Contribution from the CEA to the IAEA project on:
"Plasma-wall interaction of irradiated Tungsten and Tungsten alloys in Fusion Devices"

Title of the project:

Quantification of tritium implantation in tungsten based fusion materials:
ITER grade W, PVD W, W solid solutes (W-Ta) and particles reinforced W (as W-Y203, W-TiC)

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In collaboration with:
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And G Dinescu (Bucarest)

Acknowledgements to W. Knabl and A. Hoffmann from PLANSEE®

Outline

1. **Starting point: study of Tritium trapping in particles for biology purpose**
   - **Comparison with massive materials**
   - **First results on Tritium trapping in Dust and W ITER grade**
2. **Work to be accomplish in the CRP frame**

Origin of the activity

Fusion machines produce tritiated dust which can escape from the Torus in case of LOVA (through HEPA filters)

- **Dust characteristics :**

	size	SSA	composition	T content
Dust characteristics	100-200nm	>>10m²/g	Pure W partially surface oxidized	MBq/g

- **Open questions:**
  - **What is the behavior of these particles/of the tritium after inhalation?**
    - **Are the particles dissolved in the biologic media?**
    - **Where are the particles/the tritium located in the cell?**
  - **What are the consequences of particle ingestion in term of cyto-toxicity and of geno-toxicity?**

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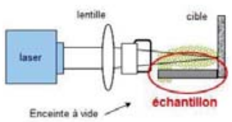
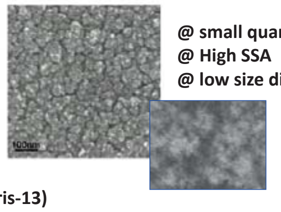
- **Tasks to be done:**
  - **ITER relevant particles production and characterization**
  - **Particle tritiation:**
    - **total quantity of tritium trapped**
    - **Tritium release**
    - **Tritium speciation**
  - **+ Biological tests**

Particles production

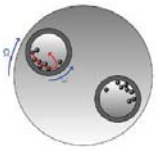
- **By laser/matter interaction (LP3 – Marseille)**

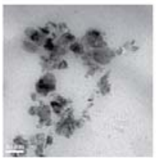




@ small quantity produced
@ High SSA
@ low size dispersion

- **By planetary milling (LSPM – Paris-13)**

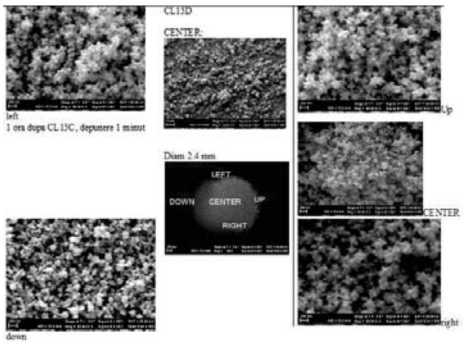




@ large quantity produced
@ low SSA
@ low size dispersion

Particles production

- **By W sputtering by Ar magnetron plasma (NILPRP – Bucarest)**



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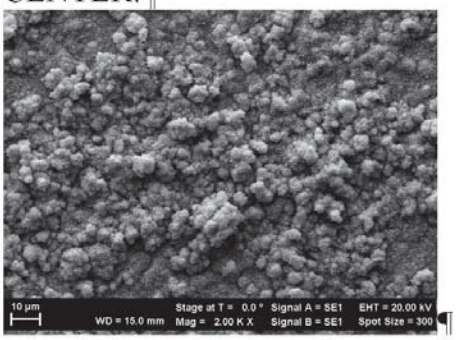
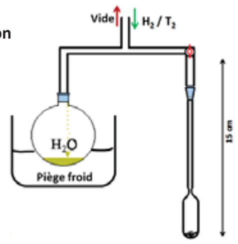


@ large quantity
@ High SSA
@ large size dispersion

Particles tritiation

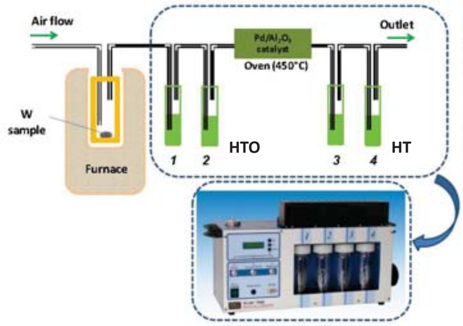
- **20-40 mg of powder treated each time**
- **Procedure:**
  - **First step: surface oxides reduction under hot hydrogen (470°C, 1,4 Bar of H_2, 10 Hours)**
  - **Second step: Tritium implantation (470°C, 1 Bar of pure T_2, 2 hours)**
  - **Third step: Tritium inventory and release analysis**

Oxides reduction



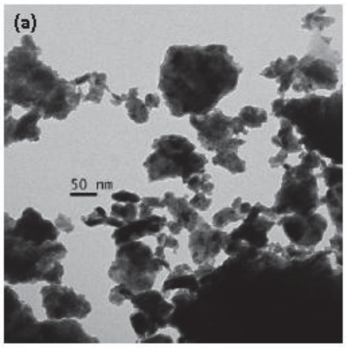
Tritium release analysis

Tritium release with T° under air or He/Ar atmosphere studied by liquid scintillation (LSC)



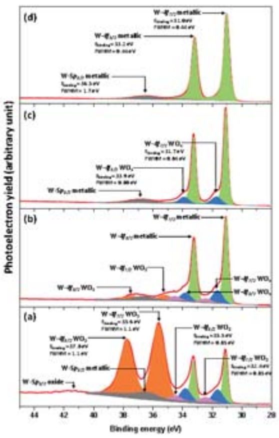
Total tritium content by full dissolution of powder and LSC

Results: SEM picture of the powder used (milled)



- 50 – 100nm
- SSA ~ from 7.5 to 15 m^2/g

Results: no oxide layer (XPS characterization)



XPS spectra for:

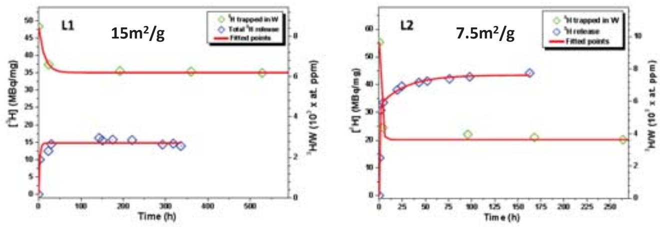
(a) Milled powder as received

(b) Same samples after surface oxides reduction

(c) Polished massive W sample (as received)

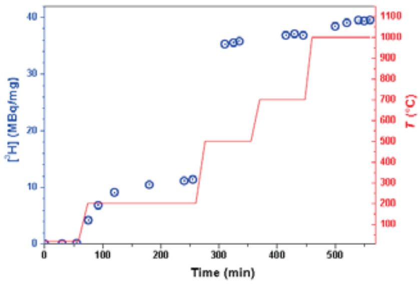
(d) Massive W sample after Ar ions etching

Results: tritium release (powder at RT under air flow)



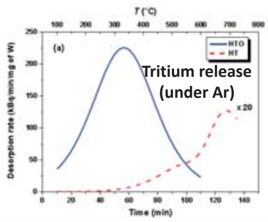
- 20 to 35 MBq/mg of tritium trapped (depending on SSA)
- Rapid release and then a steady state
- Crude extrapolation: 900Kg of W powder ~ 100g of Tritium

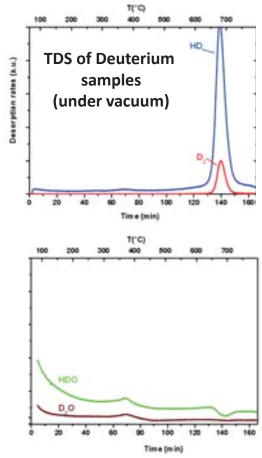
Results: tritium release with T° (air flow)



- 30% of Tritium desorbed at 200°C
- Most of Tritium released between 300 and 500°C
- After 1000°C, full dissolution of residue ~ 0,37MBq/mg

Results: Tritium and Deuterium release (samples implanted in the same conditions)

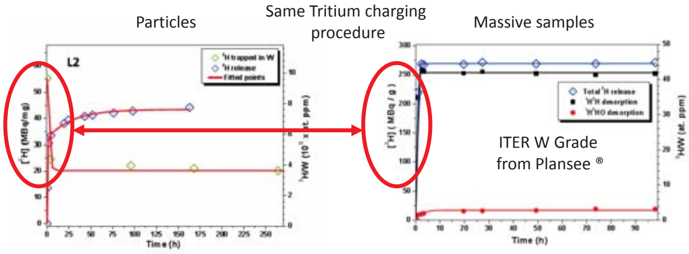




- Peak of water not observed with Deuterium implantation
- Could come from chemical surface activation processes due to beta emission inducing tritiated water formation at the surface (?).

(TDS: Thermo-Desorption Studies)

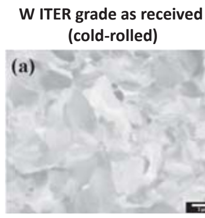
Results: comparison with massive samples

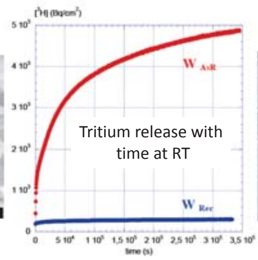


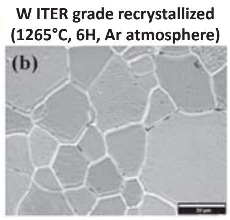
- Much larger amount of tritium released by W particles (ratio: 10^2 to 10^3)
- 20×10^3 MBq/g trapped in particles versus 11,5 MBq/g in massive samples (ratio: 10^3)

Surface effects?

Results: comparison with different types of massive samples, role of defects



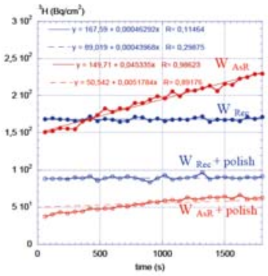




- Difference of one order of magnitude between cold rolled and recrystallized samples
  - large role of microstructural defects present in cold rolled material
- The release values much larger than expected from solubility data
  - Modeling activity ongoing

Results: assessment of implantation in massive samples under the surface

Surface activity recorded on massive W samples before and after surface polishing



Polishing remove
~1 µm of material

- **Reduction of Tritium activity when the surface layer is removed:**
  - **T is trapped at the surface (especially in cold rolled material)**

- **However, Tritium is also trapped in the bulk material**
  (~50% for recrystallized material

conclusions

An original procedure of thermal charging in gas phase has been shown to be successful for the incorporation of tritium in tungsten particles.

Different desorption tests were conducted both at room temperature and high temperature on well characterized ITER like W particles and massive samples showing a different behavior of tritium in particles and in massive samples.

A much larger amount of tritium is incorporated in W particles than in massive samples indicating important surface effects on tritium absorption, desorption and trapping in W. Moreover the behavior of tritium in massive samples was also shown to depend on the metal microstructure.

Tritium evolving from tritiated W particles was shown to desorb mostly as tritiated water – major component – with minor tritium gas species which occur at different temperatures revealing different types of tritium/tungsten interactions occurring either on the surface or in the bulk of the metal.

The strong enhancement of tritium absorption/desorption in W particles may have important implications on the management of ITER reactor.

The characterization of tritium absorption/desorption in ITER-like tungsten particles is the basis for future research on the toxicity of these particles that could be inhaled in case of accidental contamination.

Paper submitted to International Journal of Hydrogen Energy:
Authors: El-Kharbachi et al.
Tritium absorption/desorption in ITER-like tungsten particles

New activity: Tritium trapping in W alloys

- **Tritium implantation studies in different W based materials:**
  - **ITER grade W**
  - **W deposited layer by PVD (Physical vapor deposition)**
  - **W alloys:**
    - **W solide solutes as W-Ta**
    - **Particle reinforced W as W-Y_2O_3 or W-TiC**

- **Modeling the Tritium implantation in close collaboration with CRP's Marie France Barthe**

As an information, this work will start in one or two months

in parallel, we will work on:

- **T trapping on dust produced by plasma sputtering**
- **T trapping in Be dust and massive material**
- **T trapping by plasma implantation**