DE LA RECHERCHE À L'INDUSTRIE

Initiative d'excellence Aix-Marseille

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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: Dust and ITER grade materials

Contributors from CEA:

C Grisolia, E Hodille, B Rousseau, F Jambon, E Bernard, G Pieters, J Chene

And University Collaborators:

C Becquart (Orleans), MF Barthe (Lille), J Mougenot (Paris-13), Y Ferro and R Bisson (Marseille) Ph Delaporte (Marseille), D Vrel (Paris-13) And G Dinescu (Bucarest), L Begrambekov (MEPHI)

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

Activities proposed end of 2013: 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_20_3 or W-TiC

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

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

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- No real support to work on massive samples at any level (as Eurofusion)
  - 0,2PPY of support for T activity ≅ 12k€

  (comparison of T inventory of massif and layer, to be presented at the next ICFRM)

➔ Experimental activities focused on dust and supported by

- Aix Marseille University project on consequences of dust inhalation on cell
- ITER contract on behavior of dust in different media

What are the open issues raised by dust in ITER and generally in future tokamaks?

Tokamak dust properties

Due to severe Plasma Wall Interactions, fusion machines produce tritiated dust.
In ITER, safety limit: one ton of dust in the Vacuum Vessel

These dust:

- **are activated and tritiated.**
- **Are of different compositions (in ITER: pure W, pure Be and mixed materials)**
- **Have large size distribution (from 10s of nanometer to cms)**
  - **In Carbon machine, poly-disperse size distribution centered at 2-3 µm**
- **Experience large Specific Surface Area due to accretion processes**

  ➔ **<u>Enhanced surface effects</u> (compared to massive samples)**

They can escape from the Torus in case of Lost Of Vacuum Accident (LOVA)

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Open questions:

- **What is the total quantity of tritium trapped in these particles?**
- **How tritium is released in function of the ambient atmosphere? In aqueous media?**
- **In case of inhalation :**
  - **What is the behavior of these particles/of the tritium?**
  - **Are the particles dissolved in the biologic media?**
  - **Where are the particles/the tritium located in the cells?**
  - **What are the consequences inhalation in term of cyto-toxicity and of geno-toxicity?**

Tasks to be done

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Tasks to be done:

- **ITER relevant particles production and characterization (no tokamak production)**
  - **Current work on W (but Be studies (dust and massive) starting in September)**
  - **Targets :**
    - **Small particles of about 100nm for Biological test**
      - **High probability to escape HEPA filters**
    - **Larger size for comparison**
- **Particle tritiation → total quantity of tritium trapped**
- **Tritium release studies**
  - **in different media and ambiances (including biological media)**
  - **Tritium speciation**
- **+ In vitro tests**

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- \+ In vitro tests

Deuterium implantation studies: The WHISCI project (06/2015)

- how D retention in tungsten depends on:
  - Sample (temperature, grade…)
  - Ion flux (10^{18}-10^{22} D.m^{-2}.s^{-1})
  - Ion fluence (10^{21}-10^{27} D.m^{-2})
  - --> a non-linear trend on 5 orders of magnitude

Retention α Fluence$^{0.5\text{-}0.7}$

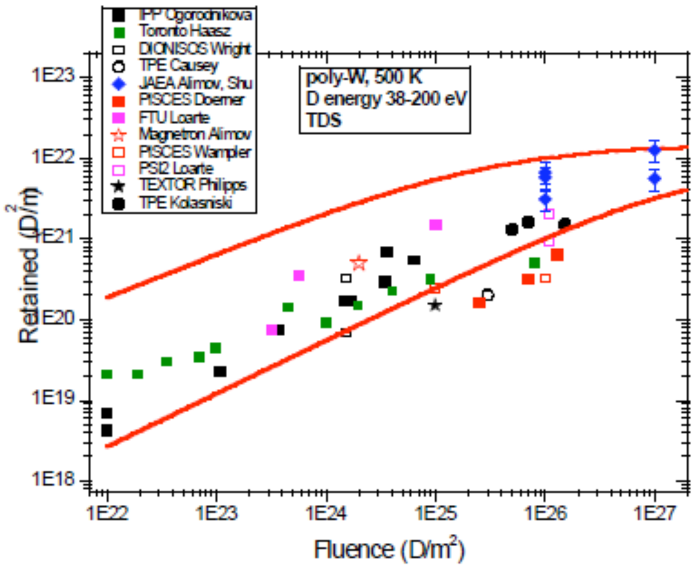
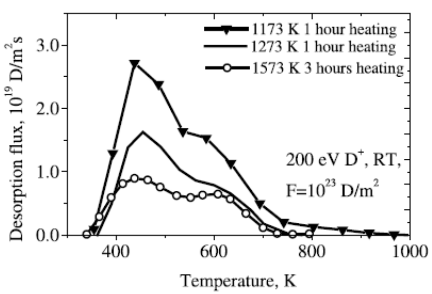
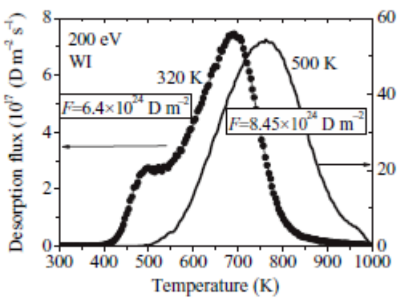


Figure 7.1.1: Version of Figure 3.5.1 with new data from U. Toronto and Alimov added.

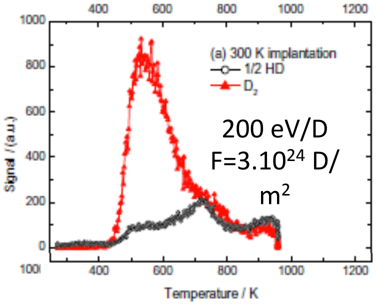
- **Problem: TPD varies from one lab to the other… what physics to put in models?**



Ogorodnikova *et al.* J. Nucl. Mater. 313-316, 469 (2003)



Ogorodnikova *et al.*, Phys. Scr. T138, 014053 (2009)

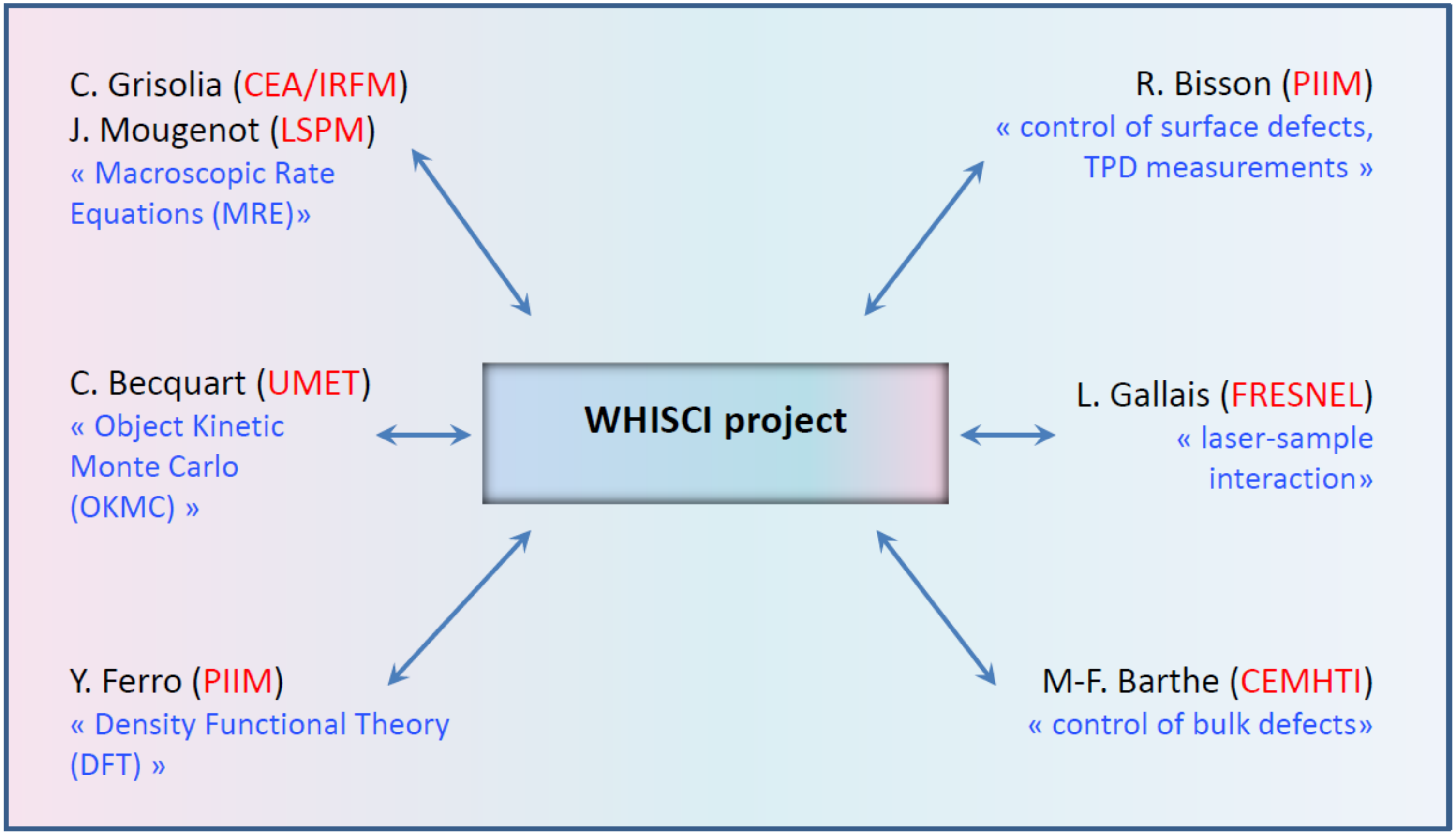


Tian *et al.*, J. Nucl. Mater. 399, 101 (2010)

TPD: Temperature Programmed Desorption

Deuterium implantation studies: The WHISCI project (06/2015)

Multi-scale modeling validated by well controlled laboratory experiments



Coordinator: Regis Bisson (PIIM Laboratory)

Strong and constant interactions in place
(starting 3 years ago)

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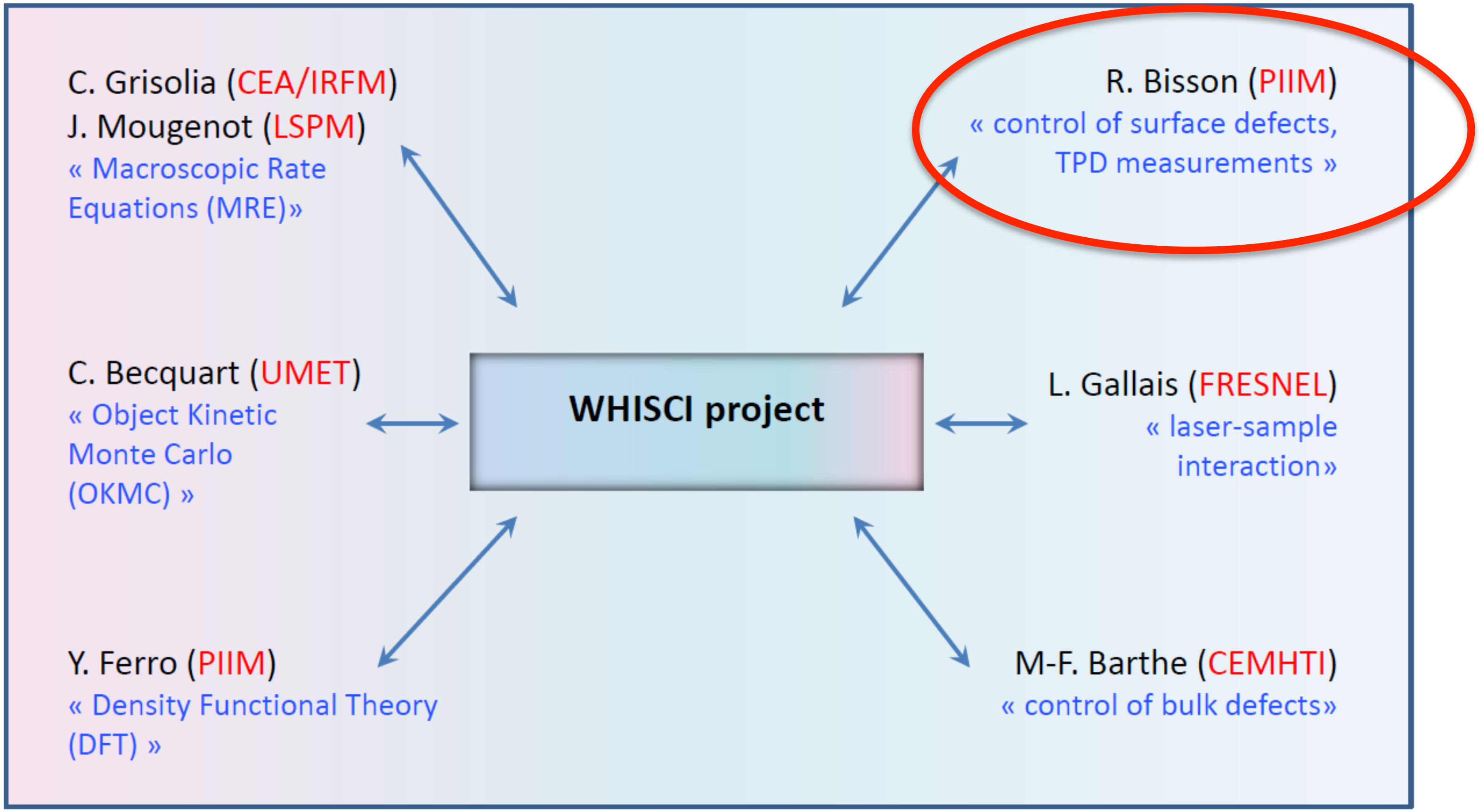

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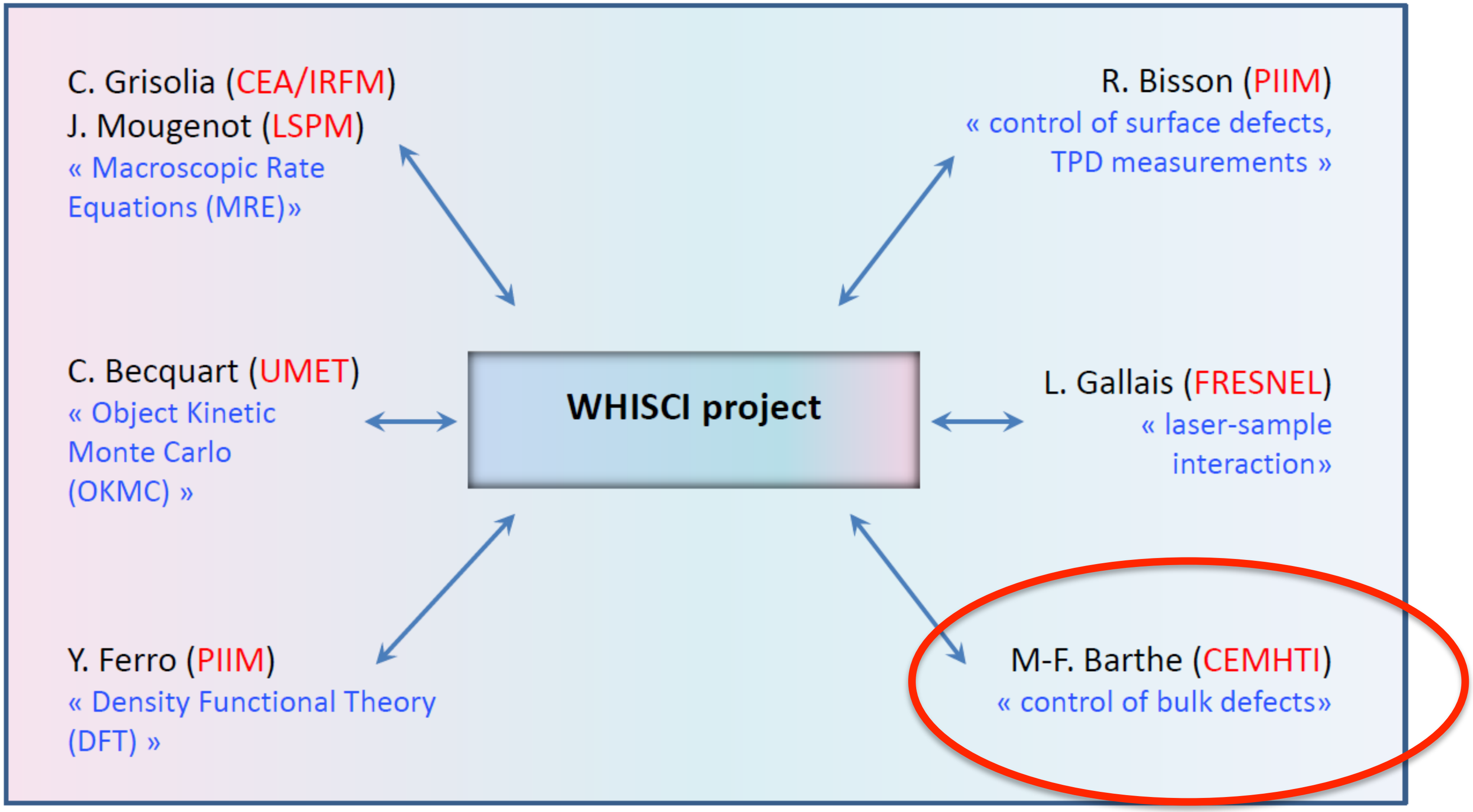

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DE LA RECHERCHE À L'INDUSTRIE

Outline

1. Particles production and characterization
2. Samples tritiation (massive & dust)
3. Tritium inventory and release
   - In gas (comparison of dust and massive samples)
   - In aqueous solution
4. Deuterium implantation studies (massive samples)
5. Positrons studies of implanted samples

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1- Particles production and characterization

Targets:

- Small particles of about 100nm for Biological tests
  - High probability to escape HEPA filters
- Larger size for comparison
- Characterization: SEM/TEM, XRD, BET (SSA), XPS

Different Processes used:

- **By impaction filtration of commercial dust, IRSN**

  (F. Gensdarmes, C. Monsanglant-Louvet)

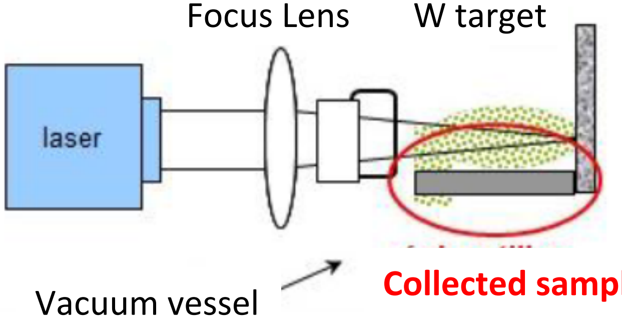
  @ Large quantity produced (g), Pure W
  @ mono-disperse, 2 types: centered at 0.7µm or at 2.9µm
  @ Low Surface Specific Area (SSA)
  @ apparent low defects density

- **By Planetary Milling, LSPM, Paris 13** (D Vrel)

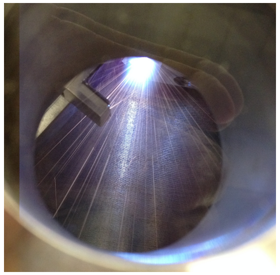
  @ Large quantity produced (g), Pure W
  @ highly poly-disperse, need complex filtration
  @ Low SSA

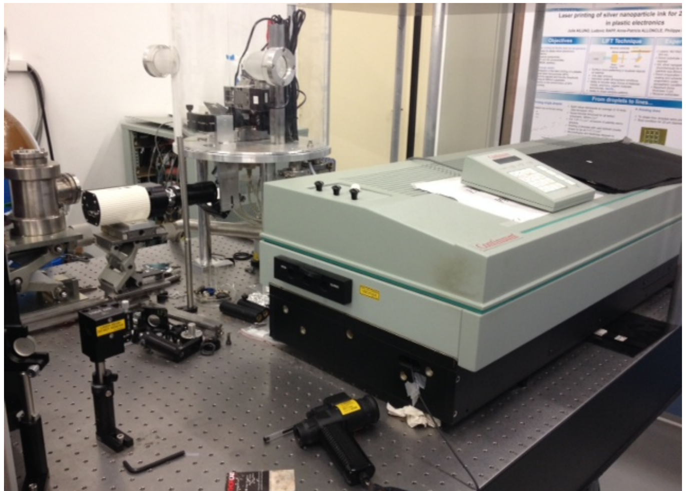
1- Particles production and characterization

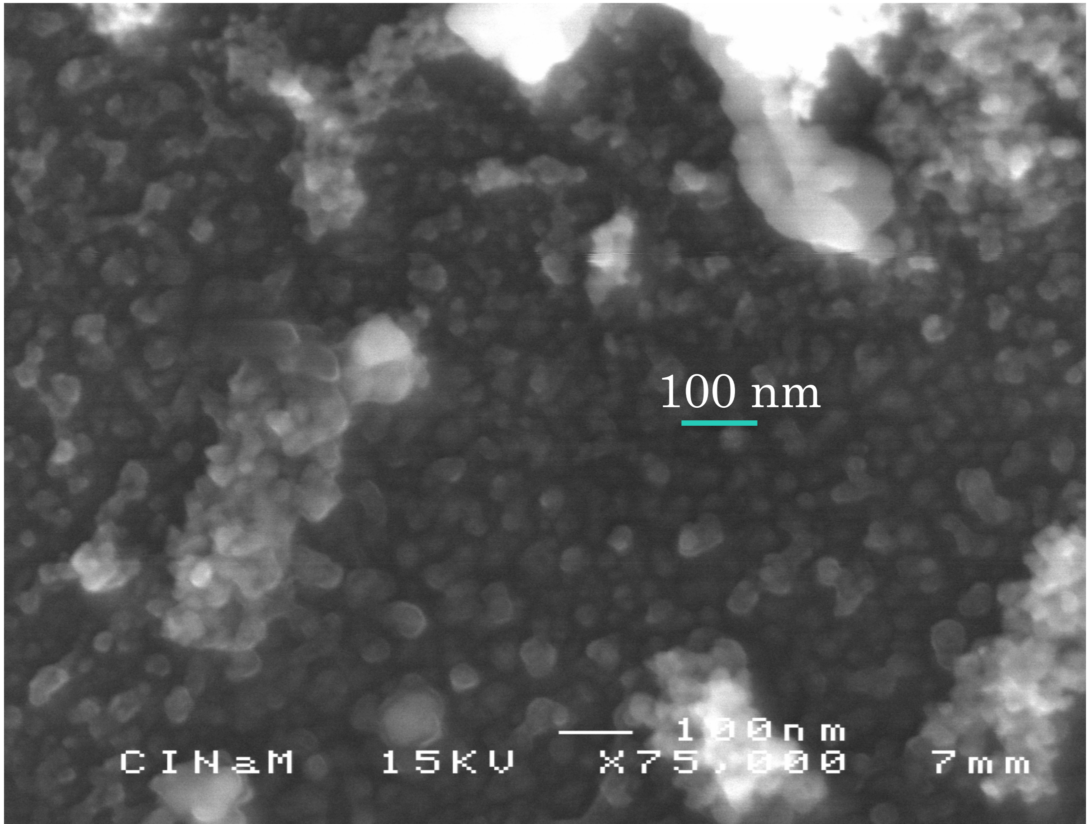
- **By laser/material interaction, LP3, AMU (P. Delaporte, E Bernard)**



- **Low production rate: 6.5 mg W particles/h, pure W**
- **Large SSA (to be measured)**
- **Mono disperse (80 nm)**



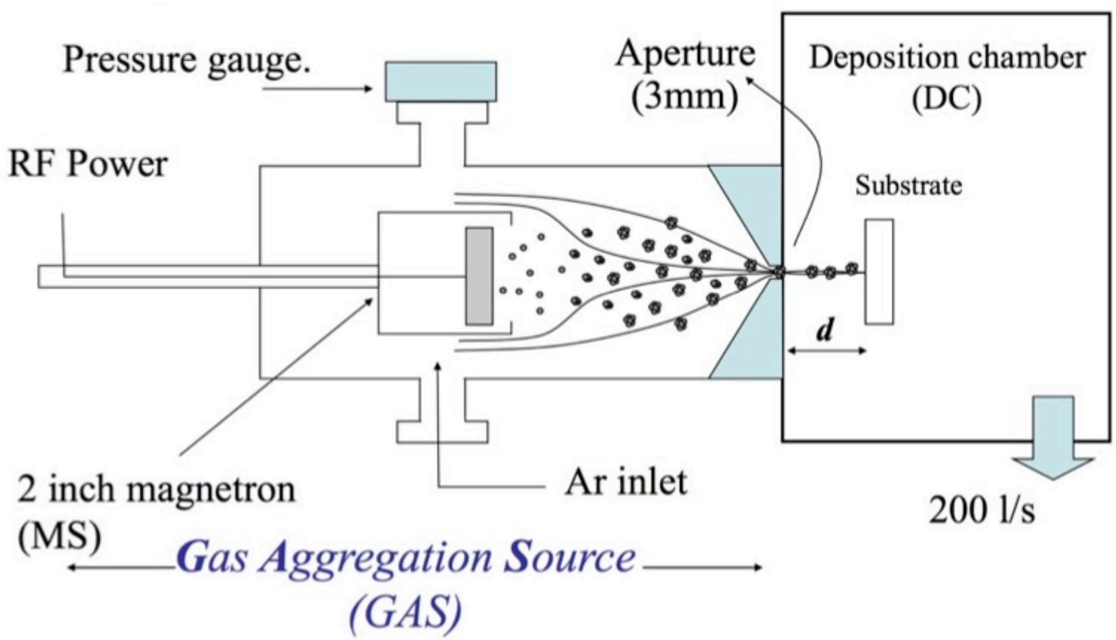


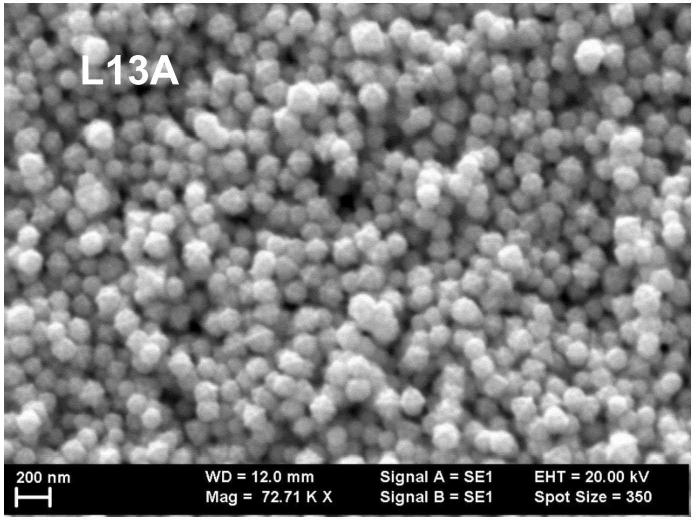


1- Particles production and characterization

- **By plasma/material interaction, NILPRP, Romania**

  (G Dinescu and T Ascente)



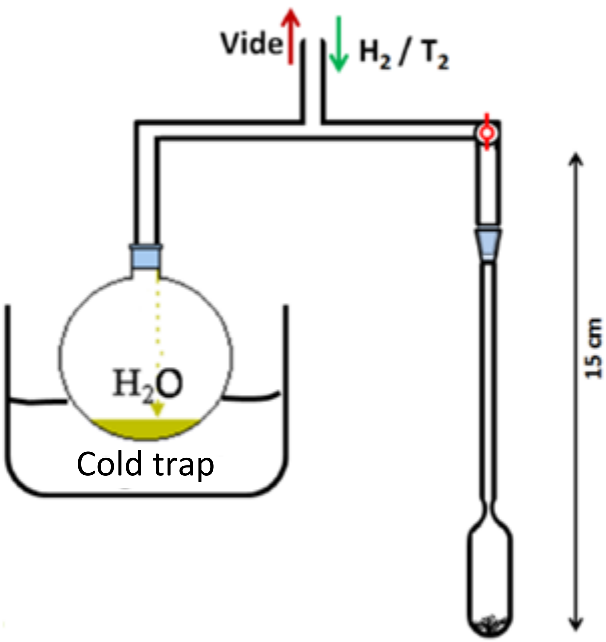


- Relatively large quantity, pure W
- Large SSA (To be measured)
- Mono-disperse (80nm)
- Mixture of α (CC) and β phase, β usually unstable

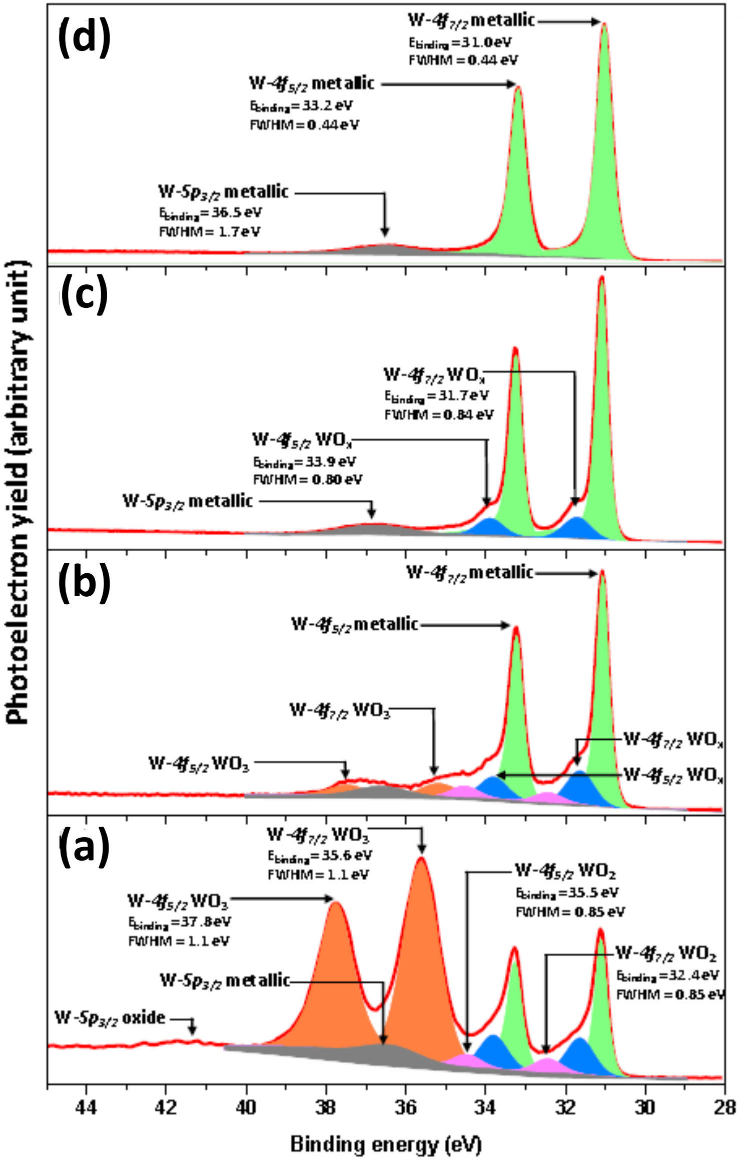
2- Samples tritiation

- 20-40 mg of powder treated each time (or 500mg of massive sample)
- 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)

Oxides reduction



2- Particles tritiation: surface oxides reduction



X-Ray Photoelectron Spectroscopy (XPS) spectra:

(d) Massive sample after Ar ions etching

(c) Polished massive W sample (as received)

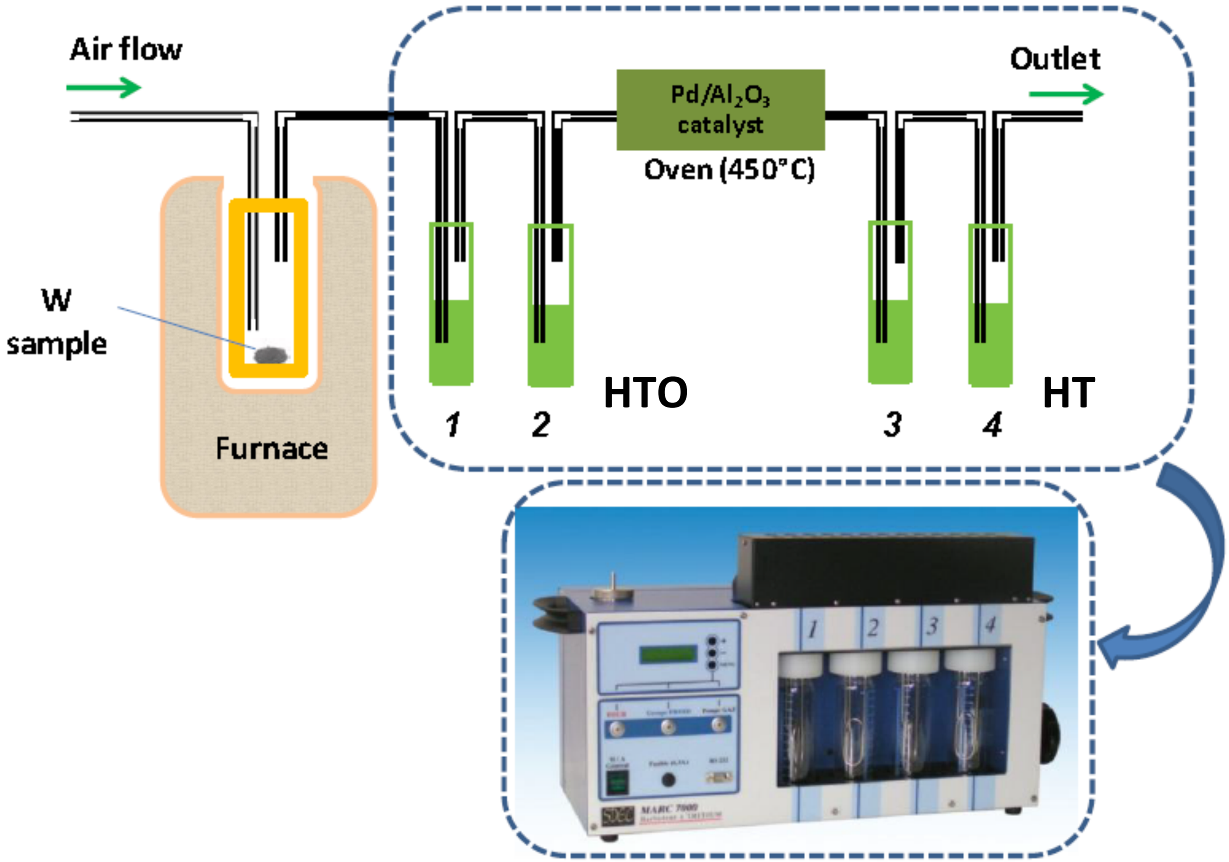
(b) Same sample after oxides reduction

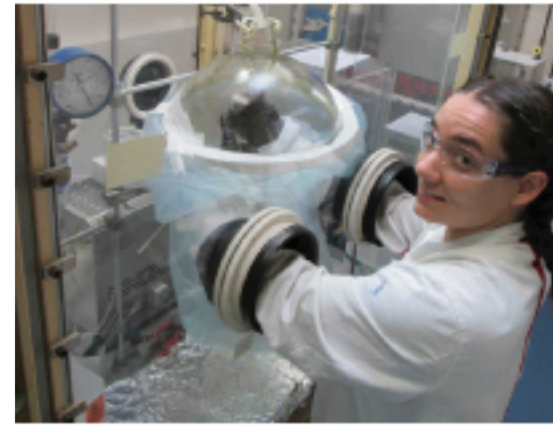
(a) Powder as received

Surface oxides removed by reduction process

3- Tritium inventory

- **Tritium release with Temperature under gas atmosphere measured by liquid scintillation (LSC)**

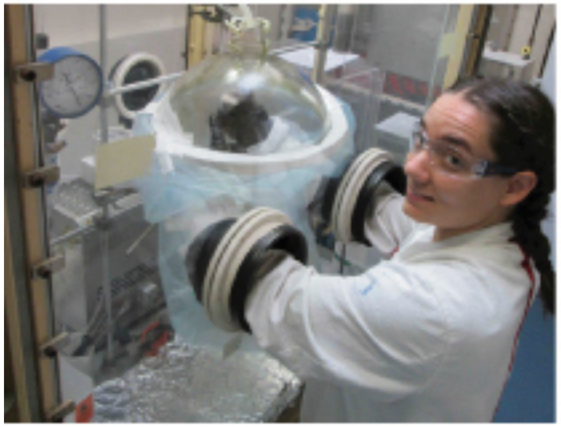


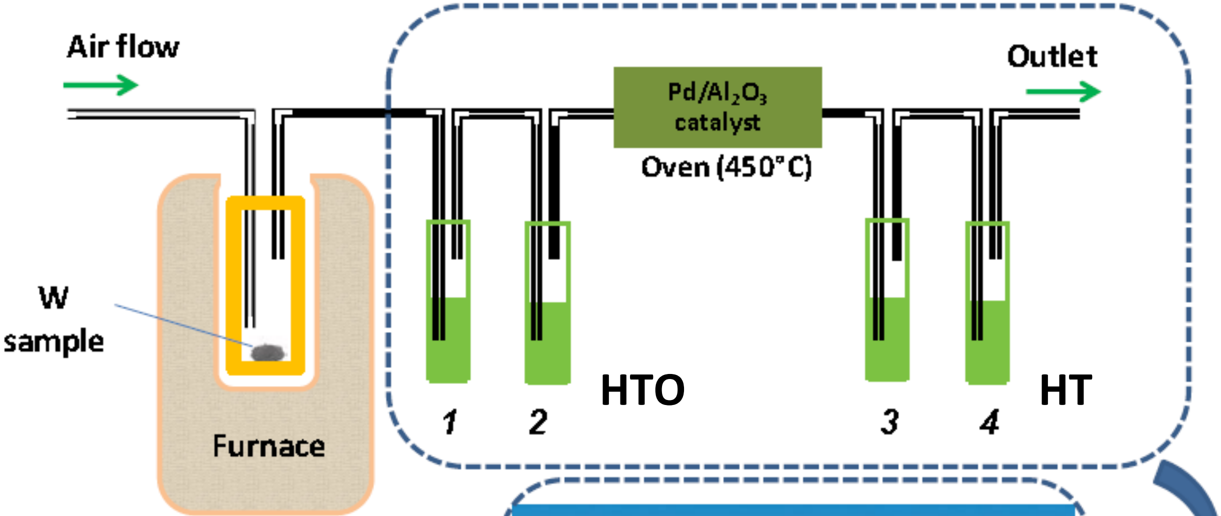


- **Total tritium content by full dissolution of powder and LSC**

3- Tritium inventory

- Tritium release with Temperature under gas atmosphere measured by liquid scintillation (LSC)





Release capability: 60TBq/year
Each experiment: 1TBq ≅ 27Ci

- Total tritium content by full dissolution of powder and LSC

3- Tritium inventory: powder stored at RT

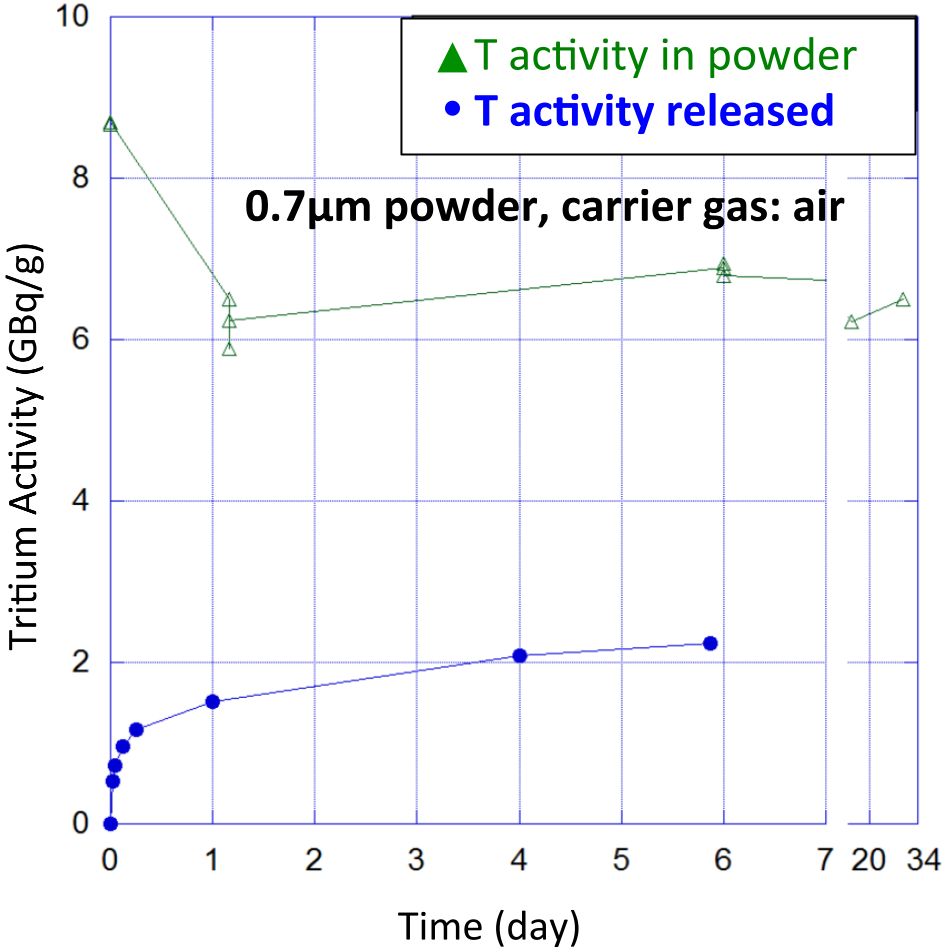
Filtration by impaction

@ Large quantity produced (g), Pure W

@ mono-disperse, 2 types: centered at 0.7µm or at 2.9µm

@ Low Surface Specific Area (SSA)

@ apparent low defects density

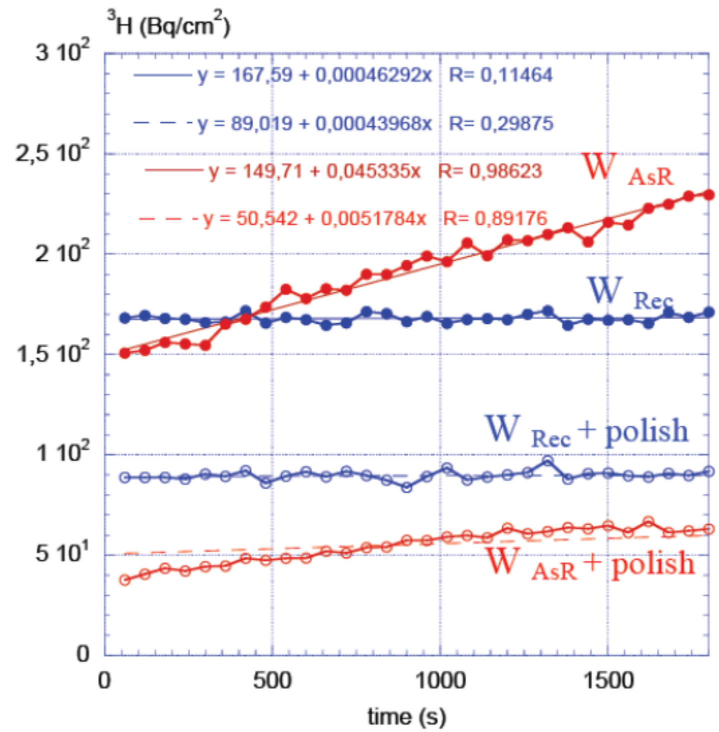


High tritium inventory: > 1Gbq/g

2-3 orders of magnitude than massive W

3- Tritium surface activity of massive samples

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

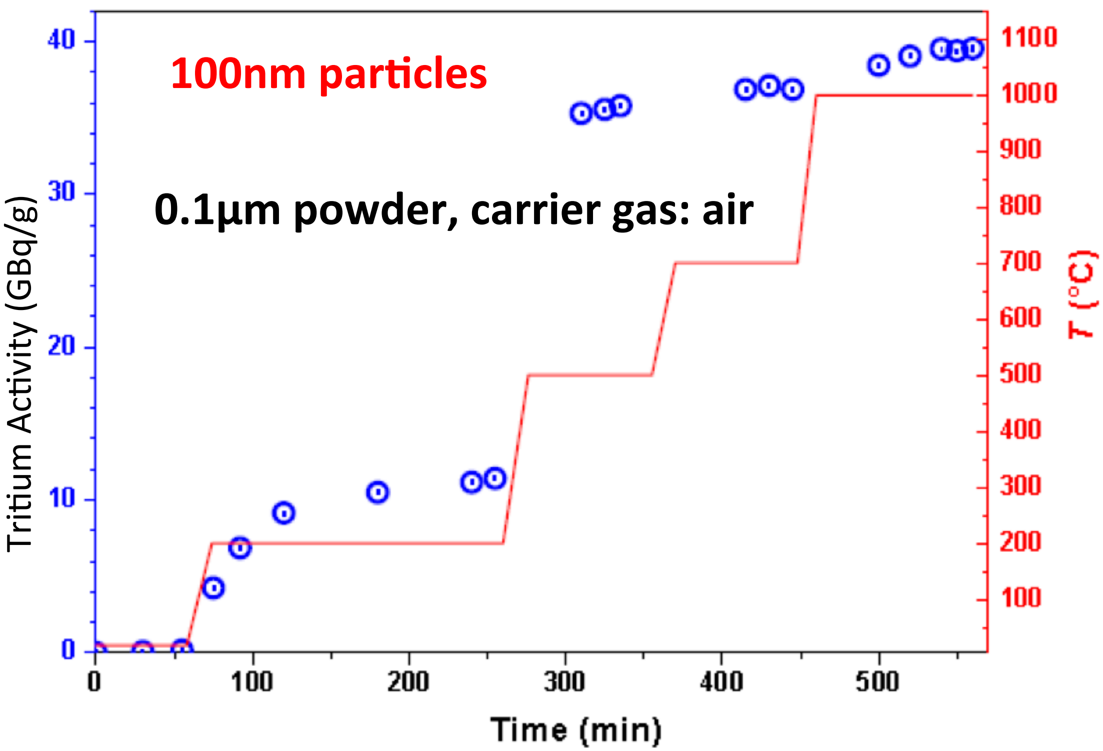


Polishing remove ~1 µm of material

- **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 (~40% for recrystallized material)**

3- Tritium release: with Temperature

Planetary milling
@ Large quantity produced (g), Pure W
@ highly poly-disperse, need complex filtration
@ Low SSA



T inventory varies with type and size of dust

- Highest T content (40GBq/g)
- Tritium trapping increases with SSA
  If density of traps homogeneous
  ➔ trap concentration $>10^{-3}$ (trap/W)
  ➔ Impossible with crystalline W

↓

Tritium trapping in powder triggered by surface effects

Temperature desorption

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

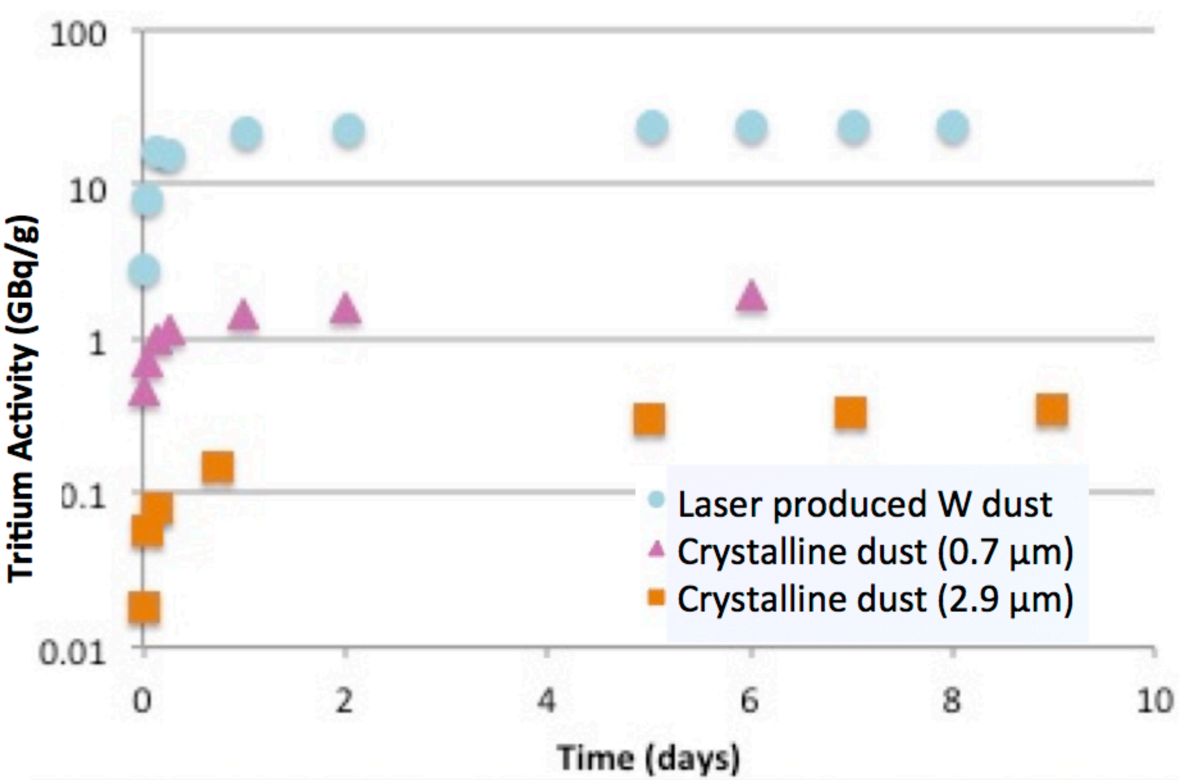
3- Tritium release: with Temperature, air flow

Filtration by impaction

@ Large quantity produced (g), Pure W
@ mono-disperse, 2 types: centered at 0.7µm or at 2.9µm
@ Low Surface Specific Area (SSA)
@ apparent low defects density

Laser production

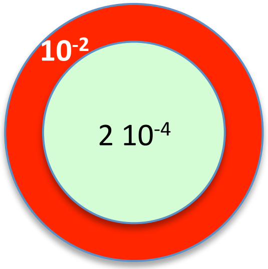
- Low production rate
- Large SSA (to be measured)
- Mono disperse (80 nm)



- T release ↗ with SSA
- Release of T in HTO form mainly due to moisture (H_20) in the carrier gas

3- Tritium inventory: Extrapolation to ITER (tentative)

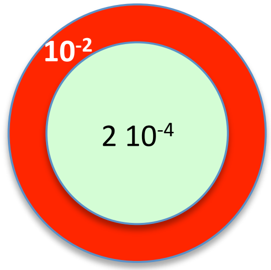
Dust Material =
bulk low defects + shell high defects



- **100nm dust**
- dust at 0.1, 10, 100 Pa during 10s after a shot,
- Constant temperature

3- Tritium inventory: Extrapolation to ITER (tentative)

Dust Material =
bulk low defects + shell high defects



- 100nm dust
- dust at 0.1, 10, 100 Pa during 10s after a shot, initially no T
- Constant temperature

Macroscopic Rate Equation Model

$$\frac{\partial C_t}{\partial t} = -C_t . \nu_{detrapping} . e^{-\frac{E_T}{k.T}} + n_{trap} . \nu_{trapping}(T) . C_m . \left(1 - \frac{C_t}{n_{trap}}\right)$$

$$\frac{\partial C_m}{\partial t} = D(T) . \frac{\partial^2 C_m}{\partial x^2} - \sum \frac{\partial C_t}{\partial t}$$

Boundary condition: $C_{m,surface} = \sqrt{P} . S(T)$

$\nu_{trapping}(T) = K . D(T)$, **K depending on W lattice constant**

$\nu_{detrapping} = 10^{13}\ s^{-1}$ **jump attempt frequency**

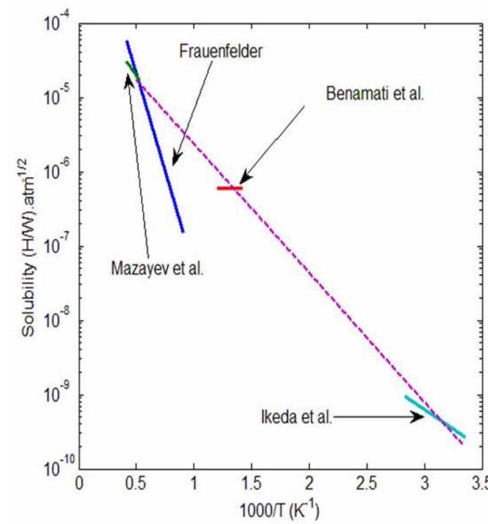
n_{trap} **= trap density**

E_T **= trapping energy**

$D(T) = 4,1\ 10^{-7} \times e^{-\frac{0,39}{k.T}}$ **en** m^2/s

MRE Parameters

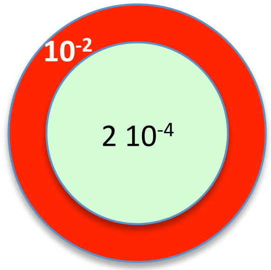
- Solubility from literature (few papers)



- Trap energies from massive samples study
- Trap densities from fit of experimental results from gas infusion (470°C, 1Ba)

3- Tritium inventory: Extrapolation to ITER (tentative)

Dust Material =
bulk low defects + shell high defects



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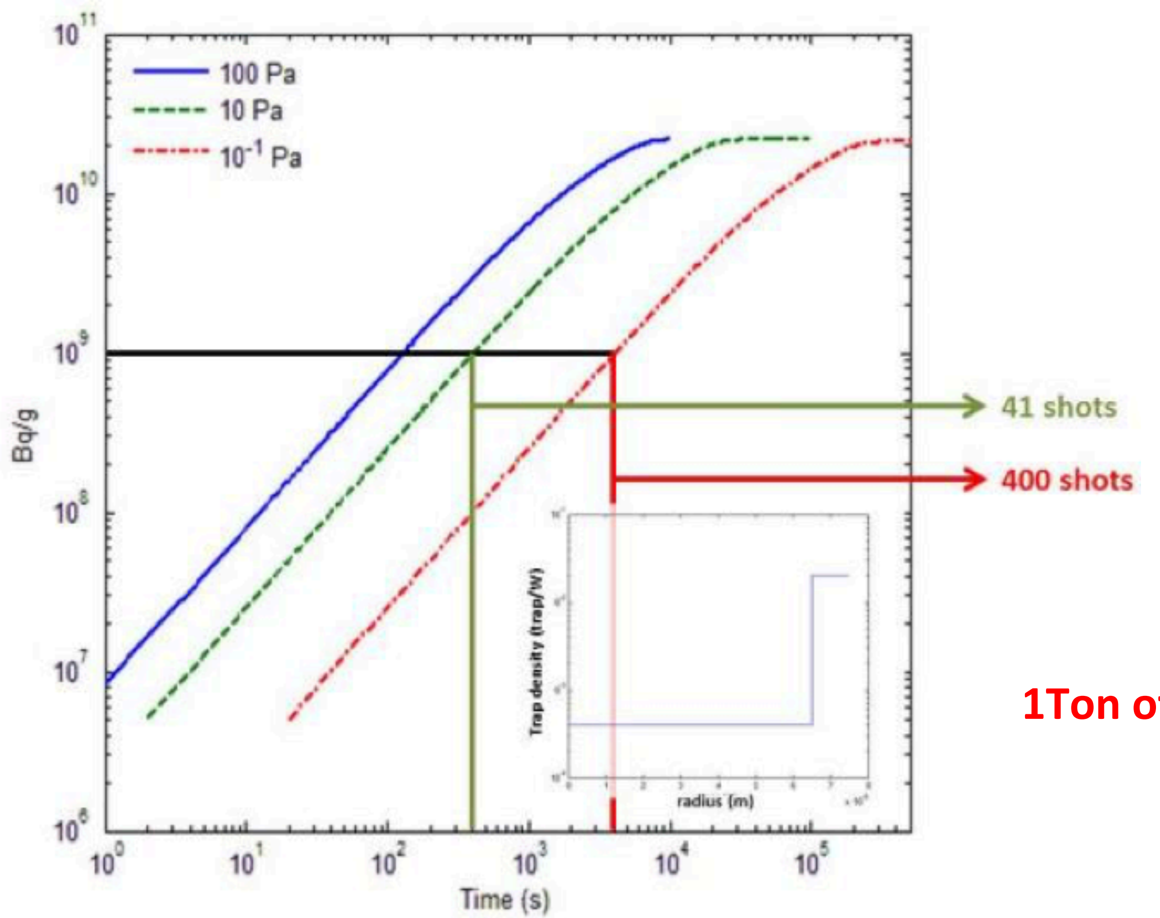


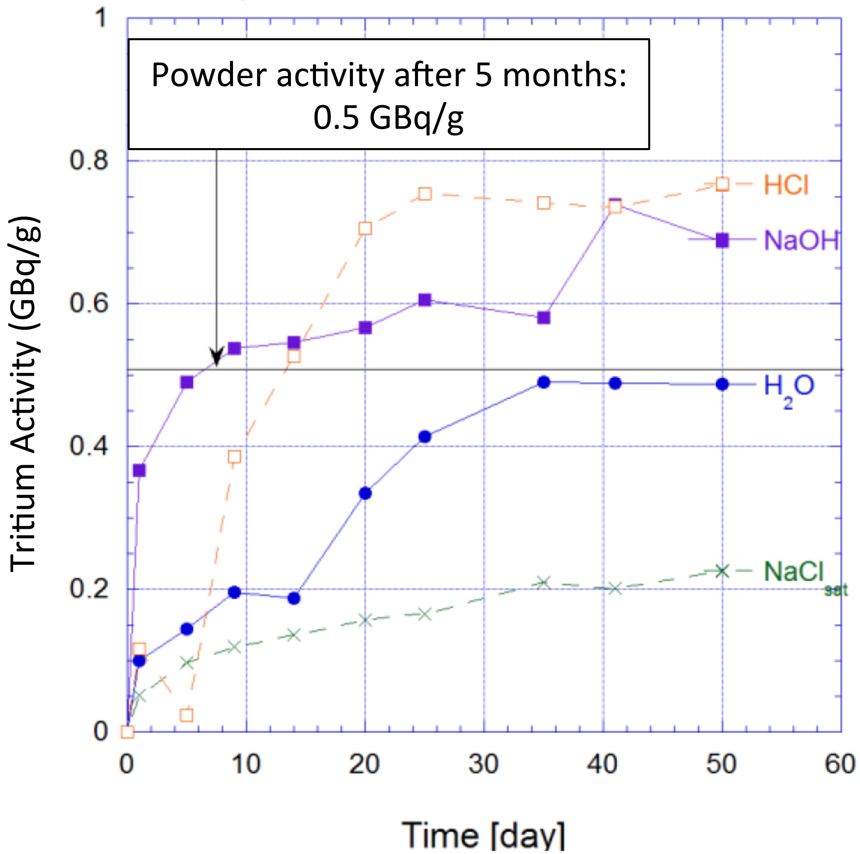
1Ton of dust ↔ 50g of T (< safety limit)
1GBq/g rapidly reached

3- Tritium release: in aqueous media

Filtration by impaction

@ Large quantity produced (g), Pure W
@ mono-disperse, 2 types: centered at 0.7µm or at 2.9µm
@ Low Surface Specific Area (SSA)
@ apparent low defects density

Tritium content in aqueous solution, 2.9µm after 5 months tritium release



- in almost all conditions, ≅ T transferred to the aqueous solution.
- $\tau_{dissolution}$ ≅ some days
- $\tau_{dissolution}$ lower in acid/basic media than in pure water.
- In agreement with litterature[1]:
  - Ph>6, W_{solid} ➔ WO_4^{-2}
  - Ph<6, W_{solid} ➔ $H_2WO_{4\ (solid)}$

[1] Anik & Osseo-Asare, J. of the Electrochem. Soc. , 149 (6) B224, 2002

Conclusions and perspectives

- Dust in tokamak are of different size and shape
- To accomplish our project, dust have been produced in purpose by different means
- In case of W dust:
  - large T inventory ↗ with dust SSA
    - T inventory > 1GBq/g (100 to 1000 times than massive sample)
    - T inventory ↗ with SSA (tritium trapping linked with surface processes)
    - T Inventory is stable at Room Temperature
    - Almost all the T released before 500°C
      - but 1% of the initial inventory remains in the dust at 1000°C
    - Extrapolation to ITER:
      - If all the one ton of dust is pure W: 50g of T (<< ITER tritium limit)
      - However, 1Gbq/g reached in a limited number of shots (less than 1000 ITER shots)
  - T release ↗ with dust SSA
  - In aqueous solution, T released due to dissolution of W dust
    - Slow process (all T released in 10-40 days)
    - More rapid in acid condition

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    - More rapid in acid condition

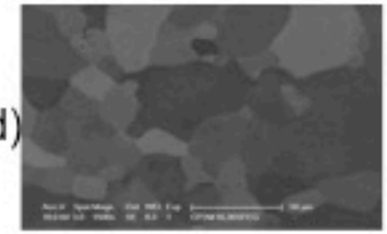
- **In September, same work starting with <u>Be dust & massive samples</u>**
- **T trapping in W layers vs W ITER grade massive samples: under study**
- **T trapping in W alloys? No support up to now!**

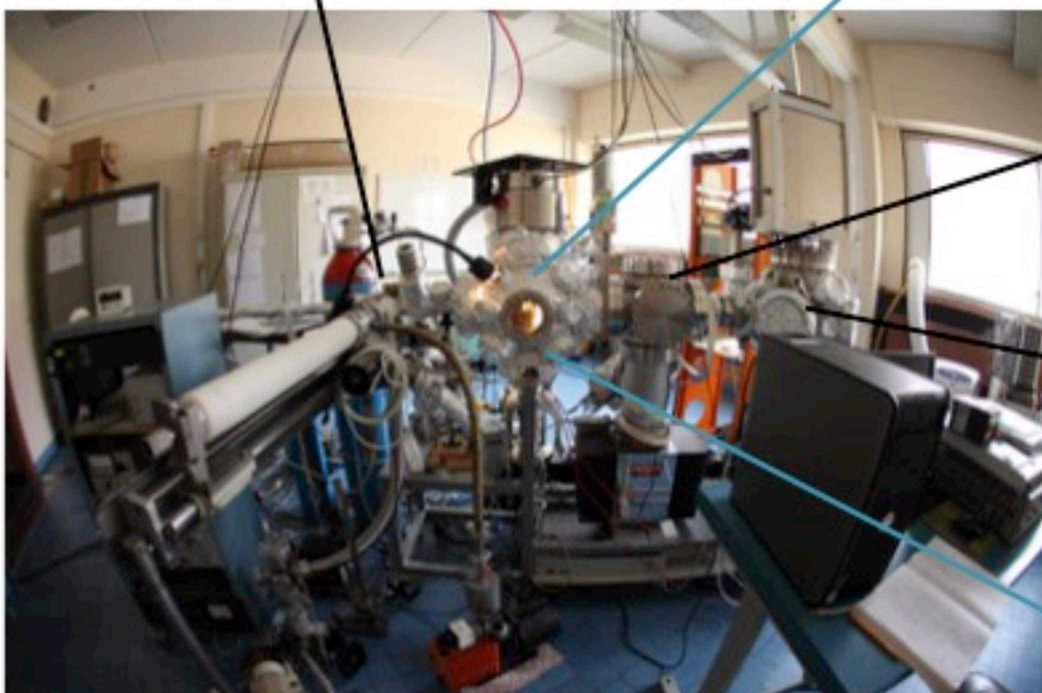
4. Deuterium implantation studies (massive samples)

Experimental set-up at AIX-Marseille University (AMU) (all in situ)

fast load-lock chamber
(base pressure: <1.10^{-9} mbar)

Polycrystalline W
(A.L.M.T. - Japan, electro-polished)





Chemical analysis
(XPS: X-ray-photoelectron spectroscopy)

plasma implantation chamber
(10^{18}-10^{20} ion.m^{-2}.s^{-1}, 20 eV – 1 keV)

ion gun implantation chamber
(10^{16}-10^{17} ion.m^{-2}.s^{-1}, 250 eV – 5 keV)

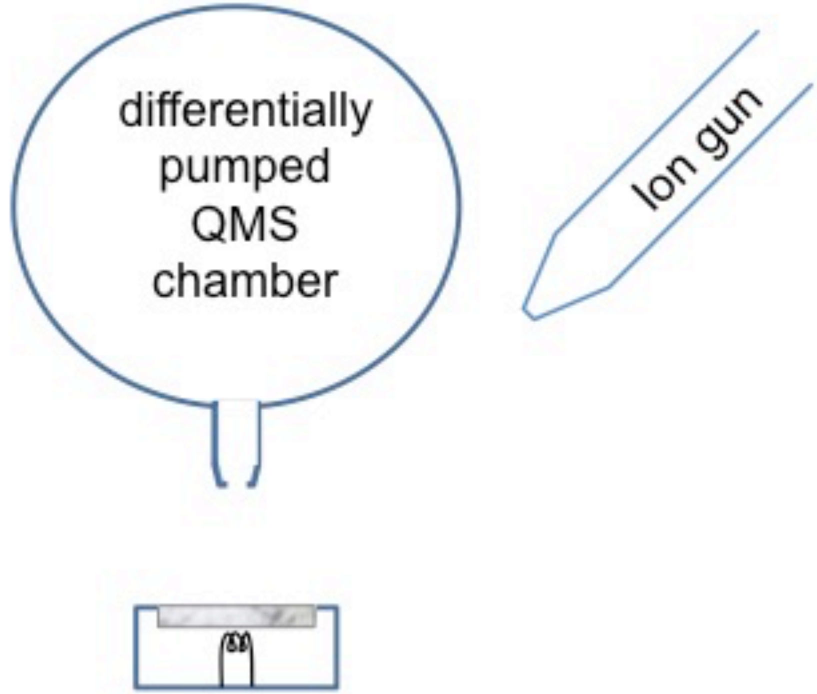
and high-sensitivity TPD
(base pressure: 1.10^{-10} mbar)

(TPD: Temperature Programmed Desorption)

4. Deuterium implantation studies (massive samples)

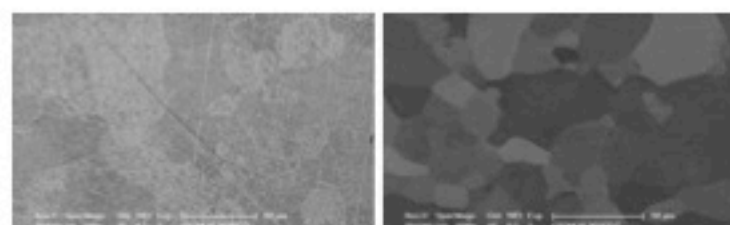
Deuterium retention in polycrystalline tungsten at low fluences

the AMU apparatus: all *in situ*



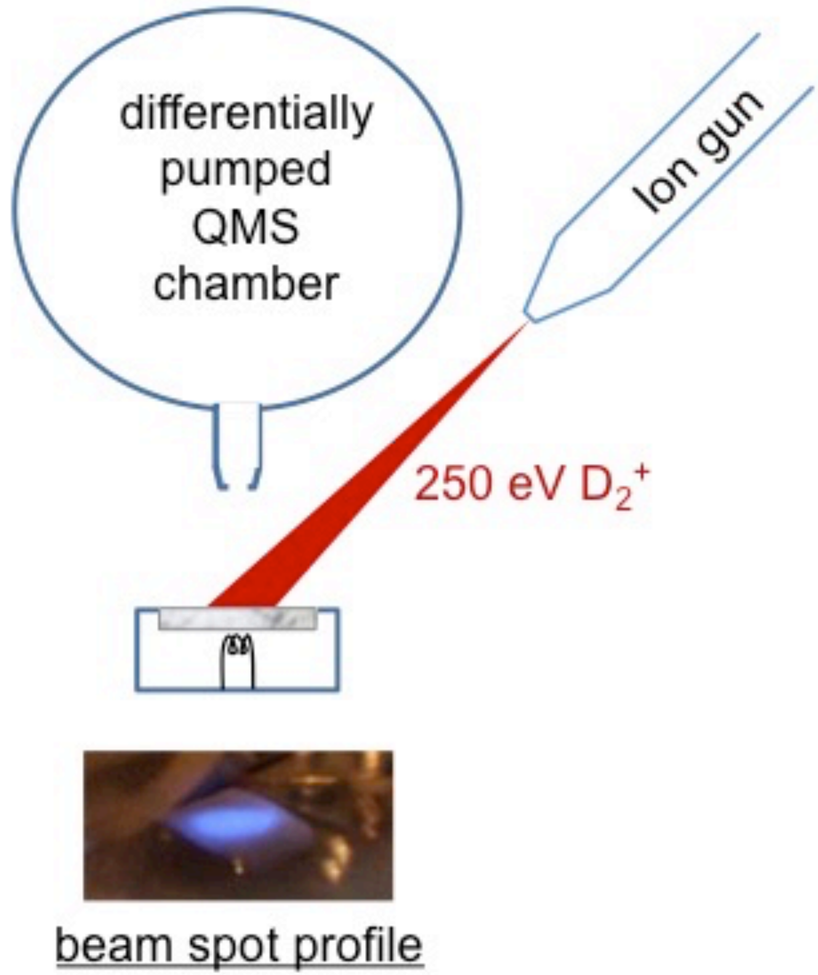
Polycrystalline sample with low defect concentration

- Recrystallized (1573K-1h) in vacuum by A.L.M.T. (Japan)
- Electro-polished in-house for mirror finish
- Annealing at 1300 K in ultra-high vacuum

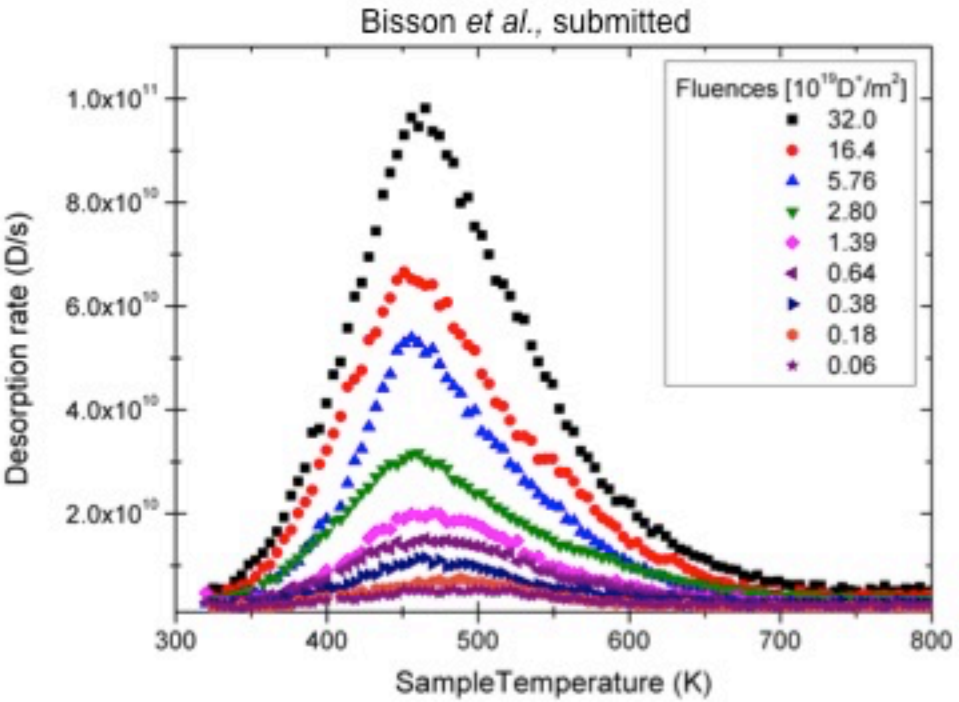


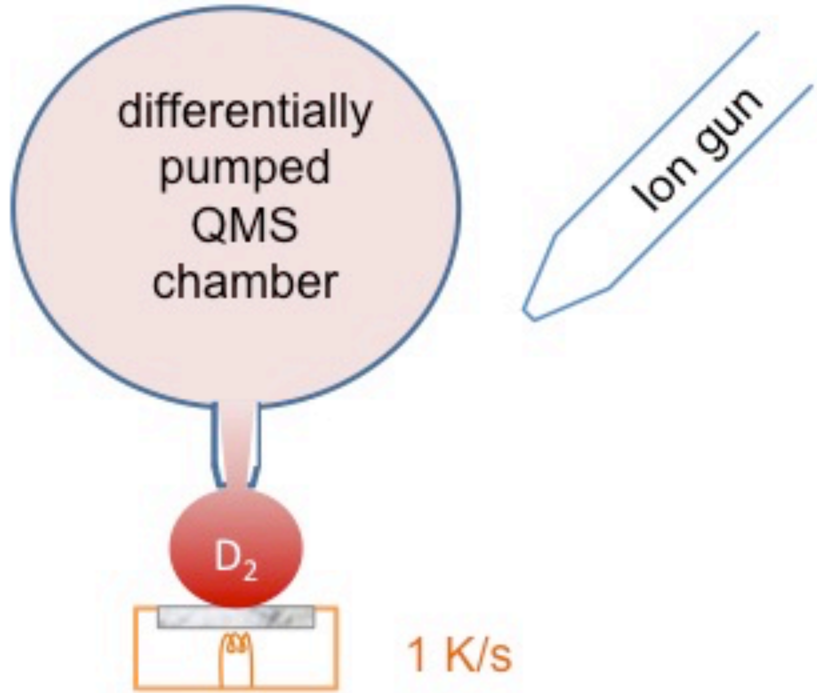
as received after electropolishing

4. Deuterium implantation studies (massive samples)



4. Deuterium implantation studies (massive samples)





- Absolute calibration of the TPD in desorption rate (D_2/s)

- Once integrated, a TPD gives an absolute deuterium retention for a given fluence (D/m^2)

checked with NRA measurements (JSI, Slovenia)

Deuterium retention in polycrystalline tungsten at low fluences

Results: fluence dependence

- repeated these TPD for fluence in the range $10^{17\text{-}21}$ D^+/m^2
- once combined with *in situ* data from Ogorodnikova *et al.* *similar sample preparation [JNM 313-316 (2003) 469].*

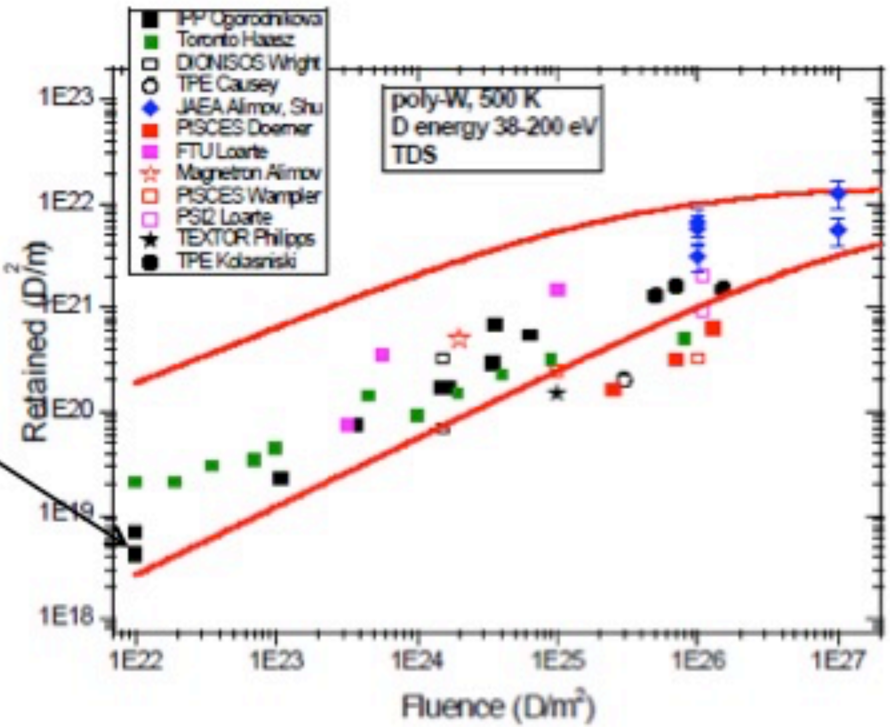


Figure 7.1.1: Version of Figure 3.5.1 with new data from U. Toronto and Alimov added.

4. Deuterium implantation studies (massive samples)

Deuterium retention in polycrystalline tungsten at low fluences

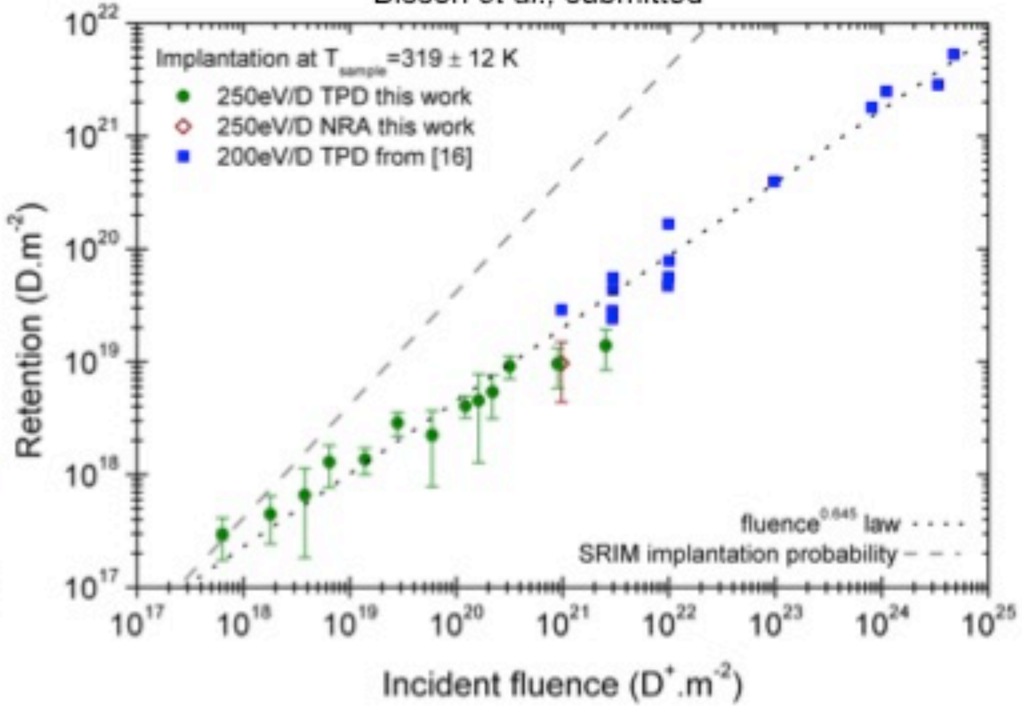
Results: fluence dependence

- repeated these TPD for fluence in the range $10^{17\text{-}21}$ D^+/m^2

- once combined with *in situ* data from Ogorodnikova *et al.* *similar sample preparation [JNM 313-316 (2003) 469]:*

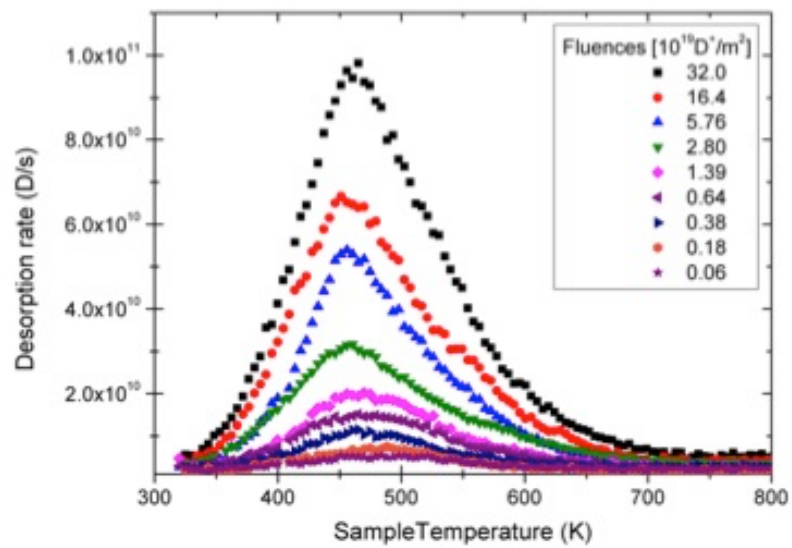
retention on the same non-linear trend on 8 orders of magnitude
(3 orders of magnitude improvement)

Bisson *et al.*, submitted



➢ **retention = a*fluence$^{0.645\pm0.025}$**
improvement on uncertainty (before 0.5-0.7)

4. Deuterium implantation studies (massive samples)



- Apparently one peak observed in PCW?
- Different from other published spectra.
- Bulk/surface retention?
  - If bulk what type of trapping site (Dislocation, GB, Vacancies)

➔ WHISCI project (well characterized samples from SCW to PCW)

(oral contribution to the next ICFRM conf. in Aachen)

Vacancy defects studied in tungsten by using Positron annihilation spectroscopy

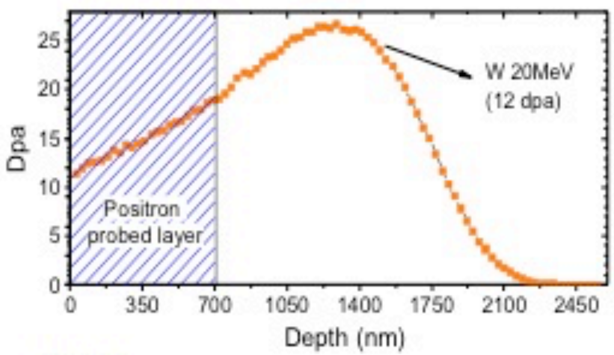
MF Barthe and co-workers

- Vacancy defects in W : Single vacancies irradiated with W ions (JANNUS Saclay)
- Positron annihilation spectroscopy & Transmission electron microscopy

5. Positron annihilation studies

Irradiation conditions

✓ **W 20 MeV**



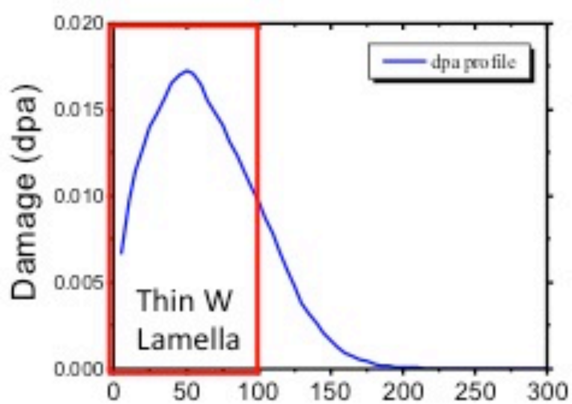
Positron probed zone

SRIM Calculations E_d=90eV

Ion	Fluence (cm^{-2})	dpa* (0-700nm) SRIM	Temperature (K)
W 20MeV	9.98×10^{12}	0.025	91
	3.70×10^{14}	1	110K
	4.49×10^{15}	12	110K
	4.49×10^{15}	12	873K

dpa* : mean value in the zone probed by slow positrons : 0-700 nm

✓ **W 1.2 MeV**



SRIM Full Cascade ~2.5×10^{-5} dpa/s

PAS ex situ

Ion	Flux ($cm^{-2}.s^{-1}$)	Fluence (cm^{-2})	dpa SRIM at RP (50 nm)
1.2MeV W	3×10^9	1.8×10^{12}	0.017

From RT to 773K

TEM in and ex situ

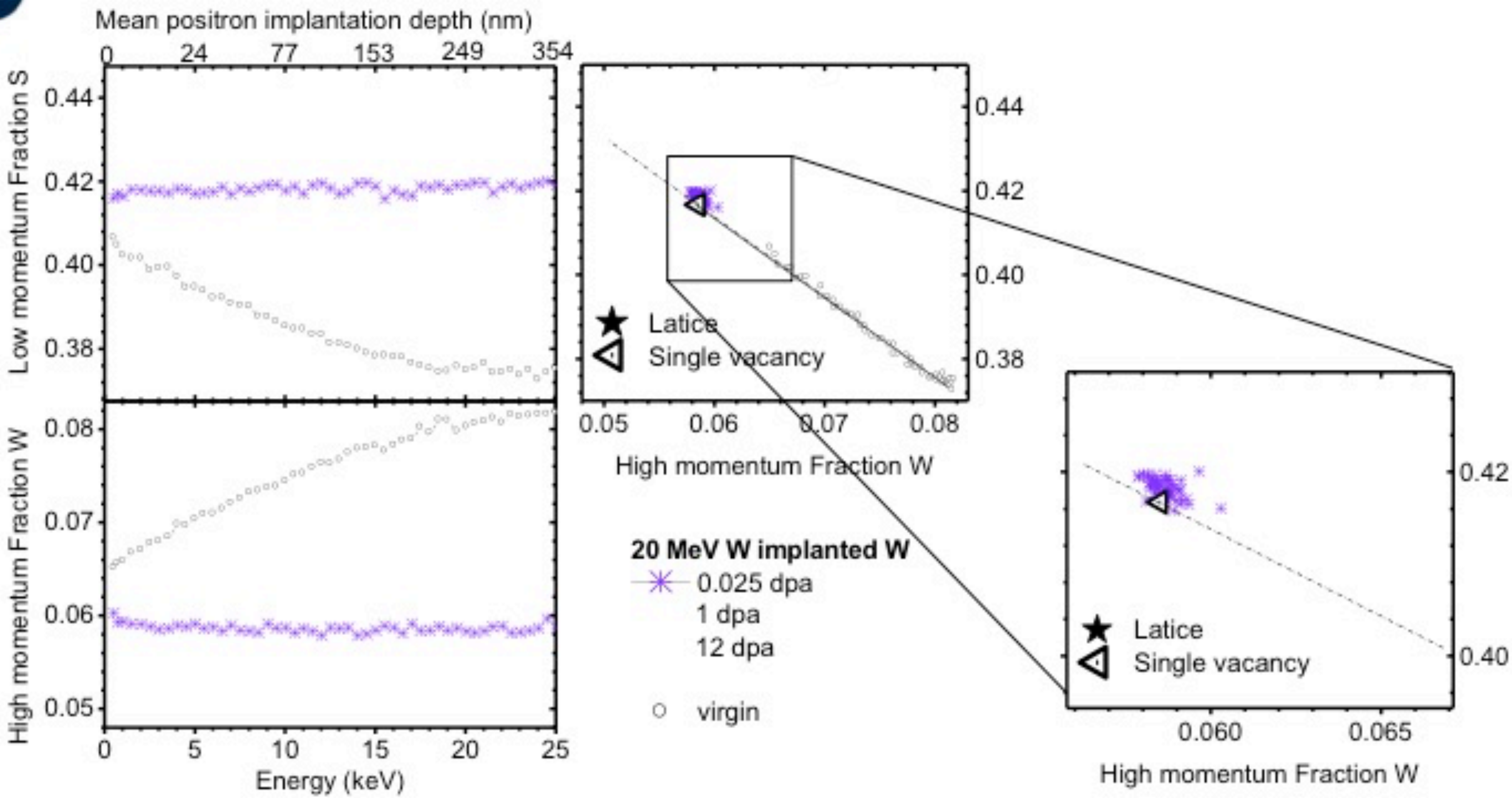
Annealed

Edge

Center

DF-AC :Double forged Annealed extracted from the center

20 MeV W irradiation: effect of low dpa (0,025)



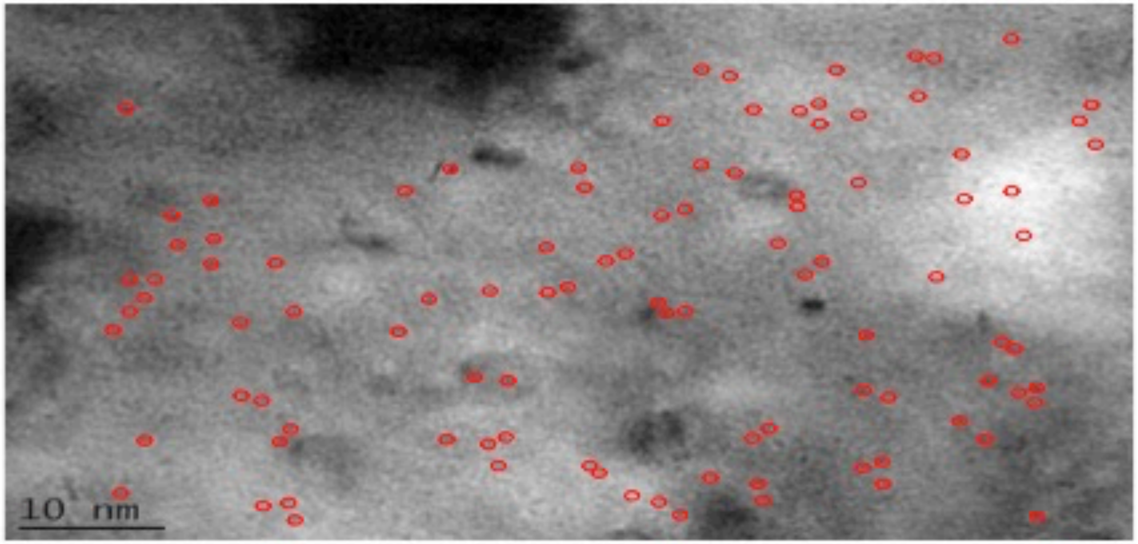
V-clusters (small 3D V-cluster+ V-loops) are created **even at low dpa**

S ≅ concentration & size of clusters

S=f(W) indication of clustering

Ex situ TEM in 1.2 MeV irradiated W (0.017dpa, RT)

N3 0.017 dpa, RT



N = 101

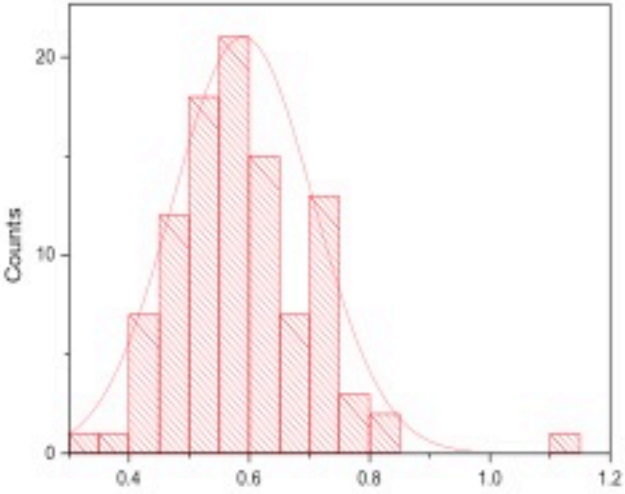


Image Size of cavities (nm)

Mean diameter : 0.59±0.12 nm

4.23×10^{-4} cavities/nm^3

Small vacancy clusters, image Ø ≤ 0.6 nm

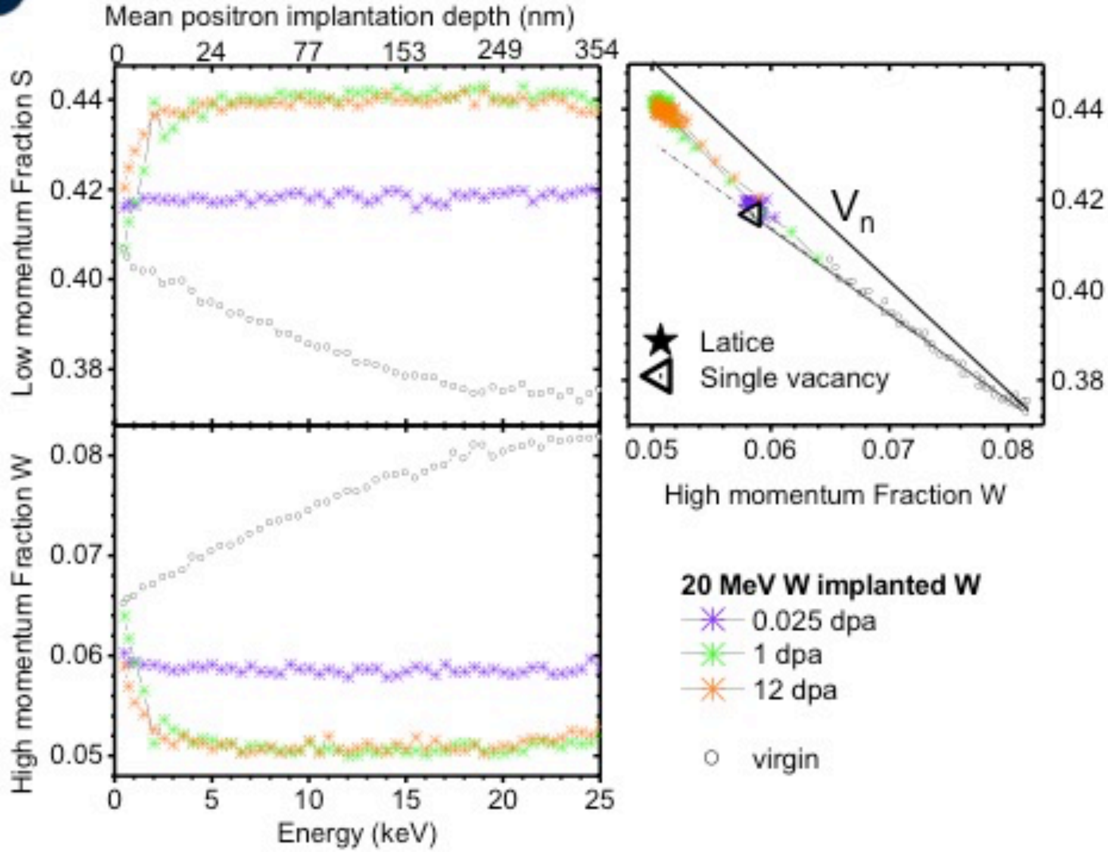
Smaller V-clusters can not be excluded

- ❑ + **Presence of loops**

5. Positron annihilation studies

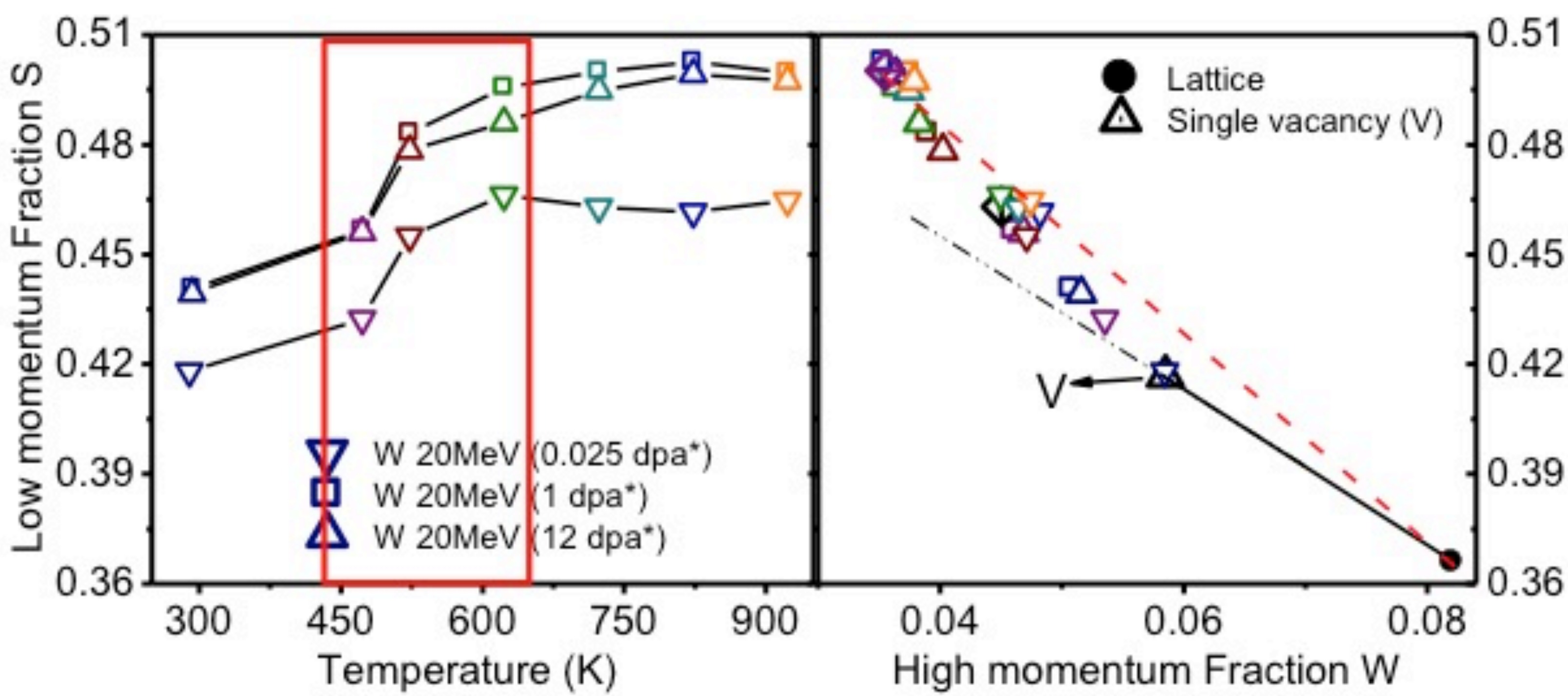
Cemhti
cnrs

20 MeV W irradiation: effect of dpa



V-clusters (small 3D V-cluster+ V-loops) are created **even at low dpa**
Size and/or concentration of vacancy clusters increases with dpa
Saturation from 1 dpa

Annealing in vacuum : effect of dpa



S ≅ concentration & size of clusters

S ↗ with T

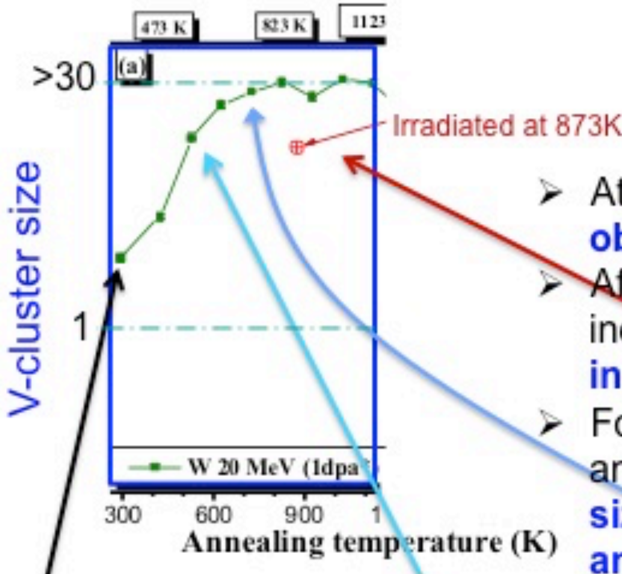
S=f(W) indication of clustering

- ∀ dpa clustering occurs in the same temperature range as for single vacancies
  → mainly due to V migration and agglomeration on small clusters
- Larger vacancy defects when irradiation dose is 1 dpa
- Vacancy cluster distribution (size and concentration) are very close for 1 and 12 dpa

5. Positron annihilation studies

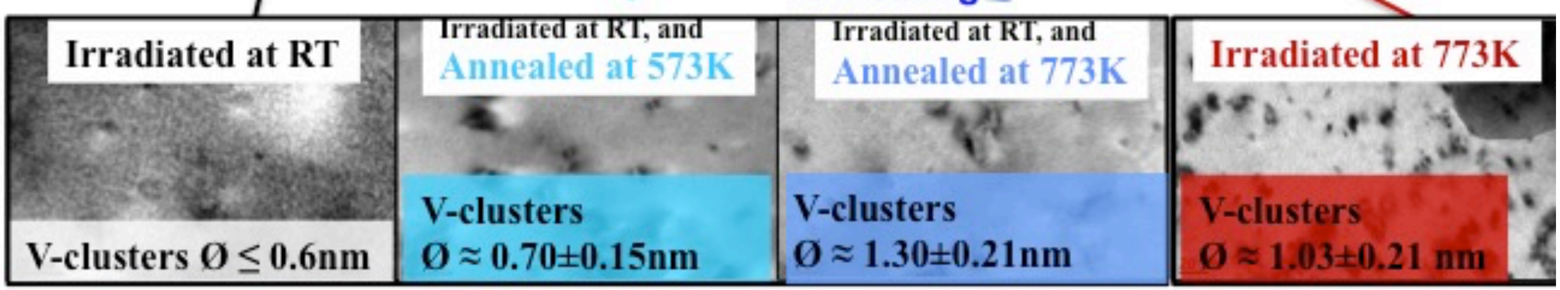
PAS and TEM results

PAS
In bulk, 1dpa



- At RT, S> S(1V) → **Vacancy clusters are observed**
- After annealing at T<873K, S and S/W increase → **The size of V-clusters increases**
- For HT irradiation, S(irr 873K)<S(irr RT and annealed at 873K) → **smaller V-clusters size or concentration than in RT irra. + annealing**

0.017 dpa



Same evolution of vacancy clusters size and concentration between PAS and TEM results

5. Positron annihilation studies

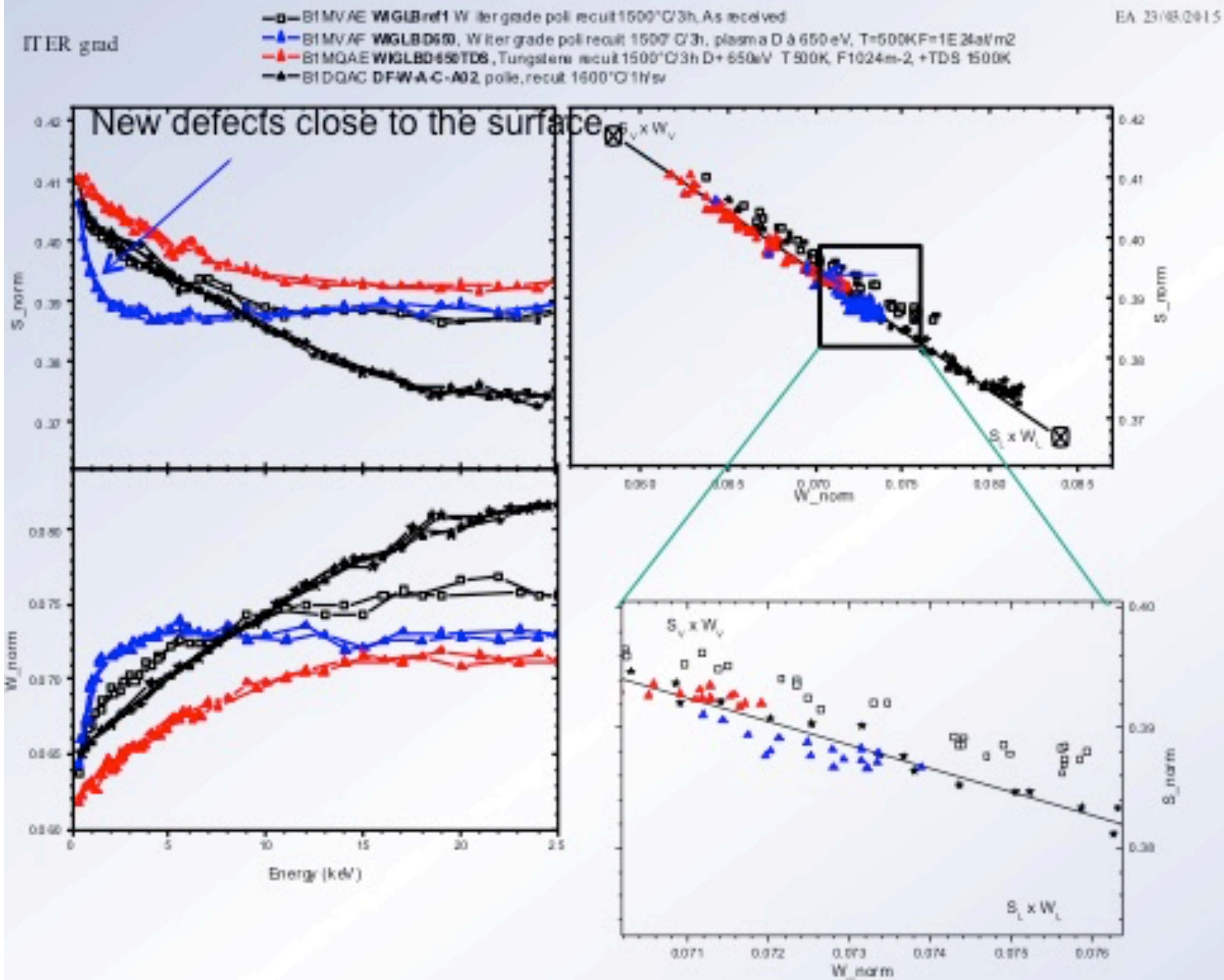
W exposed to D plasma

High flux and low energy

Positron traps

Plasma D implantation ate the MEPHI laboratory (Pr L Begrambekov)

5. Positron annihilation studies



- ✓ Before exposure: defects
- ✓ After D exposure (650K, 10^{24} m^{-2}): New defects are detected with S and W points below the V line* , V or H and vacancy complexes?
- ✓ TDS (1500K) after D exposure: new defects V with S and W points above the V line* clusters? (to be confirmed)

*V line is the SW line characteristic of annihilation in single V

Conclusions

- Light ions : single vacancies
  - ✓ from T in the range 473-623K: migration and clustering
- Heavy ions
  - ✓ at RT or lower temperature
    - ➢ low dpa : V-clusters (3D small voids +V-loops) + single vacancies
    - ➢ high dpa : V-clusters with larger size + single vacancies
      >423K clustering due to migration of single vacancies and with V-loops as precurssors?
  - ✓ Irradiation at 873K
    larger V-clusters are detected compared to low temperature irradiation with size or concentration lower than in irradiation at RT and subsequent annealing
- D Plasma
  nV-mH complexes close to the surface?

Perspectives :
TEM studies (density, effect of dpa …, loops)

High temperature helium irradiation of tungsten: *multi technique defect characterization and additional H trapping*

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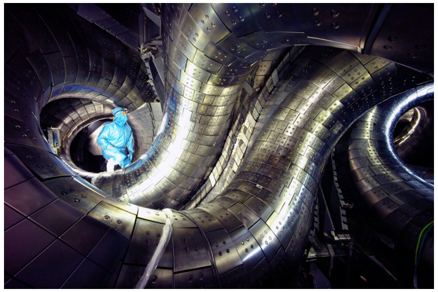
[d]CEA, IRFM, F-13108 Saint-Paul-lez-Durance, France

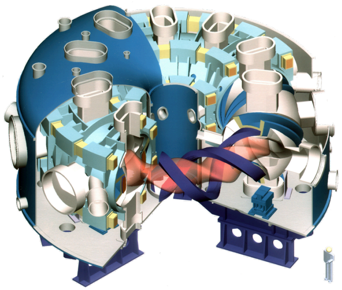
High temperature helium irradiation of tungsten:
multi technique defect characterization and additional H trapping

Experimental set-up

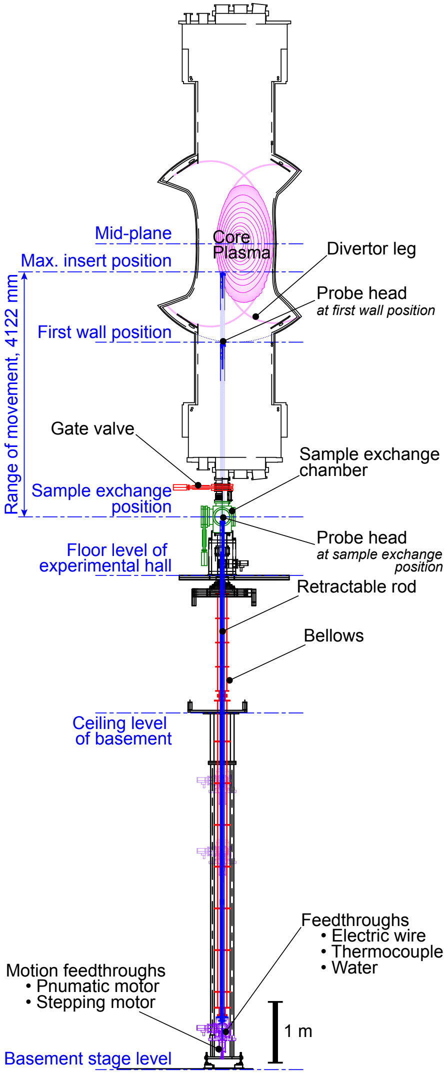
LHD and the retractable material probe

- LHD; Large Helical Device: World largest superconducting stellarator with heliotron configuration
  - A pair of continuous winding helical coils and three pairs of poloidal coils
  - R= 3.9 m, a_{eff}= 0.63 m, V~ 30 m^3, B_T~ 3 T
  - Net-current free plasma with NBI, ECH and ICH





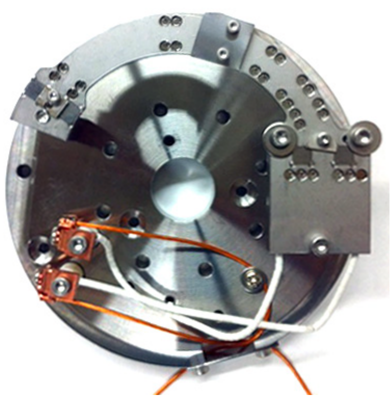
- Two retractable material probes are installed for the PWI studies
  - Plasma exposure experiments under various conditions
    - SOL Plasma, Divertor leg, CX particles
  - Electric feedthrough for controlling temperature (heater and thermocouple measurement)
  - Motion feedthrough for controlling exposure condition (shutter and insertion)

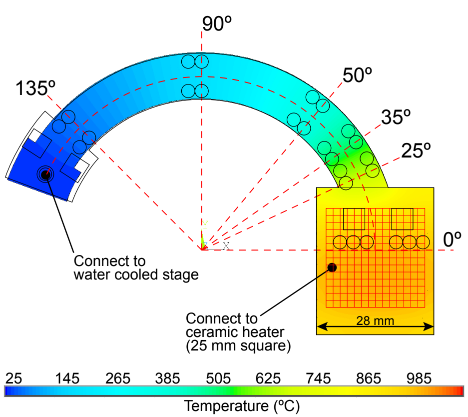


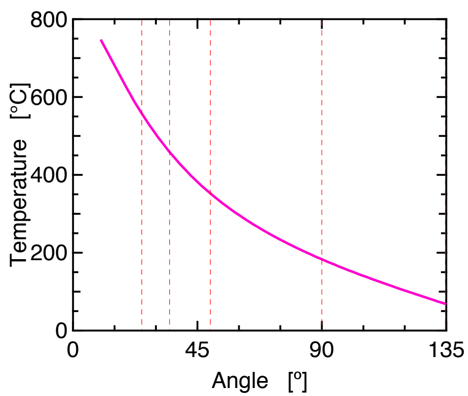
High temperature sample holder

- Controlling sample temperature during LHD plasma exposure
  - Sample holder 1: Multi temperature exposure using temperature gradient between a heater and heat sink, 65 - 600 °C
  - Sample holder 2: Higher temperature exposure with reduced thermal loss structure, 500 - 800 °C

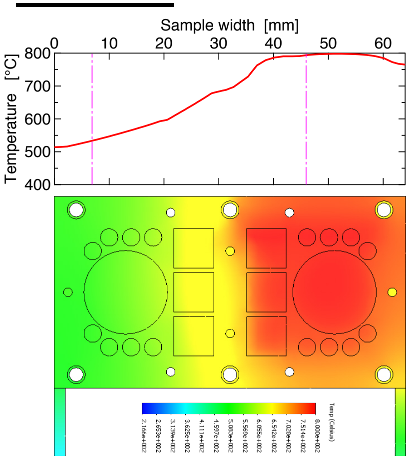
Holder 1







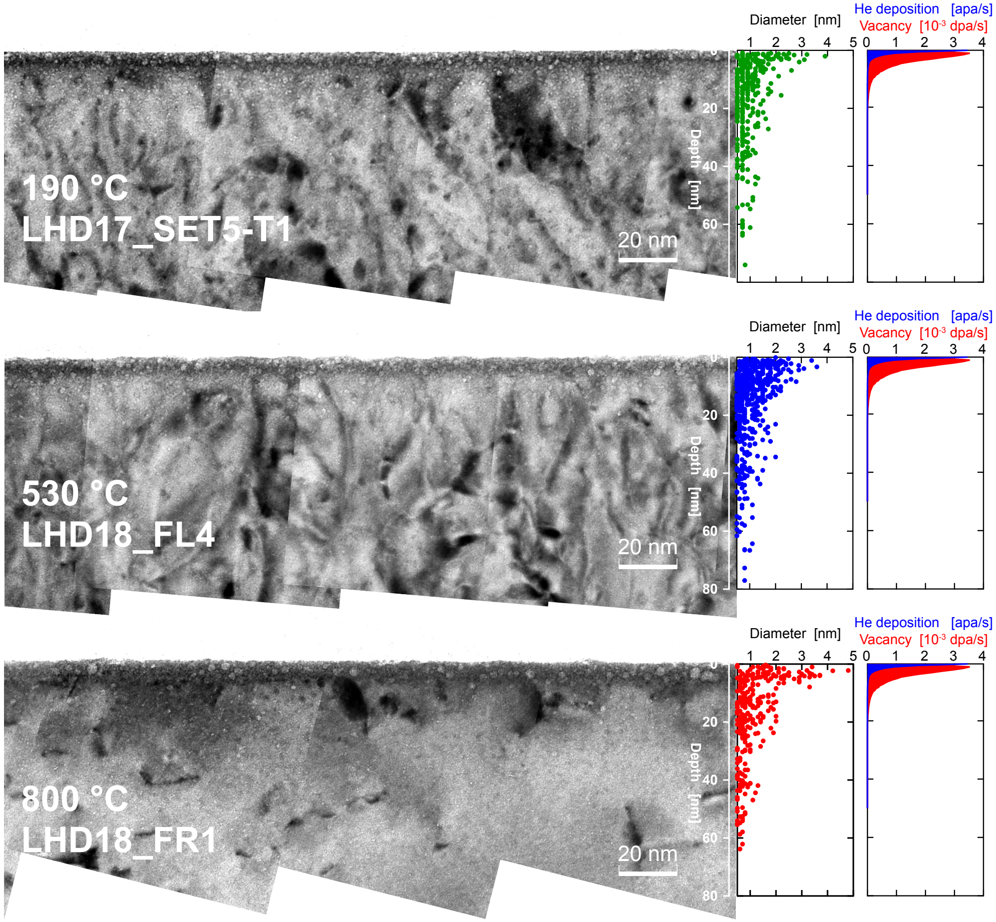
Holder 2



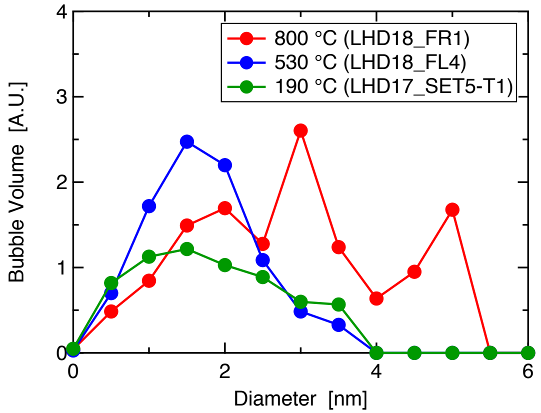
High temperature helium irradiation of tungsten:
multi technique defect characterization and additional H trapping

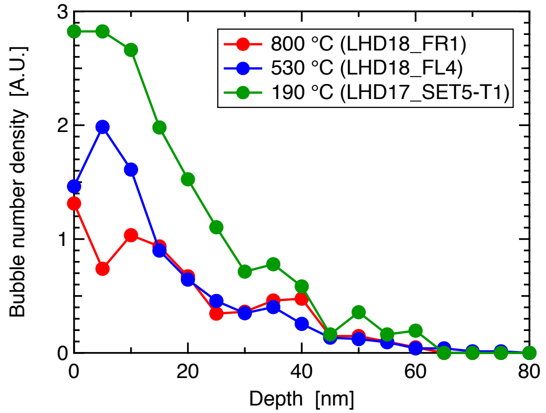
Cross-sectional TEM observation

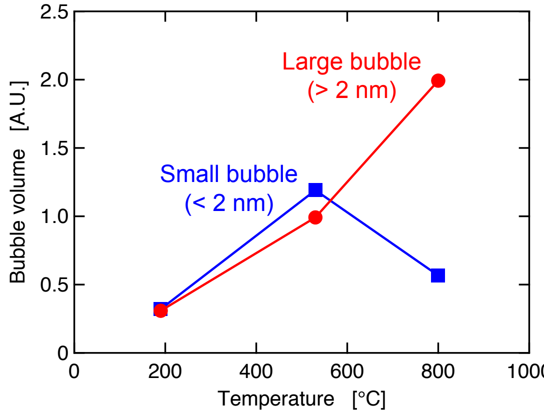
- Bubbles formation
  - Distribute up to 70 nm depth beyond the ranges of helium implantation (<15 nm) at any temperature range
    - Insensitivity to temperature implies that the vacancy play a lesser role in bubble nucleation
    - Bubbles are nucleate by accumulating helium itself
  - Increase size and decrease density as increase irradiation temperature
    - Bubbles grow efficiently capturing vacancy at temperature above 500 °C



High temperature helium irradiation of tungsten:
multi technique defect characterization and additional H trapping



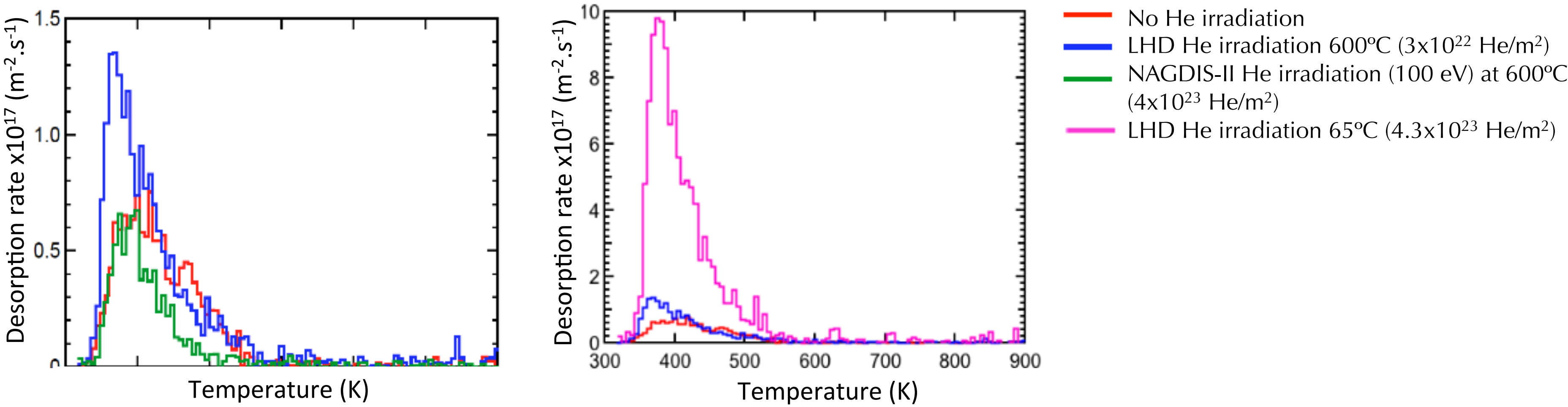




High temperature helium irradiation of tungsten:
multi technique defect characterization and additional H trapping

Additional D irradiation and TDS

❖ Desorption spectra



- Trapped D is desorbed at low temperature so little impact on long term H inventory
- Increase of D retention at low temperatures due to pre existing LHD He irradiation
    - **As the fluence increase, more D is trapped**

❖ Model: Migration of H Isotopes in MaterialS (MHIMS)

- 2 populations: trapped $C_{t,i}$ and mobile C_m
- Simulation with 2 detrapping energies and uniform trap density: Desorption from low detrapping energy trap 1 and diffusion deeper in the bulk