

# Positron studies in self-irradiated tungsten

M-F. Barthe

*CNRS, CEMHTI UPR3079, Univ. Orléans, F-45071 Orléans, France*



## TOKAMAK

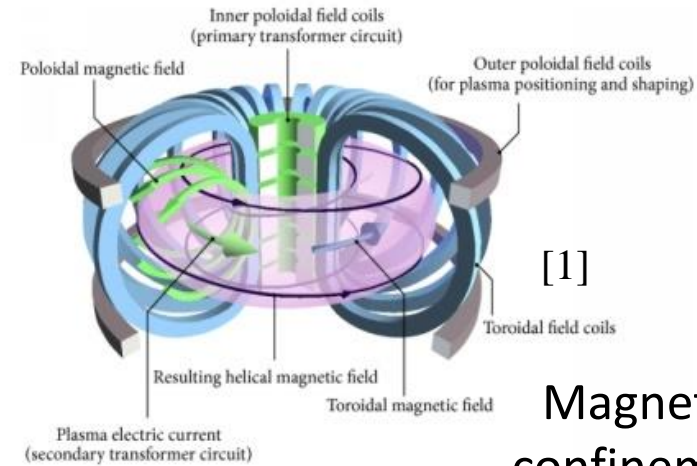
1st concept in the 1950s by [Soviet physicists Igor Tamm](#) and [Andrei Sakharov](#), inspired by a letter by [Oleg Lavrentiev](#)

### Fusion reaction



Hydrogen Plasma

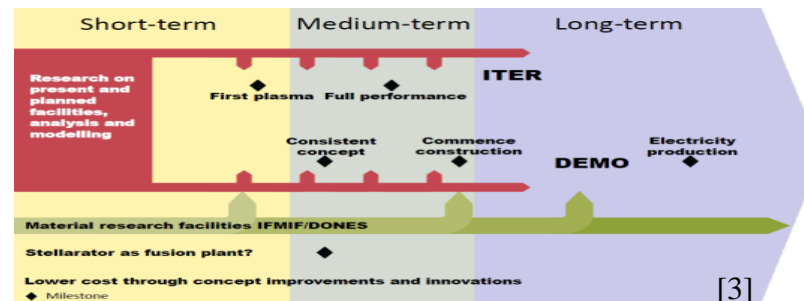
10 keV over  $100 \times 10^6 \text{C}^\circ$  ( $> \text{sun} - 25 \times 10^6 \text{C}^\circ$ )



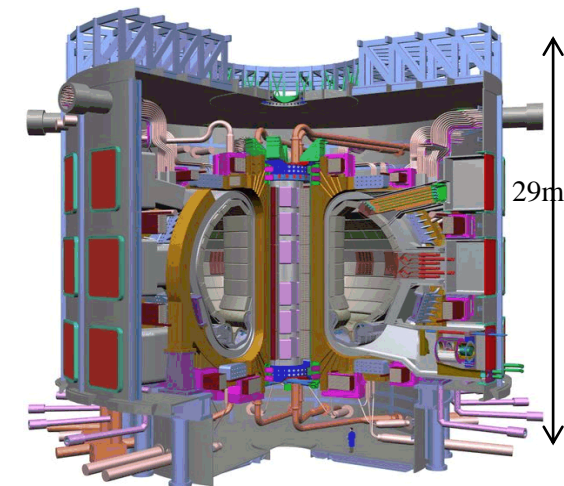
Magnetic confinement



50 operating tokamaks in the world [2]



Fusion Power Plants



ITER Tokamak

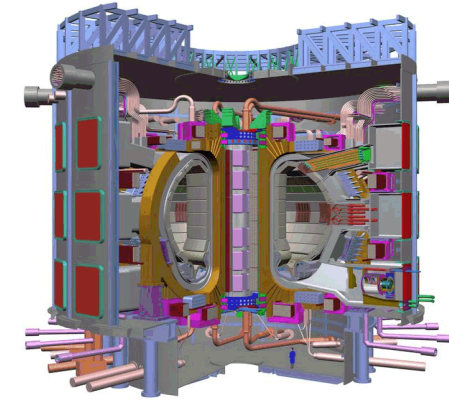
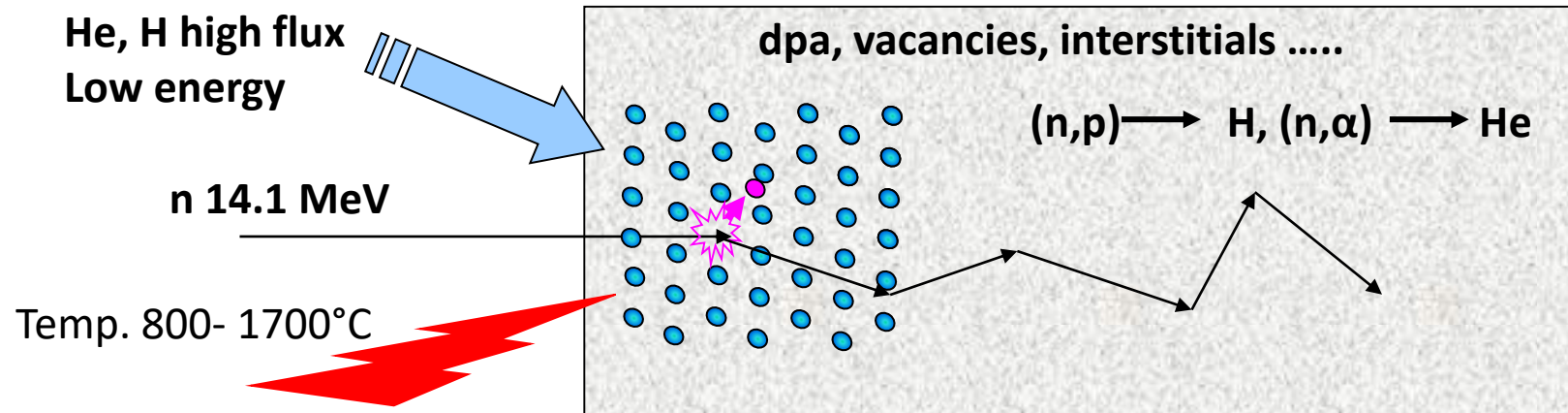
✓ ITER, DEMO : steps in the development of fusion energy production

[1] S. Li, H. Jiang, Z. Ren, C. Xu - S. Li et. al. , Abstract and Applied Analysis doi:10.1155/2014/940965, CC BY 4.0, <https://commons.wikimedia.org/w/index.php?curid=74679708>, [2] <http://www.tokamak.info/>; [3] A.J.H. Donné | SOFT, Sicily, IT | 17 September 2018

✓ ITER, DEMO : very hard conditions for materials

✓ Fusion reaction  $^2\text{H} + ^3\text{H} \rightarrow ^4\text{He}(3,5 \text{ MeV}) + n(14.1 \text{ MeV})$

Plasma and neutrons irradiations



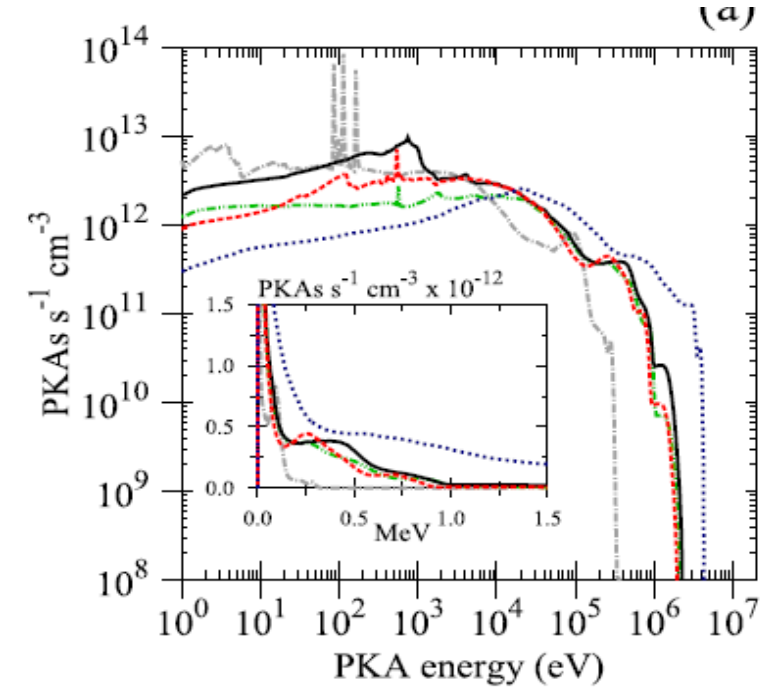
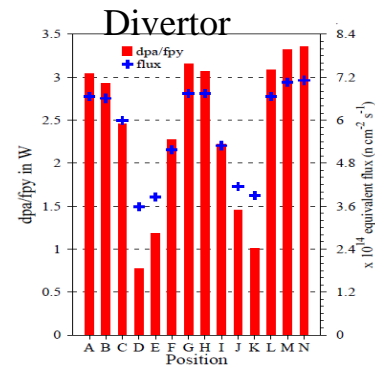
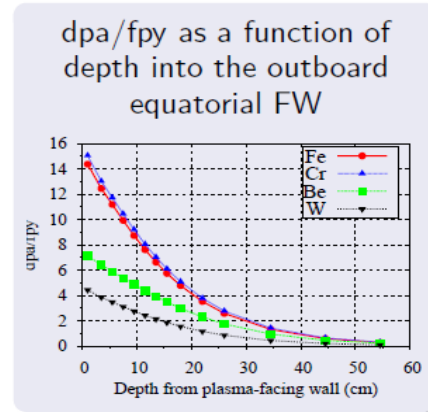
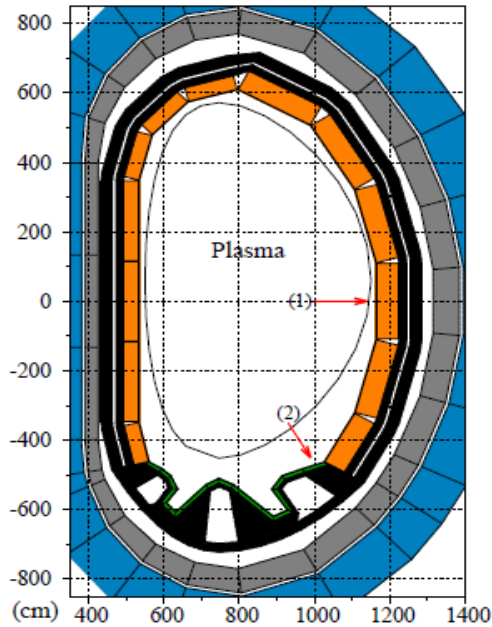
ITER Tokamak

Elastic collision  
 Pka : Primary Knocked on atom, recoil atom  
 dpa : displacements per atom

Big challenges : for thermal, electrical and mechanical properties

**Tungsten divertor (ITER) , first walls? (DEMO)**

## Calculations in DEMO



-- W = predominant recoil

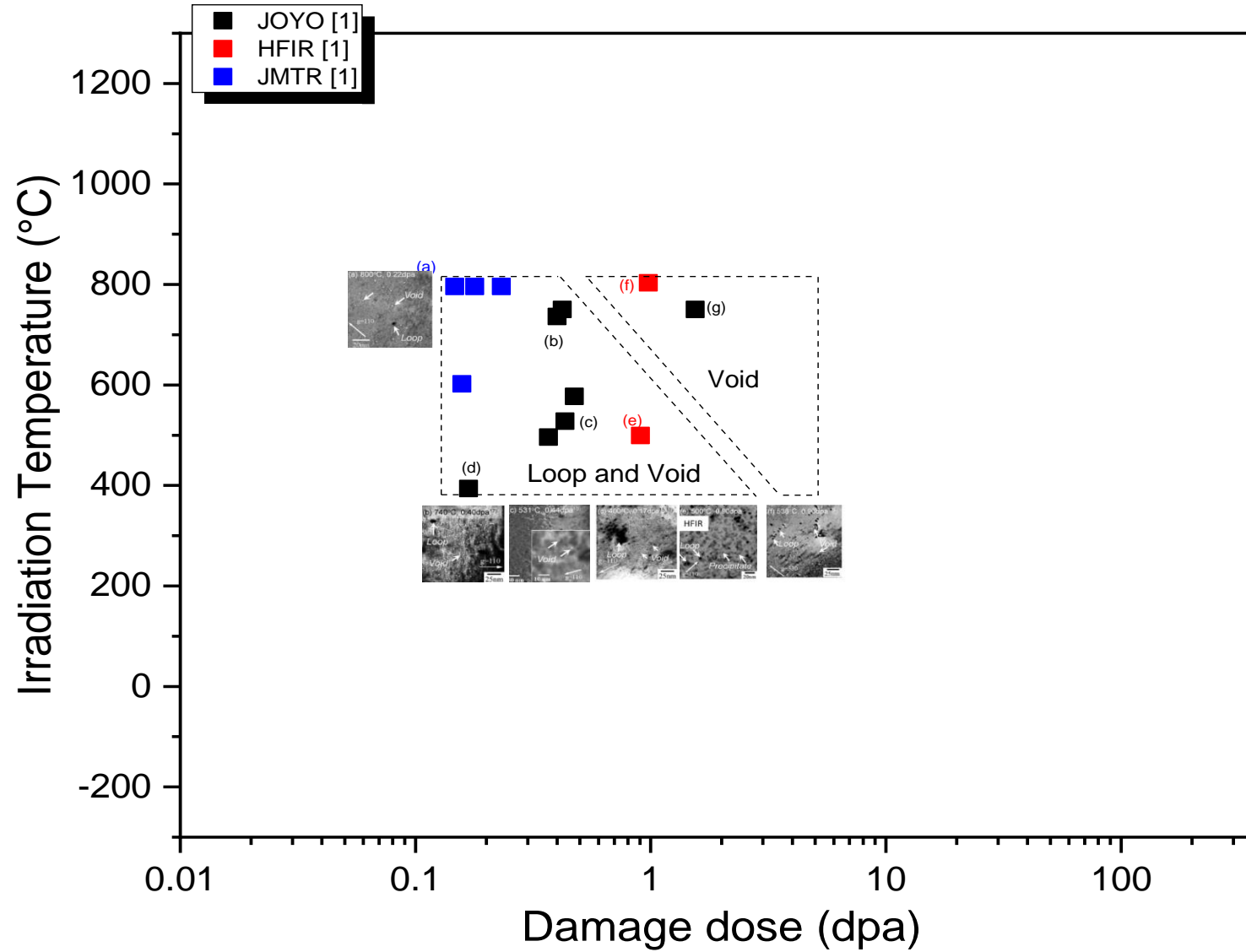
## W for FW or divertor

Damage dose max in 3 years = 12dpa  
 [He]max in 3 years = 20-30 appm  
 He/damage = 1.6-2.5 appm/dpa

- Large energy distribution (up to 1 MeV)  
 mean value at 150 keV

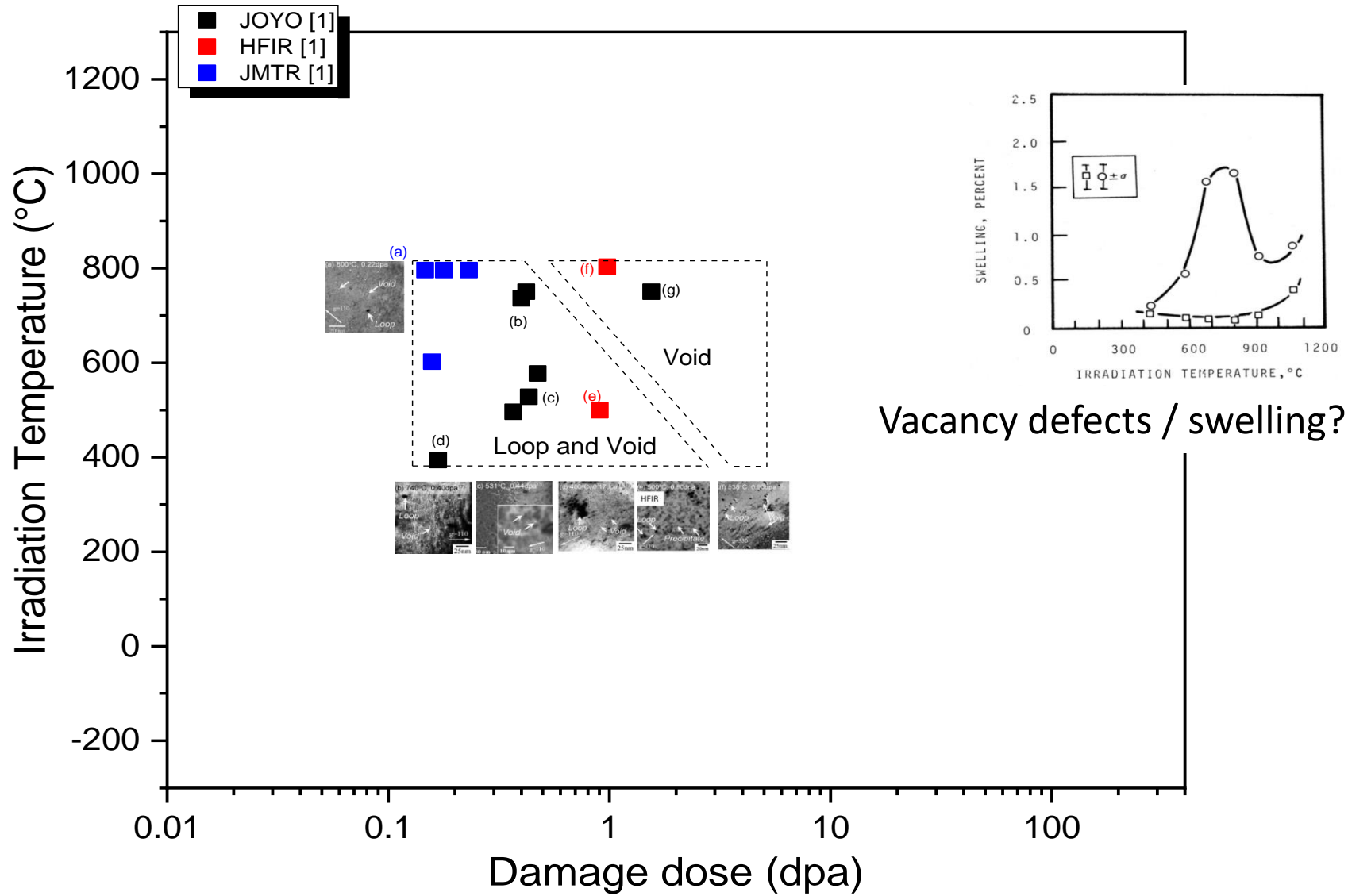


[1] M.R. Gilbert et al. / JNM 442 (2013) S755–S760, M.R. Gilbert et al. / JNM 467 (2015) 121



[1] A. Hasegawa et al. / Fusion Engineering and Design 89 (2014) 1568–157, [2] A. Barabash et al. / Journal of Nuclear Materials 283–287, (2000) 138-146. [3] M. R. Gilbert et al / (2013).Journal of Nuclear Materials 442, (2013) S755-S760

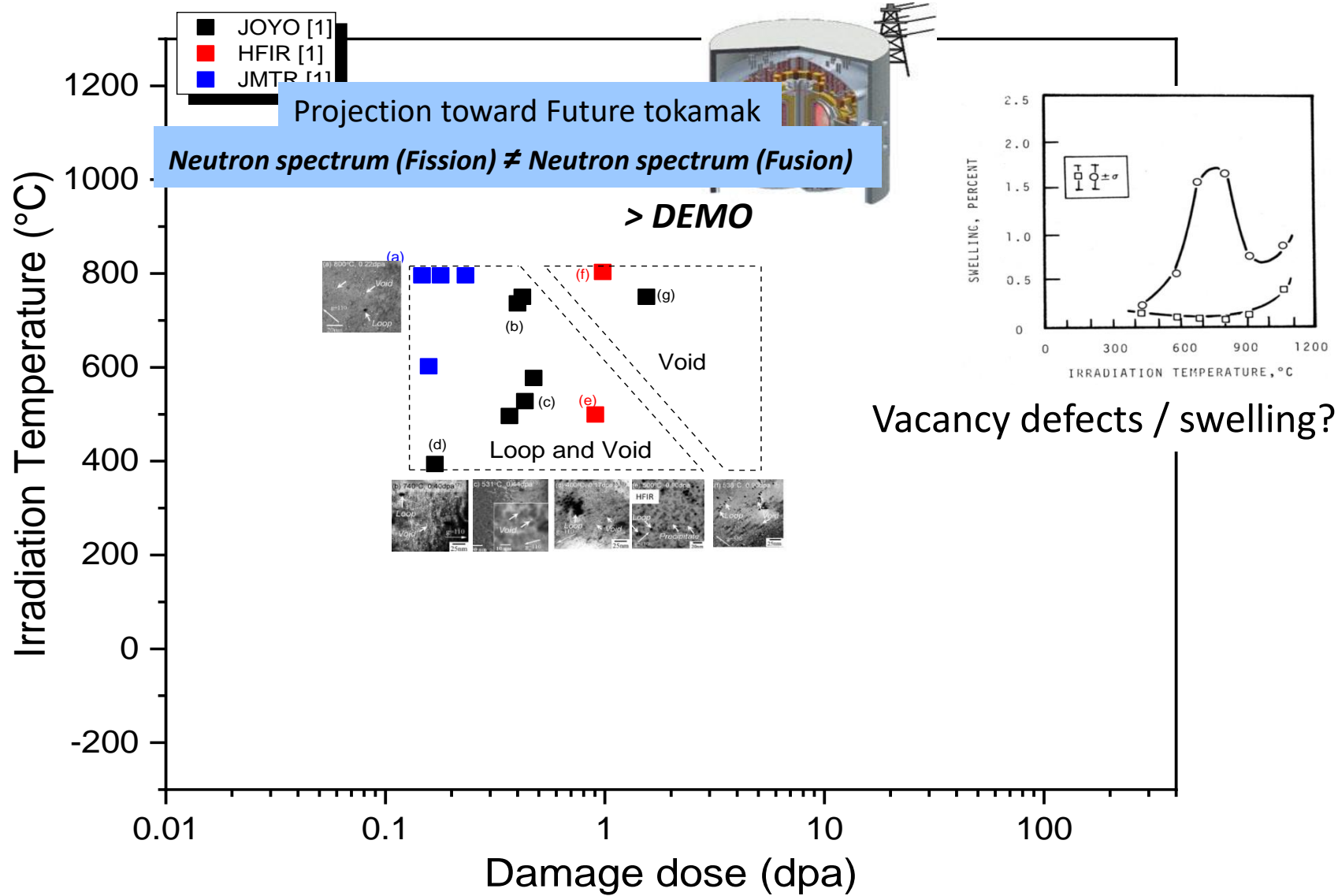




Vacancy defects / swelling?

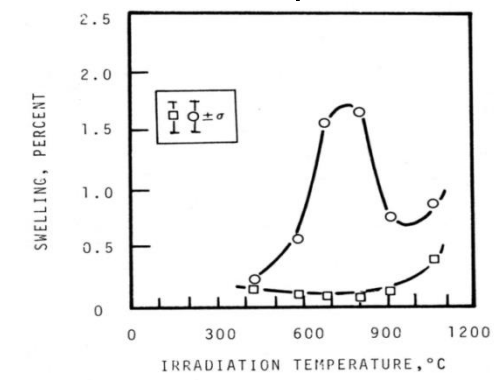
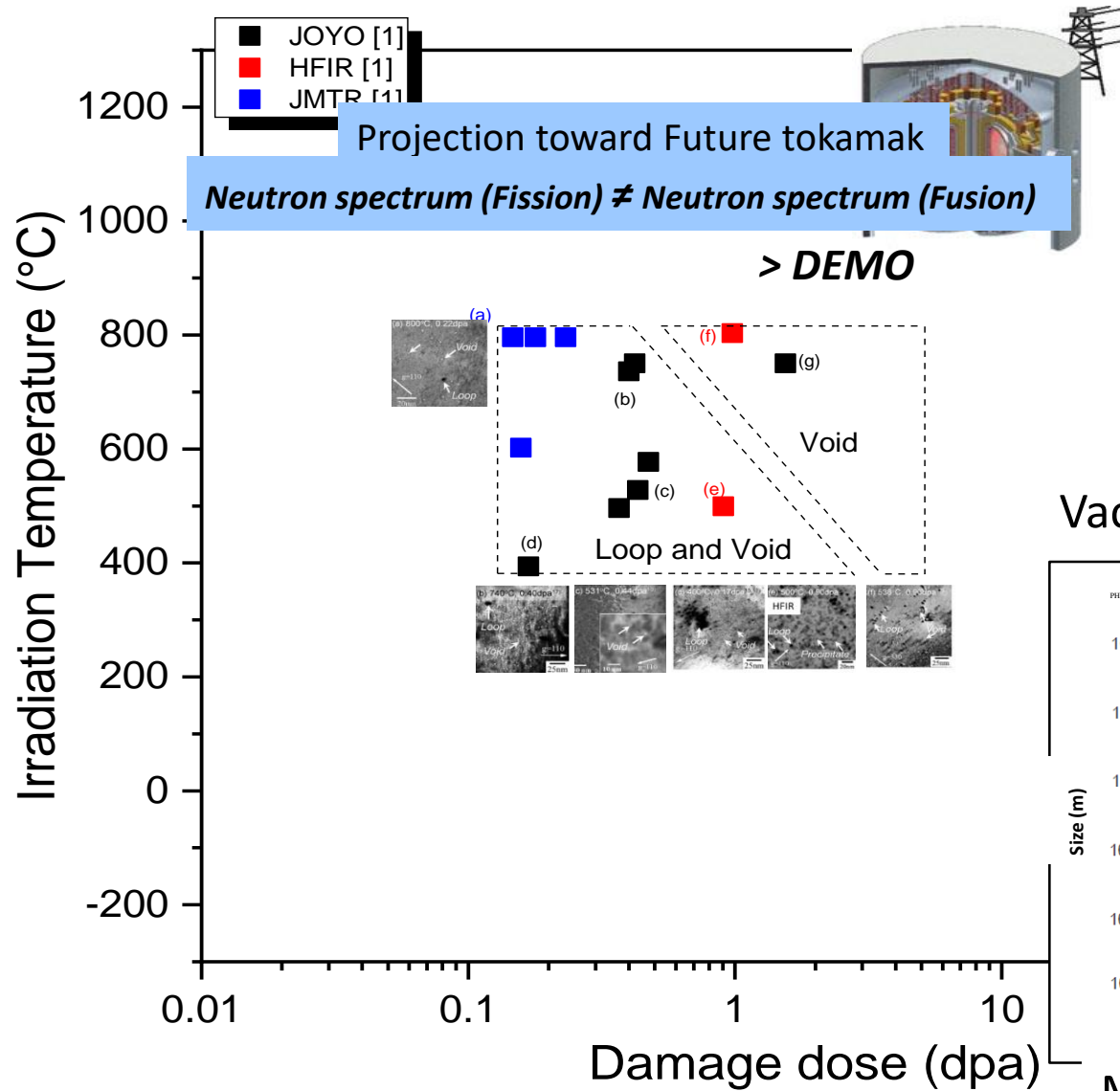
[1] A. Hasegawa et al. / Fusion Engineering and Design 89 (2014) 1568–157, [2] A. Barabash et al. / Journal of Nuclear Materials 283–287, (2000) 138-146. [3] M. R. Gilbert et al / (2013).Journal of Nuclear Materials 442, (2013) S755-S760



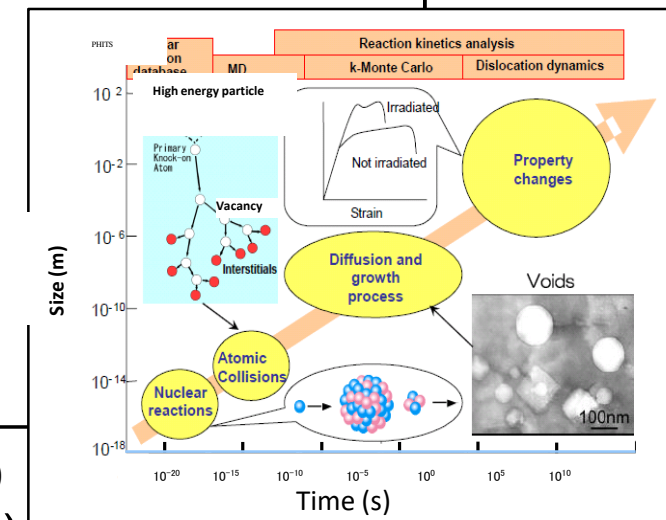


Vacancy defects / swelling?

[1] A. Hasegawa et al. / Fusion Engineering and Design 89 (2014) 1568–157, [2] A. Barabash et al. / Journal of Nuclear Materials 283–287, (2000) 138-146. [3] M. R. Gilbert et al / (2013).Journal of Nuclear Materials 442, (2013) S755-S760



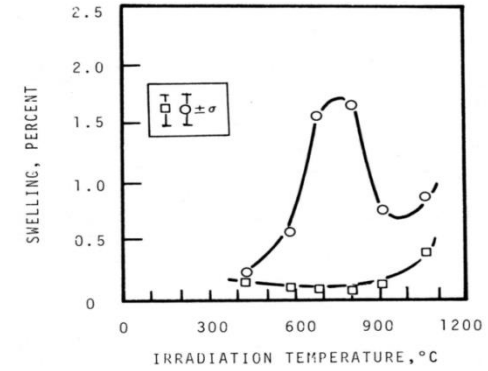
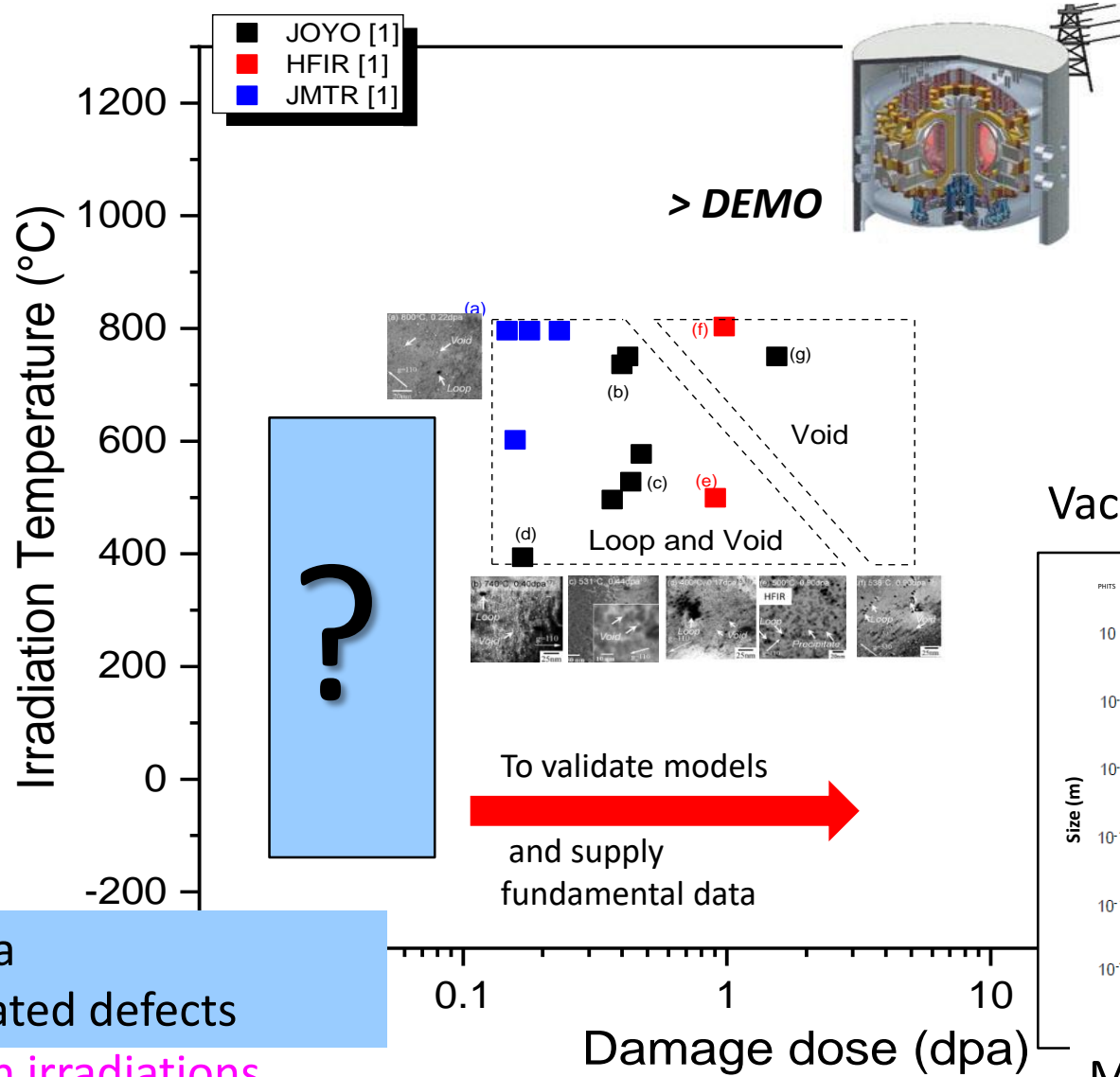
Vacancy defects / swelling?



Multiscale Modelling / experiments

[1] A. Hasegawa et al. / Fusion Engineering and Design 89 (2014) 1568–157, [2] A. Barabash et al. / Journal of Nuclear Materials 283–287, (2000) 138-146. [3] M. R. Gilbert et al / (2013).Journal of Nuclear Materials 442, (2013) S755-S760



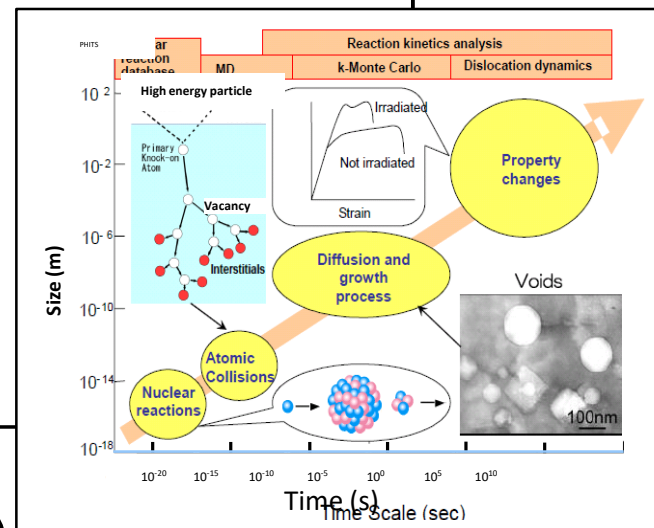


?

To validate models  
and supply  
fundamental data

Quantitative data  
On all the generated defects  
Using ion irradiations

Vacancy defects / swelling?



Multiscale Modelling / experiments

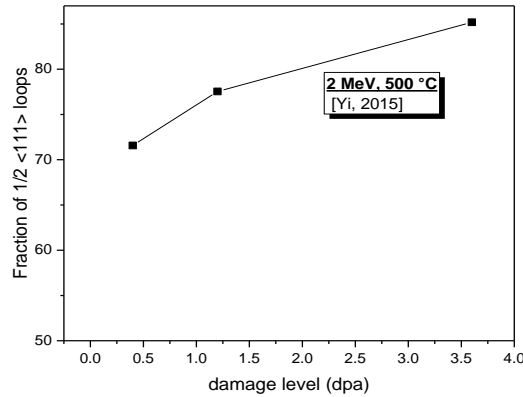
[1] A. Hasegawa et al. / Fusion Engineering and Design 89 (2014) 1568–157, [2] A. Barabash et al. / Journal of Nuclear Materials 283–287, (2000) 138-146. [3] M. R. Gilbert et al / (2013).Journal of Nuclear Materials 442, (2013) S755-S760

## Dislocation loops: $\frac{1}{2}\langle 111 \rangle$ , $\langle 100 \rangle$

2 MeV W, 0.4 dpa, 500°C [1] :

Loops  $\frac{1}{2}\langle 111 \rangle$  :  $3.3 \times 10^{22} \text{ m}^{-3}$

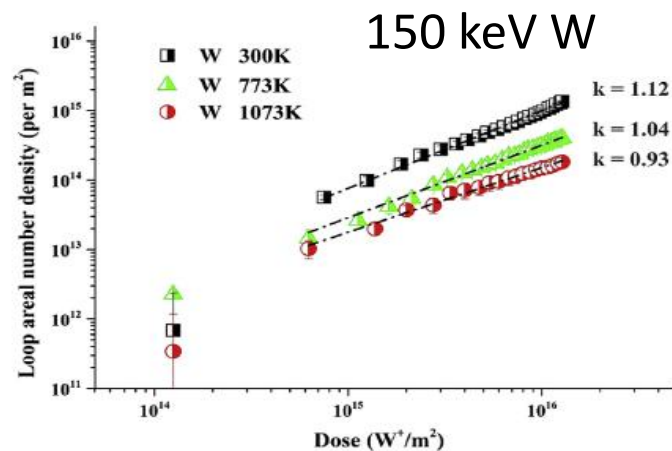
mean size 3-4 nm [1]



✓ For irradiation at 500°C, preferential formation of  $\langle 100 \rangle$  loops at low dpa

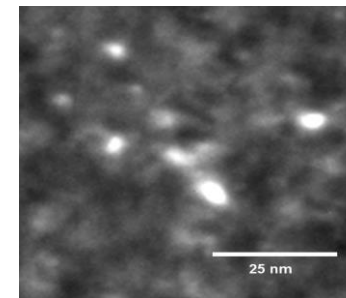
[1] X Yi et al Acta Materialia 92 (2015) 163–177

✓ Density and size depend on temperature

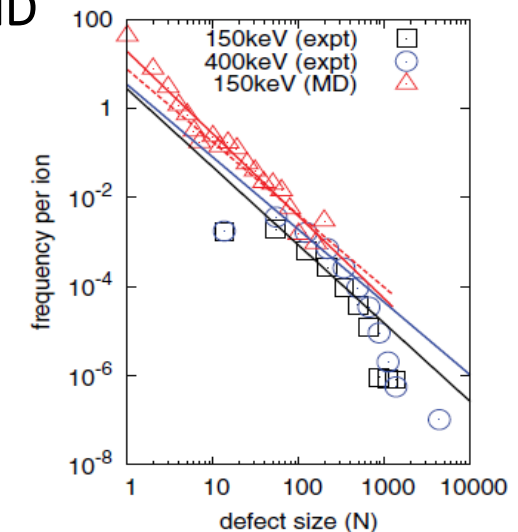


X. Yi et al. / Acta Materialia 112 (2016) 105-120

✓ Size distribution is a power law as calculated in MD



150 keV W, LN2



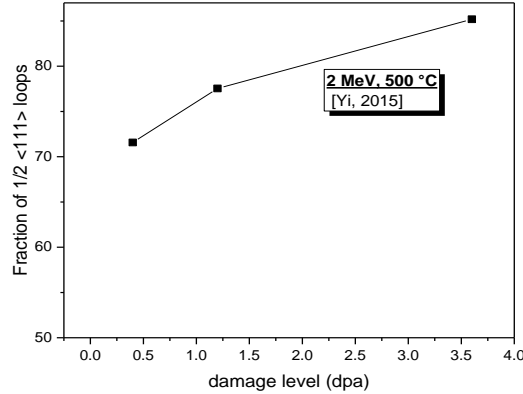
Yi, X., et al., EPL 110 (2015) 36001

Dislocation loops:  $\frac{1}{2}\langle 111 \rangle$  ,  $\langle 100 \rangle$

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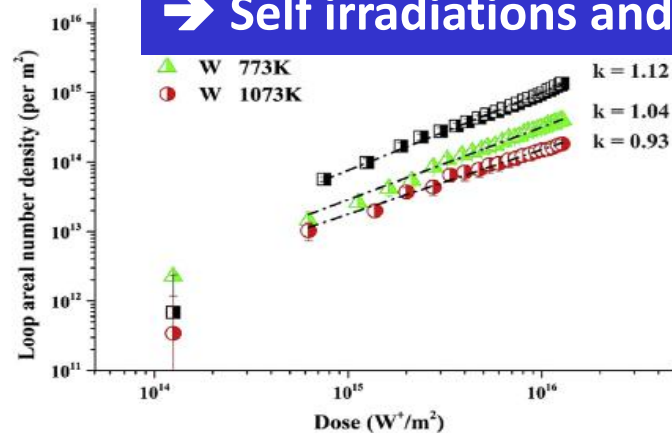


✓ For irradiation at 500°C, preferential formation of  $\langle 100 \rangle$  loops at low dpa

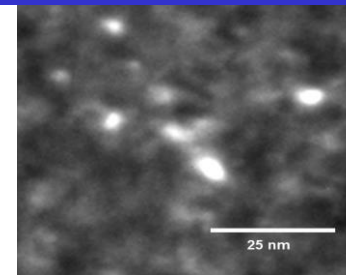
[1] X Yi et al Acta Materialia 92 (2015) 163–177

✓ Den **Less data on vacancy defects and cavities**

➔ **Self irradiations and characterization of the microstructure using TEM and PAS**

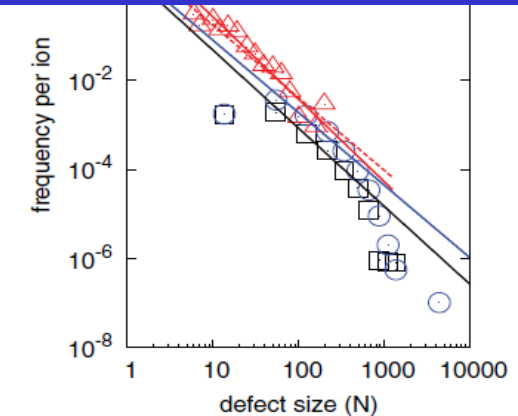


X. Yi et al. / Acta Materialia 112 (2016) 105-120



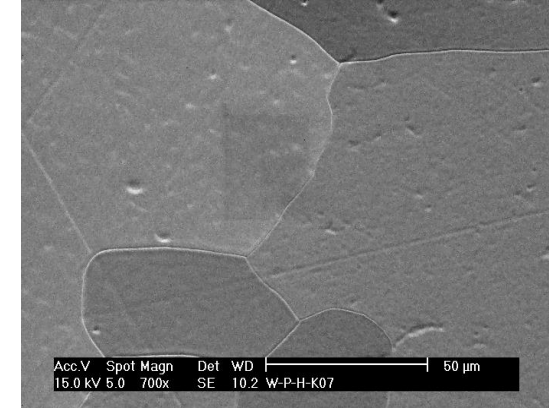
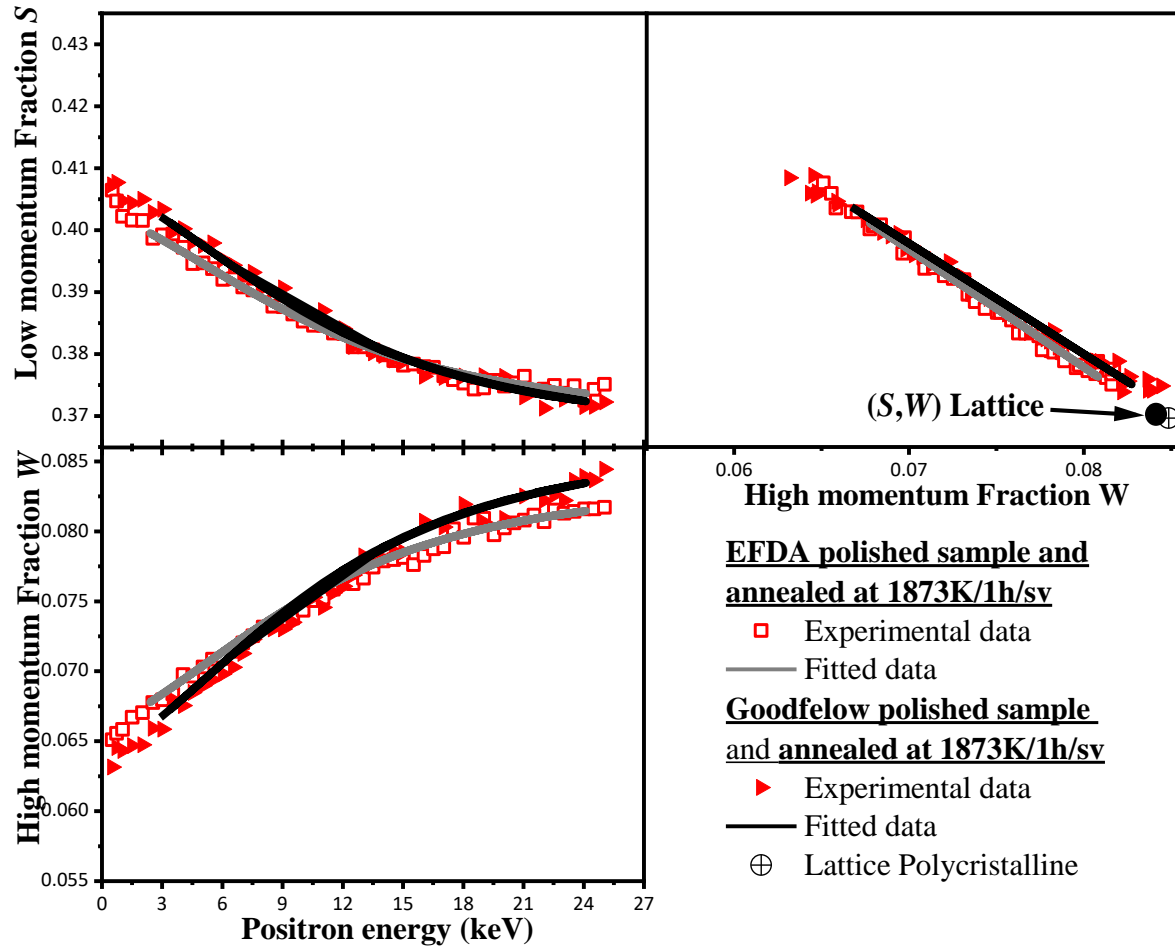
150 keV W, LN2

Yi, X., et al., EPL 110 (2015) 36001



## ✓ Objective : Vacancy defects?

- Quantitative data with the best precision to be shared with modeling
  
- Experimental investigations of damage induced by ion irradiations
  - Effect of damage dose
  - Effect of temperature
  - Role of impurities
  
- Development of a characterization methodology
  - combine characterization techniques PAS and TEM



## Virgin sample

- $L_{eff}^+ = \sim 100\text{nm}$
- Lifetime : 105-108 ps = lattice theoretical value 101 [1] - 108 [2] ps

## Detection limit DB-PAS

- $C_V < 10^5 \mu\text{m}^{-3}$

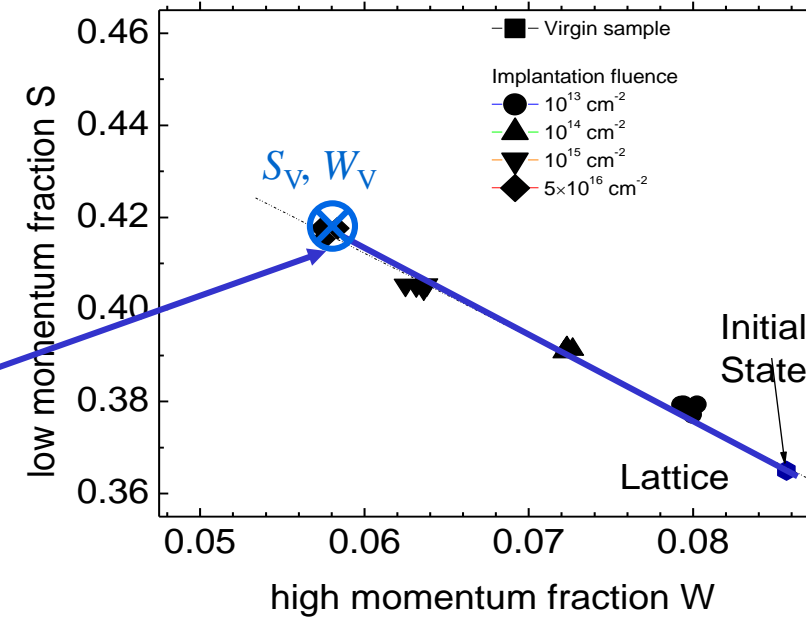
$$S_L = 0.367(4), W_L = 0.084(5)$$

[1] A. Yabuuchi, M. Tanaka and A. Kinomura / Journal of Nuclear Materials 542 (2020) 152473. [2] T. Troev, E. Popov, et al, NIMB 267, (2009), 335

Virgin sample : annealed  $1500^\circ\text{C}/1\text{h}/\text{ArH}_2$   
 $^3\text{He}$  800 keV irradiation (range  $1.6\ \mu\text{m}$ )  
 Fluence from  $10^{14}$  ( $10^{-3}$  dpa) to  $5 \times 10^{16}\ \text{cm}^{-2}$  (0.1 dpa)

➔ One single type of defect

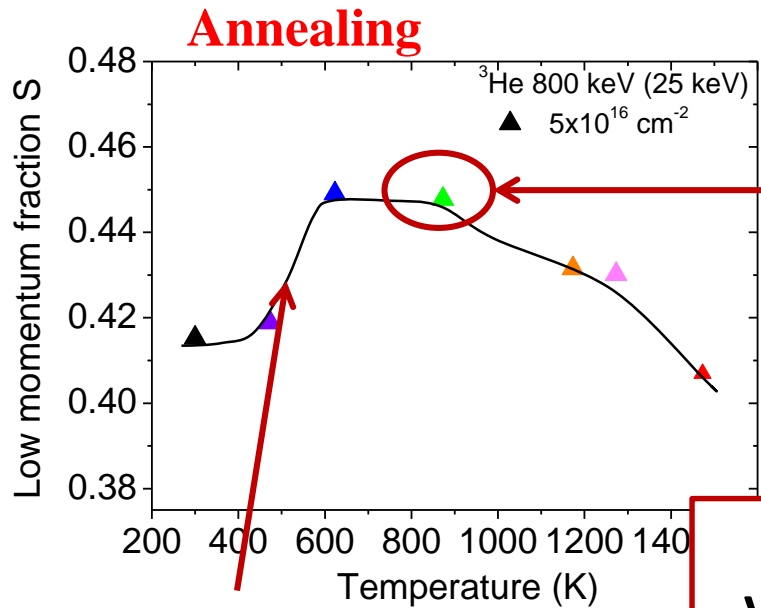
Positron Lifetime                      In agreement with calculations  
 $\tau = 200 \pm 0.4\ \text{ps}$   $I = 98 \pm 0.2\%$  , [3]                       $\tau_{\text{VCalc}} = 200\ \text{ps}$  [2]  
 PLEPS    **Single vacancy (V)**



[1] A Debelle , MF Barthe et al, JNM.376 (2008) 216; [2] T. Troev, E. Popov, et al, NIMB 267, (2009), 335; [3] P.E. Lhuillier, MF Barthe et al., PSS ,C6, (2009), 2329, [4] C.Becquart,et al JNM 403, Issues 1-3, 2010, 75-88

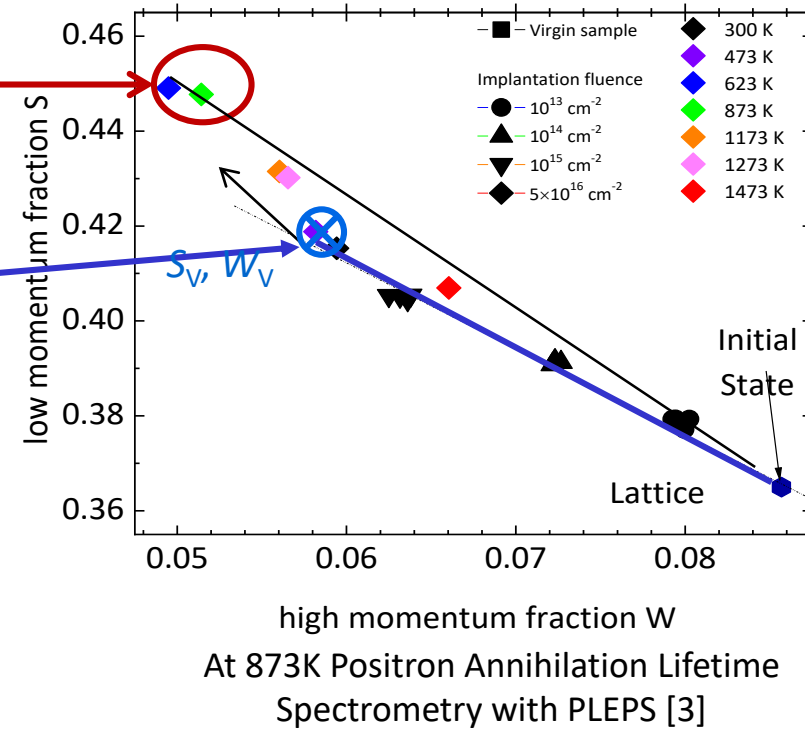
Virgin sample : annealed 1500°C/1h/ArH<sub>2</sub>  
<sup>3</sup>He 800 keV irradiation  
 Fluence 5x10<sup>16</sup> cm<sup>-2</sup> and annealing

→ One single type of defect  
**Single vacancy (V)**



V migration, 473-623K  
 E<sub>m</sub>(V) = 1.66 eV [4]

**V-clusters**  
**V<sub>N</sub> with N>30 [2]**



Mean lifetime	1 <sup>st</sup> component		2 <sup>nd</sup> component	
$\tau_m$ (ps)	$\tau_1$ (ps)	$I_1$ (%)	$\tau_2$ (ps)	$I_2$ (%)
331	176	54	458	45

[1] A Debelle, MF Barthe et al, JNM.376 (2008) 216; [2] T. Troev, E. Popov, et al, NIMB 267, (2009), 335; [3] P.E. Lhuillier, MF Barthe et al., PSS,C6, (2009), 2329, [4] C.Becquart, et al JNM 403, Issues 1-3, 2010, 75-88

## New studies

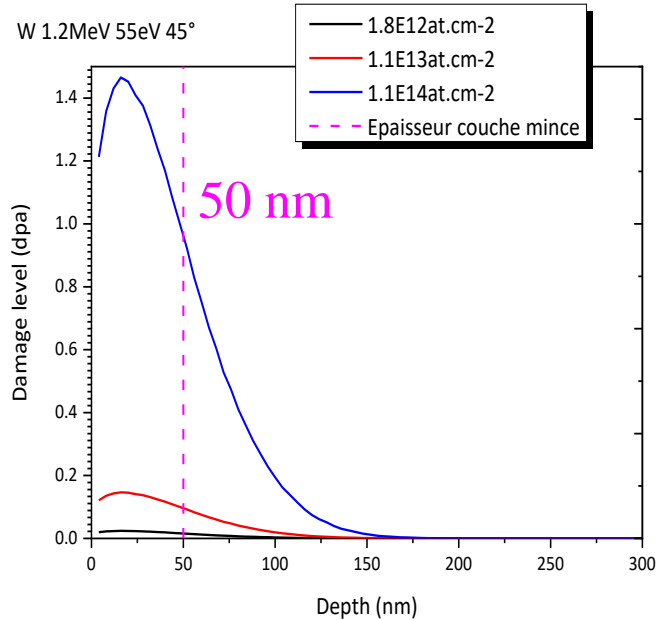
# Self irradiation damage



Before irradiation : Annealing at 1600°C/1H/Vacuum

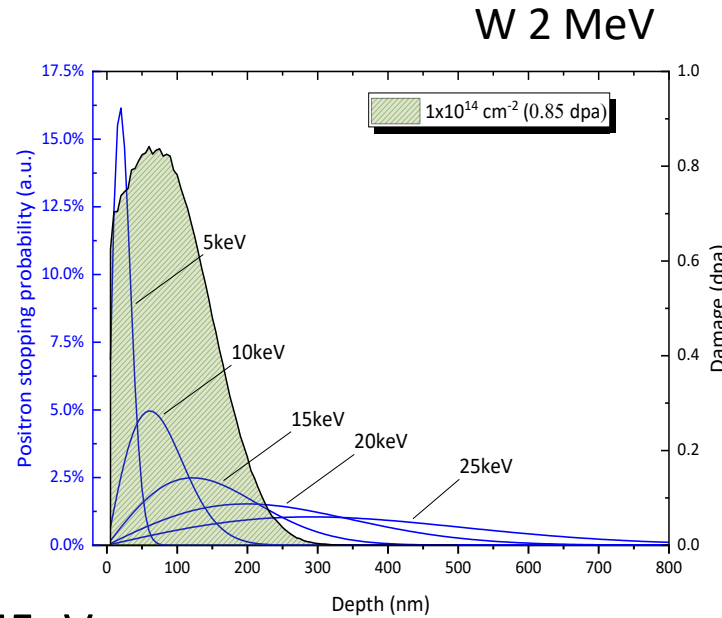
## Irradiation with W ions, SRIM calculations (KP)

- **1.2 MeV** in thin and thick samples:



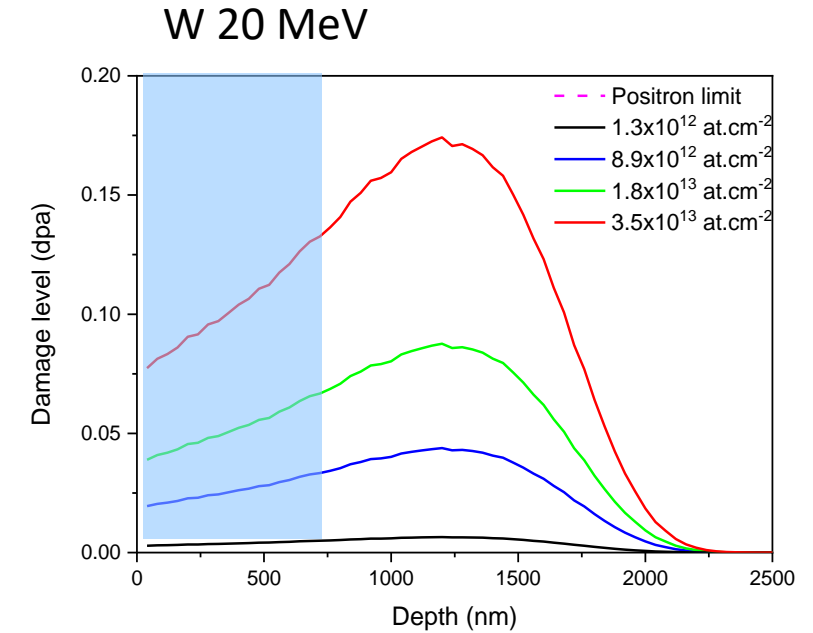
Dpa\* : mean dpa value in the thin foil

- **2 or 20 MeV** in thick samples:



$E_d = 55\text{eV}$

Dpa\* : at the maximum of the peak

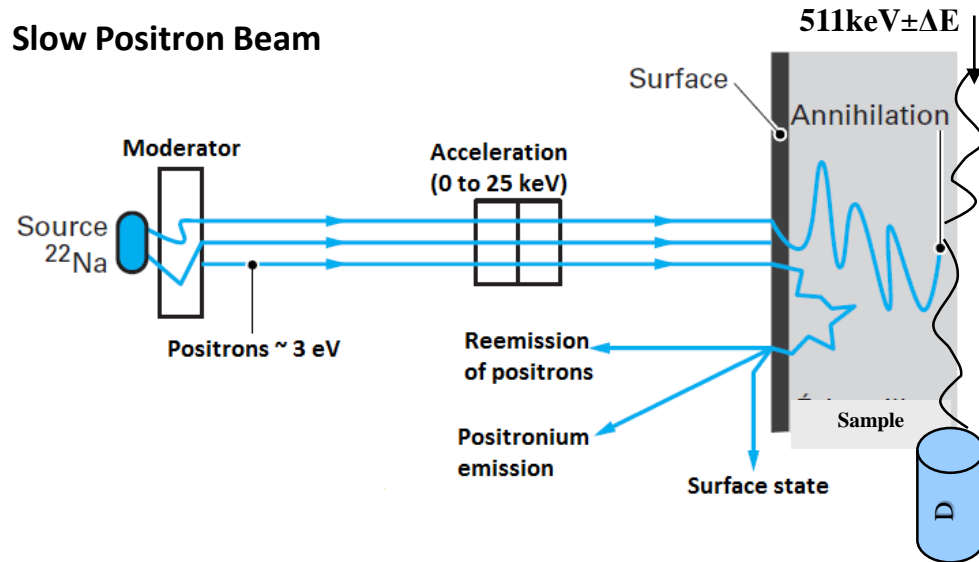


Dpa\* : mean dpa value in **700nm** surface region,

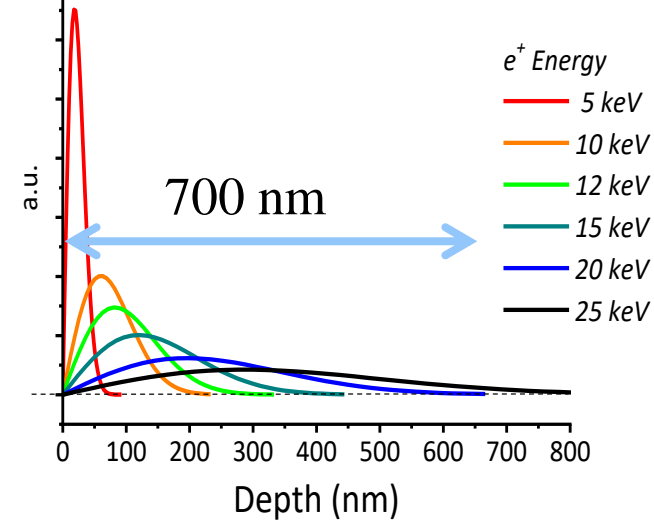
*in situ and post-mortem TEM observations  
+ PAS (only post-mortem)*

*post-mortem characterizations : PAS and TEM*

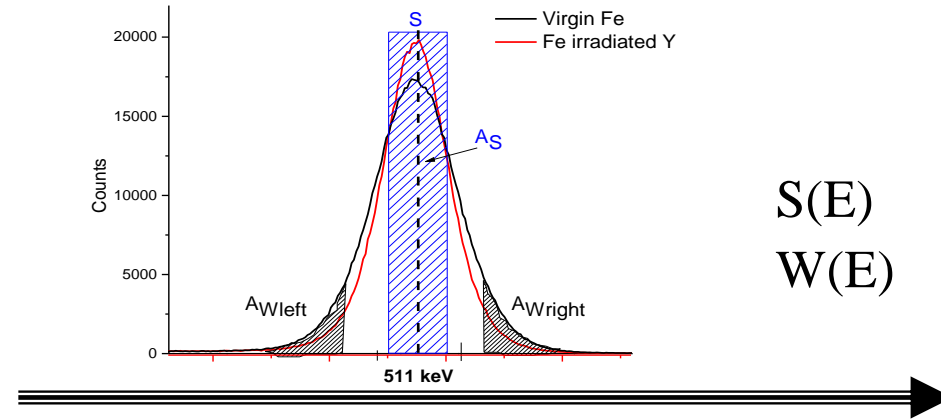
## Slow Positron Beam



## Positron implantation profile in W



DB with continuous Slow positron beam in CEMHTI (Orléans)

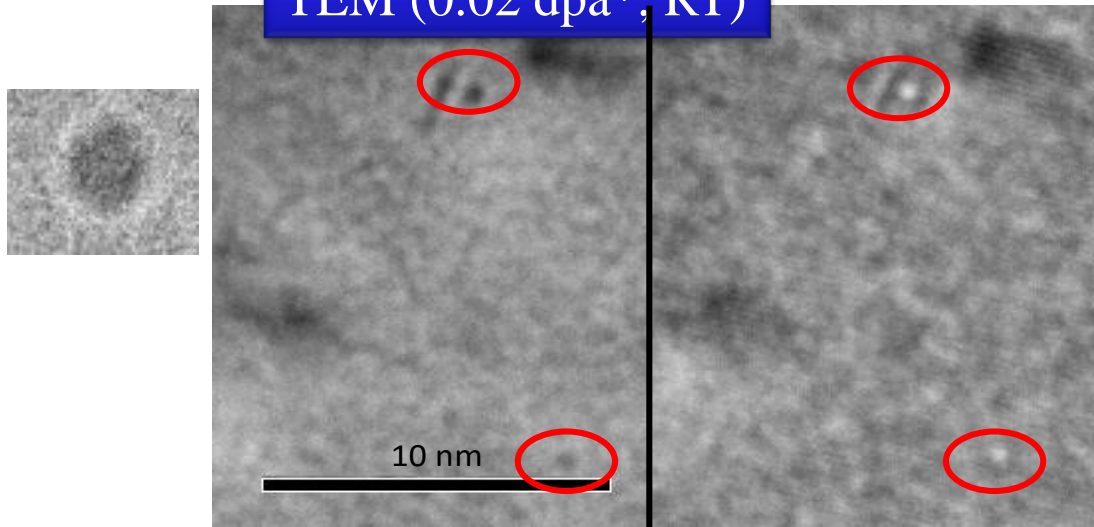


## Self irradiation damage

- ✓ At low dpa and low temperature

TEM (0.02 dpa\*, RT)

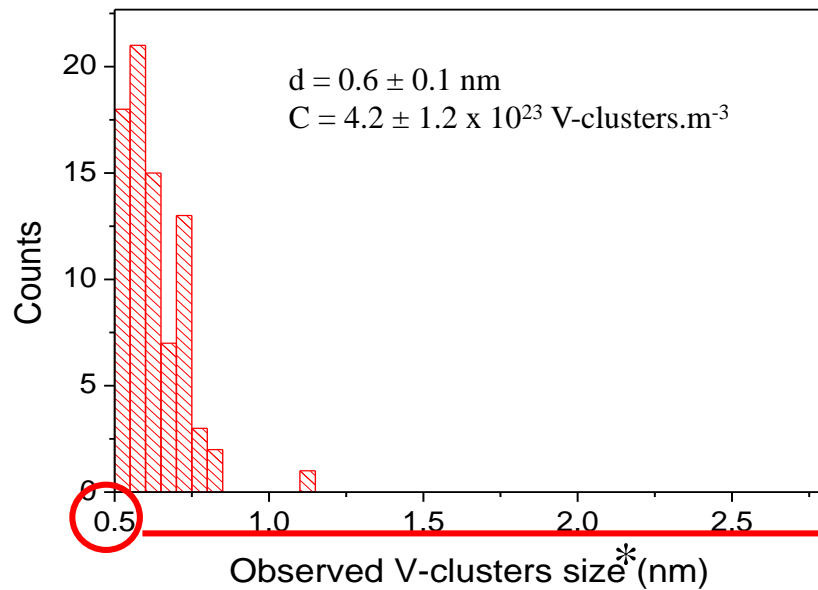
In thin lamella (3 mm disk) irradiated with 1.2 MeV W



Overfocused

Underfocused

- First observation of cavities in such irradiation conditions, at low dpa and low temperature



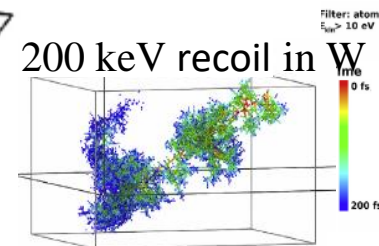
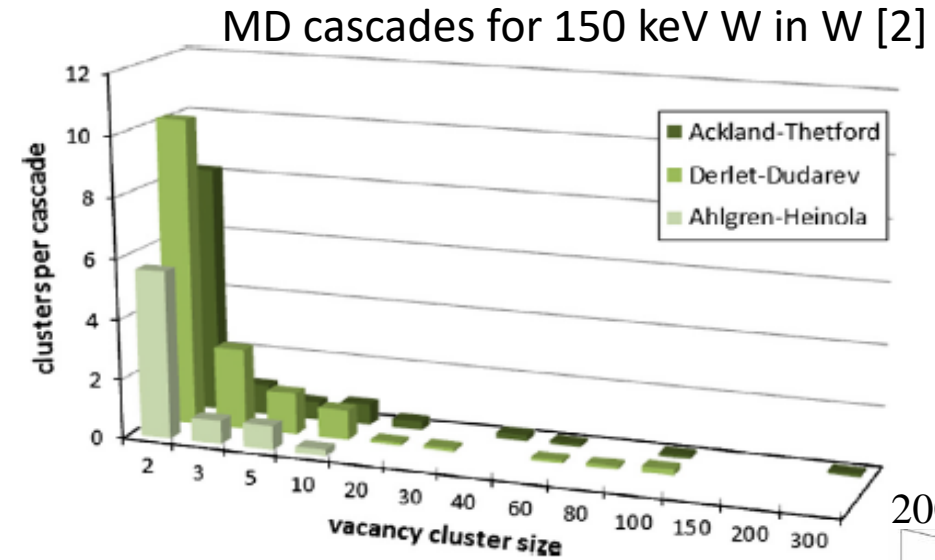
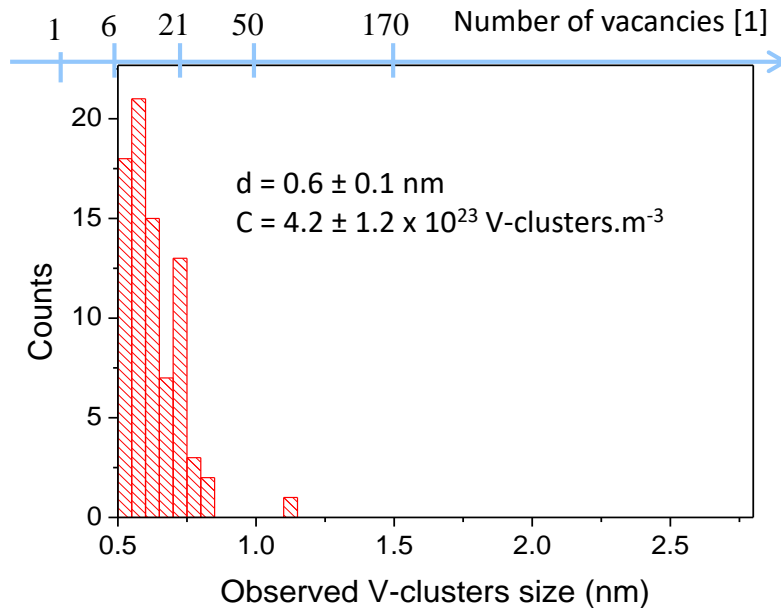
- Cavities or Vacancy clusters
- Non symmetrical distribution

➔ 0.5 nm is the detection limit of TEM

\* Mean diameter of cavity

TEM (0.02 dpa\*, RT)

In thin lamella irradiated with 1.2 MeV W



➤ Vacancy clusters are formed in the cascades

➔ Vacancy clusters are generated in the cascades

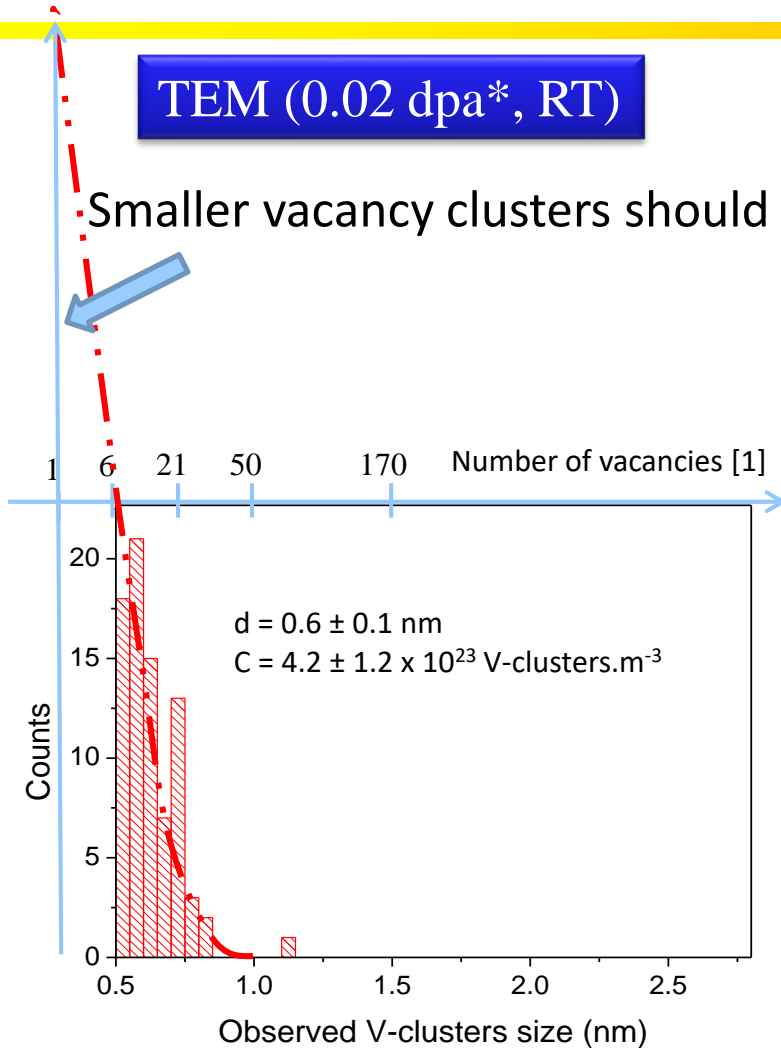
[1]  $N=(d/(2x_{r_w}))^3$

[2] A. Sand et al JNM 455 (2014) 207–211

[3] M. W. Thompson, Philosophical Magazine, (1960) 5:51, 278-296, [4] C. Becquart, et al JNM 403, Issues 1-3, 2010, 75-88

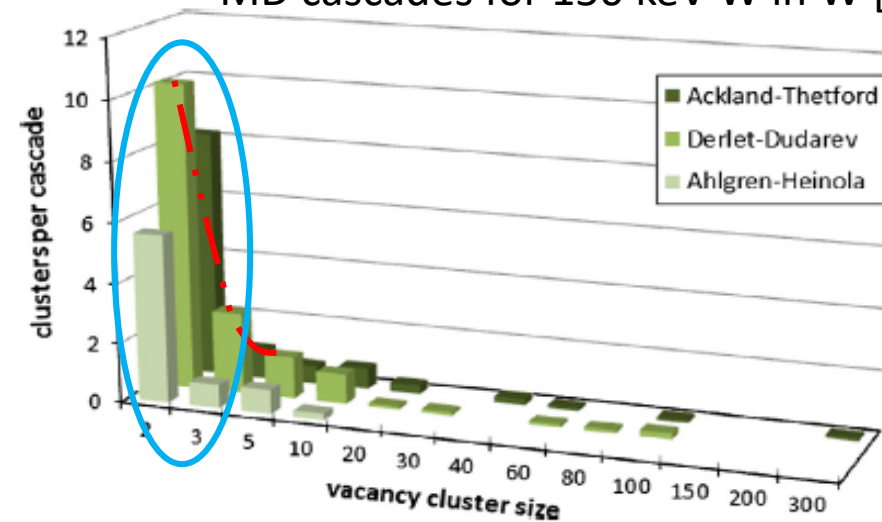
TEM (0.02 dpa\*, RT)

Smaller vacancy clusters should exist



In thin lamella irradiated with 1.2 MeV W

MD cascades for 150 keV W in W [2]



➤ Vacancy clusters are formed in the cascades

➔ Vacancy clusters are generated in the cascades

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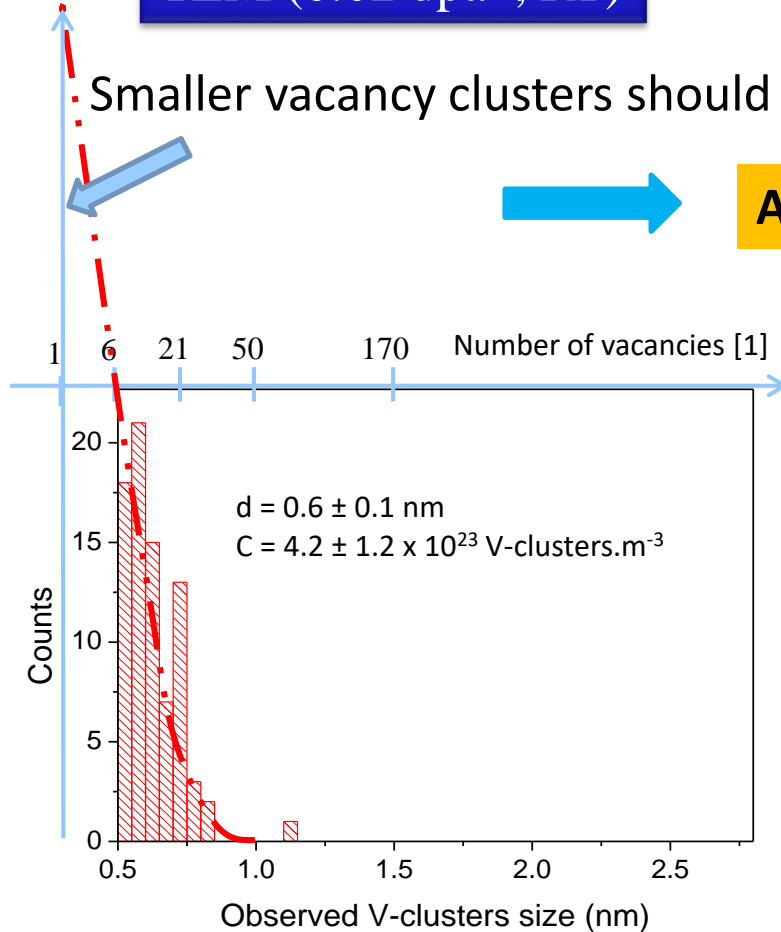
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TEM (0.02 dpa\*, RT)

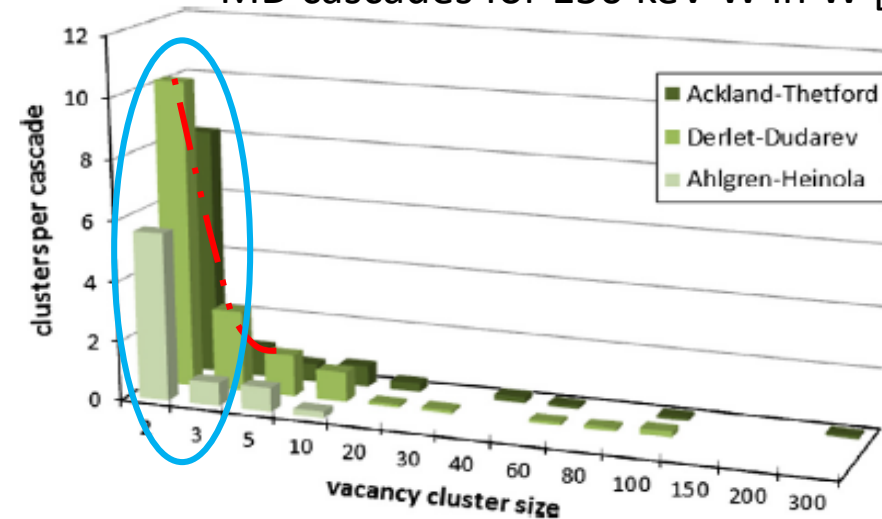
Smaller vacancy clusters should exist

In thin lamella irradiated with 1.2 MeV W

A large part of vacancy defects are not visible with TEM



MD cascades for 150 keV W in W [2]



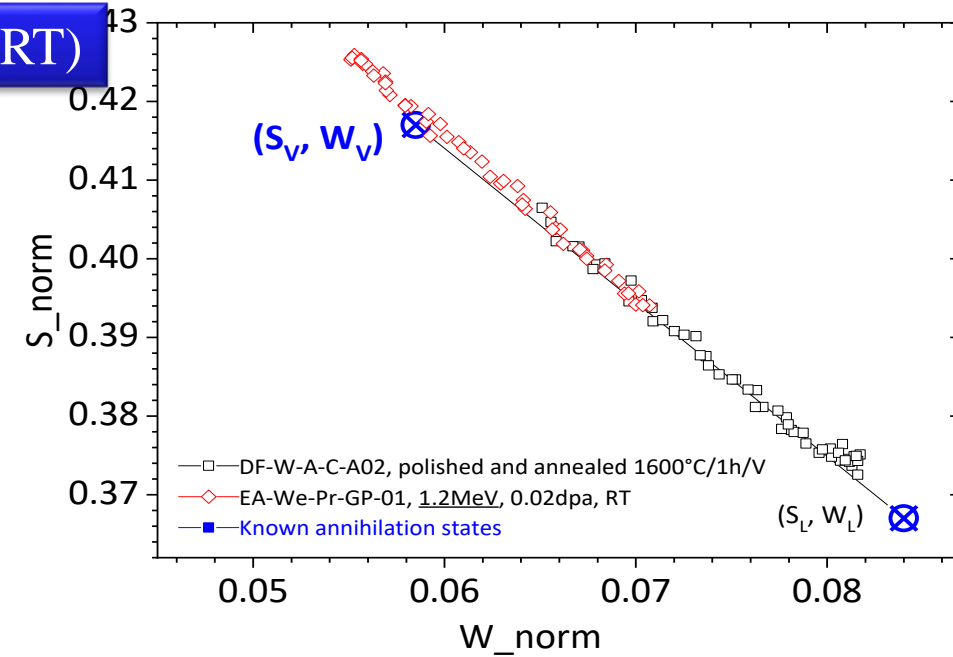
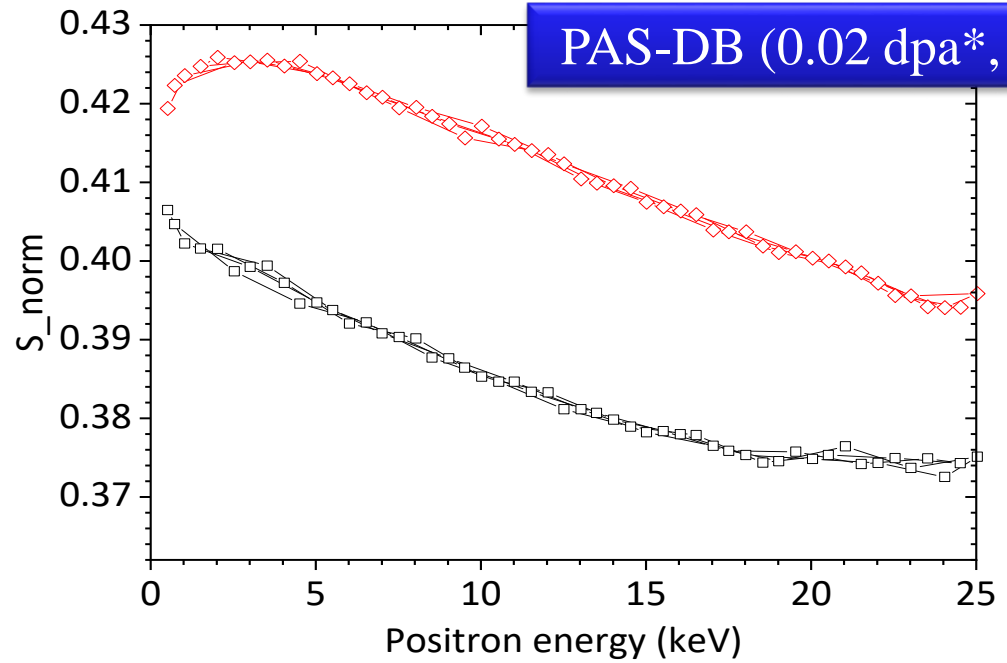
- Vacancy clusters are formed in the cascades
- $E_{mig}(V)=1.66$  eV [3,4] → no clustering expected at RT

→ single vacancies and vacancy clusters are generated in the cascades

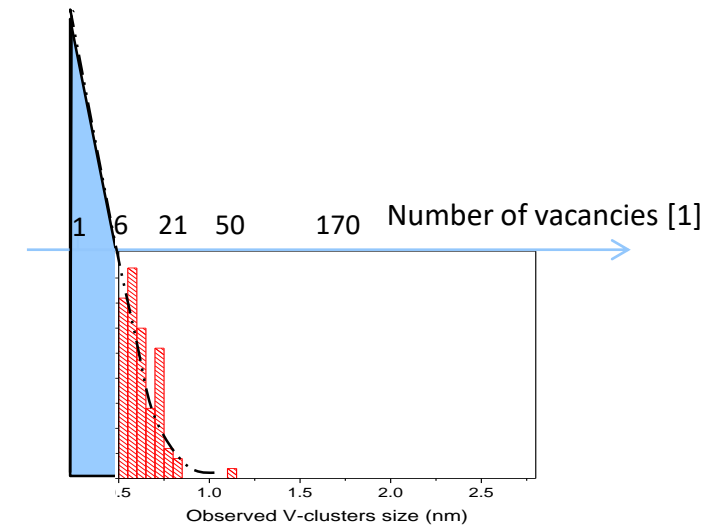
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- ✓ Maximum of damage in the first 50 nm
- ✓ Majority of **Single vacancy and small clusters** as expected from MD + **vacancy clusters** as expected from TEM

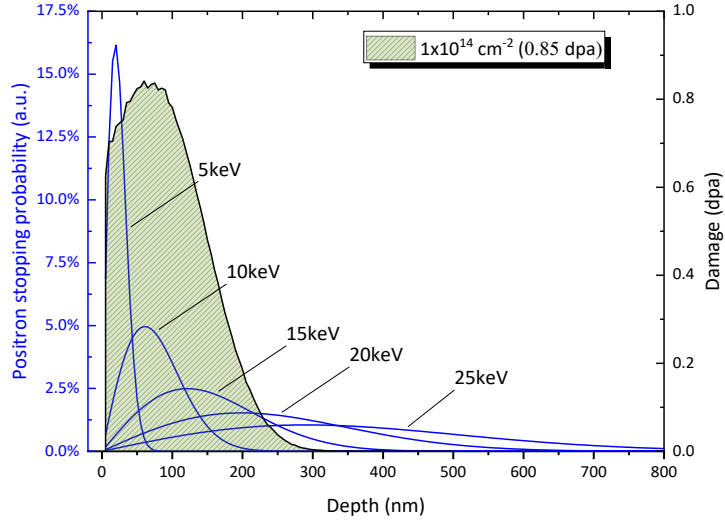




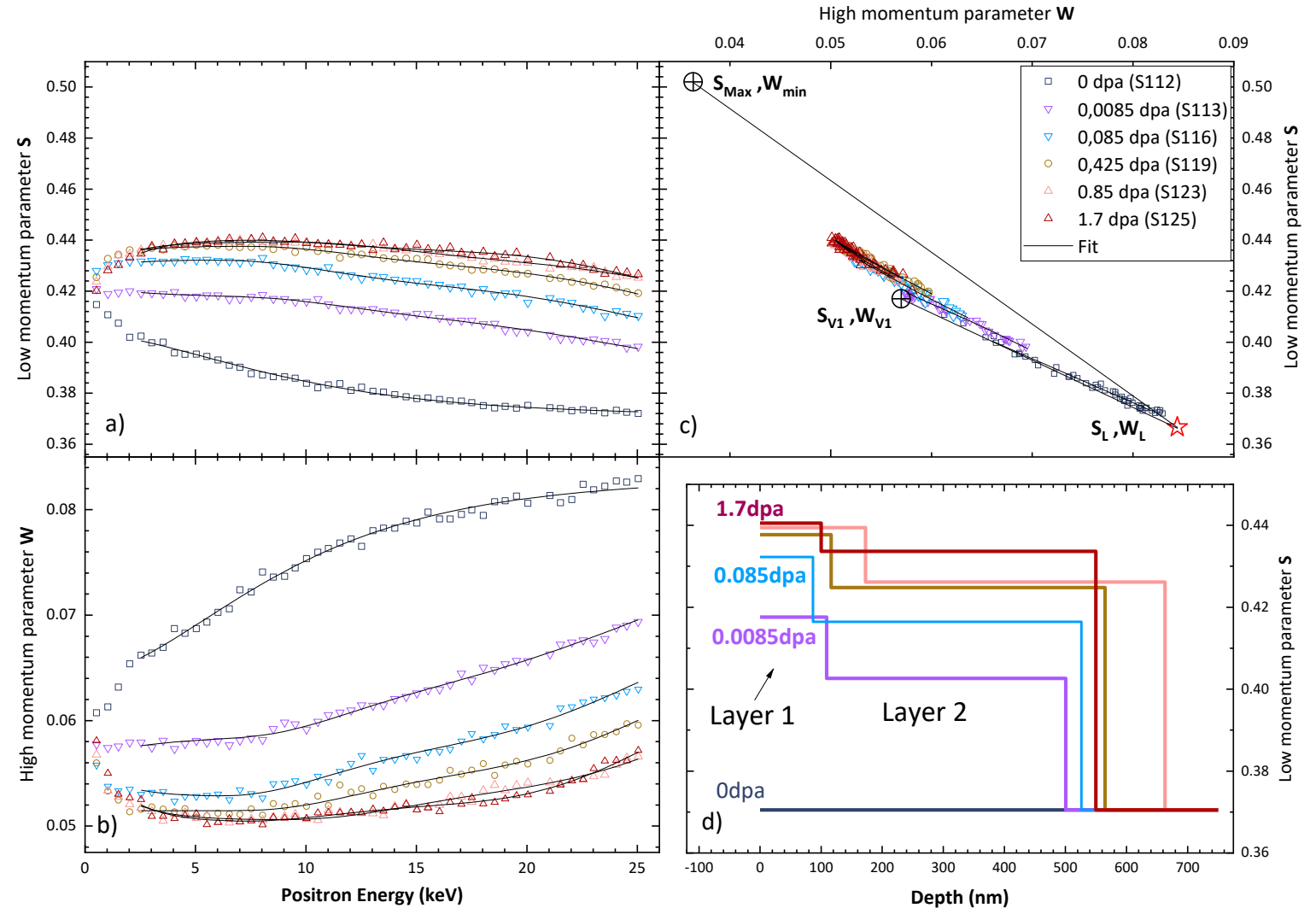
## Self irradiation damage

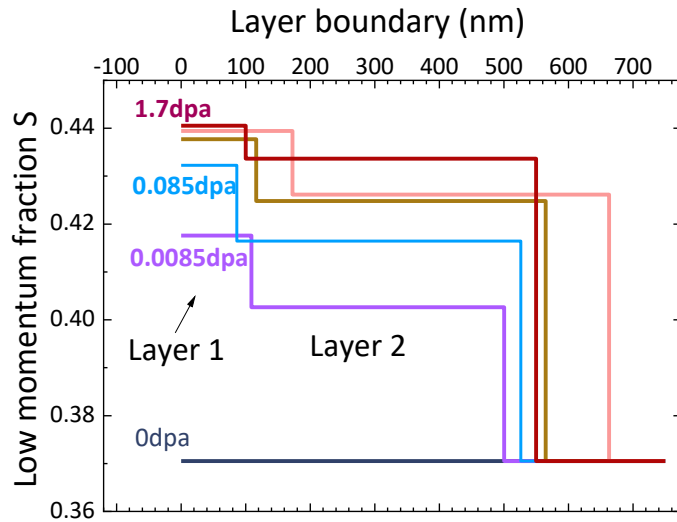
- ✓ At low dpa and low temperature
- ✓ As a function of dpa at RT

In 2 MeV self-irradiated W at RT



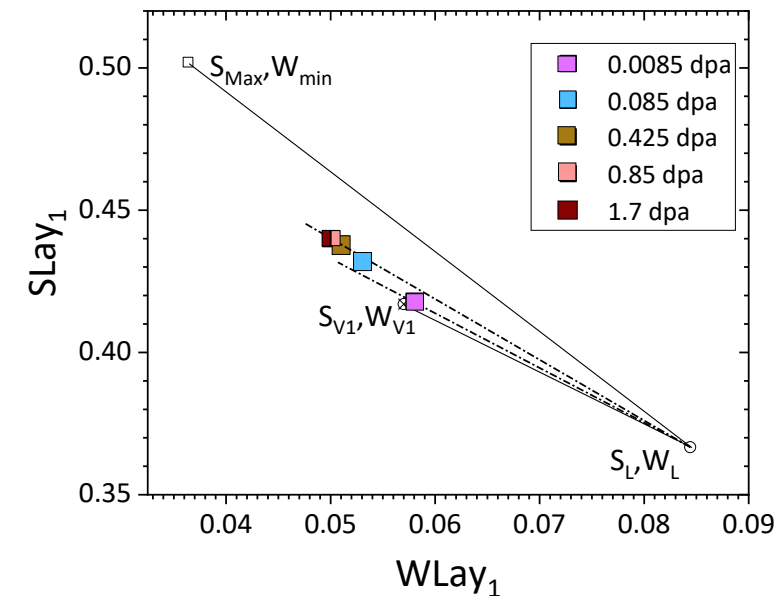
- ✓ From 0.0085- 1.7 dpa
- ✓ S increases W decreases
- ✓ 2 damaged layers close to the SRIM depth profile



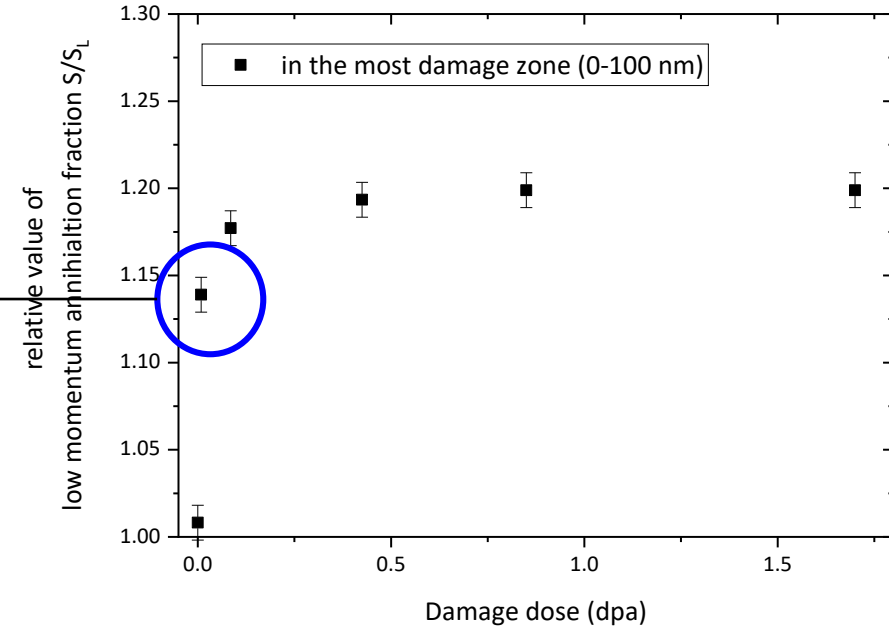


		Damage dose (dpa)				
		0.0085	0.085	0.425	0.85	1.7
Surface	$S_{\text{Surf}}$	0.419(1)	0.430(1)	0.432(1)	0.431(1)	0.431(1)
	$W_{\text{Surf}}$	0.057(1)	0.054(1)	0.053(1)	0.054(1)	0.054(1)
Layer 1	$S_{\text{Lay}}$	0.418(1)	0.432(1)	0.438(1)	0.440(1)	0.440(1)
	$W_{\text{Lay}}$	0.058(1)	0.053(1)	0.051(1)	0.050(1)	0.050(1)
	$(S_{\text{Lay}} - S_L) / (W_{\text{Lay}} - W_L)$	1.96(4)	2.10(4)	2.15(4)	2.15(4)	2.15(4)
	$L^+_{\text{Lay}}$ (nm)	14(4)	4-6	4-5	5-7	5-7
	Thickness (nm)	100(5)	90(5)	110(10)	120-170	120-170
Layer 2	$S_{\text{Lay}}$	0.402(1)	0.417(1)	0.425(1)	0.428(5)	0.428(5)
	$W_{\text{Lay}}$	0.067(1)	0.059(1)	0.057(1)	0.054(2)	0.054(2)
	$L^+_{\text{Lay}}$ (nm)	60	50	45	30	30
	Thickness (nm)	510(10)	510(20)	570(10)	610(45)	610(45)

- **Thickness** of the first damaged layer : 100 nm as maximum in SRIM
- For 0- 0.425 dpa,
  - $S_{\text{Lay}1}$  increases and  $W_{\text{Lay}1}$  decreases
  - Effective diffusion length  $L^+_{\text{Lay}1}$  decreases → density of traps increases
  - The slope  $(S_{\text{Lay}} - S_L) / (W_{\text{Lay}} - W_L)$  increases with dpa → vacancy clusters fraction increases
- From 0.85 dpa,  $S_{\text{Lay}1}$ ,  $W_{\text{Lay}1}$  and  $L^+_{\text{Lay}1}$  remain constant



PAS : RT, DBS /SPB



$$C_V^{tot} = C_V + \sum_{n=2}^{5-7} nC_{V_n} = \frac{K_{tot}}{\mu_V}, \quad K_{Tot} = \frac{D^+}{L_{eff}^+} - \lambda_L$$

Damage (dpa)	L <sup>+</sup> <sub>Lay1</sub> (nm)	C <sub>V</sub> <sup>tot</sup> (m <sup>-3</sup> )	Fraction of atomic density
0.0085	14 ± 4	9.2x10 <sup>25</sup>	0.0015
0.085	5	> 1.1x10 <sup>27</sup>	> 0.018

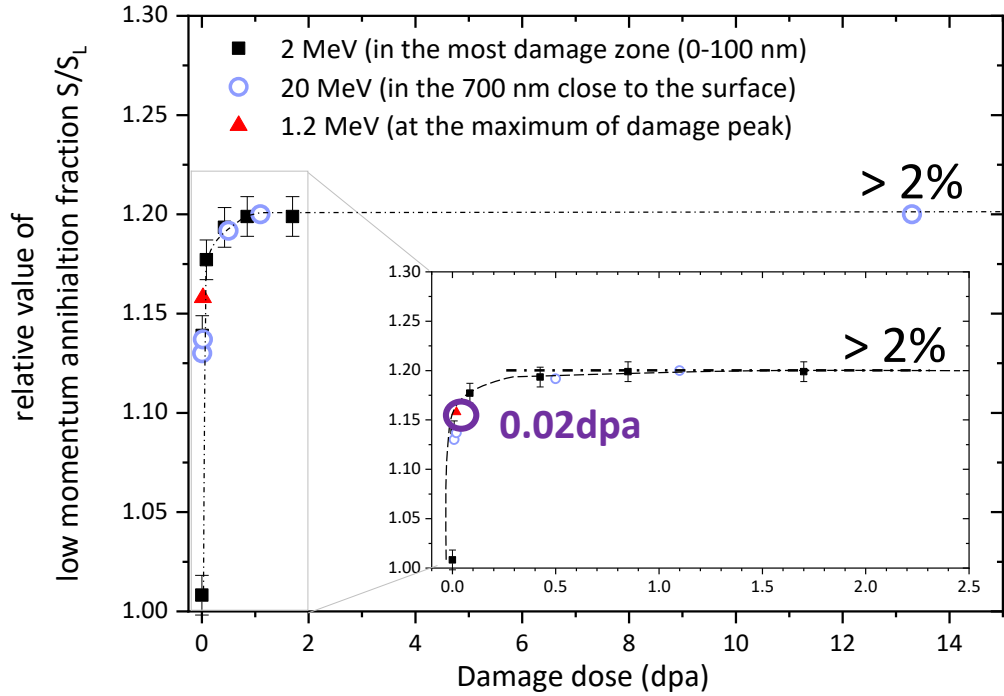
for tungsten

$$D^+ = 1.26 \times 10^{-4} \text{ m}^2/\text{s} \text{ [1]}$$

$$\lambda_L = 1/\tau_L, \tau_L = 101-105 \text{ ps} \text{ [1]}$$

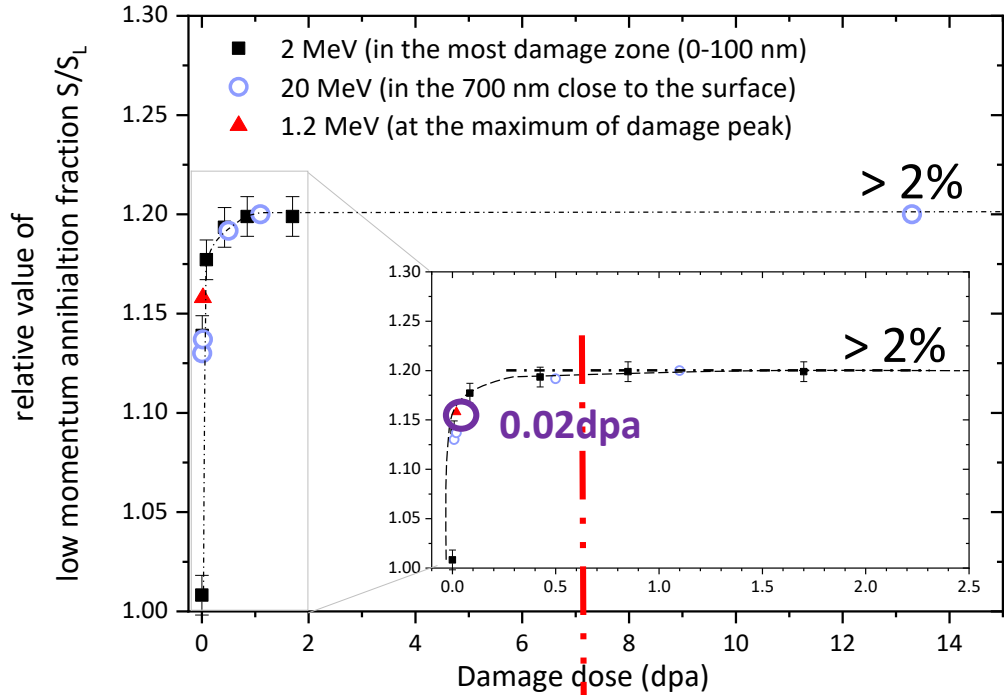
$$\mu_V = 6 \pm 3 \times 10^{-15} \text{ cm}^3 \cdot \text{s}^{-1} \text{ [1]}$$

[1] A. Hollingsworth, MF Barthe et al to be published in JNM



For 2 MeV or 1.2 or 20 MeV self-irradiated W : same behaviour  
 → saturation of S and W

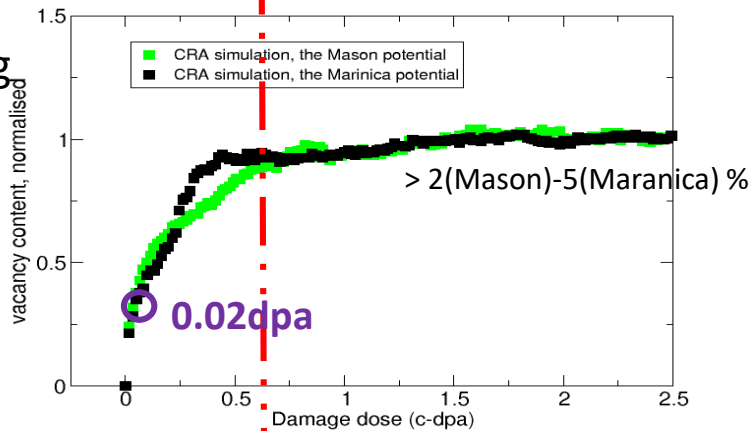
→ Saturation of positron signal or of the damage distribution ?



→ For 2 MeV or 1.2 or 20 MeV self-irradiated W : same behaviour  
 → saturation of S and W

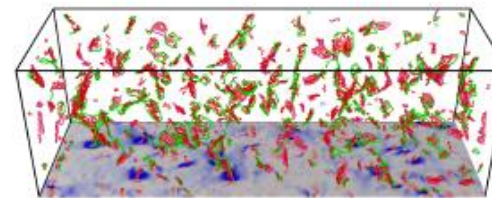
→ Saturation of positron signal or of the damage distribution ?

Simulations using the **creation-relaxation algorithm (CRA)** : 2 interatomic potentials [2]



Damage threshold :  $\approx 0.5$  dpa

Frenkel pairs accumulation



0.05 dpa

→ Saturation of the damage distribution close to 0.5 dpa

Box size : 20.2x20.2x63.2 nm<sup>3</sup>

Vacancy (blue)

Interstitial (red)

clusters with > 3 point defects are shown

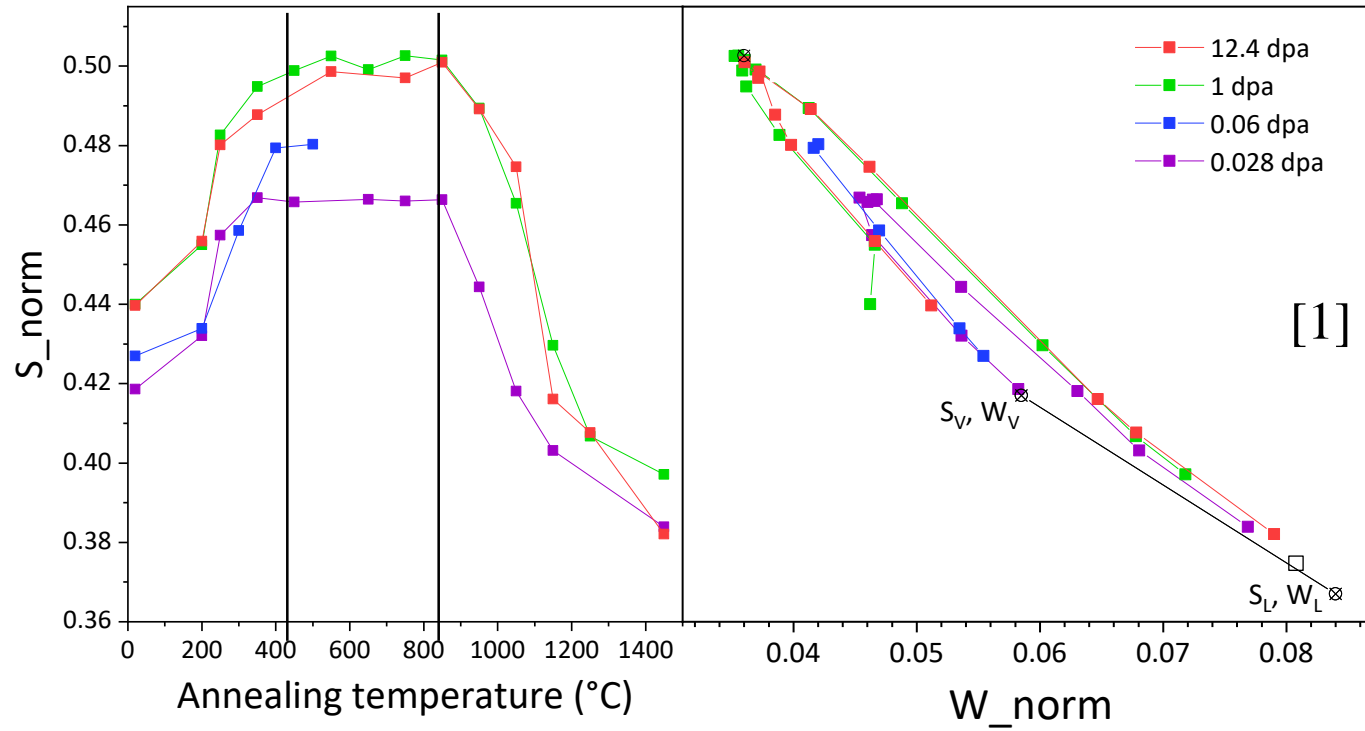
1/2<111> (green), <100> (pink) dislocation lines.

[1] A. Hollingsworth, MF Barthe et al to be published in JNM

[2] P.M. Derlet and S.L. Dudarev, Physical Review Materials **4**, 023605 (2020).

## Self irradiation damage

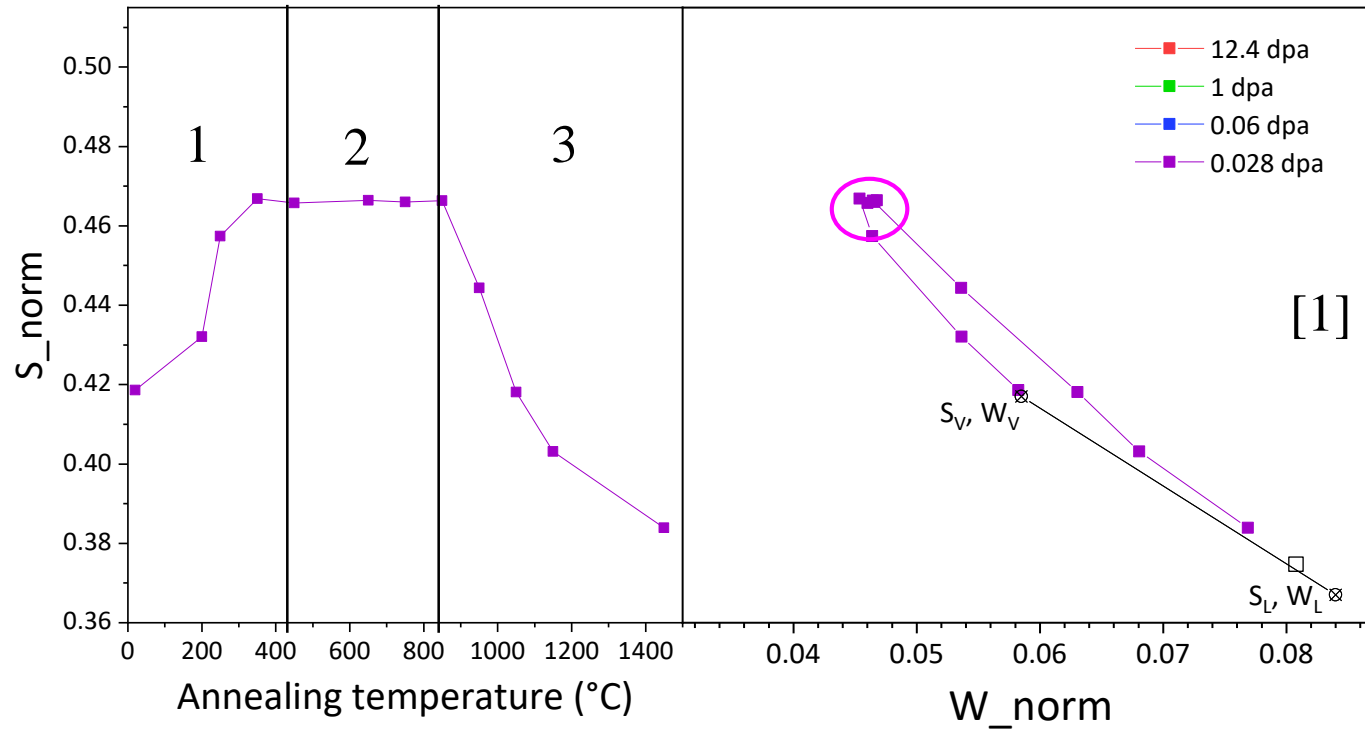
- ✓ At low dpa and low temperature
  - ✓ As a function of dpa RT
- ✓ After annealing as a function of dpa



➤ For all dpa levels 3 annealing stages

[1] M Sidibe PhD thesis Orleans University 2014





➤ For all dpa levels 3 annealing stages

✓ RT- 600°C

▪ S increase and W decrease

▪  $|(S-S_L)/(W-W_L)|$  increases

➔ agglomeration to form **large Vacancy clusters**

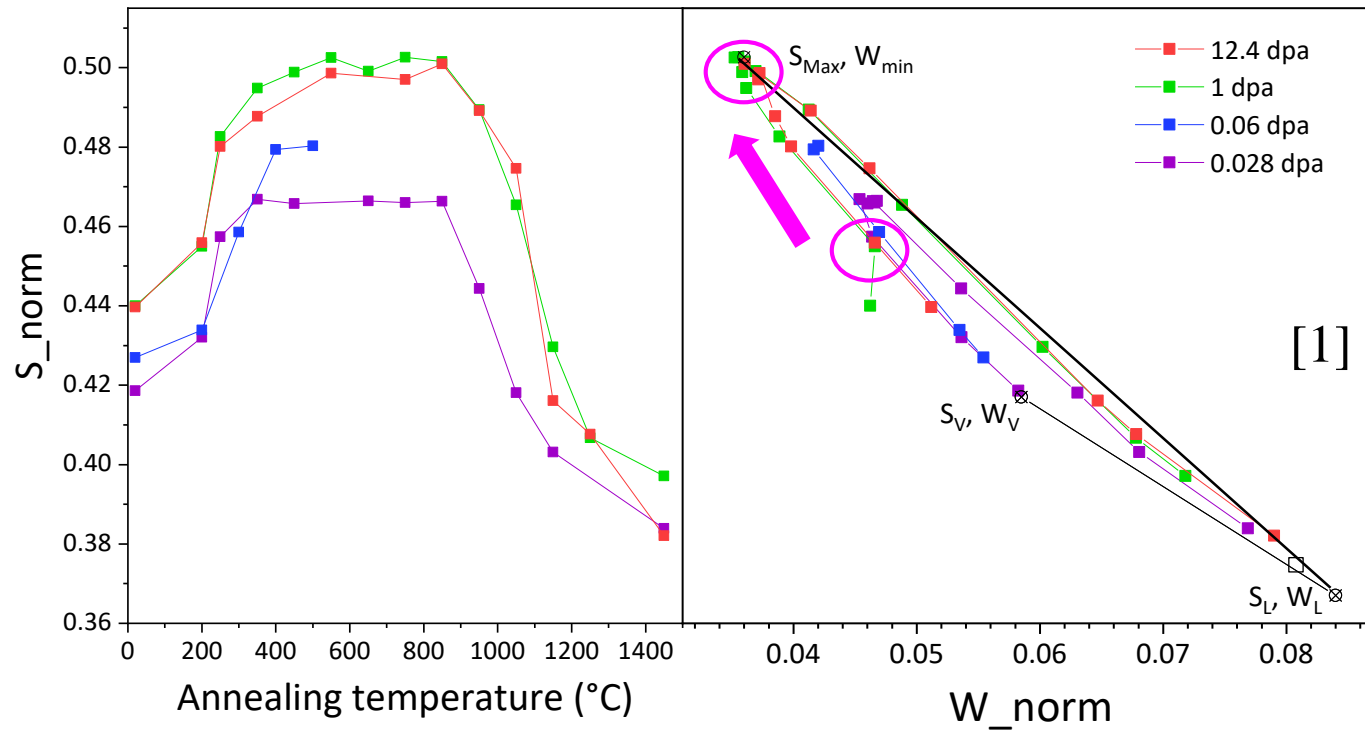
➤ 600-850°C

➤ No change

➤ 850-1500°C

➤ Damage recovery

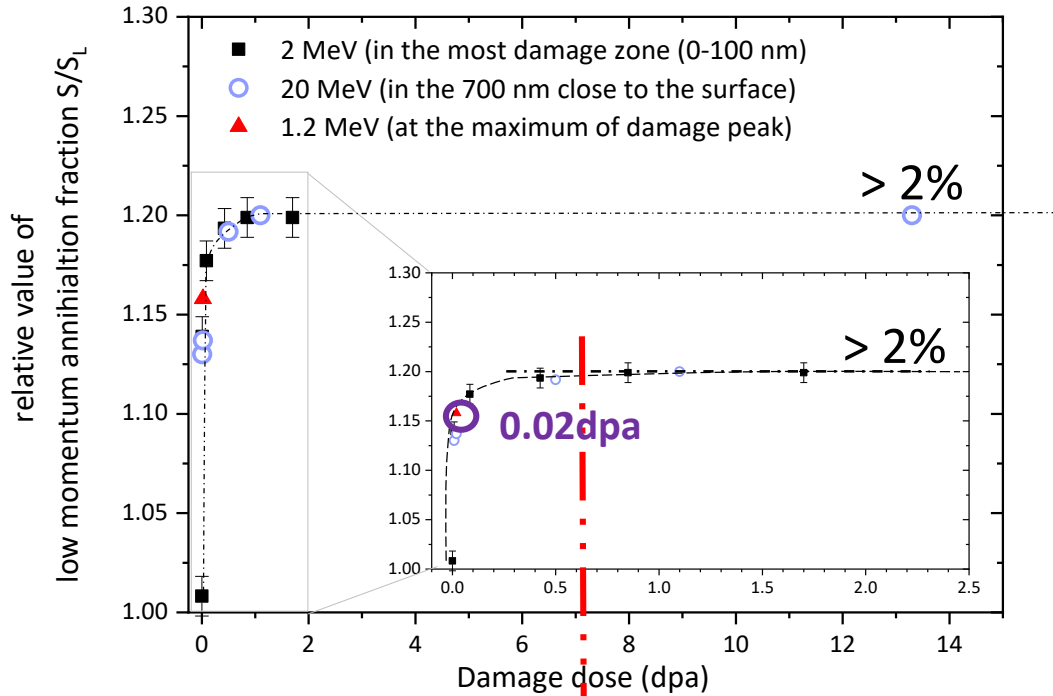
[1] M Sidibe PhD thesis Orleans University 2014



- For all dpa levels 3 annealing stages
  - ✓ RT- 600°C
    - S increase and W decrease
    - $|(S-S_L)/(W-W_L)|$  increases
  - ➔ agglomeration to form large Vacancy clusters
  
- 600-850°C
  - No change
- 850-1500°C
  - Damage recovery
  
- For low dpa (0.03 and 0.06 dpa)
  - $|(S-S_L)/(W-W_L)|$  and **size of vacancy clusters increases** when damage dose increases
- For high dpa (1 and 12 dpa)
  - No differences for 1 and 12 dpa
  
- $S_{Max}, W_{min} \rightarrow$  **New annihilation State ?**

**Defects distribution is equivalent for dpa level  $\geq$  1 dpa**

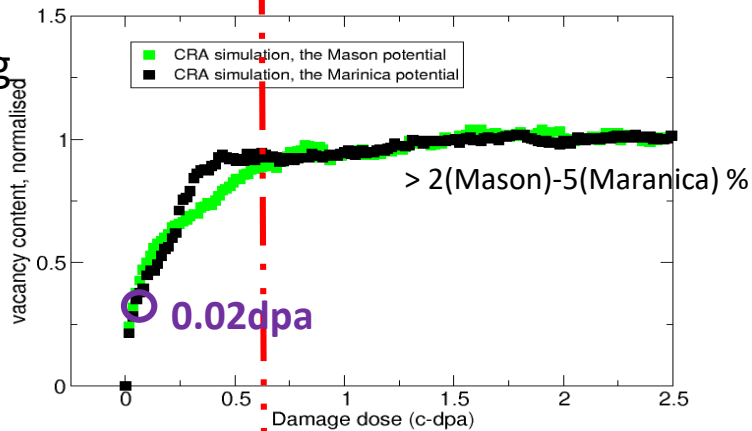
[1] M Sidibe PhD thesis Orleans University 2014



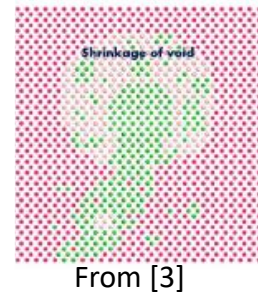
→ For 2 MeV or 1.2 or 20 MeV self-irradiated W : same behaviour  
 → saturation of S and W

→ Saturation of positron signal or of the damage distribution ?

Simulations using the **creation-relaxation algorithm (CRA)** : 2 interatomic potentials [2]



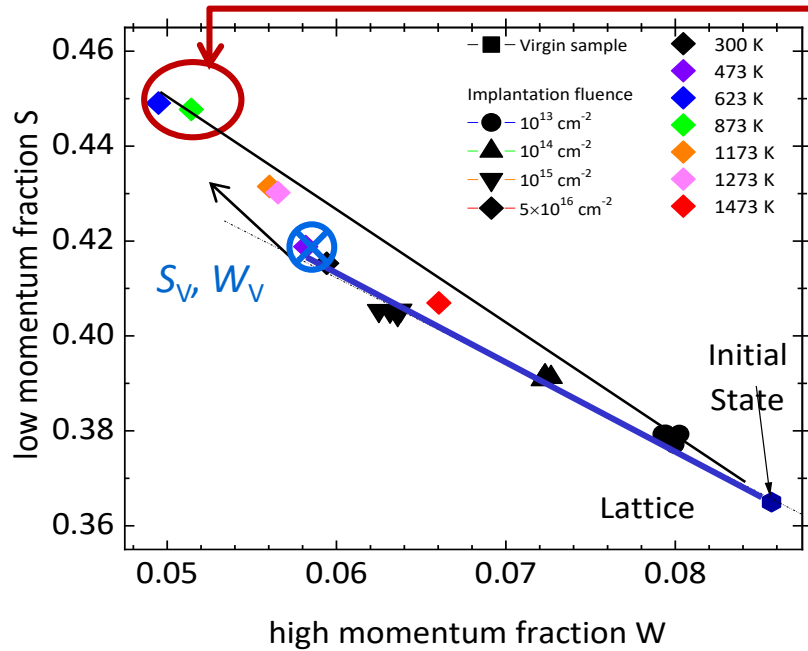
Damage threshold :  $\approx 0.5$  dpa



→ Saturation of the damage distribution close to 0.5 dpa

Due to point defects recombination and cascade overlapping inducing vacancy clusters shrinkage as shown in Fe [3]

[1] A. Hollingsworth, MF Barthe et al to be published in JNM  
 [2] P.M. Derlet and S.L. Dudarev, Physical Review Materials **4**, 023605 (2020)  
 [3] Agarwal et al., Sci. Adv. 2020; **6** : eaba8437 29 July 2020.



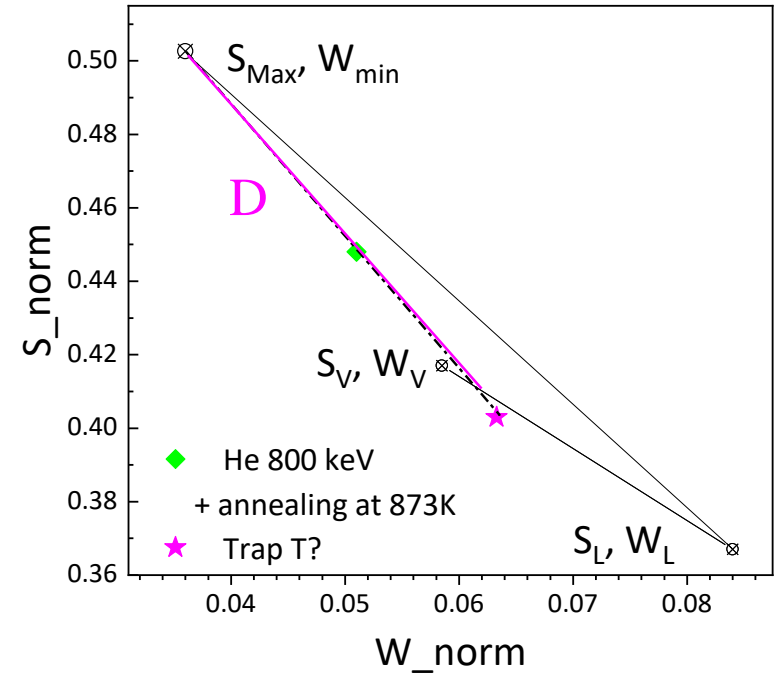
Mean lifetime	1 <sup>st</sup> component		2 <sup>nd</sup> component	
$\tau_m$ (ps)	$\tau_1$ (ps)	$I_1$ (%)	$\tau_2$ (ps)	$I_2$ (%)
331	176	54	458	45

At 873K Positron Annihilation Lifetime Spectrometry with PLEPS [1]

**V-clusters**  
 $V_N$  with  $N > 30$  [2]

T?

- $S_{Max} = S_{V_N}$  and  $W_{min} = W_{V_N}$   
 With  $N = \text{or} > 30$
- $S_T = 0.403(5)$  and  $W_T = 0.063(5)$
- Slope of D line  
 $|(S_{Max} - S_T) / (W_{min} - W_T)| \approx 3.66$



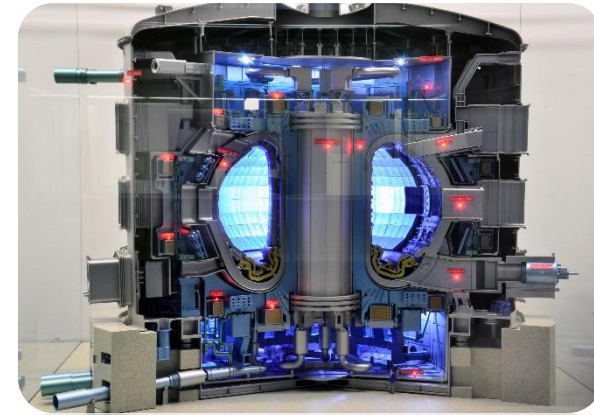
[1] P E Lhuillier et al Phys. Status Solidi C 6, No. 11, 2329–2332 (2009) / DOI 10.1002/pssc.200982114

## Self irradiation damage

- ✓ At low dpa and low temperature
- ✓ As a function of dpa at RT
- ✓ **Role of purity**

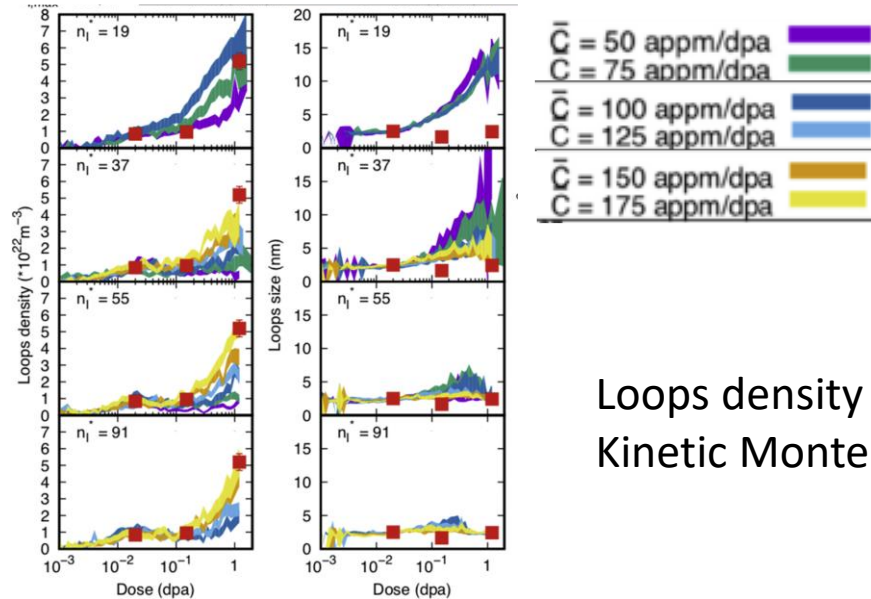
## Source of impurities

- H, He from Plasma and transmutations<sup>[2]</sup>
  - bubbles, fuzz...
- Transmutation elements (Re, Os, Ta..) <sup>[3]</sup>
  - precipitates...
- Reactor's environment light elements **impurities (C, N, O...)** <sup>[4-5]</sup>
  - Impurity-defects complexes



ITER TOKAMAK  
(Start of the assembly on **28 July 2020**)

?

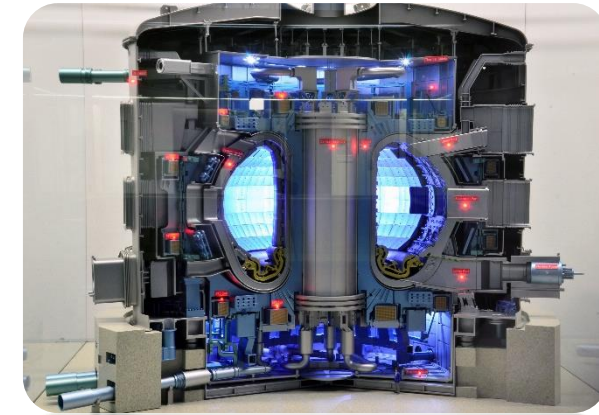


Loops density and size as a function of dpa OKMC (Object Kinetic Monte Carlo) simulations [1]

[1] N. Castin, et al J. Nucl. Mater. 527 (2019); 1.Hirai, T. et al.. Nuclear Materials and Energy 9, 616–622 (2016). 2 Greenwood.al 212–215, 635–639 (1994). 3 Computational Materials Science 184, 109932 (2020). 3 Liu. Computational Materials Science 50, 3213–3217 (2011). 4 You, Y.-W. et al.. RSC Adv. 5, 23261–23270 (2015). 5 Alkamees, A. et al.. Journal of Nuclear Materials 393, 508–512 (2009).

## Source of impurities

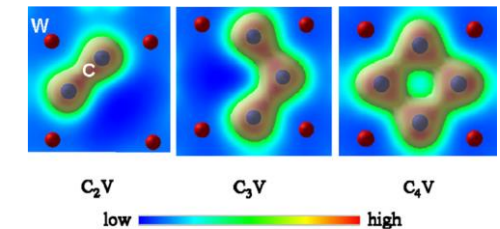
- H, He from Plasma and transmutations<sup>[2]</sup>
  - bubbles, fuzz...
- Transmutation elements (Re, Os, Ta..) <sup>[3]</sup>
  - precipitates...
- Reactor's environment light elements **impurities (C, N, O...)** or LEs <sup>[4-5]</sup>
  - ?** → Impurity-defects complexes



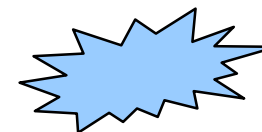
ITER TOKAMAK  
(Start of the assembly on **28 July 2020**)

Vacancy interactions with LEs  
DFT calculations

X	$E_m^X$ (eV)	$E_b^{V-X_1}$ (eV)	$E_{diss}^{V-X_1}$ (eV)
H	0.21 (TIS-TIS) [24]	1.24 [25]	1.45
C	1.46 (TIS-OIS) [20]	1.93 [20]	3.39
N	0.73 (TIS-OIS) [26]	2.48 [21]	3.21
O	0.17 (TIS-TIS) [22]	3.05 [22]	3.22



*Light element impurities* (H,C,N,O..)



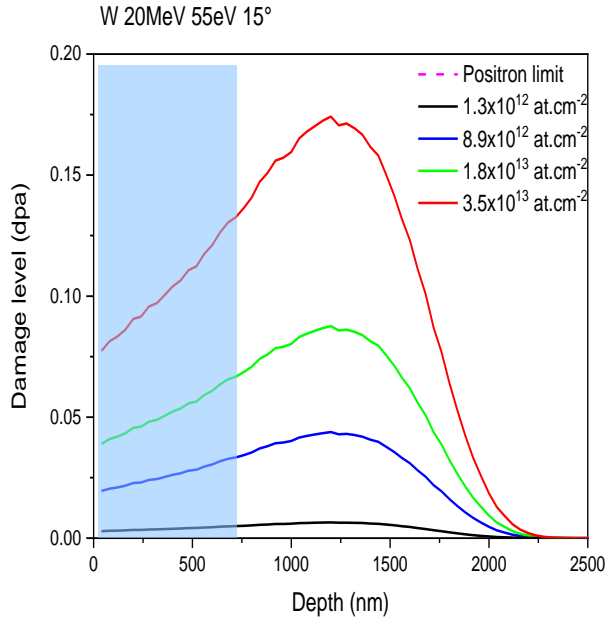
**defects**(single vacancy, vacancy-complexes and SIA).

[1] N. Castin, et al J. Nucl. Mater. 527 (2019); 1 .Hirai, T. et al.. Nuclear Materials and Energy 9, 616–622 (2016). 2 Greenwood, al 212–215, 635–639 (1994). 3 *Computational Materials Science* **184**, 109932 (2020). 3 Liu. *Computational Materials Science* 50, 3213–3217 (2011). 4 You, Y.-W. et al.. *RSC Adv.* 5, 23261–23270 (2015). 5 Alkamees, A. et al.. *Journal of Nuclear Materials* 393, 508–512 (2009). 6 A. Vehanen et al *Phys Rev B* 25 (1982) 762

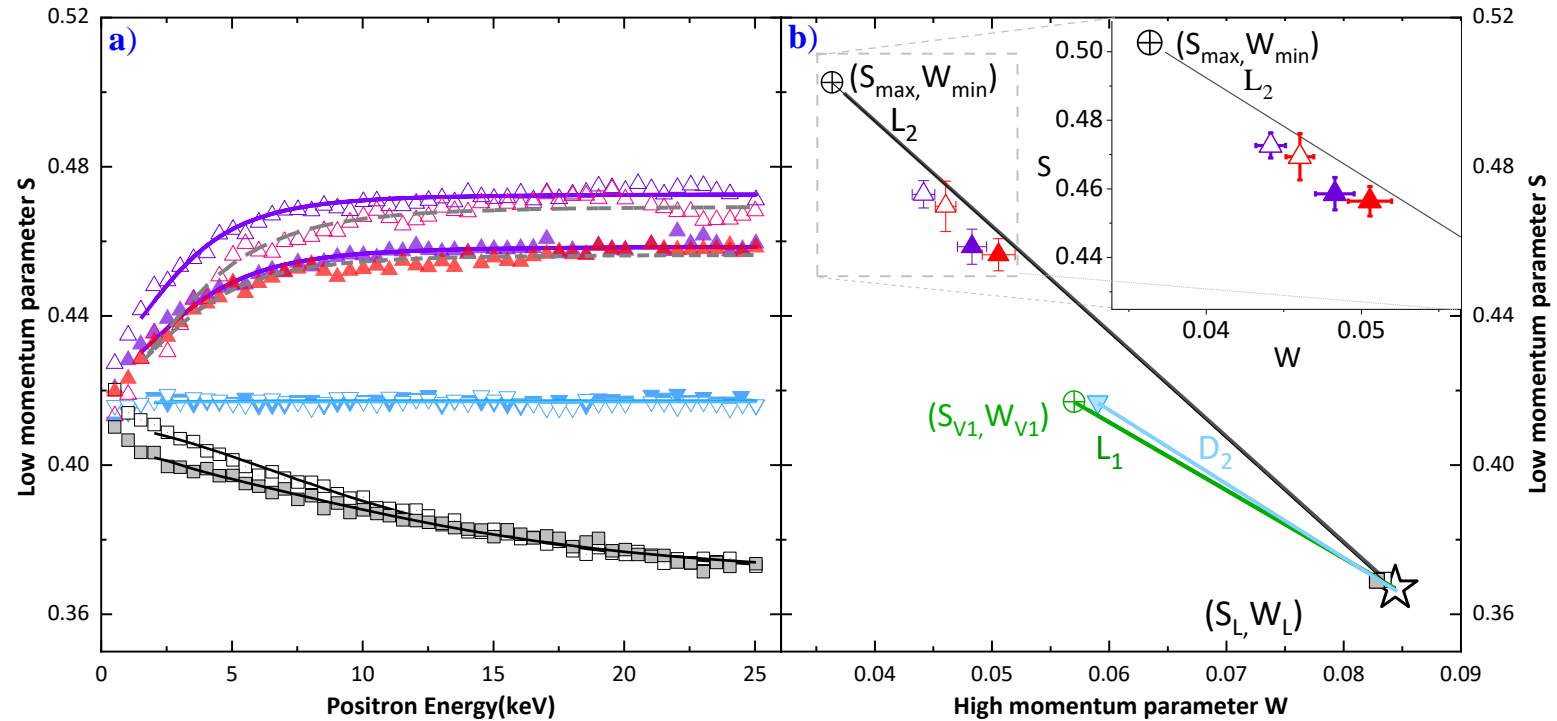
Polycrystalline W :  
2 purities : 6N-XHP and 3N-HP

Larger vacancy clusters in  
6N-XHP than in 3N-HP

VEPFIT	dpa	Temp°C	3N-HP	6N-XHP
—	0		□	□
—	0.01	RT	▼	▼
—	0.02	500	▲	▲
- - -	0.02	700	▲	▲

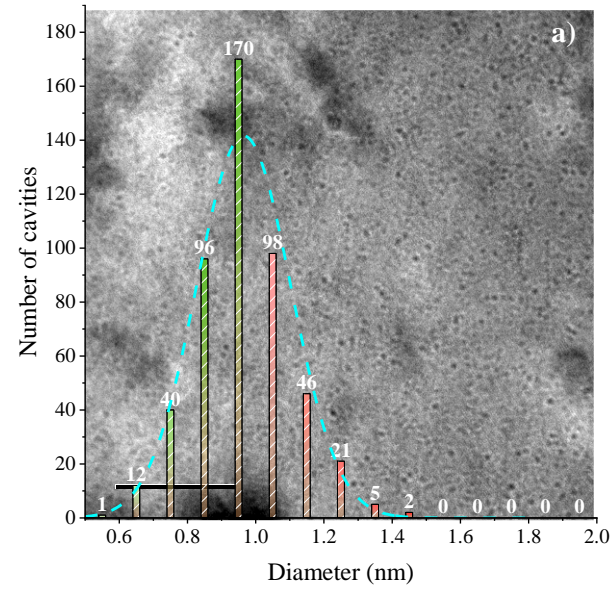


Dpa\* : mean dpa value in **700nm** surface region,

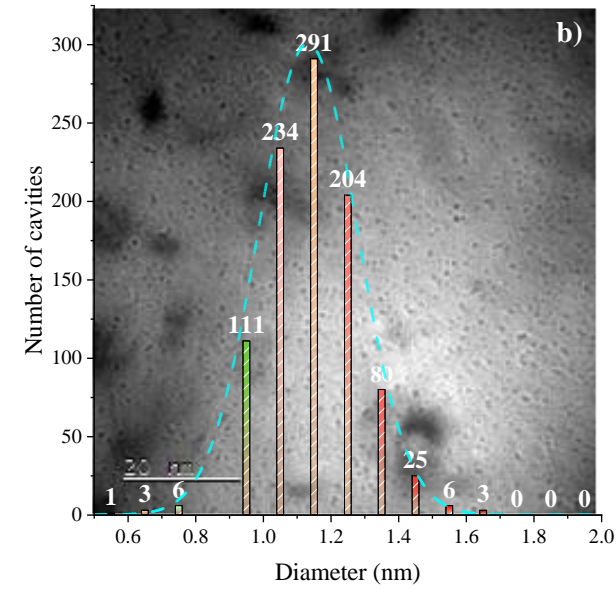




1.2 MeV W 0.02dpa 500°C

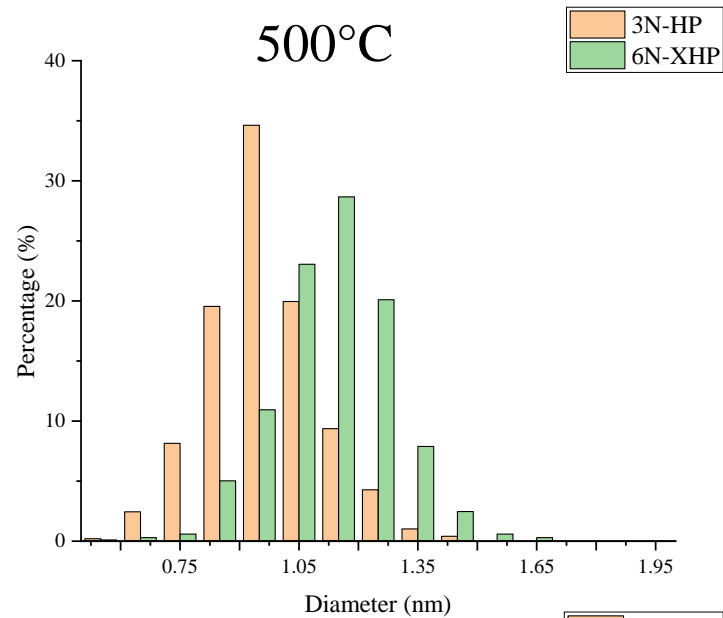


3N-HP

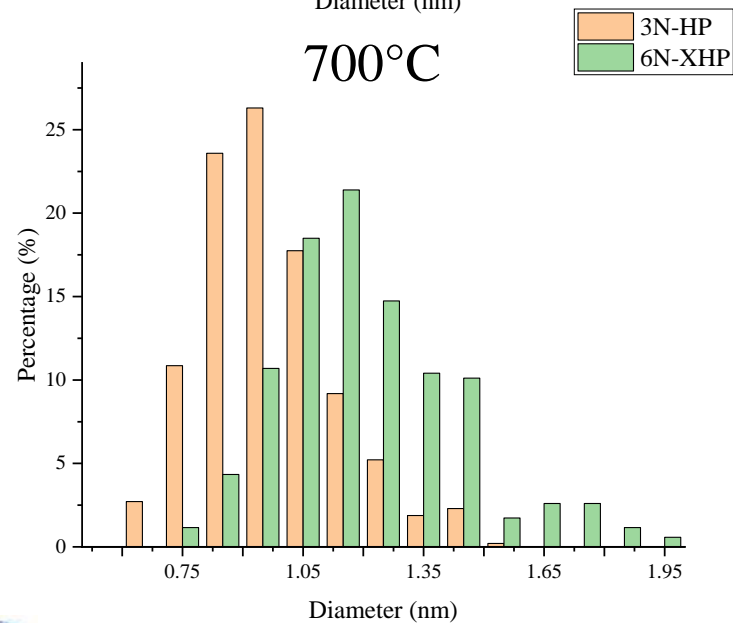


6N-XHP

JEOL ARM200F cold field emission gun microscope and Over-focus (+300 nm) images

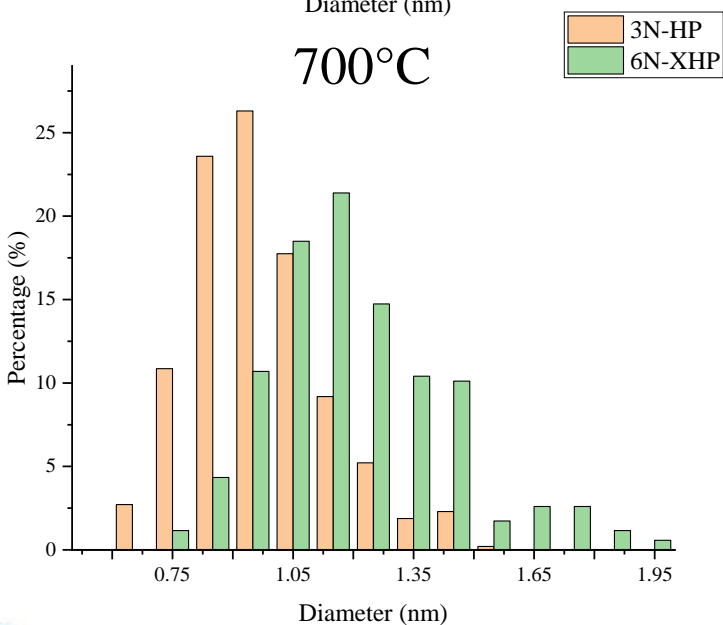
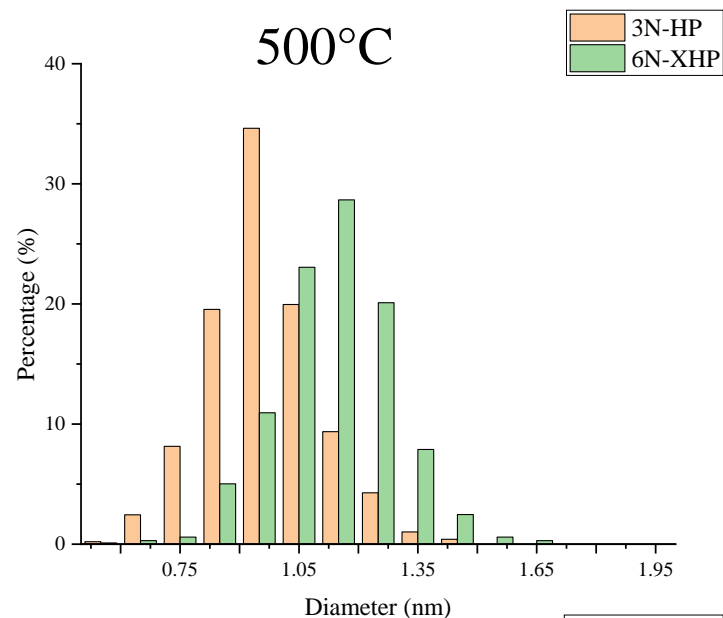


➤ Larger cavities in **6N-XHP** samples compare to **3N-HP**



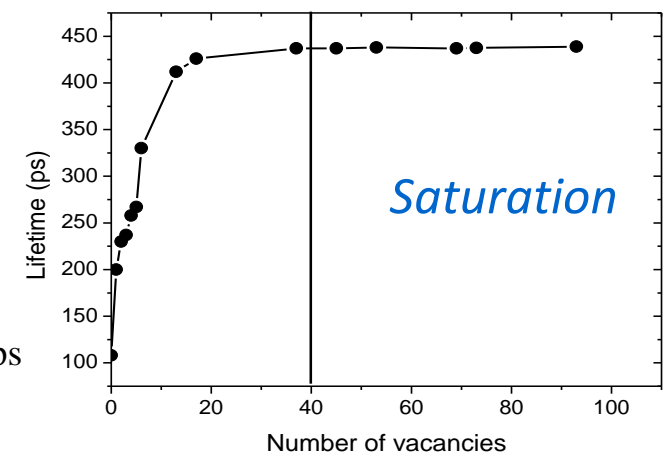
Damage level (dpa)	0.02			
Irradiation Temperature (°C)	500		700	
Purity	3N-HP	6N-XHP	3N-HP	6N-XHP
Thickness (nm)	55 ± 20 (EFTEM)	90 ± 30 (EFTEM)	35 ± 20 (evaluated)	60 ± 50 (evaluated)
Mean diameter(nm)	<b>0.97 ± 0.19</b>	<b>1.13 ± 0.21</b>	<b>0.97 ± 0.25</b>	<b>1.22 ± 0.25</b>
Density (10 <sup>24</sup> m <sup>-3</sup> )	1.73 ± 0.71	1.56 ± 0.52	0.70 ± 0.42	0.60 ± 0.53
Swelling %	0.09 ± 0.02	0.12 ± 0.04	0.04 ± 0.02	0.08 ± 0.05

Z. Hu, MF Barthe et al , Journal of Nuclear Materials 556 (2021) 153175



Theory (DFT) [1]

$$\tau_{\text{Lattice}} = 108 \text{ ps}$$



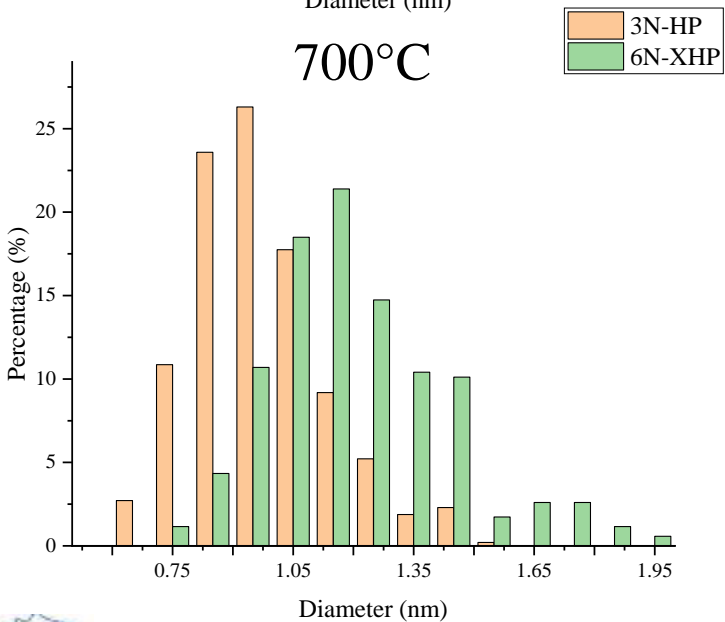
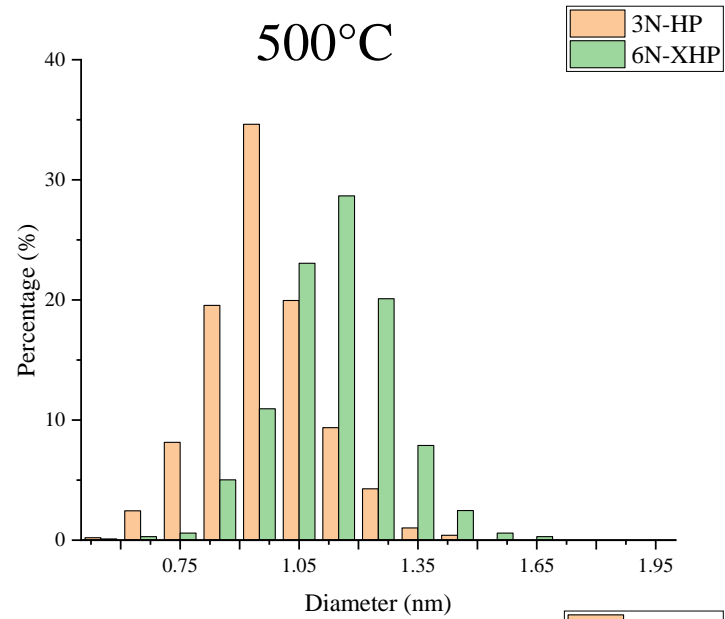
*diam > 1 nm → clusters V<sub>N</sub> N > 40*

➤ Larger cavities in **6N-XHP** samples compare to **3N-HP**

Damage level (dpa)	0.02			
Irradiation Temperature (°C)	500		700	
Purity	3N-HP	6N-XHP	3N-HP	6N-XHP
Thickness (nm)	55 ± 20 (EFTEM)	90 ± 30 (EFTEM)	35 ± 20 (evaluated)	60 ± 50 (evaluated)
Mean diameter (nm)	<b>0.97 ± 0.19</b>	<b>1.13 ± 0.21</b>	<b>0.97 ± 0.25</b>	<b>1.22 ± 0.25</b>
Density (10 <sup>24</sup> m <sup>-3</sup> )	1.73 ± 0.71	1.56 ± 0.52	0.70 ± 0.42	0.60 ± 0.53
Swelling %	0.09 ± 0.02	0.12 ± 0.04	0.04 ± 0.02	0.08 ± 0.05

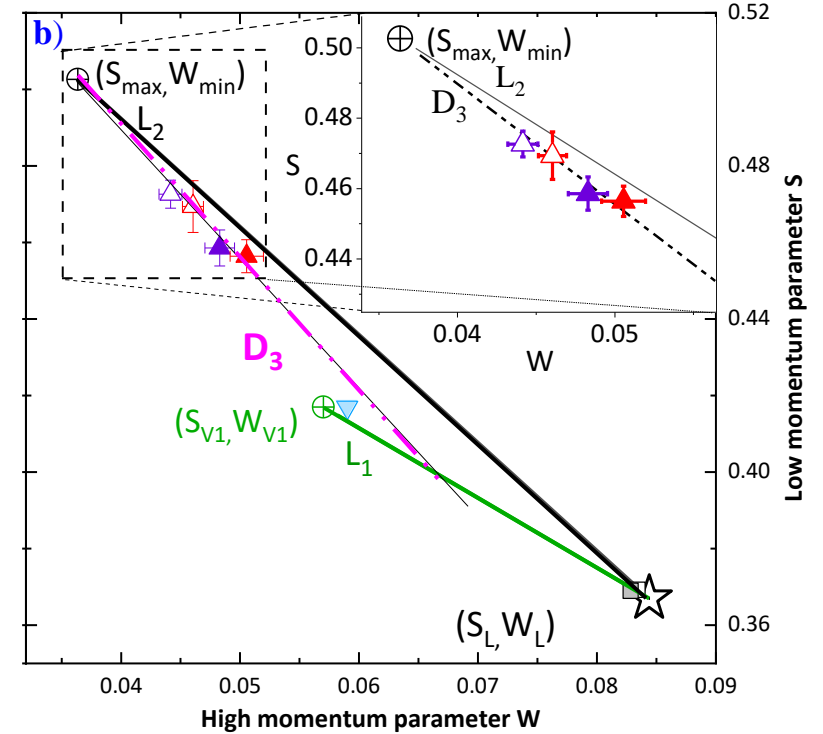
Z. Hu, MF Barthe et al, Journal of Nuclear Materials 556 (2021) 153175

[1] T. Troev, E. Popov, P. Staikov, N. Nankov, T. Yoshiie, NIMB 267, 3, 335



- Larger cavities in **6N-XHP** samples compare to **3N-HP**
- as in PAS
- **D<sub>3</sub>** line : trapping competition in large vacancy clusters  $V_N$  and an unknown trap **T?** ,  $|(S_{Max} - S_T)/(W_{min} - W_T)| \approx 3.5$
- **T?** annihilation characteristics on **D<sub>3</sub>** line

dpa	Temp°C	3N-HP	6N-XHP
0		■	□
0.01	RT	▼	▽
0.02	500	▲	△
0.02	700	▲	△



Sample	Purity %wt	Damage level(dpa)	Temperature °C	S	W	$L_{eff}^+$ (nm)
G-35	99.95	0	-	0.369(2)	0.0828(4)	132(6)
J-17	99.9999	0	-	0.369(2)	0.0834(4)	105(4)
G-04	99.95	0.01	RT	0.417 (2)	0.0599(6)	0.9(3)
J-05	99.9999			0.417(2)	0.0590(9)	0.7(2)
G-05	99.95	0.02	500	0.459(2)	0.0483(9)	15(2)
J-11	99.9999			0.473(4)	0.0429(8)	9(1)
J-06				0.474 (3)	0.043 (1)	12(2)
J-15				0.473(4)	0.044 (1)	12(2)
G-02	99.95	0.02	700	0.456(4)	0.051(1)	14(2)
J-03	99.9999			0.469(7)	0.0461(9)	17(2)

Close to Lattice values, very low concentration of defects

Single vacancy as the main vacancy defects

No difference in effective diffusion length for both purities and both irradiation temperatures

$$L_{eff}^+ \approx 14 \text{ nm}$$

→ A high total trapping rate  $K_{tot} \approx 8 \times 10^{11} \text{ s}^{-1}$

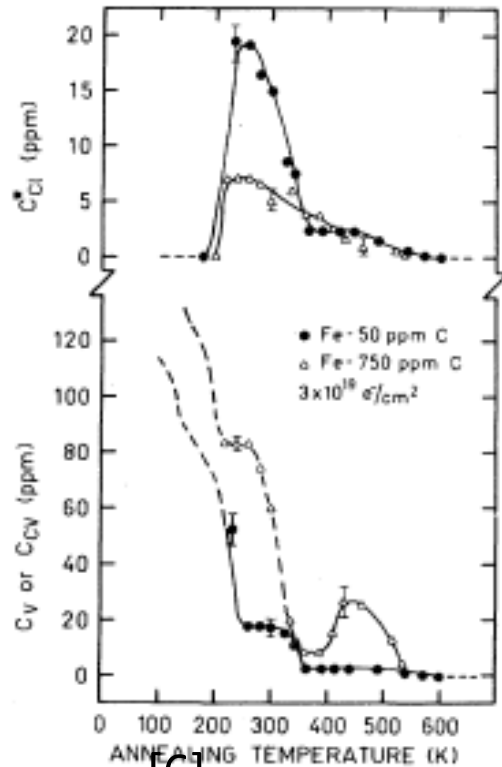
$$\rightarrow K_{tot} \approx K_{cav} + K_T$$

For 500°C

$$K_{cav} = 3 \times 10^{11} \text{ s}^{-1}. \text{ using } \mu_{cav} = 1.6 \times 10^{-13} \text{ m}^3 \cdot \text{s}^{-1} \text{ [1]}$$

$$\rightarrow K_T = 5 \times 10^{11} \text{ s}^{-1}. = C_T \times \mu_T \text{ if } \mu_T = \mu_V = 6 \pm 3 \times 10^{-15} \text{ m}^3 \cdot \text{s}^{-1} \text{ [1]} \rightarrow C_T \approx 7 \times 10^{25} \text{ m}^{-3} \text{ (i.e. about 1200 at. ppm).}$$

[1] Hautojarvi P., Corbel C., Positron Spectroscopy of Defects in Metals and Semiconductors, Proc. Int. Sch. Phys. Enrico Fermi. 125 (1995) 491–532.



[6]

For lower C concentration

→ Higher concentration of V in  $V_N$  clusters

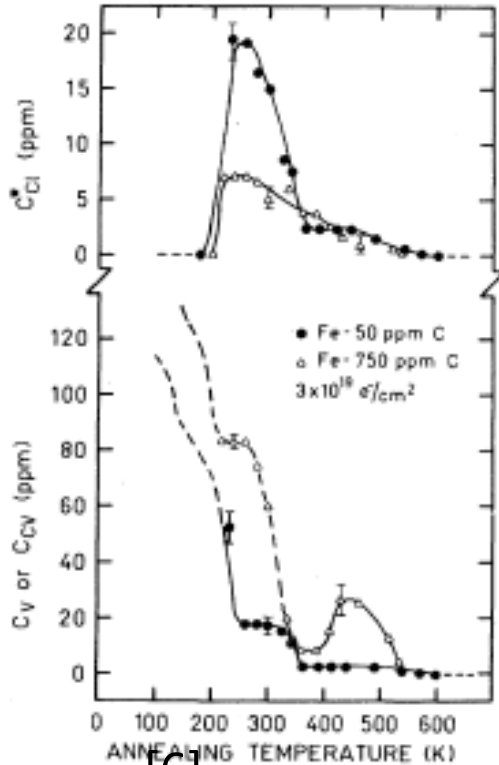
→ Carbon trapping at V prevent agglomeration

→ Lower concentration V-C complexes

➤ Larger cavities in **6N-XHP** samples compare to **3N-HP**

As in carbon doped iron [6]

[1] N. Castin, et al J. Nucl. Mater. 527 (2019); 1 .Hirai, T. et al.. Nuclear Materials and Energy 9, 616–622 (2016). 2 Greenwood.al 212–215, 635–639 (1994). 3 *Computational Materials Science* **184**, 109932 (2020). 3 Liu. *Computational Materials Science* 50, 3213–3217 (2011). 4 You, Y.-W. et al.. RSC Adv. 5, 23261–23270 (2015). 5 Alkhamees, A. et al.. *Journal of Nuclear Materials* 393, 508–512 (2009). 6 A. Vehanen et al Phys Rev B 25 (1982) 762



[6]

For lower C concentration

➤ Larger cavities in **6N-XHP** samples compare to **3N-HP**

As in carbon doped iron [6]

➔ Higher concentration of V in  $V_N$  clusters

➔ Carbon trapping at V prevent agglomeration

➔ Lower concentration V-C complexes

Irradiation at 500°C

X	at. ppm		$E_m^X$ (eV)	$E_b^{V-X_1}$ (eV)	$E_{diss}^{V-X_1}$ (eV)
	3N-HP	6N-XHP			
<del>H</del>	910	Ud	0.21 (TIS-TIS) [24]	1.24 [25]	<del>1.45</del>
<b>C</b>	460	Ud	1.46 (TIS-OIS) [20]	1.93 [20]	<b>3.39</b>
<b>N</b>	130	Ud	0.73 (TIS-OIS) [26]	2.48 [21]	3.21
<b>O</b>	345	Ud	0.17 (TIS-TIS) [22]	3.05 [22]	3.22

**In W**

T? = vacancy complexes with C,O, N

[1] N. Castin, et al J. Nucl. Mater. 527 (2019); 1 .Hirai, T. et al.. Nuclear Materials and Energy 9, 616–622 (2016). 2 Greenwood.al 212–215, 635–639 (1994). 3 *Computational Materials Science* **184**, 109932 (2020). 3 Liu. *Computational Materials Science* 50, 3213–3217 (2011). 4 You, Y.-W. et al.. *RSC Adv.* 5, 23261–23270 (2015). 5 Alkamees, A. et al.. *Journal of Nuclear Materials* 393, 508–512 (2009). 6 A. Vehanen et al *Phys Rev B* 25 (1982) 762

TEM / PAS (slow positron beams + lifetime and Doppler) complementary techniques to characterize vacancy defects from single to clusters

→ PAS more adapted for single vacancy or small size complexes detection

## ▪ Self Damage

✓ In RT W irradiated W : small vacancy clusters (TEM) + a majority of single vacancies (PAS)

✓ Saturation of S and W characteristics

✓ Annealing leads to agglomeration of vacancies to form large vacancy clusters

→ damage saturation when damage dose  $>0.5$  dpa as in modeling due to point defect recombination and cascade overlapping

✓ In 500°C W irradiated W :

✓ **Cavities distribution depends on W purity**

✓ **Larger vacancy clusters** (PAS and TEM) for 6N-XHP

✓ A new trap **T?**

→  $T = V\text{-Imp} (C, O, N)$

Perspectives : DFT calculations in collaboration with KTH (SW)

SIMS analysis



# Thank you for your attention

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