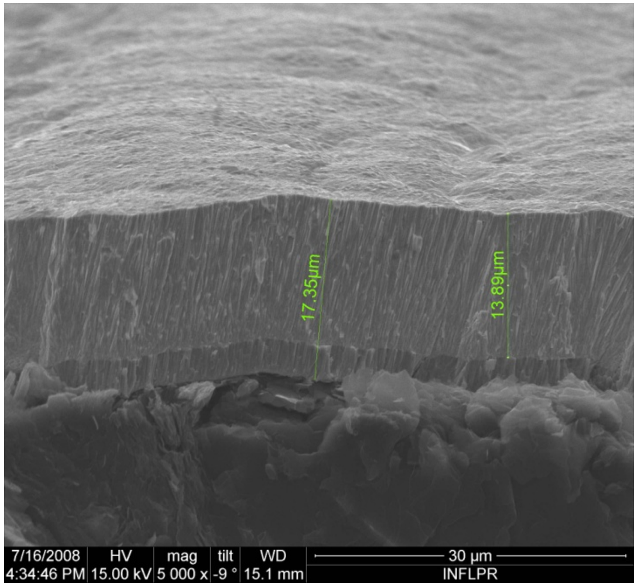
- High energy ion bombardment
  U_{HV} = 30-70 kV; t ~ 20 μs; f = 25 Hz

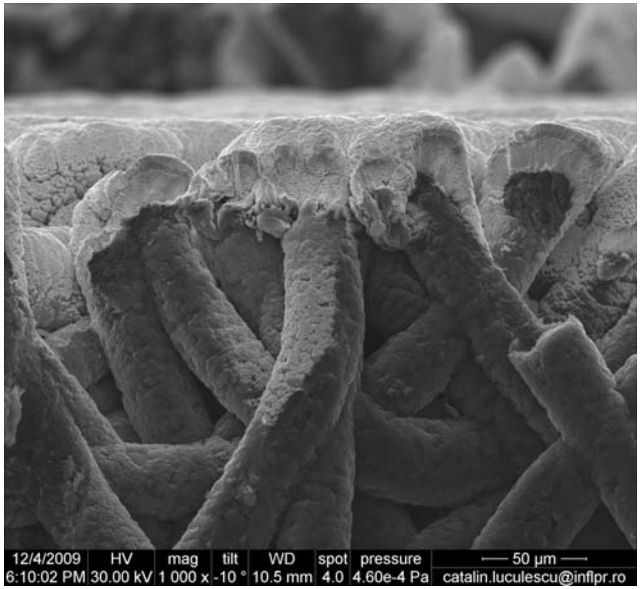
r = 2 nm/s ⇒ for 40 ms → d_{calc} = 0.08 nm
- increase the surface mobility
- high densification of the coating

- Nano-crystalline structure

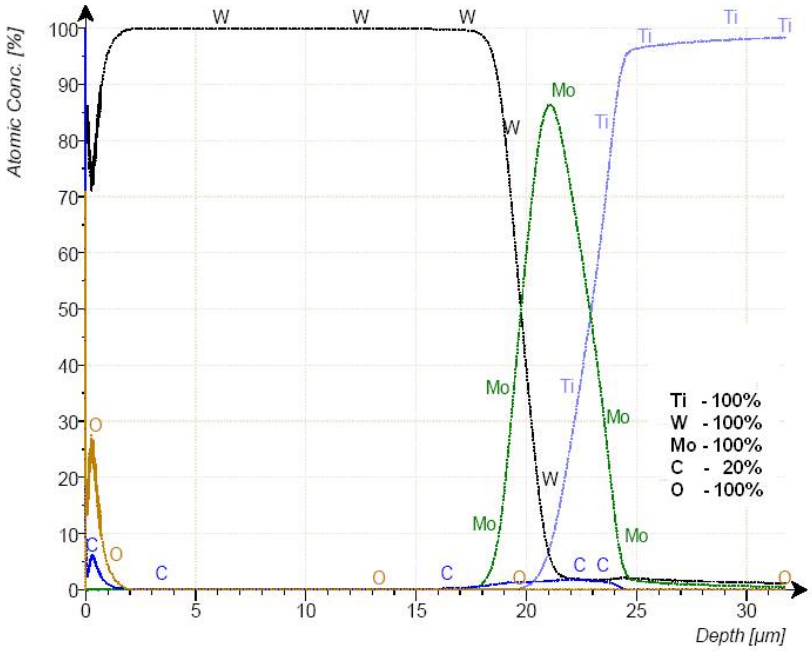
- A Mo interlayer is used to adjust the mismatch of thermal expansion coefficients between *CFC* ($\alpha_{CFC} = 10\cdot10^{-6}\ K^{-1}$ perpendicular to fiber and $0\text{-}1\cdot10^{-6}\ K^{-1}$ parallel to fiber plane) and W ($\alpha_W\ 4.5\cdot10^{-6}\ K^{-1}$). α_{Mo} is $7.2\cdot10^{-6}\ K^{-1}$.

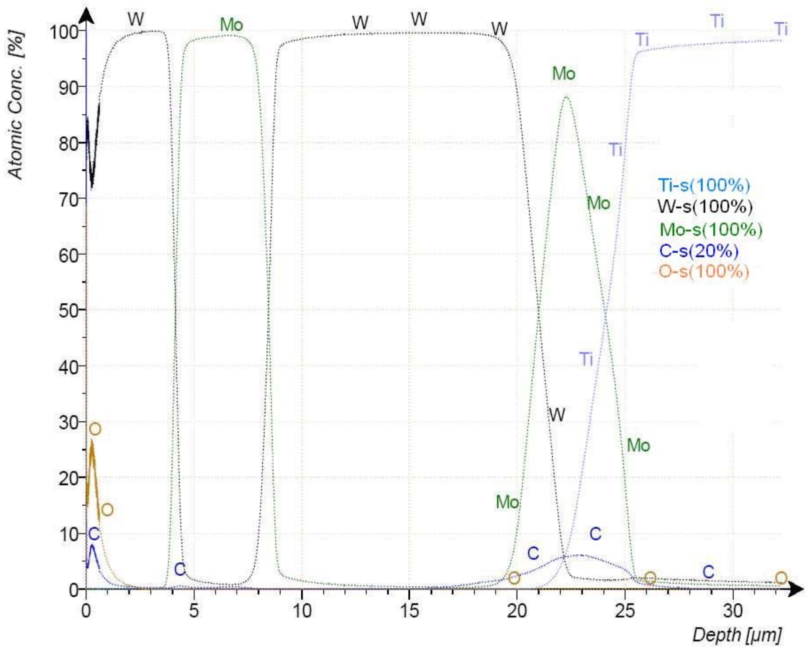
- Due to the high energy ion bombardment a stress relief occurs into the coating and consequently relative thick coatings (10-30 μm) can be produced.



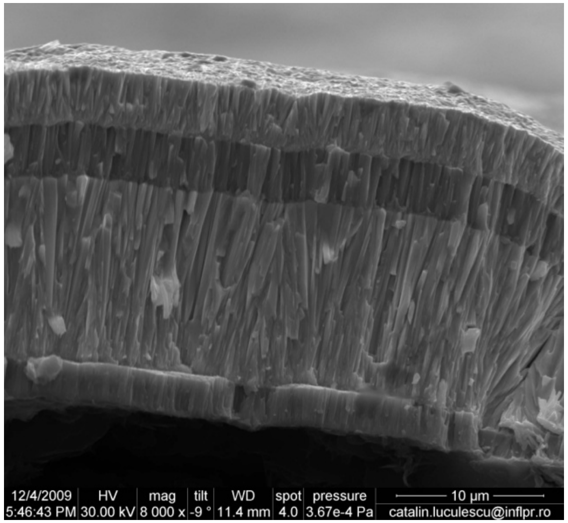


GDOES depth profile





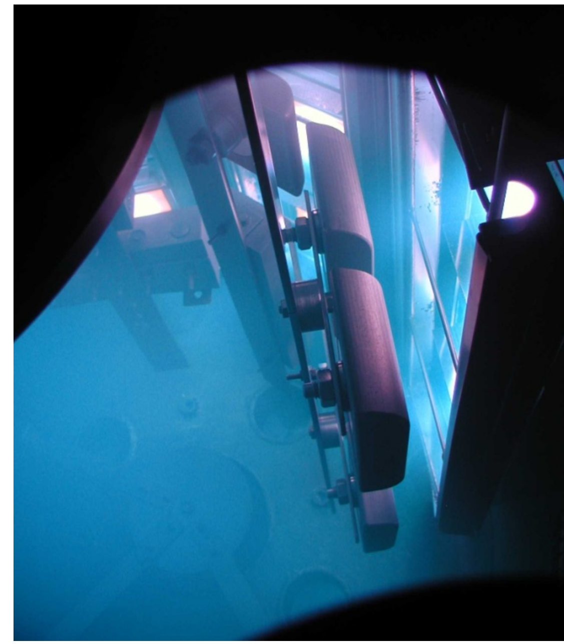
GDOES depth profile for a W coating of 20 µm and a marker tile



➢Structure of markers:

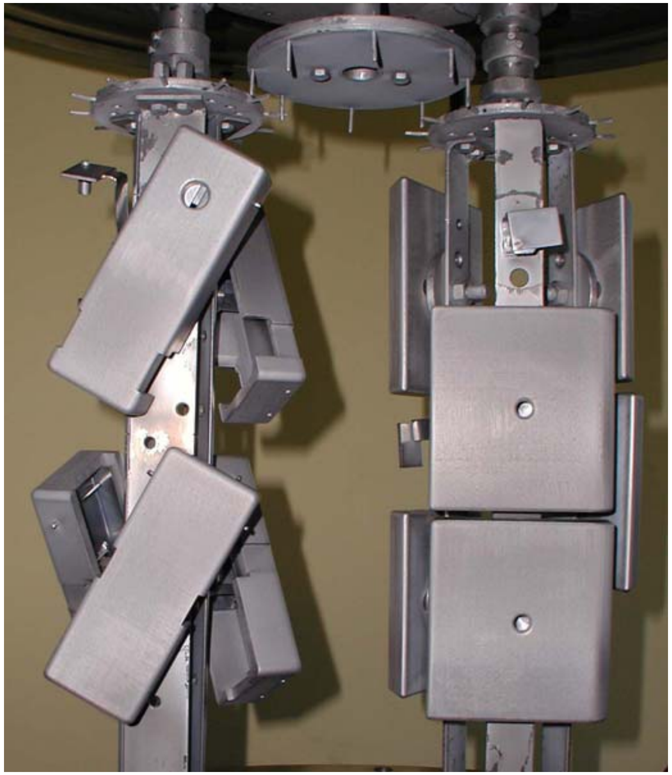
- 2-3 µm Mo
- 12-14 µm W
- 3-4 µm Mo
- 3-4 µm W

Industrial Scale Production of W Coatings on *CFC* Tiles



- Development of the *CMSII* technology from laboratory to industrial scale: about 2 years.
- Qualification of the W coating technology for each type of tiles: about 6 months
- Production of W coating including *HHF* (High Heat Flux) tests of 10% for ~ 3,000 tiles: about 20 months

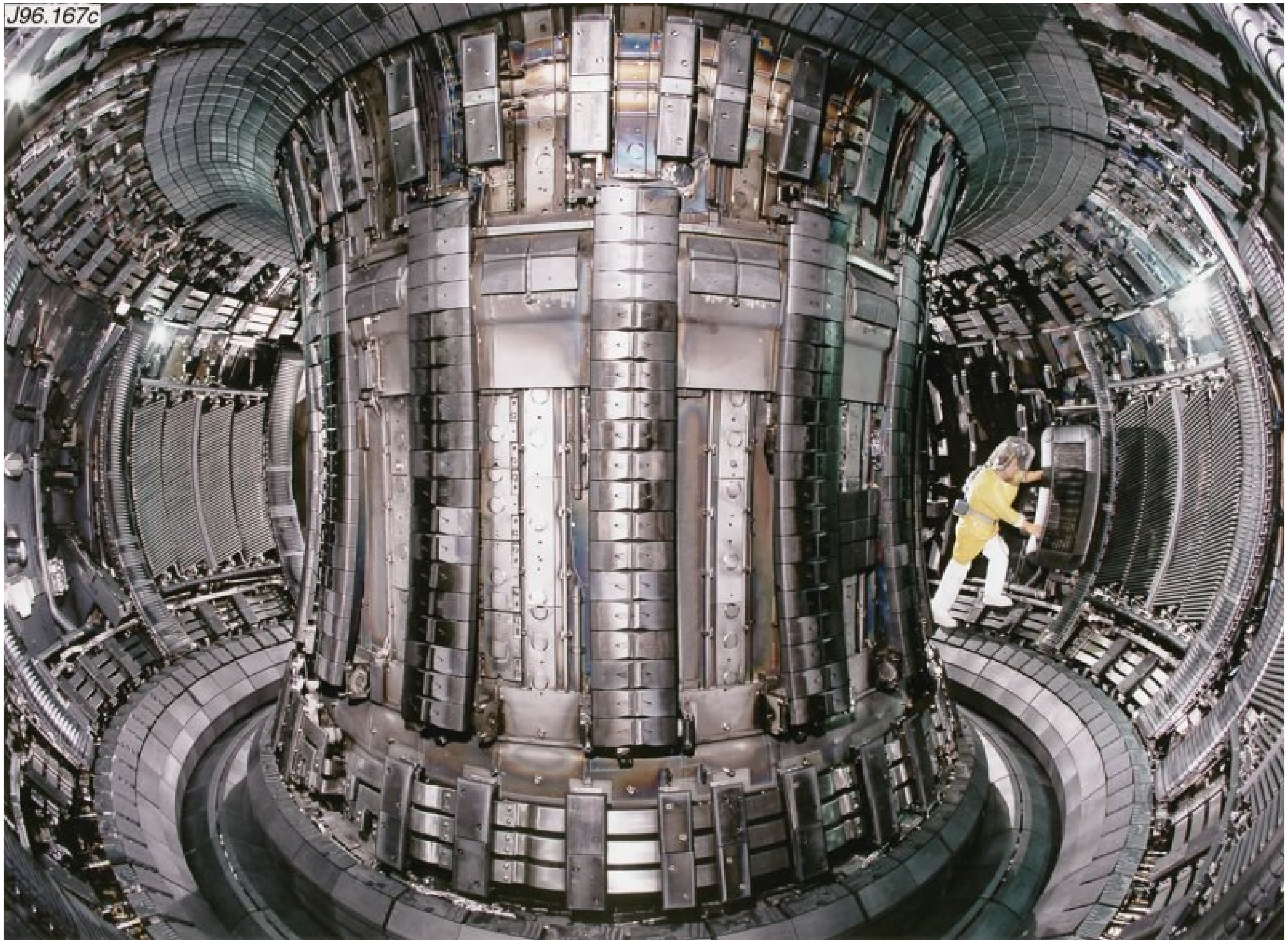
W Coated *CFC* Tiles for the main chamber & divertor



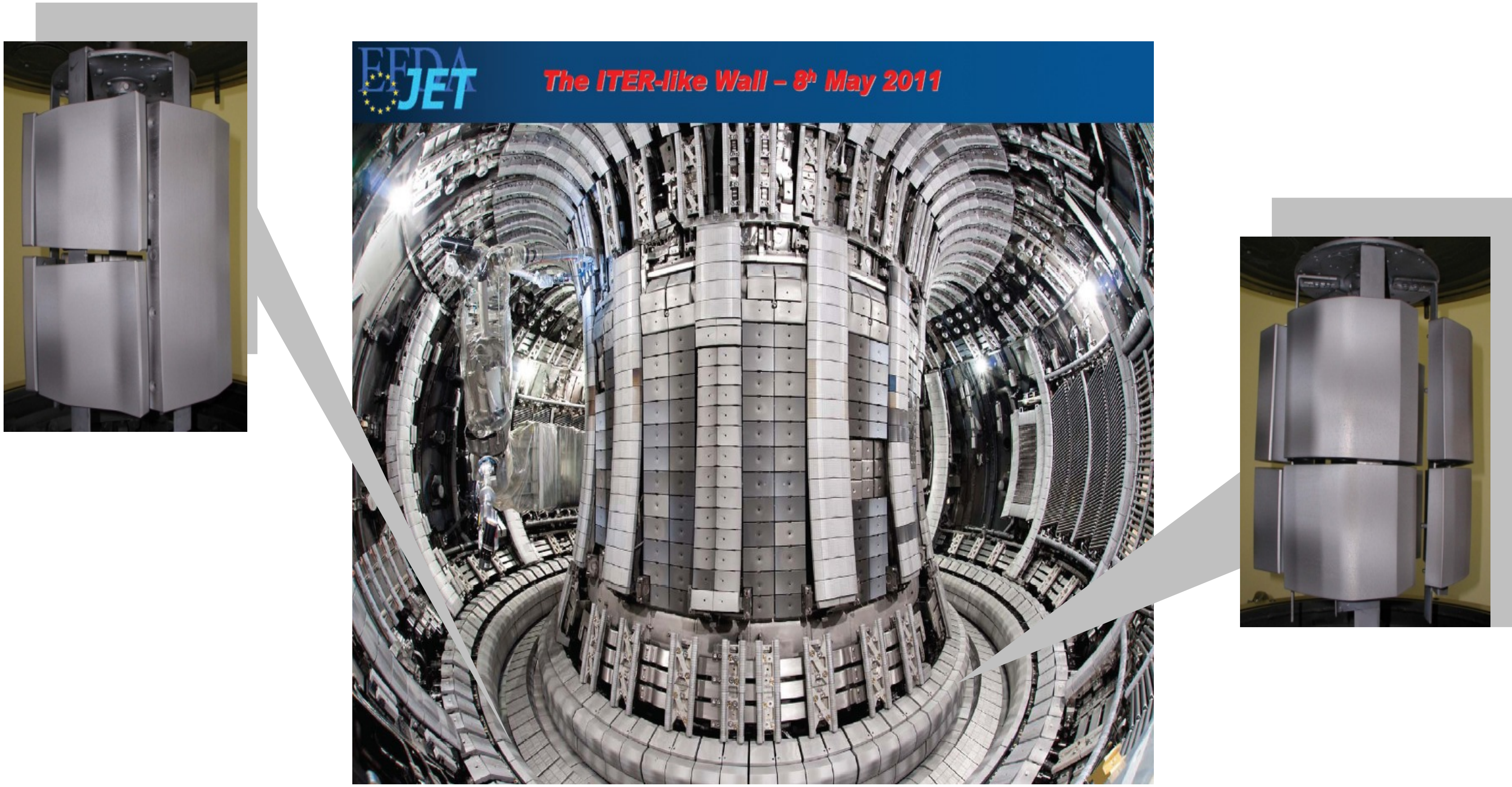
Diagnostic covers and shinethrough protection tiles from the main chamber; coating thickness 10-15 μm

G 6 and G7 divertor tiles coated with 20-25 μm W in series production

Interior of *JET* tokamak with *CFC* tiles



Interior of *JET* tokamak with metallic wall



- About 1800 *CFC* tiles were W coated by *CMSII* technology with layers of 10-15 μm or 20-25 μm
- Most of them (about 80%) are installed on *JET*. The remaining are saved as spares.

W coating of *FGG* tiles for *ASDEX* Upgrade

- About 1,000 *FGG* (Fine Grain Graphite) components have been coated by *CMSII* technology and partially installed in tokamak.
- Coating thickness: 10-15 μm

HHF tests of W-coated *CFC* at *NILPRP*

- During the R&D and production phases of *ITER like Wall* (ILW) only a minimum number of high heat flux tests were carried out on the *GLADIS* hydrogen beam test facility at *IPP* Garching.

  The reasons:
  - tight time constraints of the *ILW* project
  - limited time of *GLADIS* available for *ILW*
  - costs of the tests

A more realistic testing program seems to be necessary in order to evaluate the lifetime of W coated *CFC* tiles:

- Large number of pulses (>2000)
- Suitable power densities on surface

Prediction concerning:
- the lifetime of the coatings
- the maximum thermal load acceptable
- the coatings failure mechanism

↳ In order to test the W coatings at a number of pulses relevant for *JET*, a new *HTTF* was designed, manufactured and commissioned at MEdC.

The main objectives for this project were:

- to test the W coatings deposited on *CFC* and *FGG* tiles for *JET* and *ASDEX Upgrade*, respectively, at a large number of pulses (> 3,000)
- to improve the *CMSII* technology with respect to the thermo-mechanical properties of W coatings

Heating: Electron beam

→ Estimated beam power: 1 kW / 1.3kW

→ Accelerating voltage: ~ 20 kV

→ Estimated beam spot: ellipse with axes 18/12 mm ⇒ ~ 170 mm^2

→ Power density: up to 9 MW/m^2

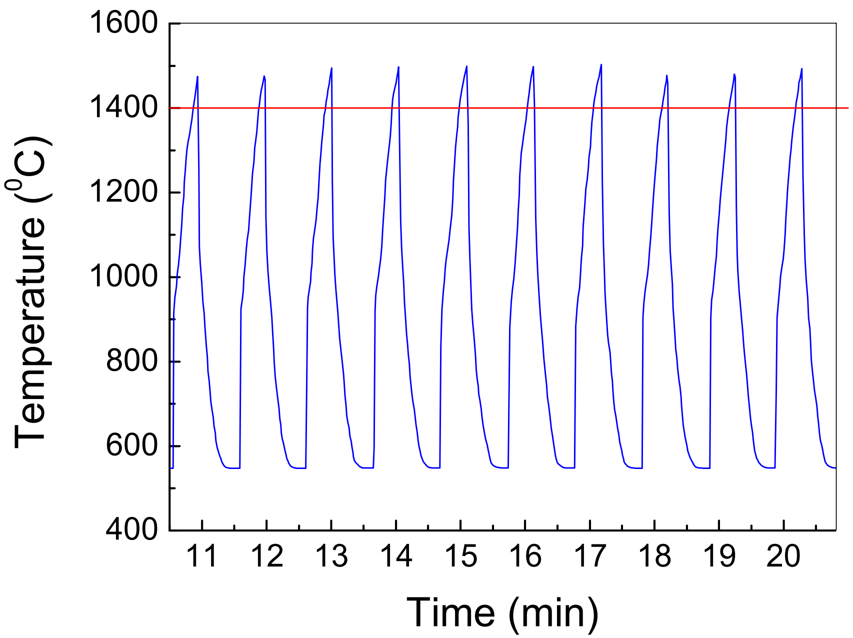
→ Pulse duration: 24s

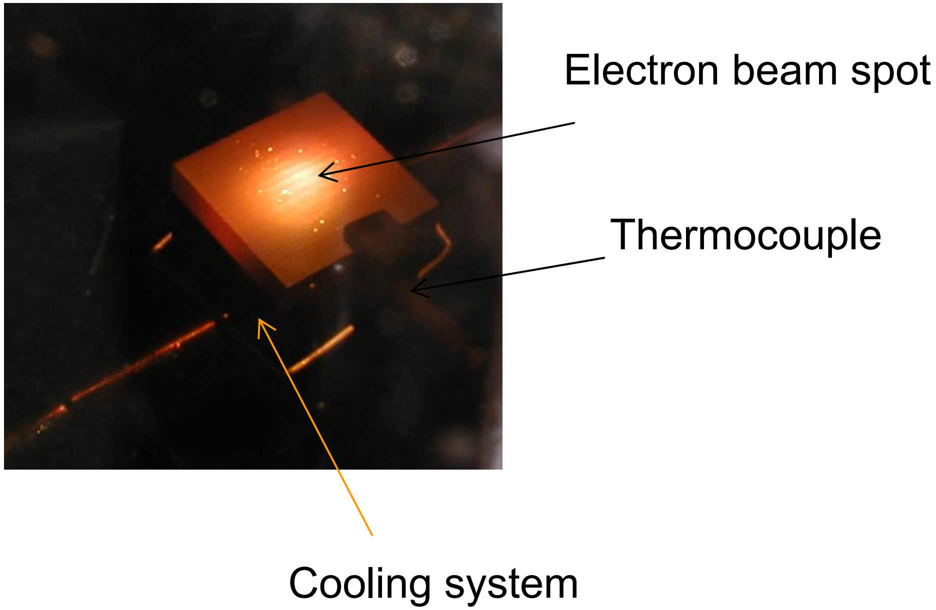
→ Pause between pulses: 35-50 s

→ $T_{coating} \leq 2{,}000$ °C

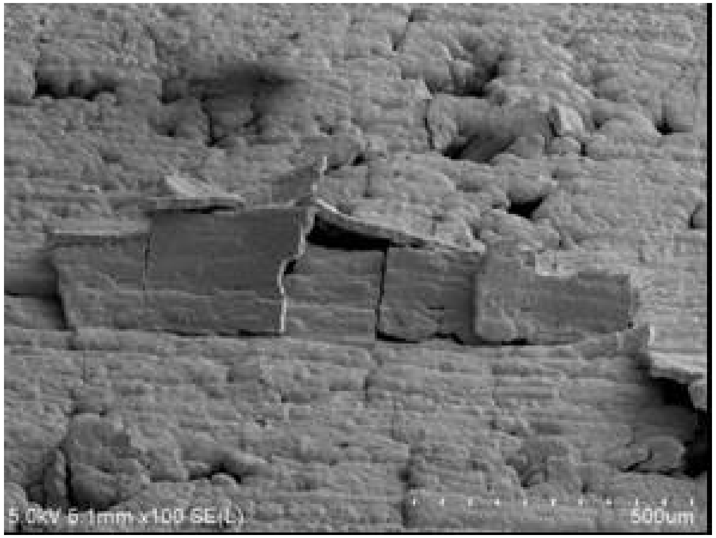
Testing program:

- W coated *CFC* samples with 10 μm and 20 μm thickness
- Testing temperature: 1250° and 1450°C → Power densities of 6.9 MW/m^2 respectively 8.4 MW/m^2
- Minimum temperature ~200°C
- Pulse duration 25s, pause between pulses 40-60s

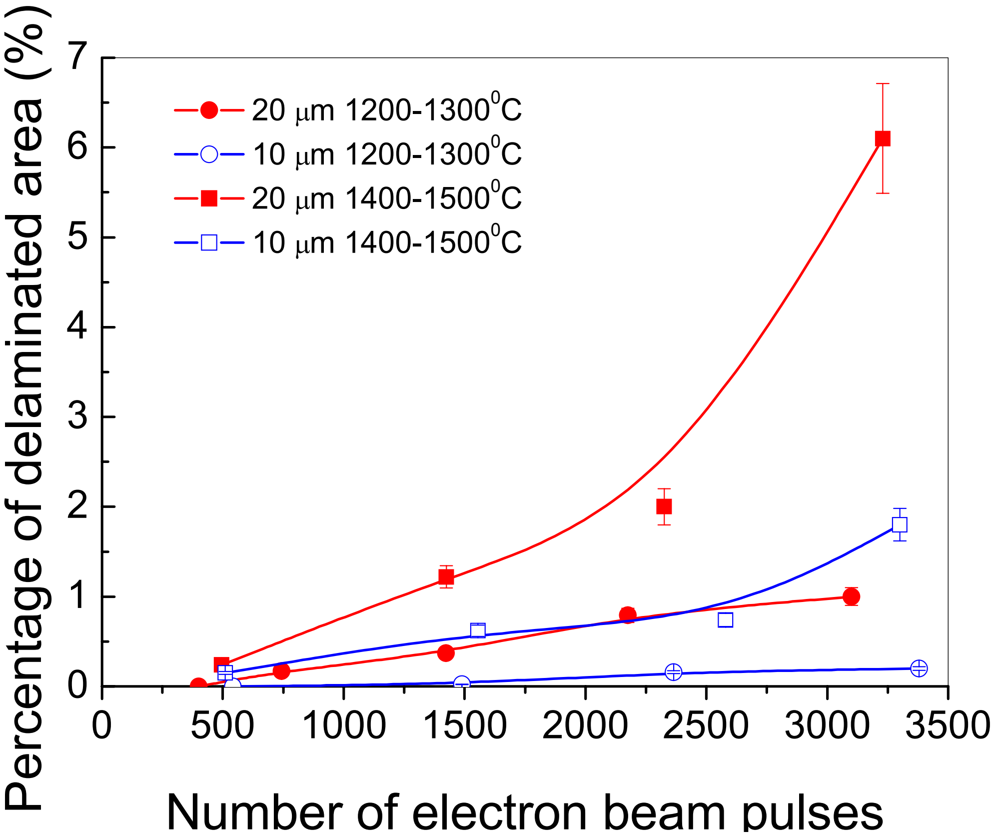




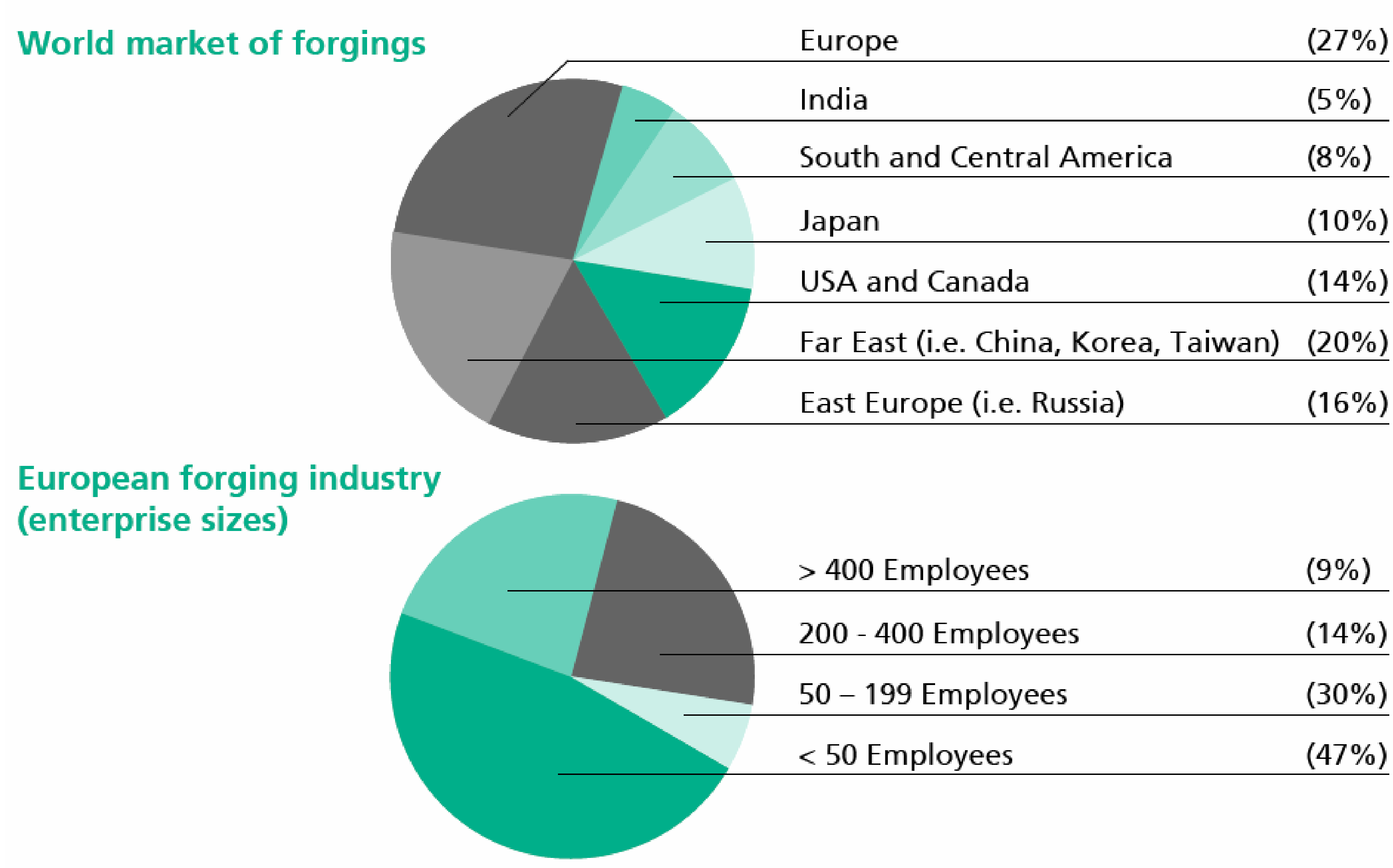
<u>Results</u>:



20 µm W coating after 3,230 pulses



Combined treatment of laser alloying and plasma nitriding



European forging industry is world market leader ⇒ 78.800 people ⇒ 3.4 billion products/year ⇒ annual turnover of 9.4 billion Euros. (Data: European Forging Association – *EUROFORGE*)

- The European forging industry – dominated by SMEs – is steadily forced to:
  - Reduction of the production costs
  - Increase production volume
  - Increase the part quality.

- One way to meet these challenges is the application of innovative surface treatments to increase the forging dies lifetime by 100-200% and thus to reduce the overall die costs by ~ 30%.

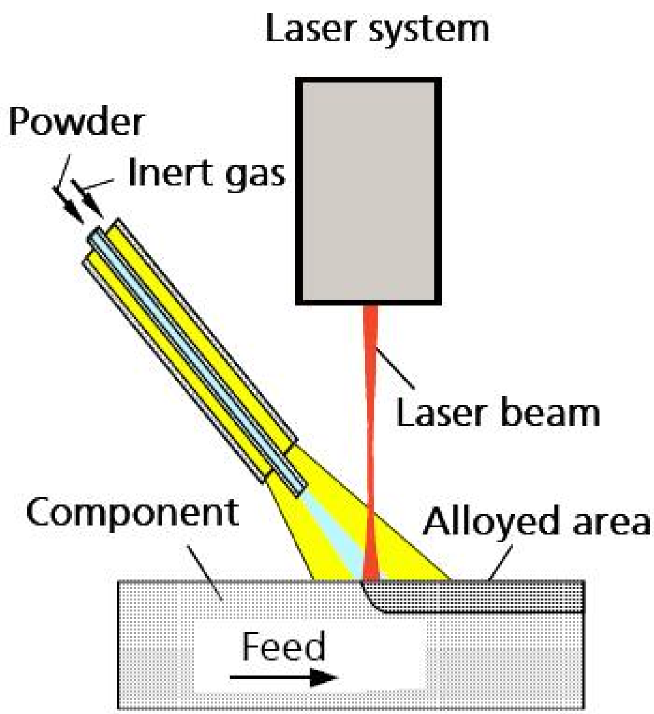
- During operation a forging die is subjected to:
  - Thermal stress: 500 ºC – 800 ºC
  - Very high mechanical stress (impact and friction)
  - Oxidation

- This regime ⇒ erosion, plastic deformation, cracking, adhesion of the forged material

- Innovative surface treatment: combination between laser alloying or dispersing and nitriding.

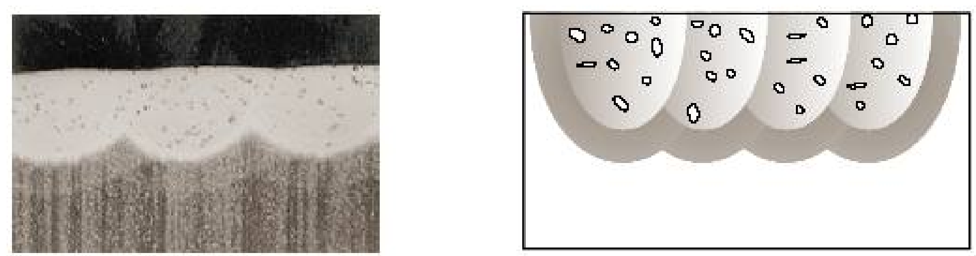
- In this way a surface layer with unique properties can be produced:
  - very good fatigue resistance (due to the laser treatment)
  - good wear and oxidation resistance (due to the nitriding of the surface alloyed layer)

Schematic representation of Laser Alloying and Laser Dispersing



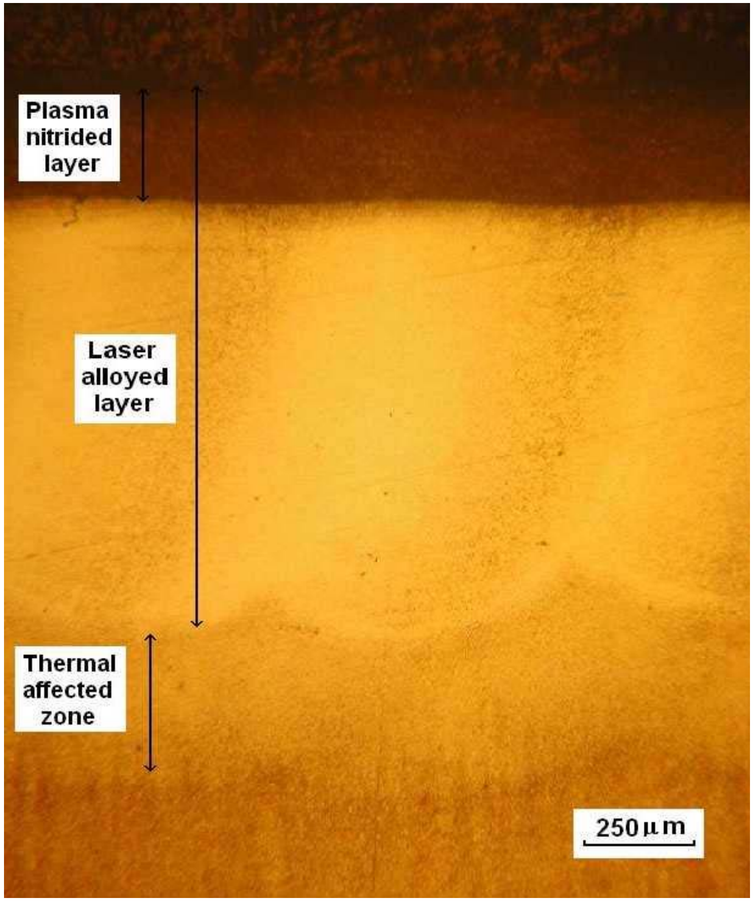
Schematic representation

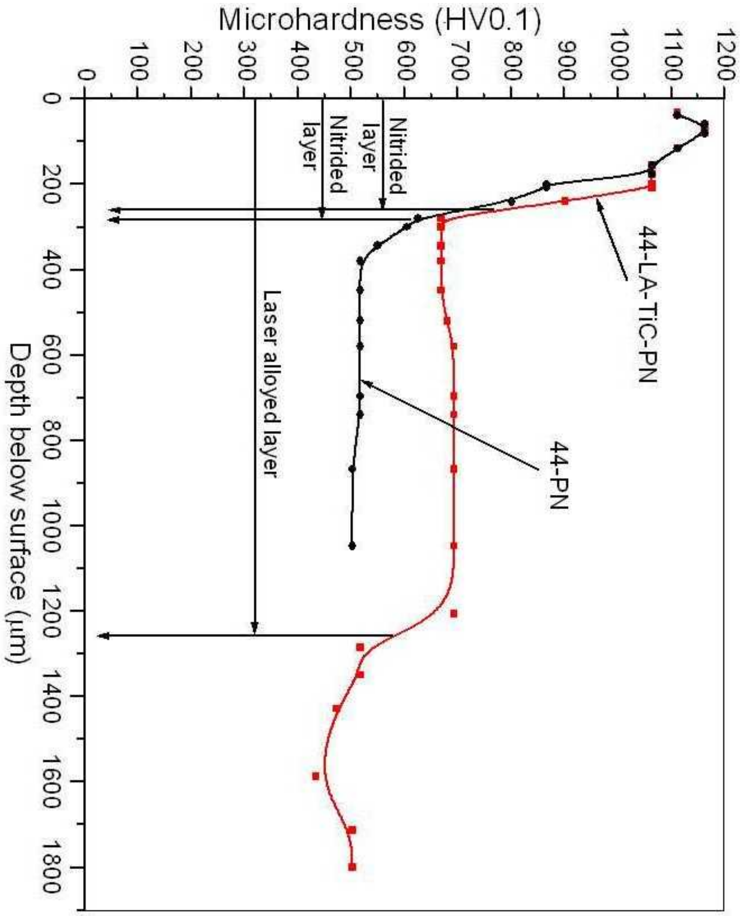
Laser alloying



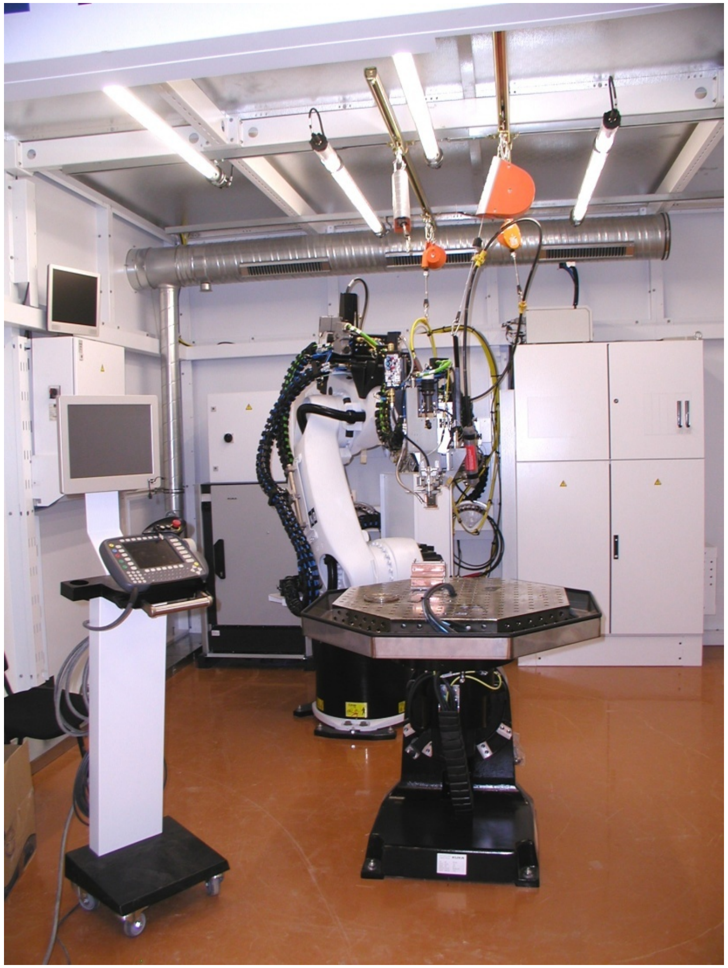
Laser dispersing

Micrograph and microhardness depth profile of a surface layer produced on W1.2344 steel by Combined Treatment (TiC/*LD* + *PN*)

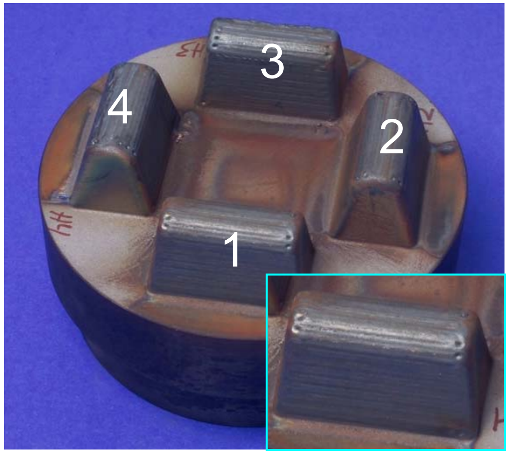




Laser alloying equipment and treated components

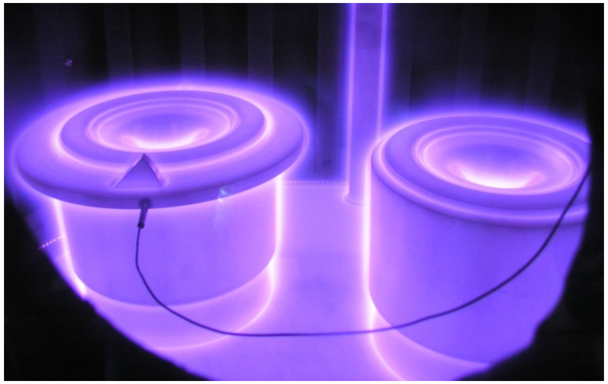


During laser alloying



After laser alloying

Forging dies subjected to Combined (*LA+PN*) treatment



During plasma nitriding

After plasma nitriding

Die surface after 1700 forging cycles
Treatment: Salt bath nitrocarburising

Die surface after 1700 forging cycles
Treatment: Combined treatment TiC/LD+PN

Conclusions

- Combined Magnetron Sputtering and Ion Implantation proved to be a suitable technique to coat with W, carbon based materials for fusion applications

- More than 2000 *CFC* tiles and 1000 *FGG* tiles have been coated for *JET* and respectively for *Asdex* upgrade tokamaks

- A *HHF* equipment for testing materials at high temperature and pulsed regimes has been built and used to evaluate the lifetime of W coatings

- A combined treatment (Laser Alloying and Plasma Nitriding) to increase the lifetime of forging dies has been developed and applied. An increase with 120% of their lifetime have been obtained