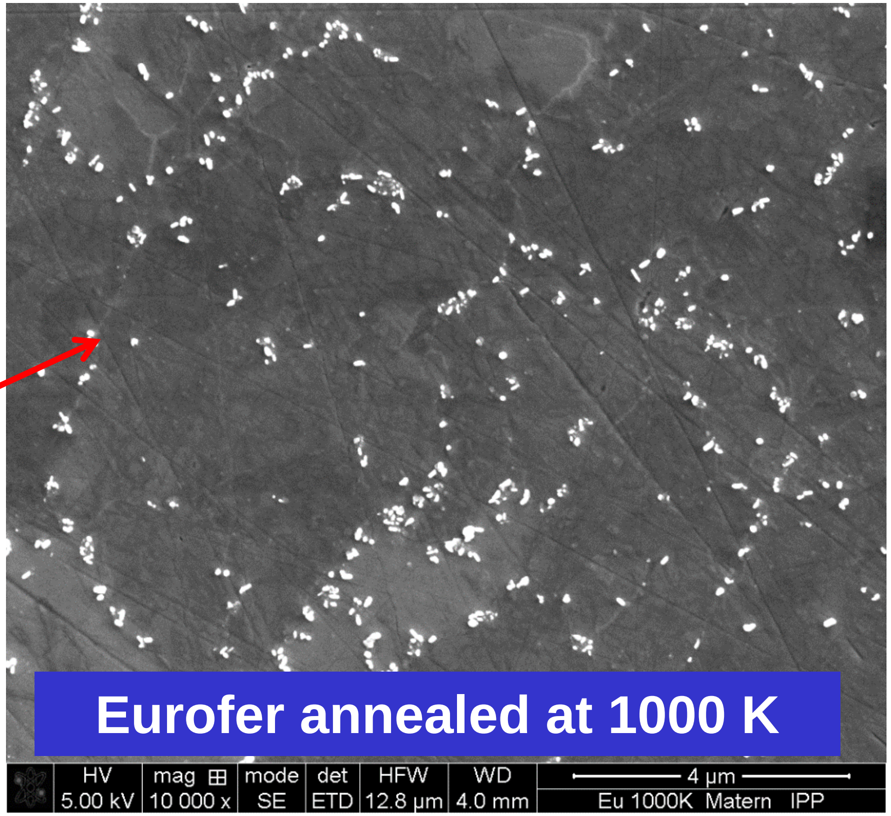
Interaction of low energy deuterium plasma with reduced-activation ODS steels irradiated with 20 MeV W ions

O. V. Ogorodnikova[1], Z. Zhou[2], K. Sugyama[3], Yu. Gasparyan[1], V. Efimov[1], M. Balden[3]

[1]National Research Nuclear University MEPhI, Moscow, Russia
[2] University of Science and Technology Beijing, Beijing 100083, China
[3]Max-Planck Institute fuer Plasmaphysik, Garching, Germany

Samples: Eurofer 9Cr

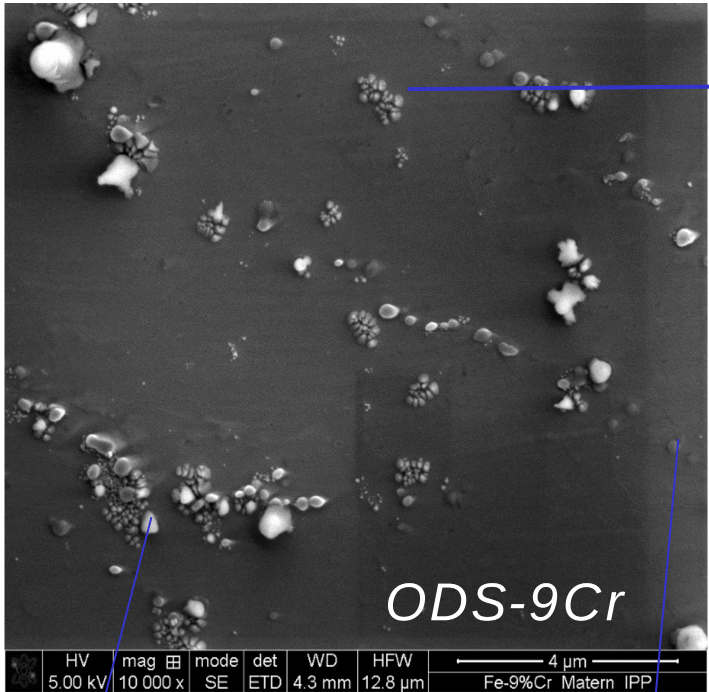
$M_{23}C_6$



Cr precipitates on grain boundaries

Eurofer: The precipitates are $M_{23}C_6$ which can contain tantalum, tungsten, chromium, vanadium etc. The density of precipitates on grain boundaries increases after annealing of Eurofer up to 1000 K.

ODS strengthened by dispersed oxides of Y, Ti or Al



SiO

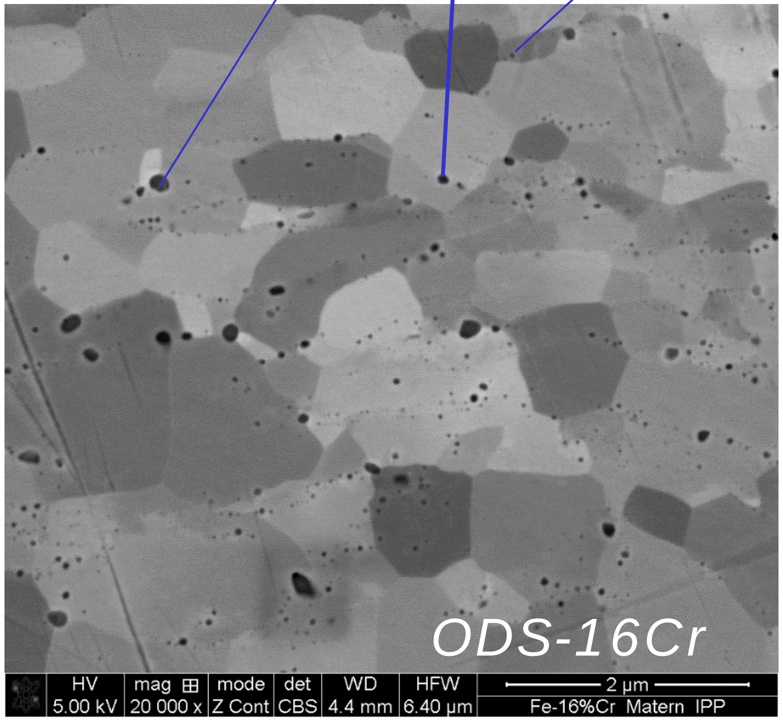
Contain Ti and some of them W

Very small dispersoids contain Y

Ti-Cr-W-Y

Ti-Cr-W

Ti-Cr-Y



ODS: **The structure is very inhomogeneous with Ti –Cr precipitates, probably, in the form of oxides, some of them contain Y (small ones), some W (big ones) and there is also SiO dispersoids (biggest ones)**

$Y_2Ti_2O_7$ (EDS, TEM): several nm, Y_2TiO_5

Material compositions

Sample	Compositions
ODS-9Cr	Fe-9Cr-1.5W-0.1C-0.15Si-0.5Ti-0.35Y_2O_3
ODS-12Cr	Fe-12Cr-2W-0.15Si-0.5Ti-0.35Y_2O_3
ODS-14Cr (Ti)	Fe-14Cr-2W-0.15Si-0.5Ti-0.35Y_2O_3
ODS-14Cr (Al)	Fe-14Cr-2W-0.15Si-4.0Al-0.35Y_2O_3
ODS-16Cr	Fe-16Cr-2W-0.15Si-0.5Ti-0.35Y_2O_3
Eurofer	Fe–9Cr–1W-0.1C-0.2V–0.14Ta–0.03N

- ***Fe-(9-12)Cr has ferritic/martensitic structure***
- ***Fe-(14-16)Cr has ferritic structure***

Pre-irradiation with 20 MeV W ions

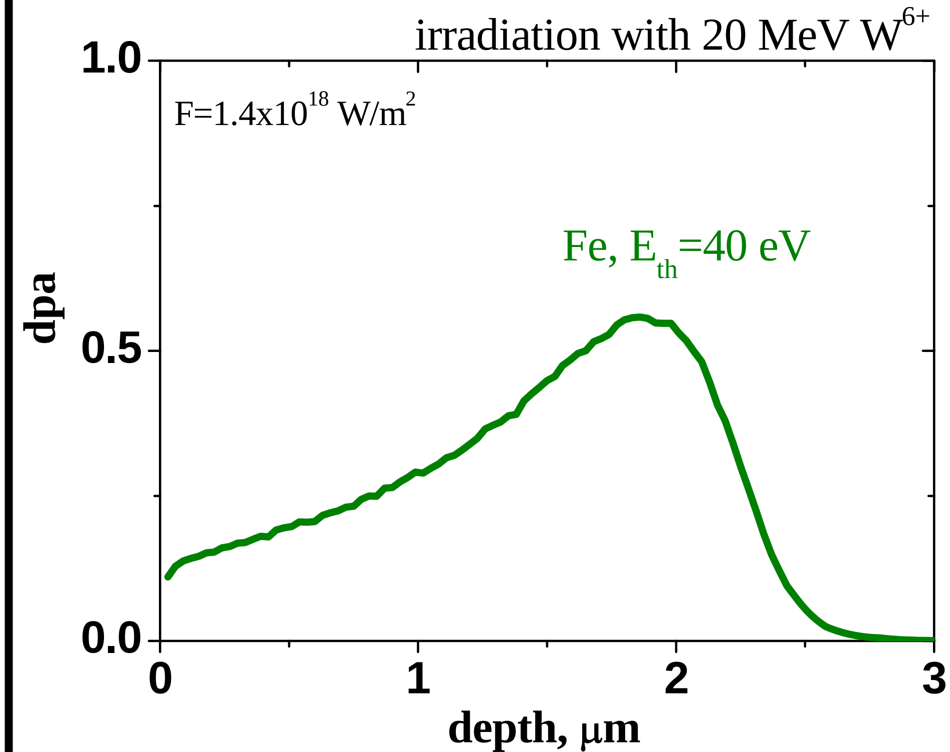
Self-ion irradiation was done at IPP

20 MeV W^{6+}, RT

Pre-irradiation with 20 MeV W ions

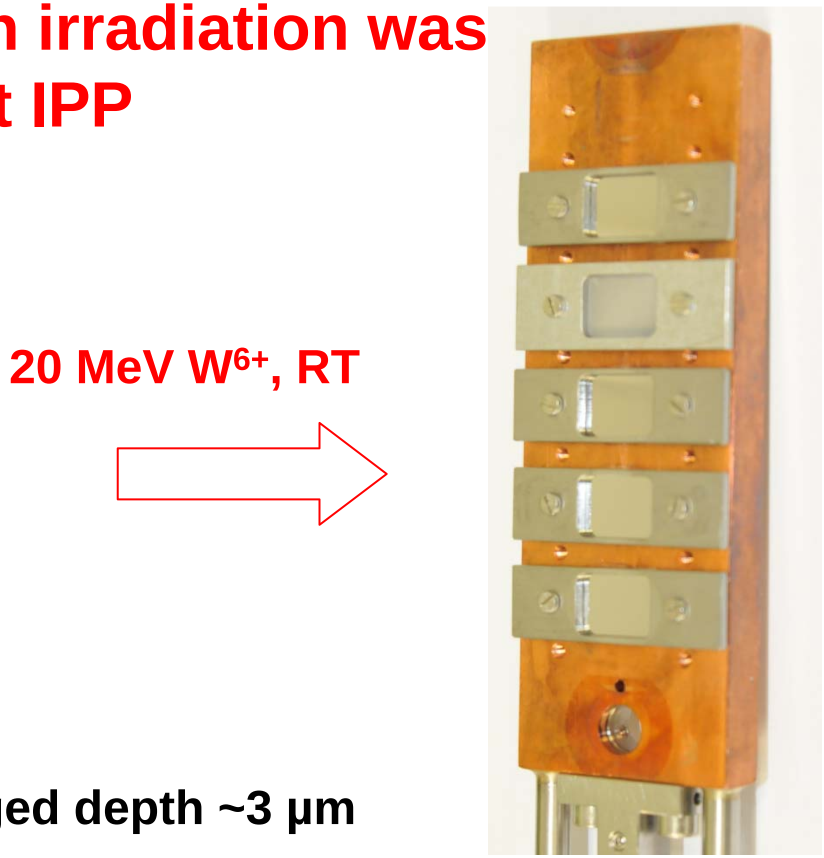
Self-ion irradiation was done at IPP

- **Damaged depth ~3 μm**
- **Beam sweep unit for homogeneous irradiation of larger areas (20 mm)**
- **Well defined fluence control by 4 corner Farraday cups**

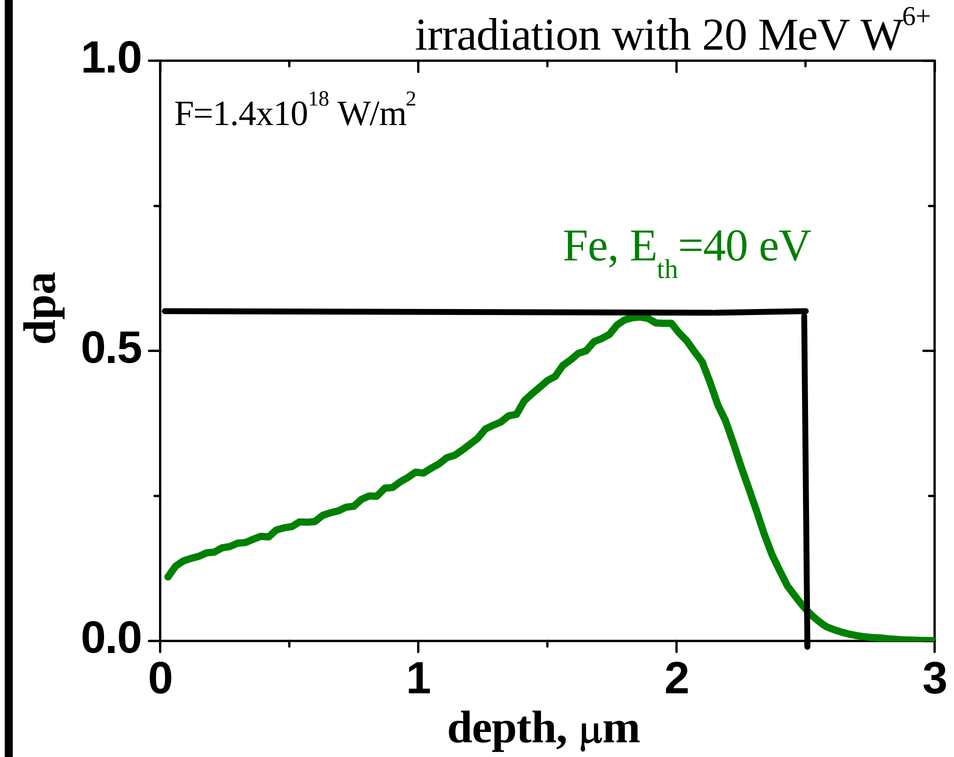


Pre-irradiation with 20 MeV W ions

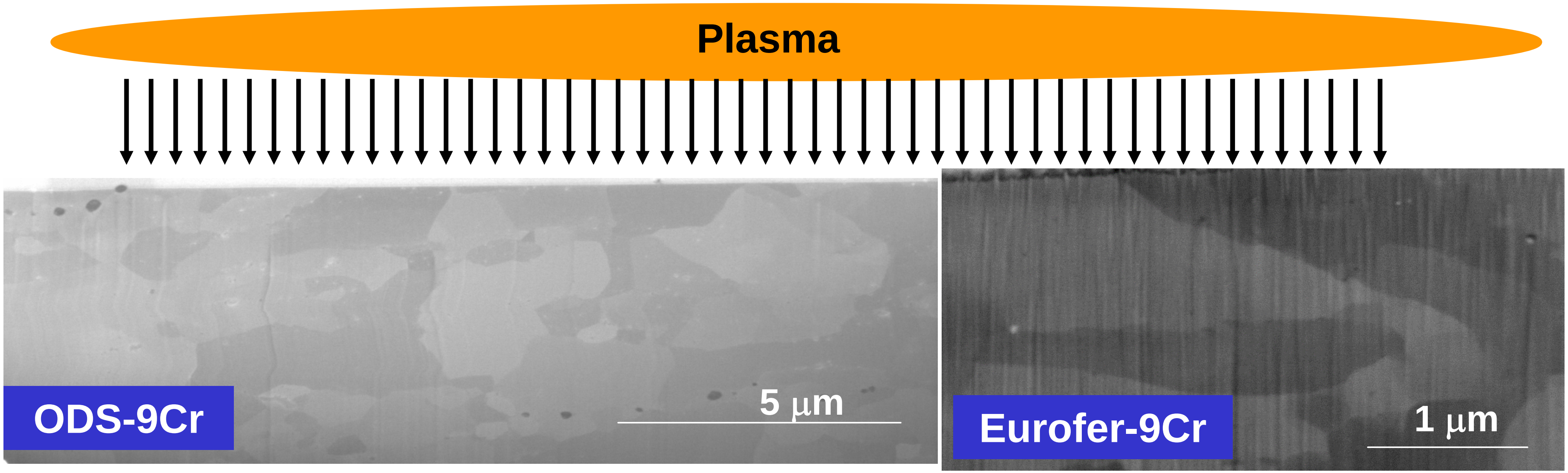
Self-ion irradiation was done at IPP



- **Damaged depth ~3 μm**
- **Beam sweep unit for homogeneous irradiation of larger areas (20 mm)**
- **Well defined fluence control by 4 corner Farraday cups**

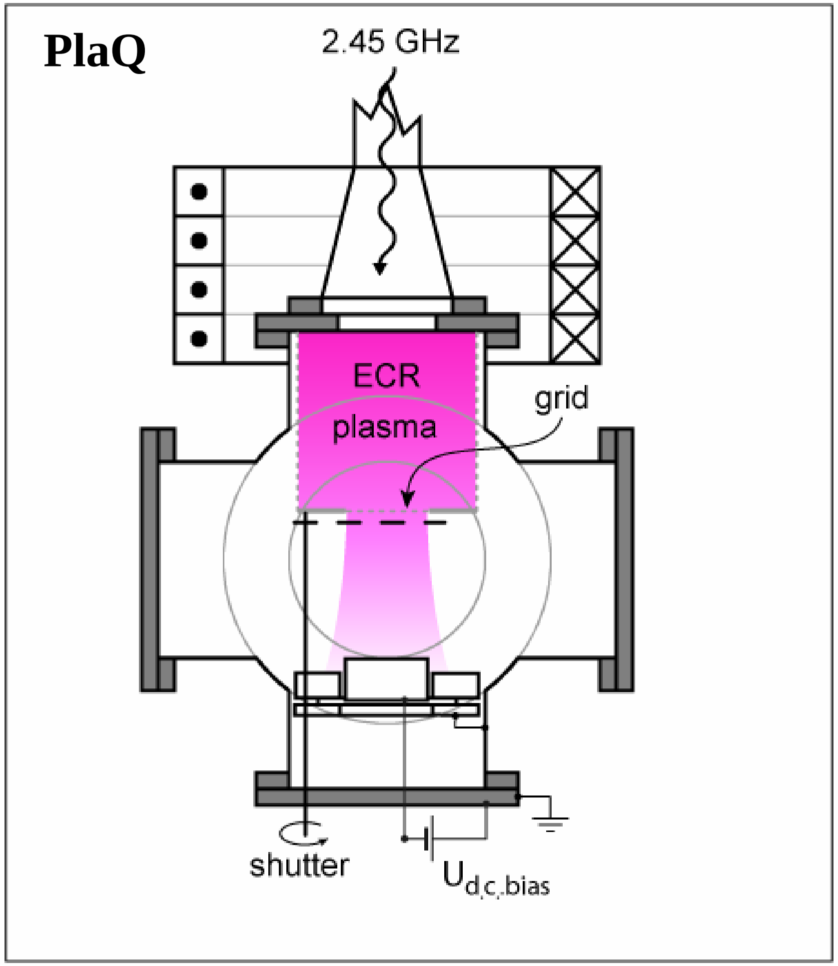


Plasma exposure: undamaged and damaged samples



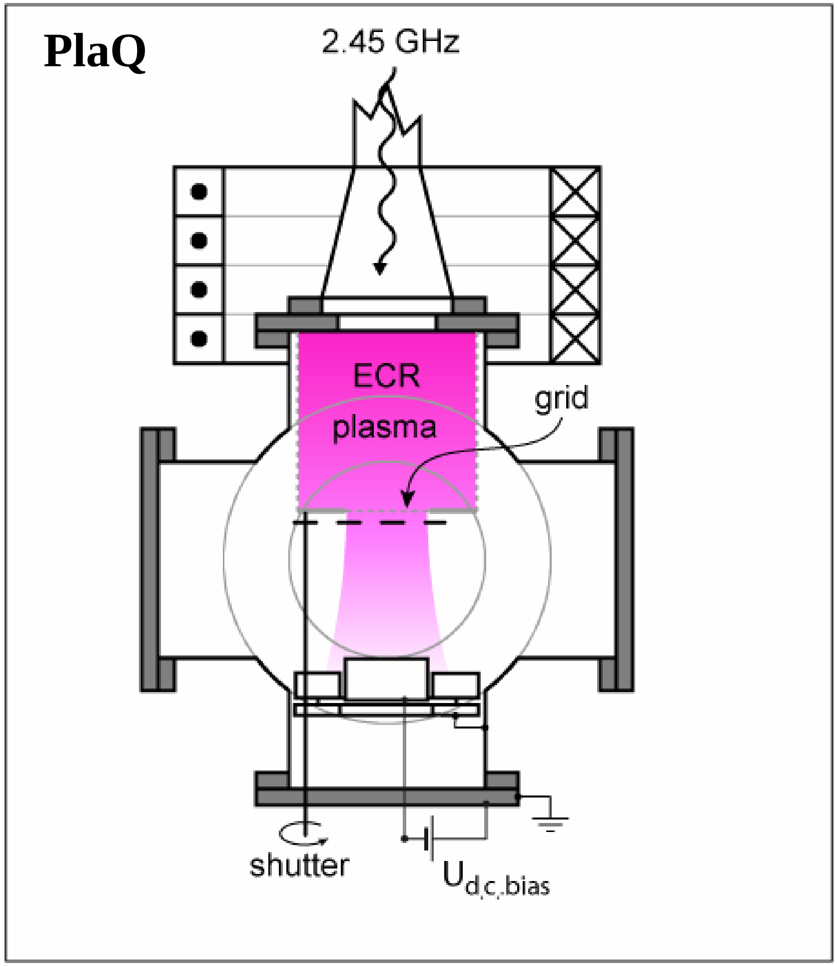
Cross section by FIB

Exposure of samples to D plasma



- **Ion flux:~10^{20} D/m^{2}s**
- **Ion energies: 20-200 eV D^+**
- **Temperatures: 290 K - 700 K**
- **Fluence up to $3x10^{25}$ D/m^2**

Exposure of samples to D plasma

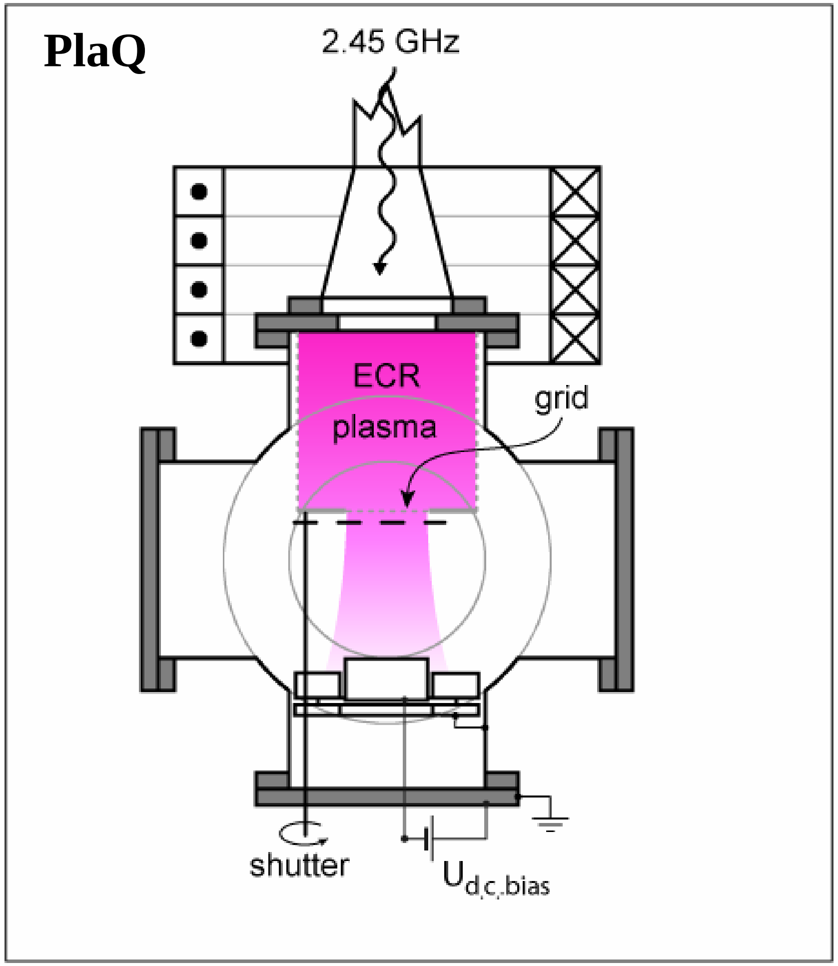


- **Ion flux:~10^{20} D/m^{2}s**
- **Ion energies: 20-200 eV D^+**
- **Temperatures: 290 K - 700 K**
- **Fluence up to 3×10^{25} D/m^2**

Post-morten analysis

1) HSEM: surface modification

Exposure of samples to D plasma



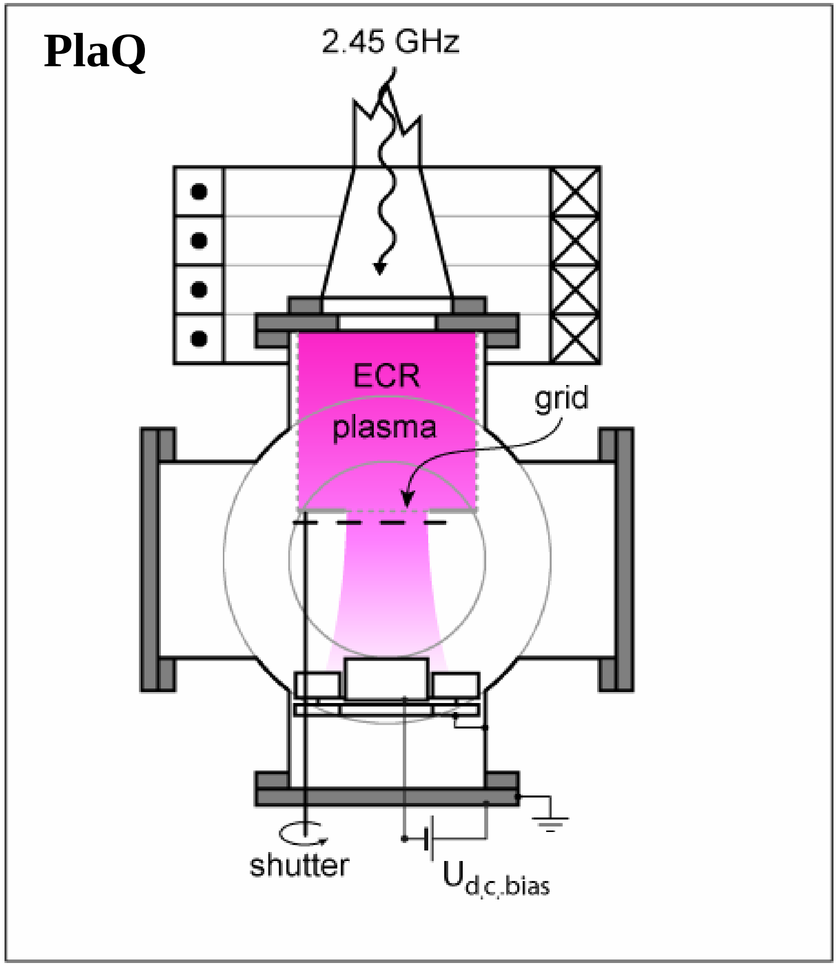
- **Ion flux:~10^{20} D/m^{2}s**
- **Ion energies: 20-200 eV D^+**
- **Temperatures: 290 K - 700 K**
- **Fluence up to 3x10^{25} D/m^2**

Post-morten analysis

1) HSEM: surface modification

2) NRA: Depth profile of D up to 6 μm

Exposure of samples to D plasma

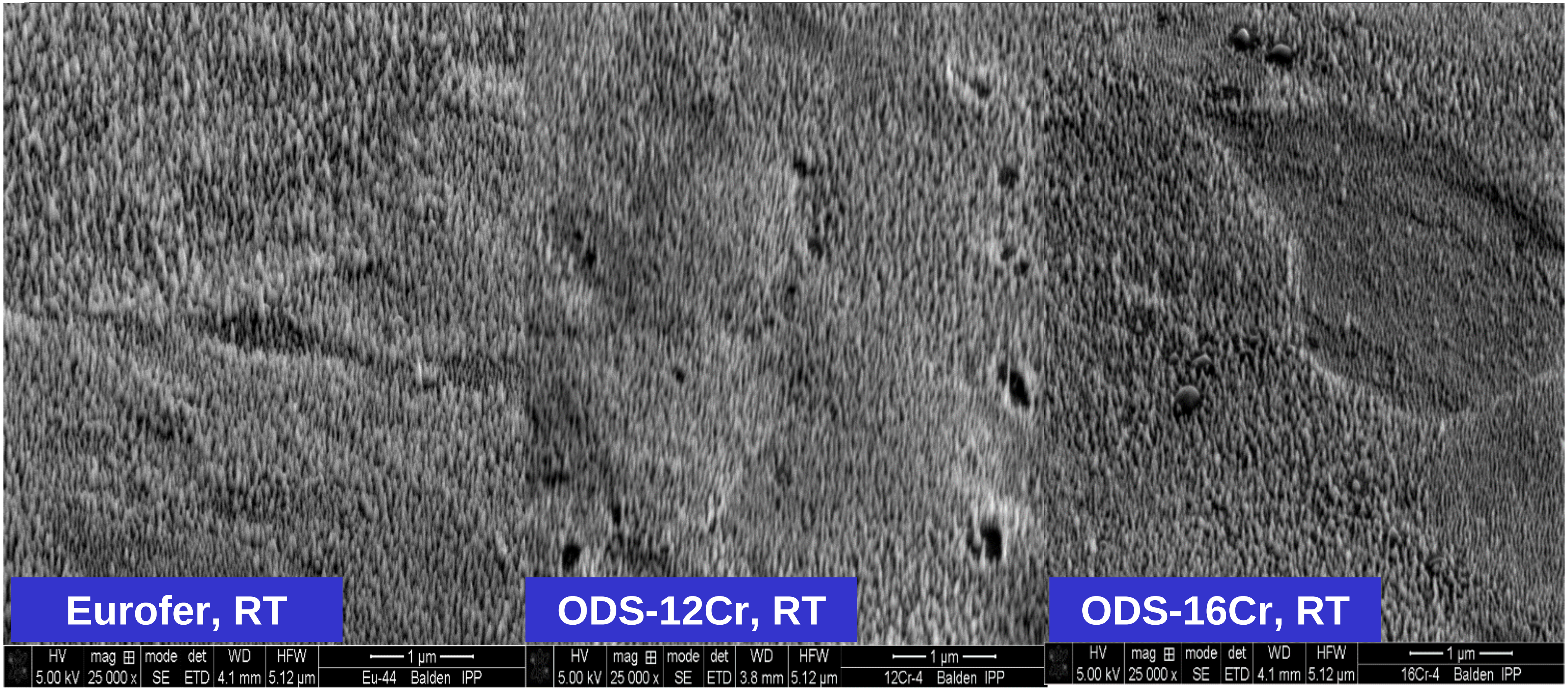


- **Ion flux:~10^{20} D/m^{2}s**
- **Ion energies: 20-200 eV D^+**
- **Temperatures: 290 K - 700 K**
- **Fluence up to $3x10^{25}$ D/m^2**

Post-morten analysis

1) HSEM: surface modification
2) NRA: Depth profile of D up to 6 μm
3) TDS: Total D retention and kinetics

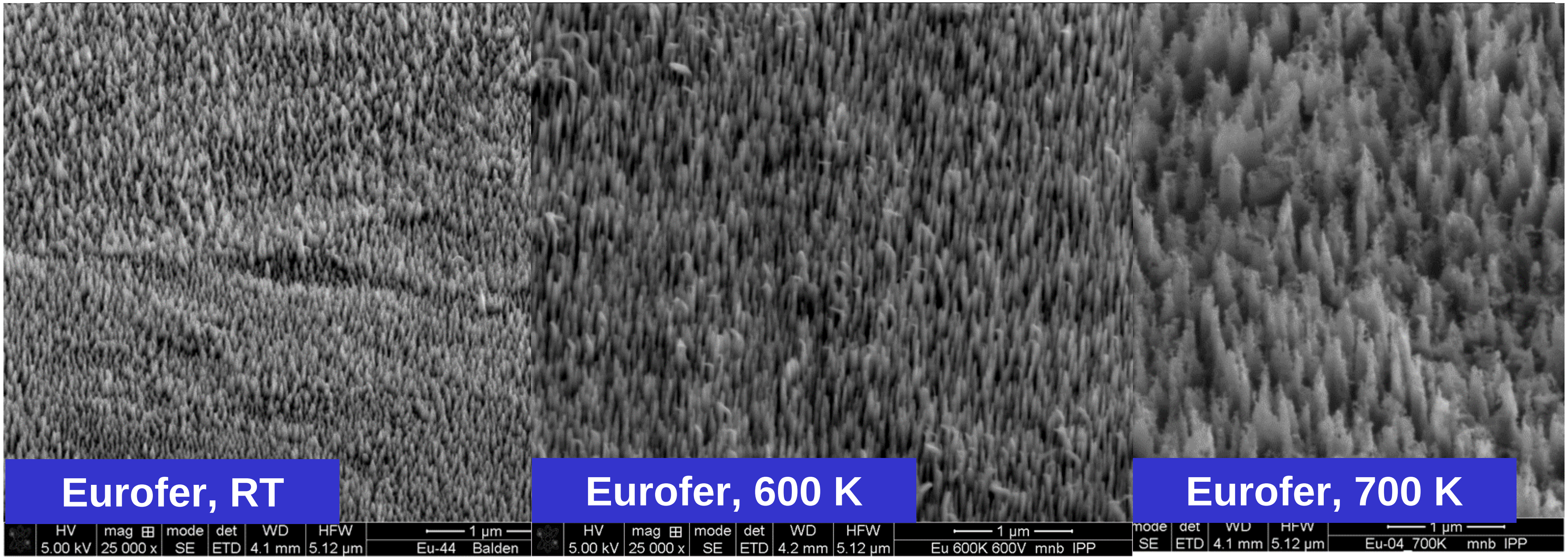
Surface morphology: Eurofer vs ODS, 200 eV



Nano-roughness modification (fuzz-like) due to preferential sputtering of light elements: cylindrical cones enriched in W or Ta (EDS and RBS).

No visible difference in surface modification for different RAMF steels and for pre-damaged samples was observed.

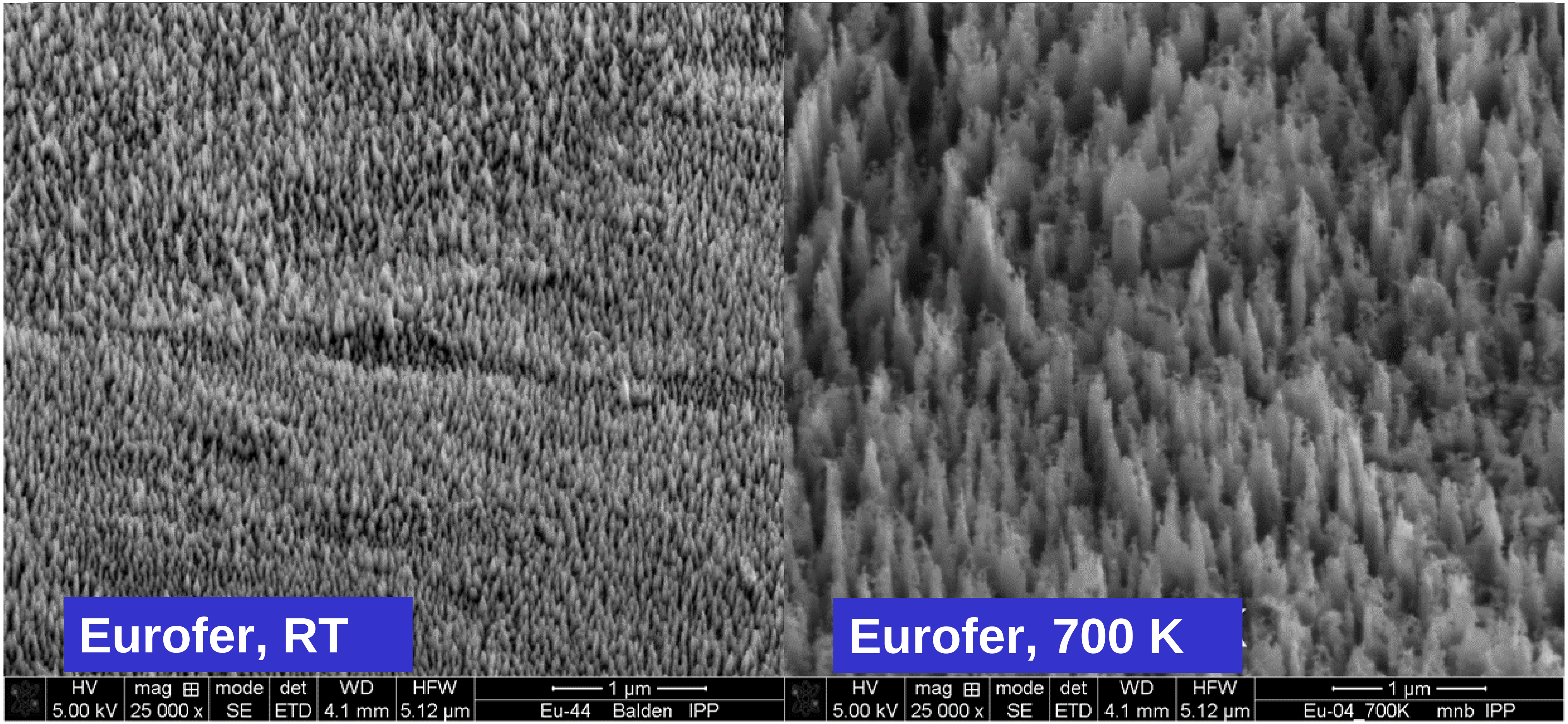
Surface morphology: temperature effect, 200 eV



Increase of nano-roughness (growth of ‘fuzz’) with increasing of the temperature

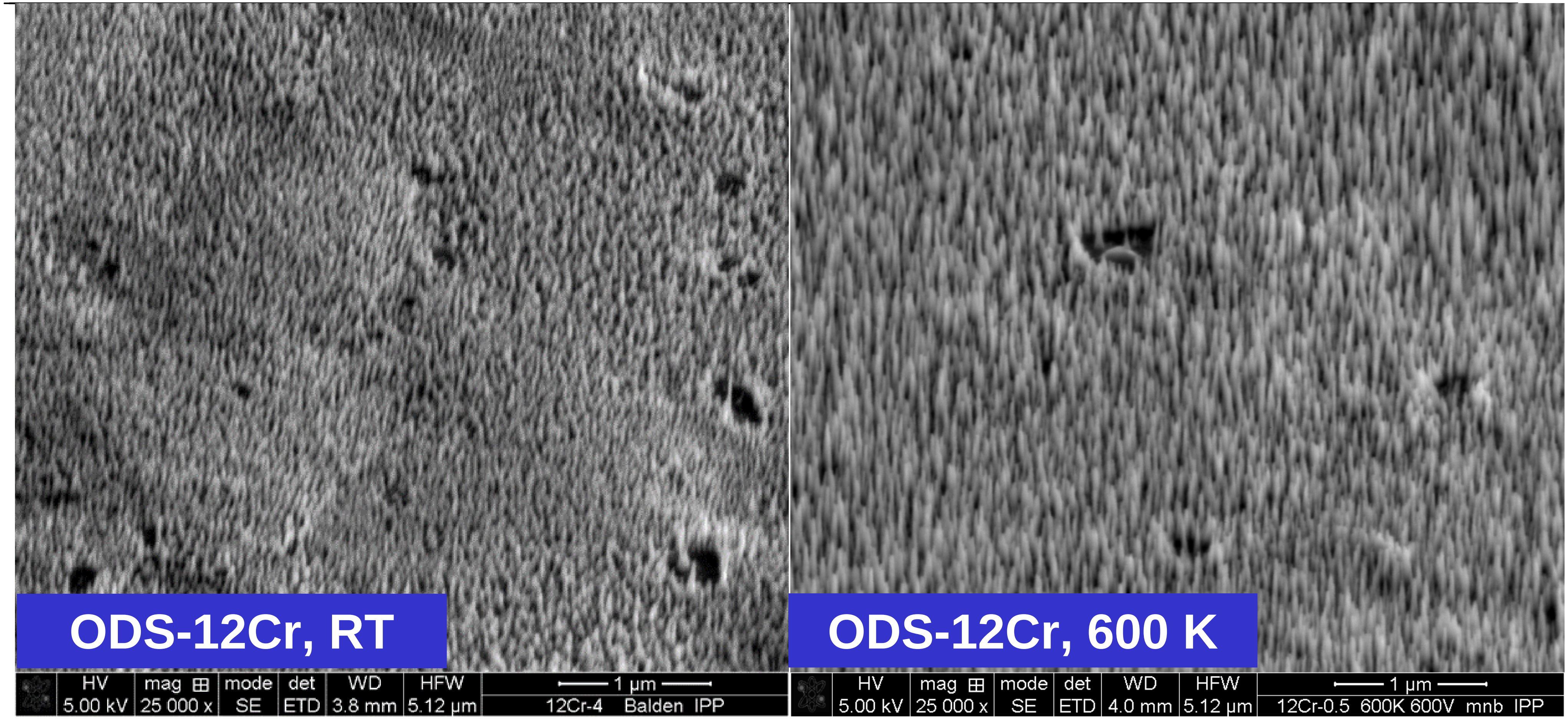
Increase of sputtering yield with increasing of the temperature (weaker bonds, migration of Fe and precipitates at 700 K).

Surface morphology: Eurofer, 200 eV



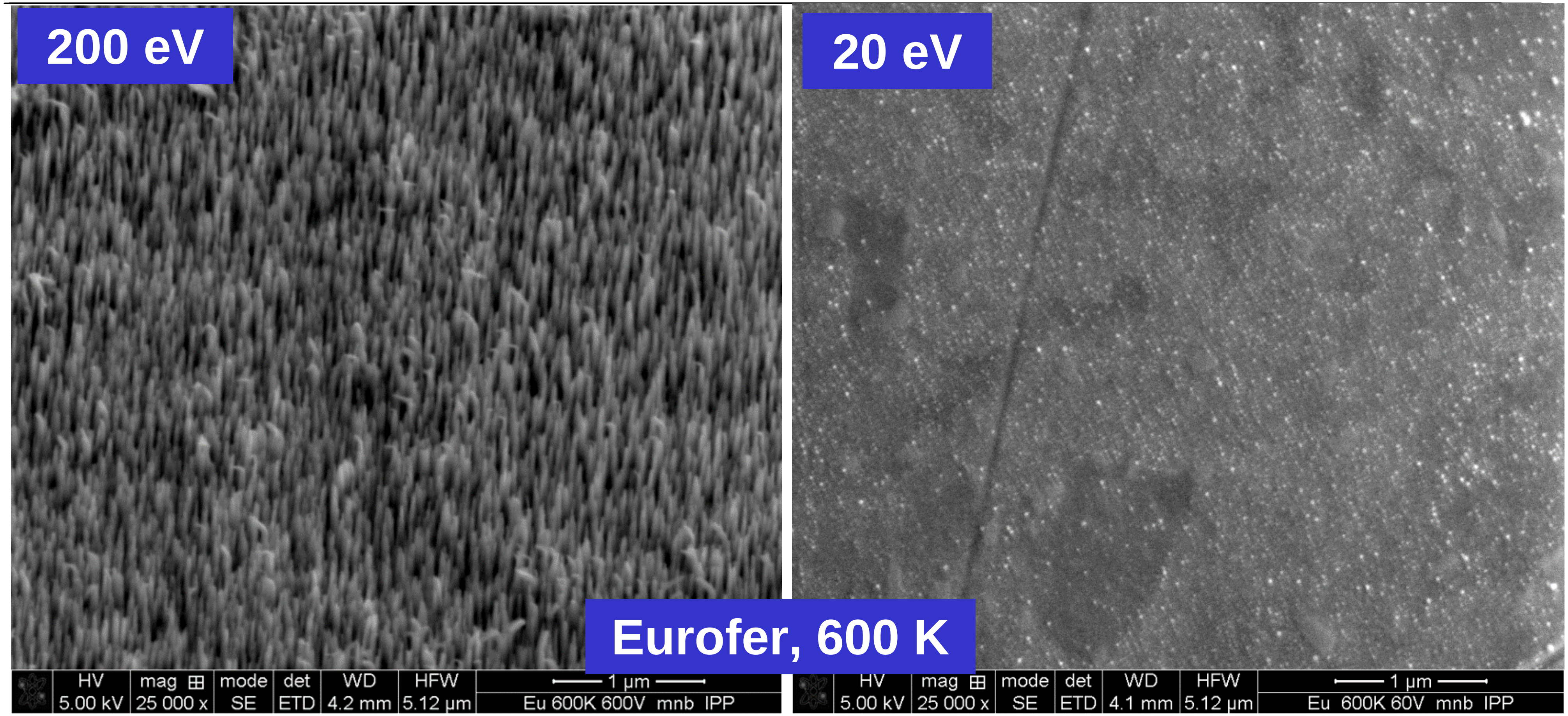
Sputtering yield = f(E_i, Tem)

Surface morphology: ODS, 200 eV



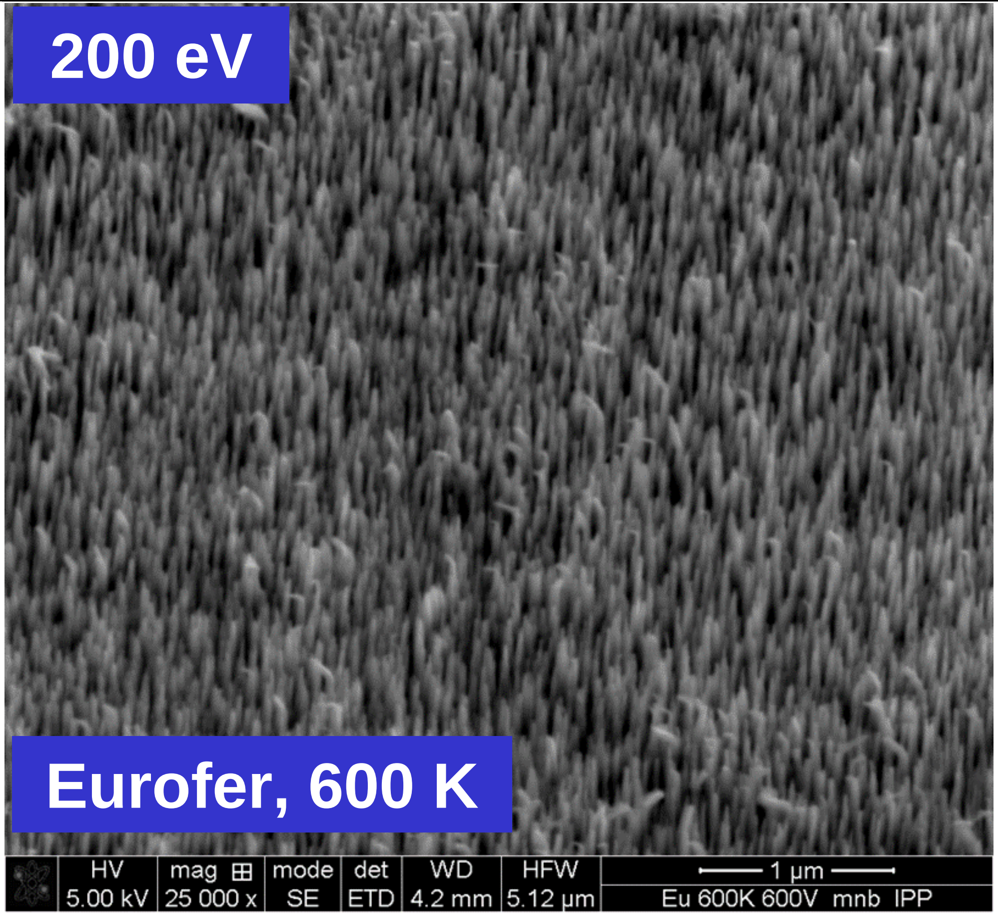
Sputtering yield = f(E_i, Tem)

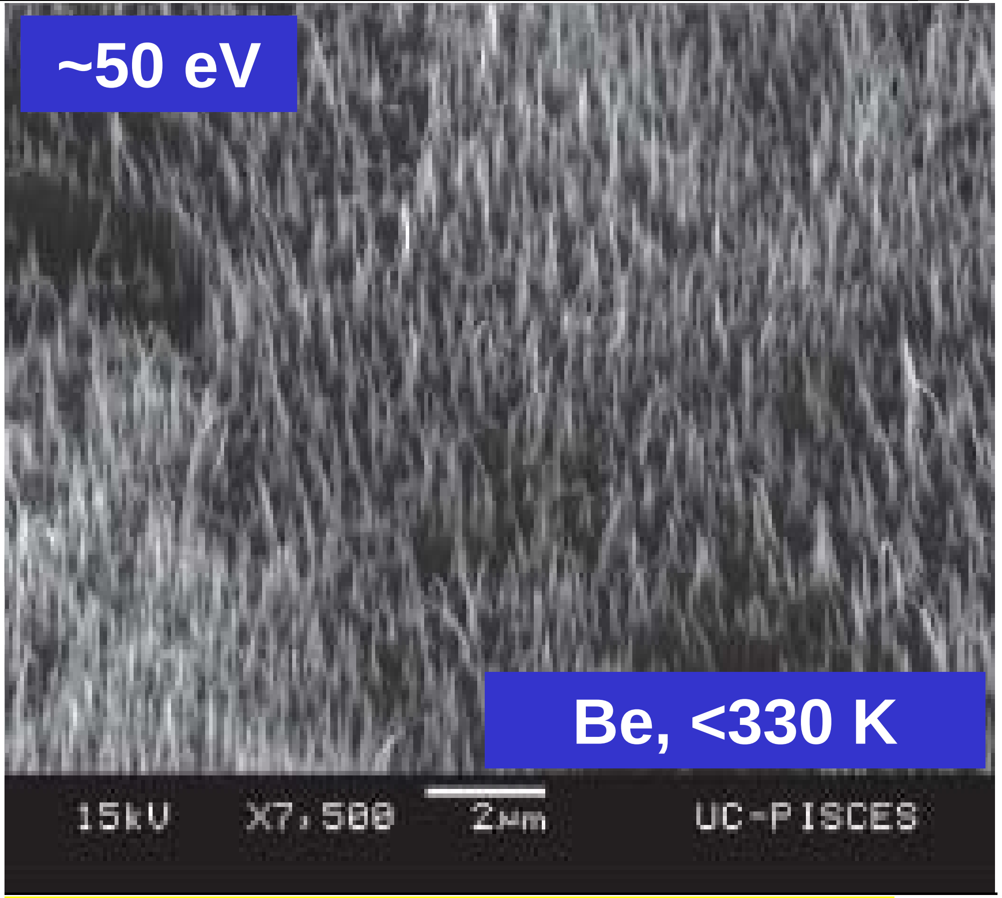
Surface morphology: energy effect



Even subthreshold D energy of 20 eV can cause sputtering and 'fuzz' growth at elevated temperatures

Comparison with Be in PISCES

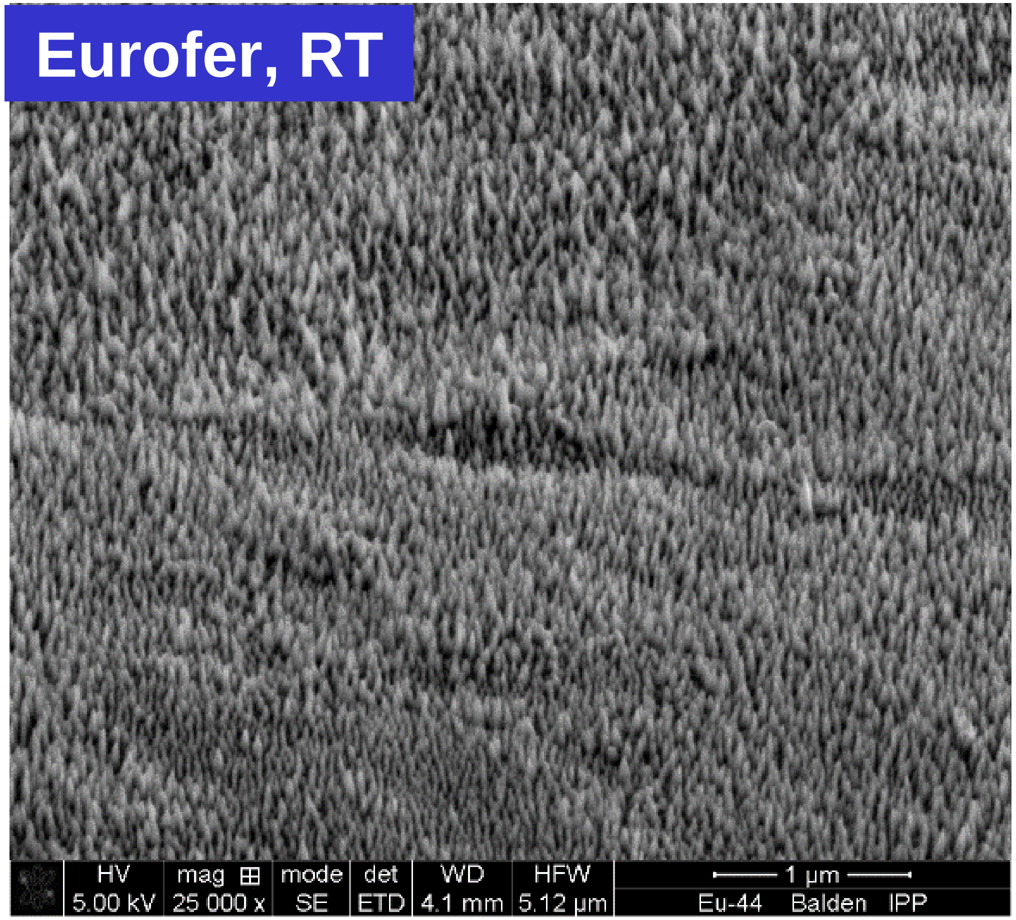


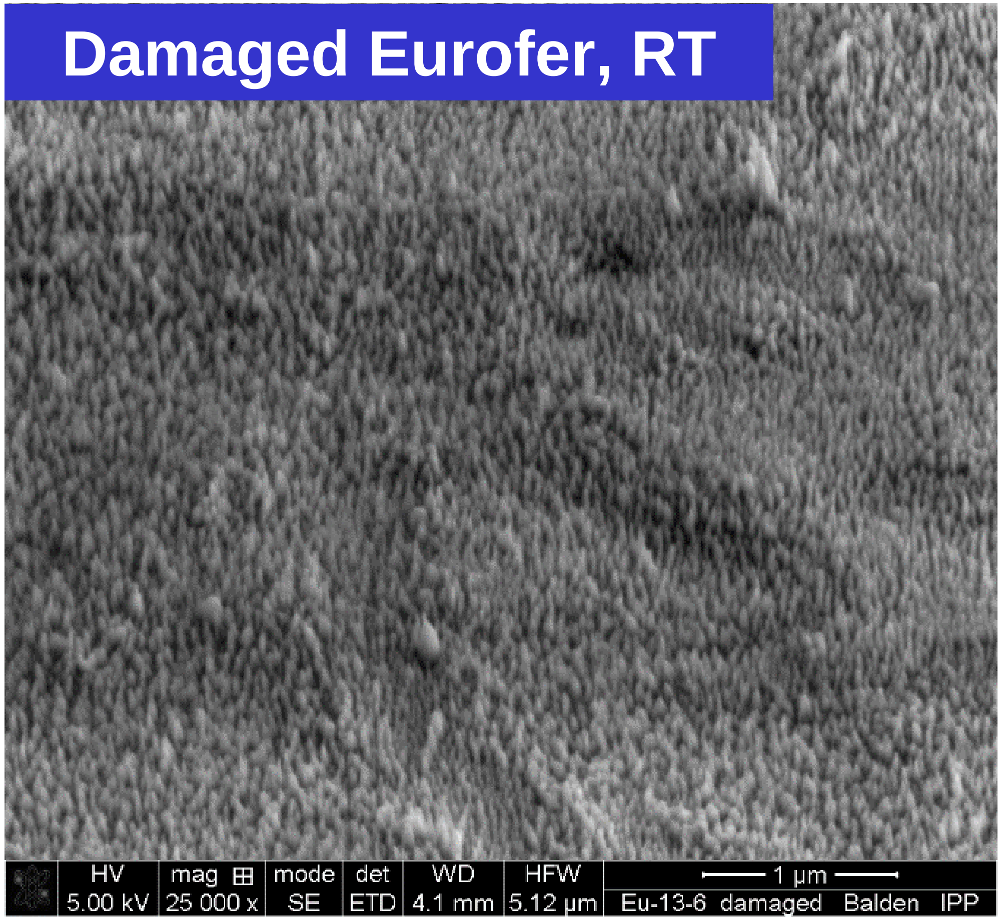


R. P. Doerner, 20th PSI Conference, Aachen, Germany, May 2012

Similar surface morphology was observed for Be in PISCES

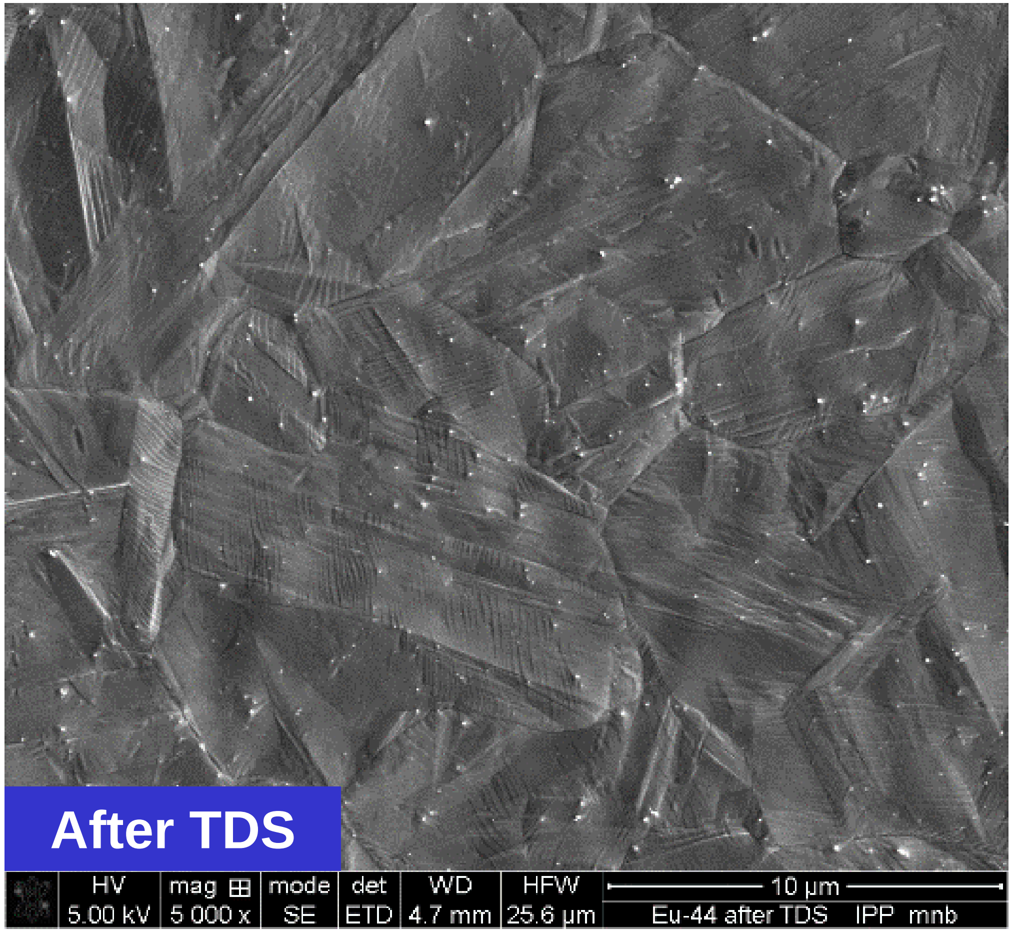
Annealing of 'fuzz'

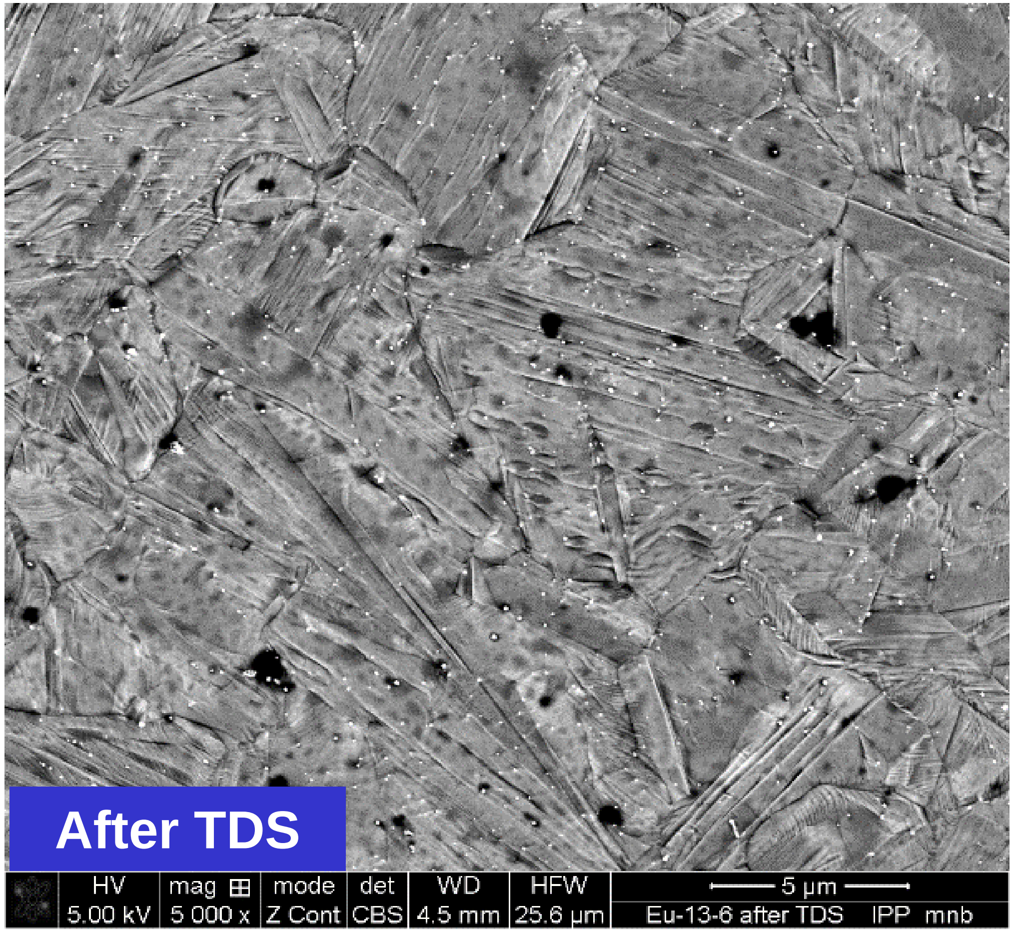




Stable at keeping at RT

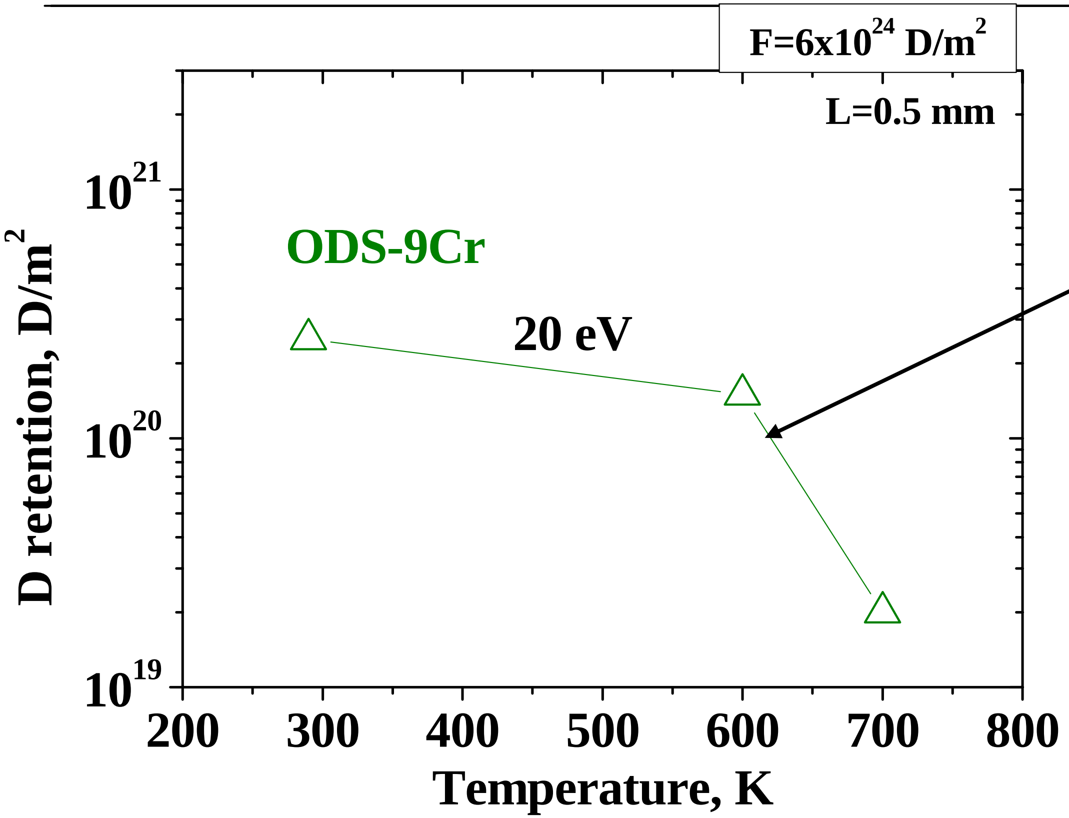
Annealing of ‘fuzz’

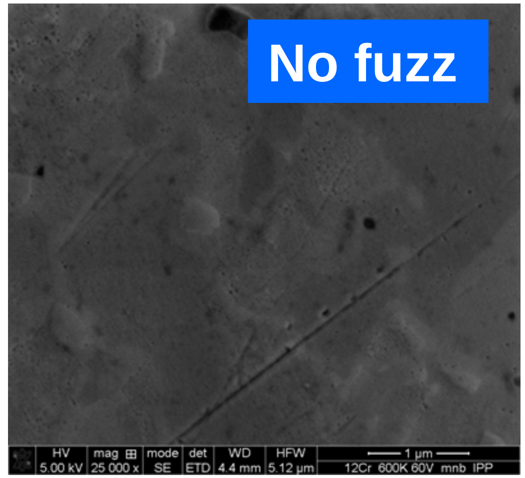




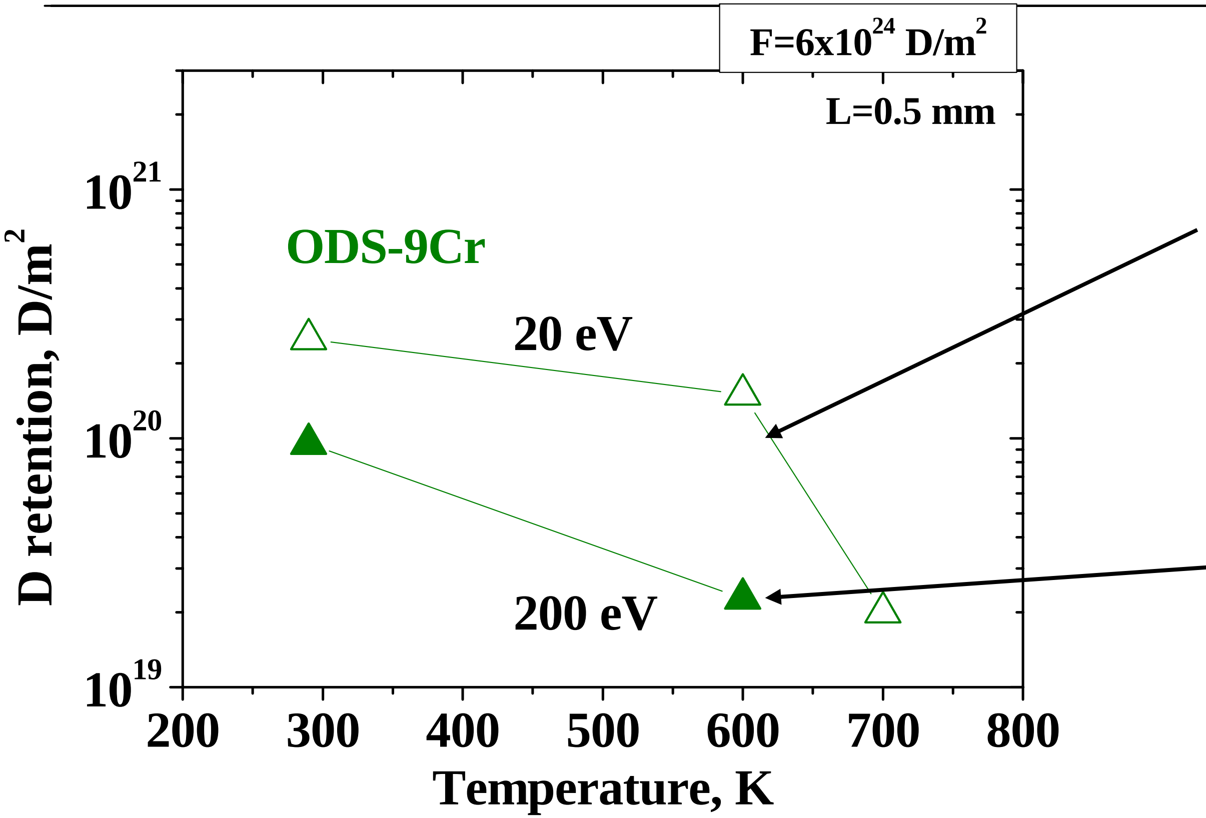
After annealing by TDS up to 1000 K, ‘fuzz’ disappear

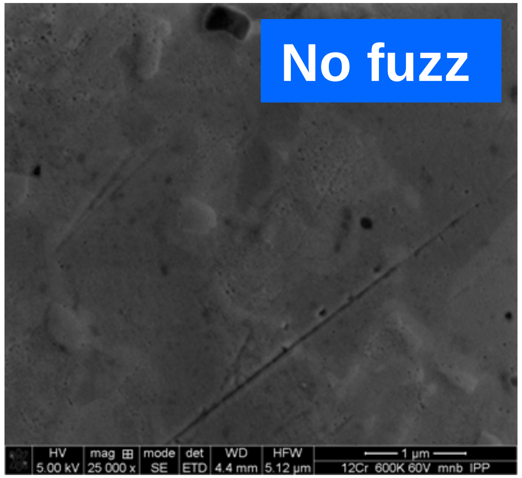
Does formation of 'fuzz' influence the D retention?

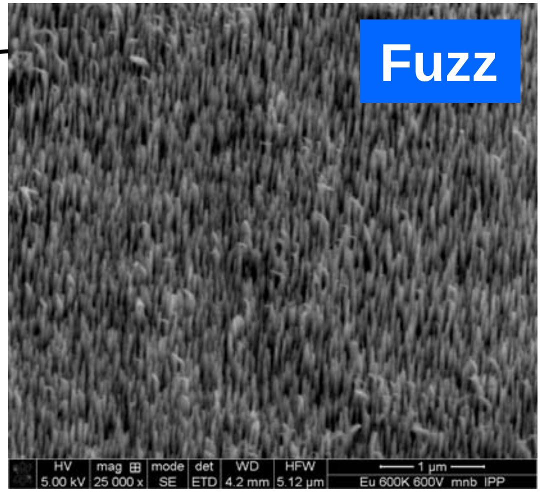




Does formation of ‘fuzz’ influence the D retention?

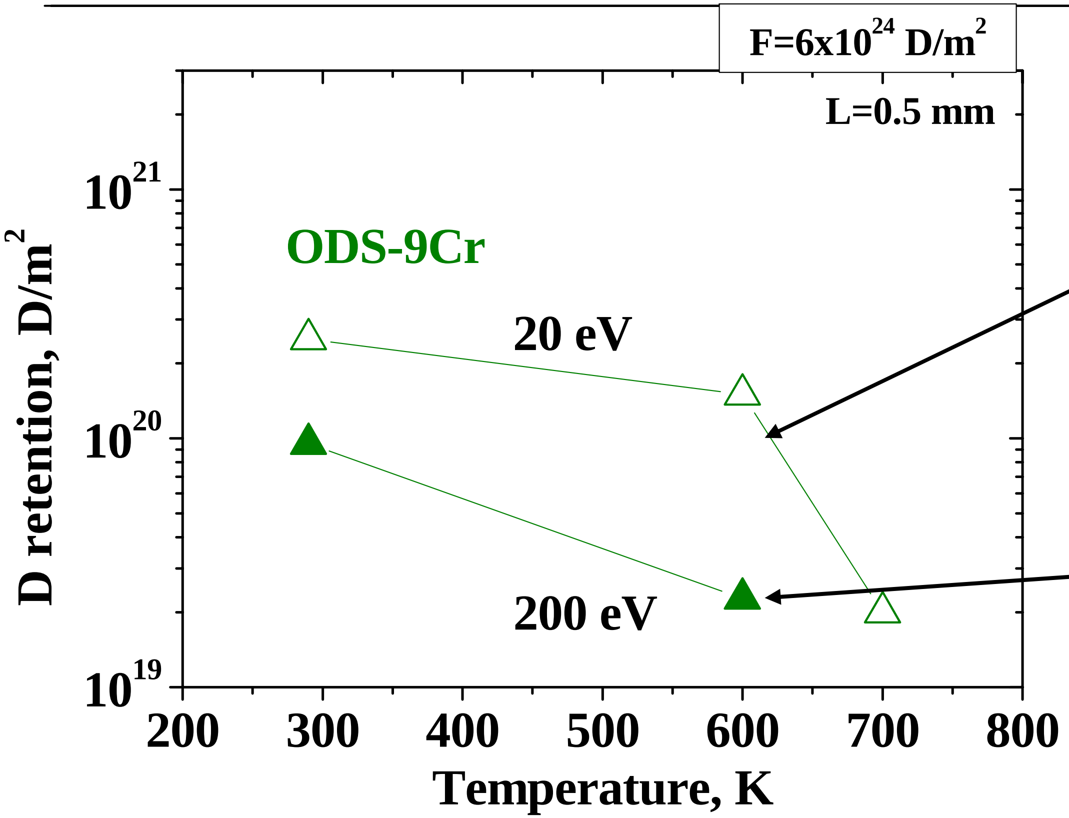


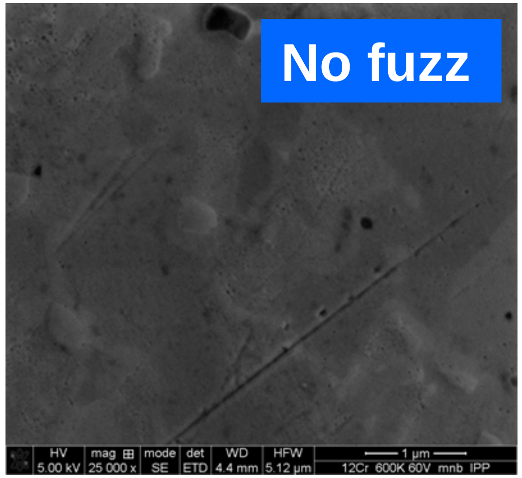


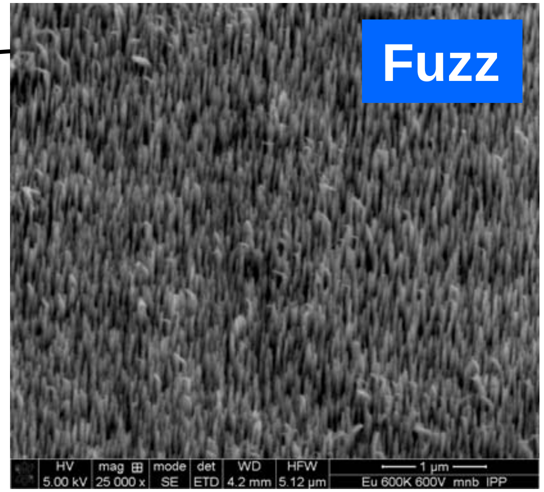


❖ **A reduction of the D retention in ODS with formation of ‘fuzz’**

Does formation of ‘fuzz’ influence the D retention?



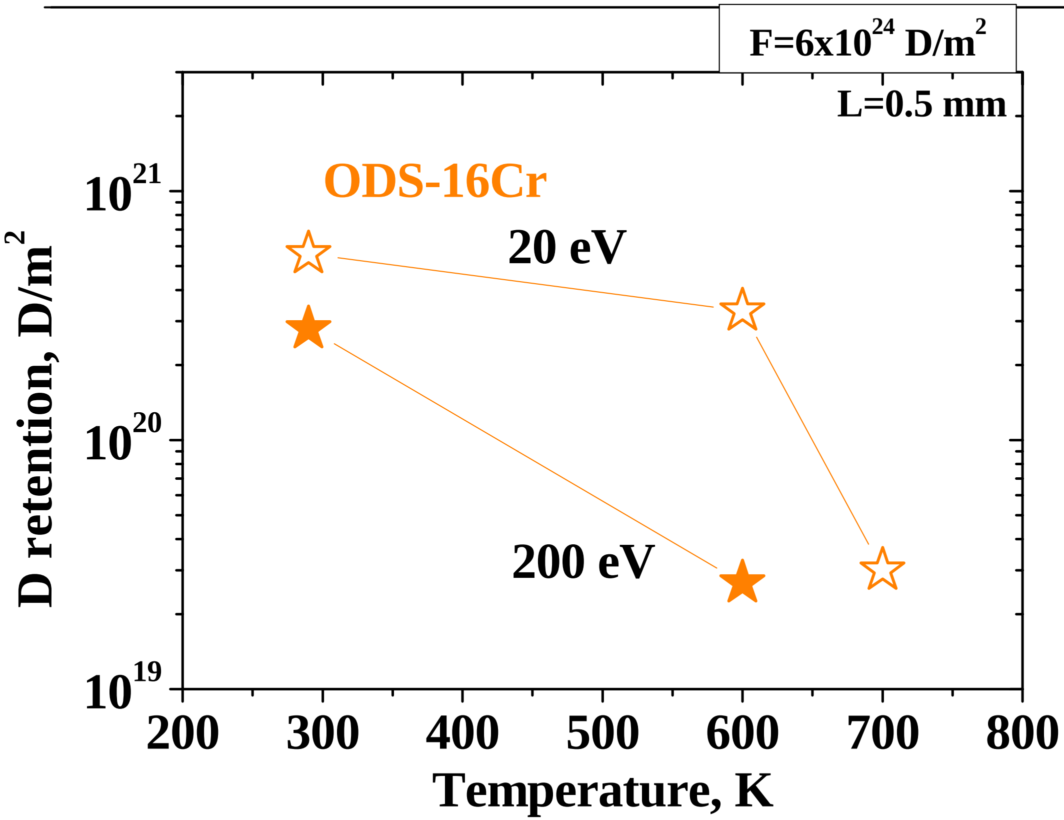




- ❖ A reduction of the D retention in ODS with formation of ‘fuzz’

$$Ret\_total = Ret\_surf + Ret\_bulk$$

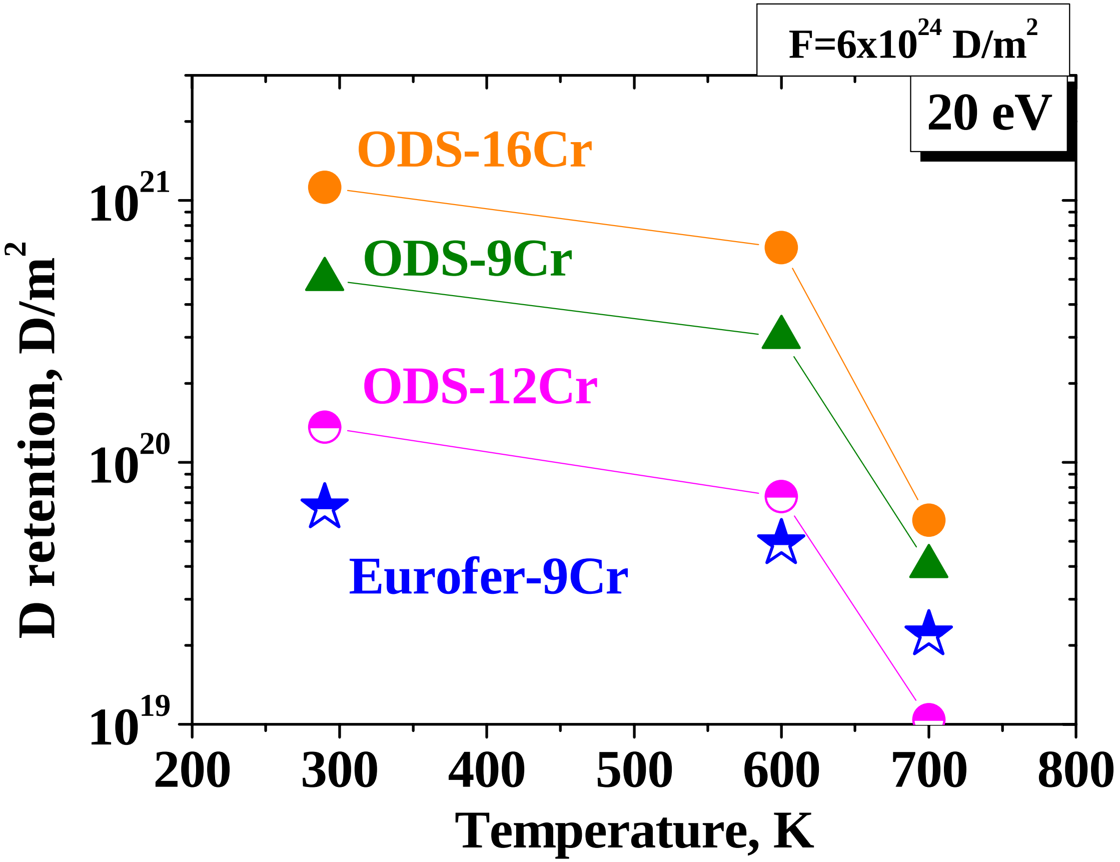
Does formation of ‘fuzz’ influence the D retention?



❖ **A reduction of the D retention in ODS with formation of ‘fuzz’**

Ret_total = Ret_surf + Ret_bulk

The D retention in undamaged Eurofer & ODS



❖ The D retention in ODS is higher compared to Eurofer

D retention

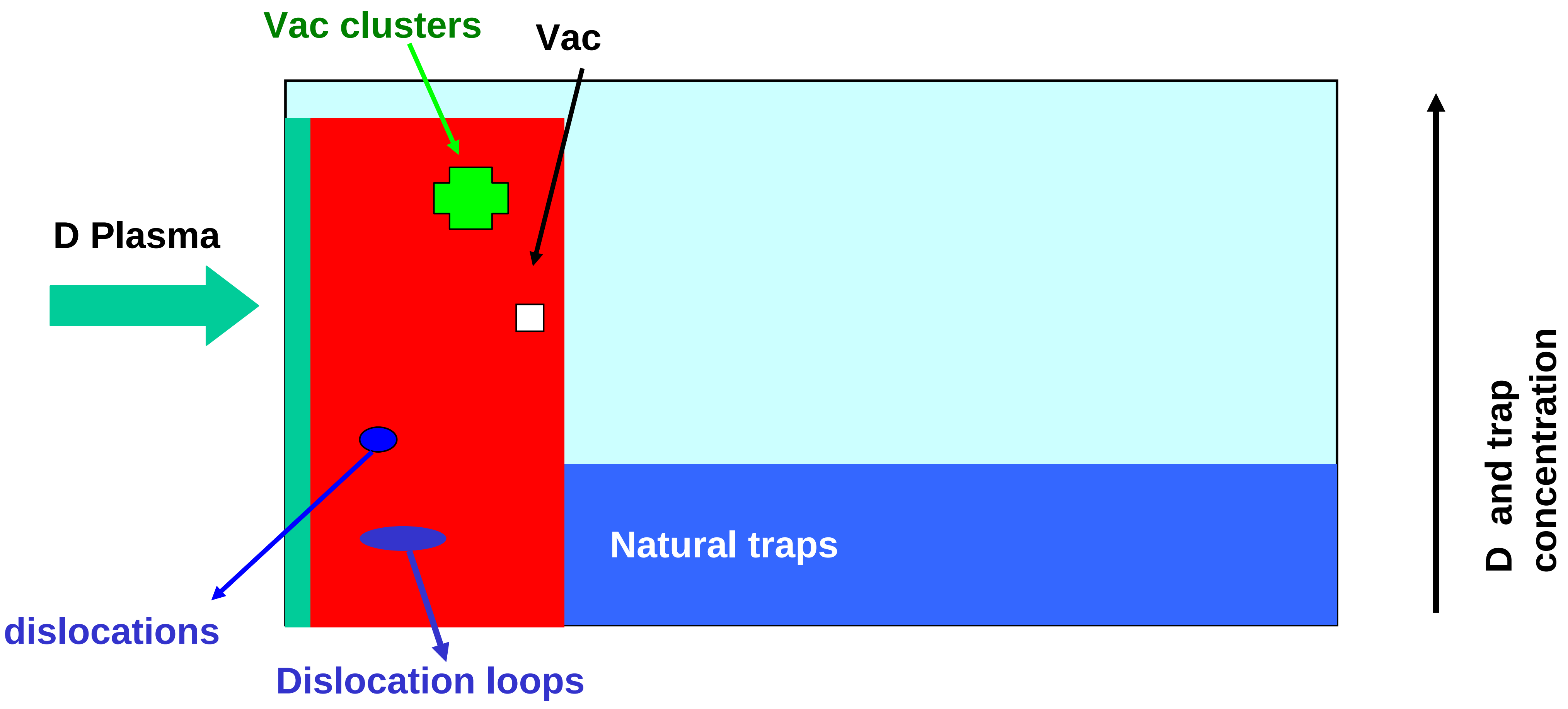
NRA: D depth profile measurement up to 6 mkm

TDS: total retention

Ret_TDS = Ret_surf + Ret_bulk

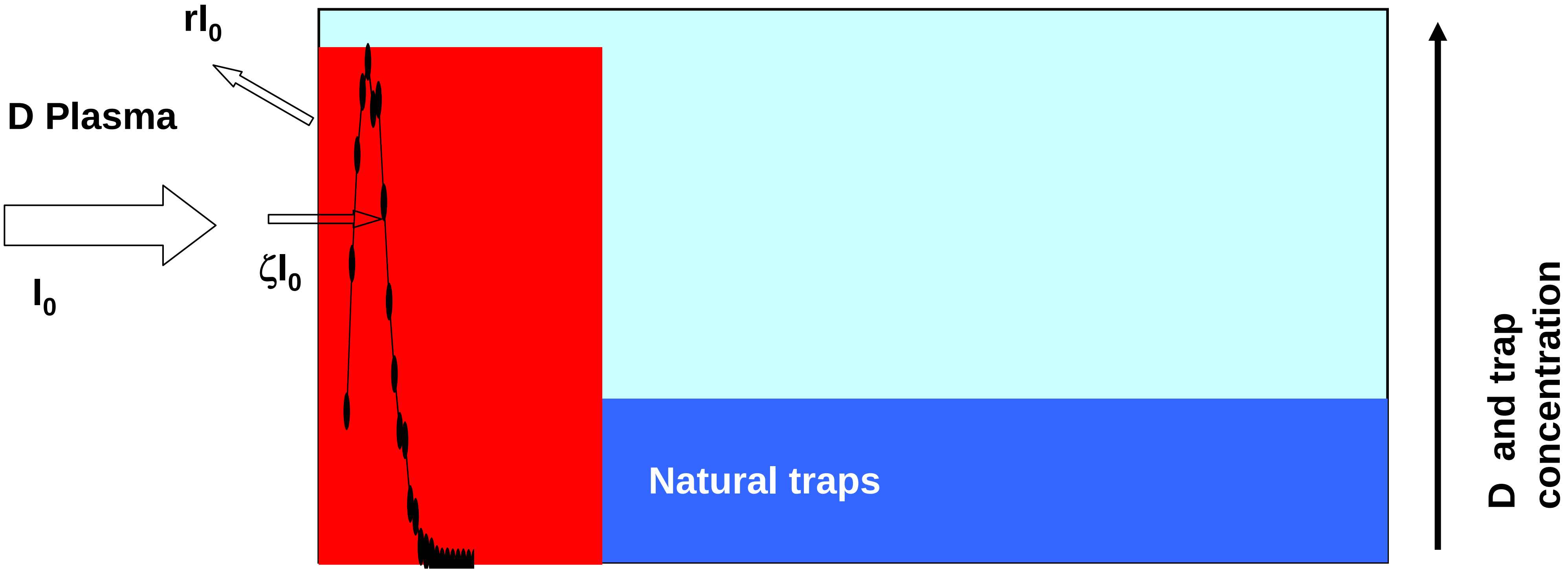
Radiation damage produced by 20 MeV W ions

Radiation damage up to ~ 1 dpa, RT



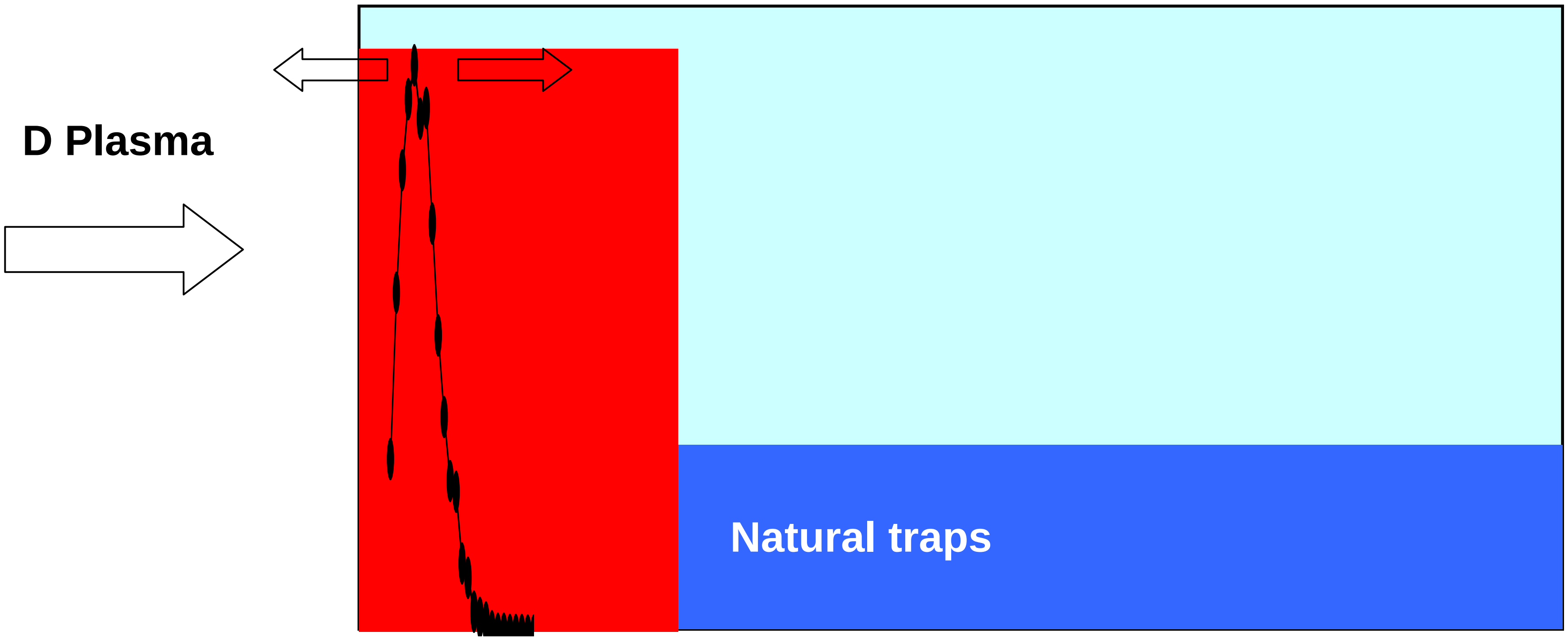
Deuterium decoration of radiation damage

Radiation damage up to ~ 1 dpa, RT



Deuterium decoration of radiation damage

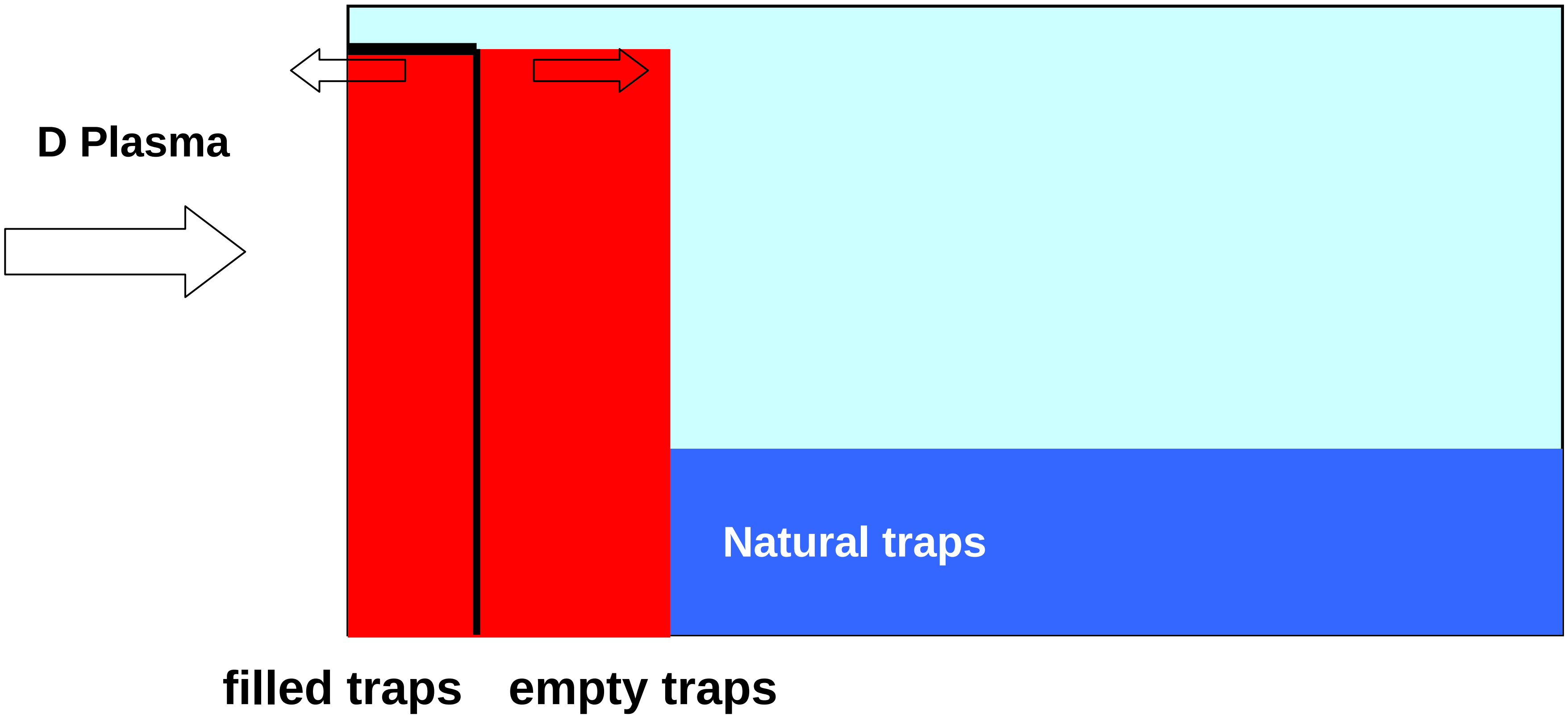
Radiation damage up to ~ 1 dpa, RT



Deuterium decoration of radiation damage

Radiation damage up to ~ 1 dpa, RT

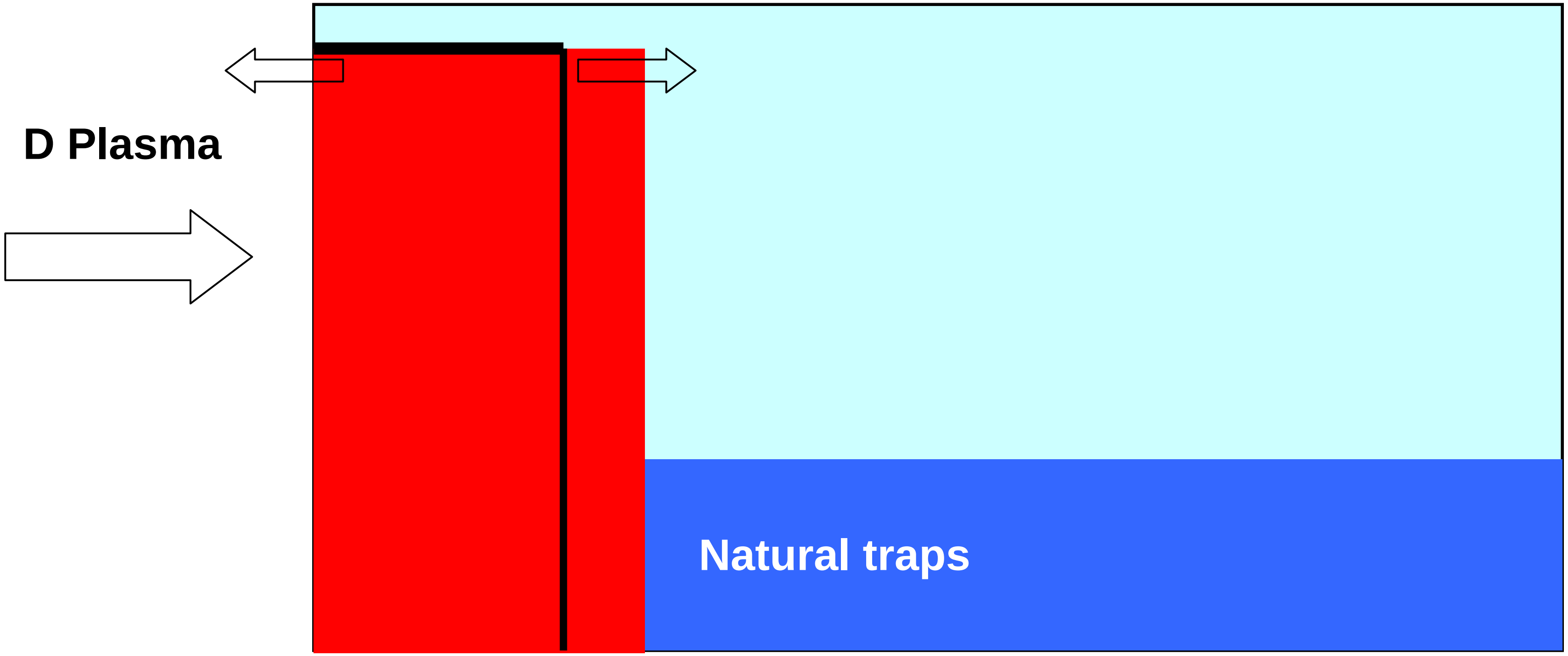
$$x(t)=\mathrm{sqrt}(2*D*t*K_s*\mathrm{sqrt}(\zeta I_0/2s\mu))/(\textit{density of filled traps}))$$



Deuterium decoration of radiation damage

Radiation damage up to ~ 1 dpa, RT

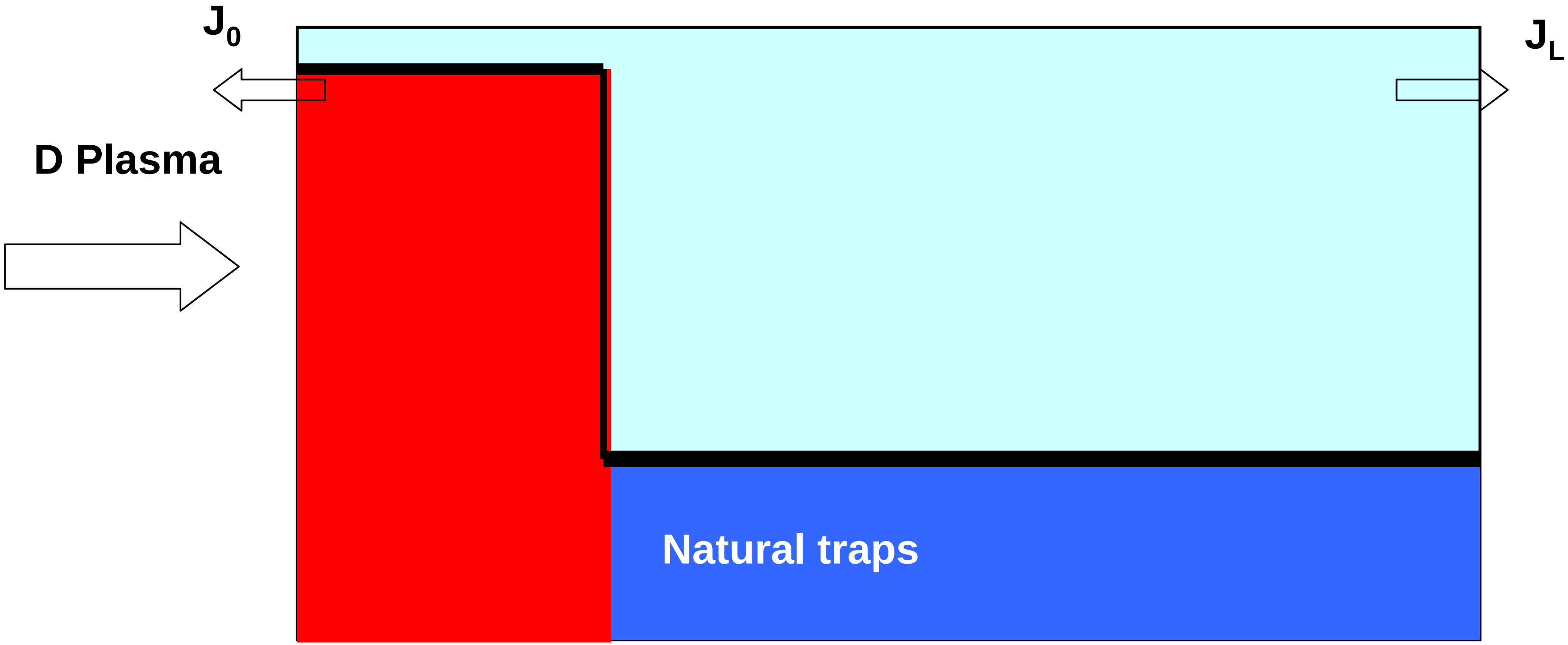
$$x(t)=\mathrm{sqrt}(2*D*t*K_s*\mathrm{sqrt}(\zeta I_0/2s\mu))/(\textit{density of filled traps}))$$



Deuterium decoration of radiation damage

Radiation damage up to ~ 1 dpa, RT

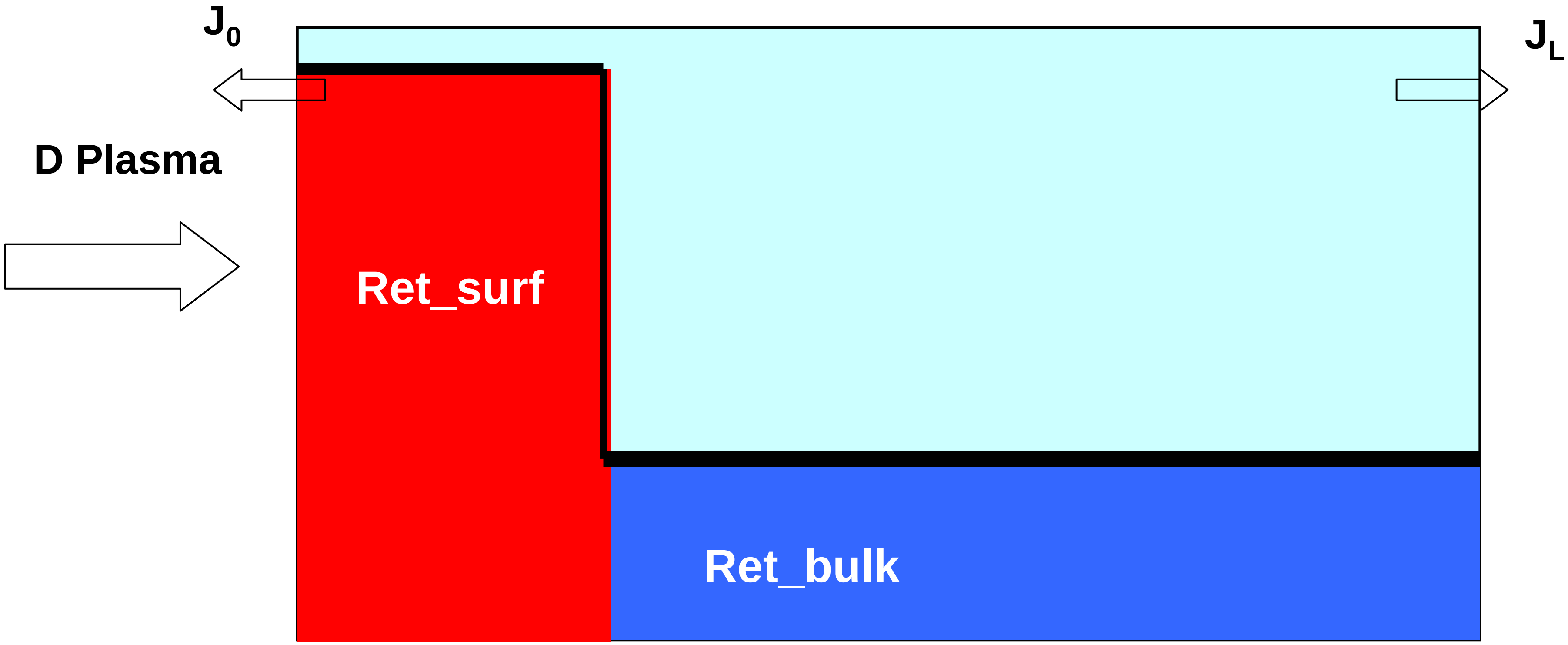
$$x(t)=\mathrm{sqrt}(2^*D^*t^*K_s{}^*\mathrm{sqrt}(\zeta I_0/2s\mu))/(\textit{density of filled traps}))$$



Deuterium decoration of radiation damage

Radiation damage up to ~ 1 dpa, RT

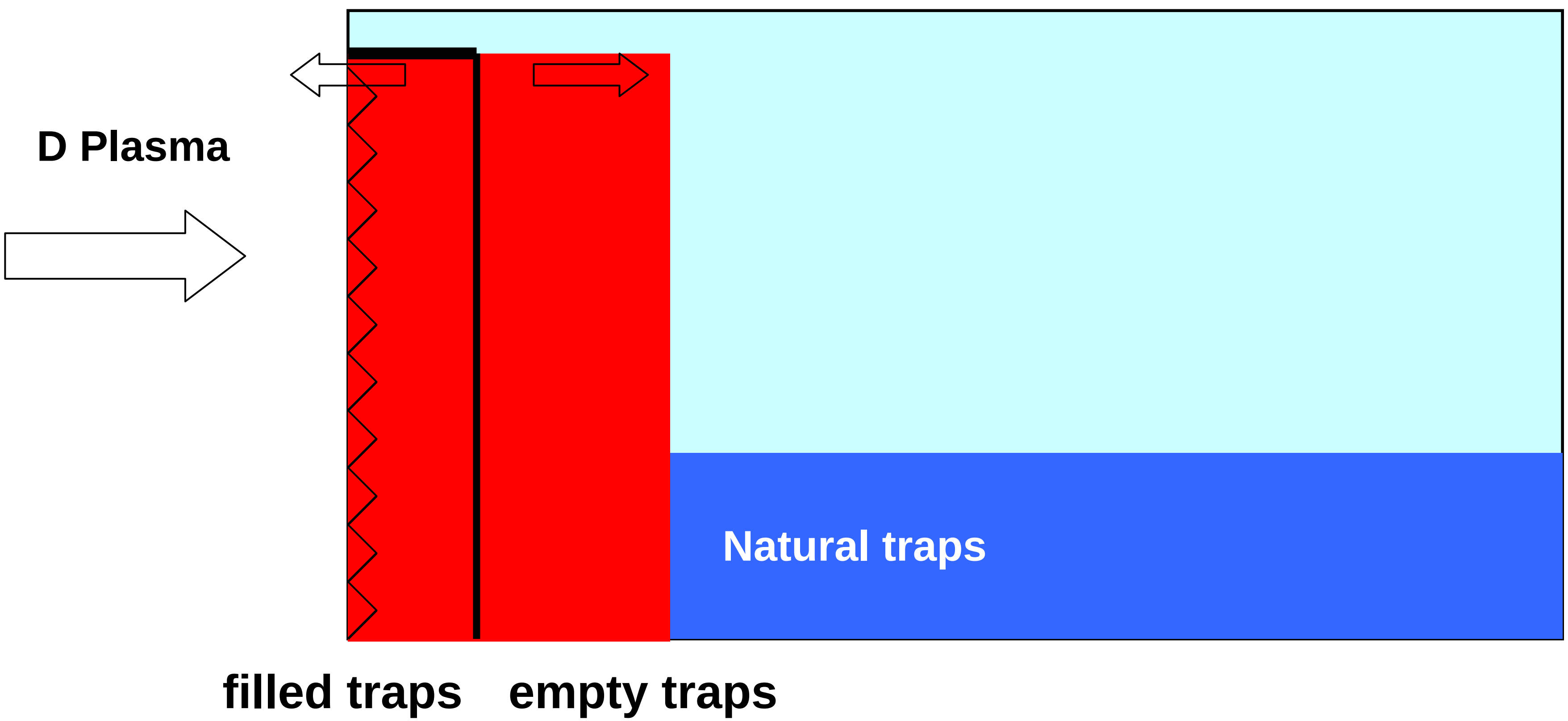
$$x(t)=\mathrm{sqrt}(2*D*t*K_s*\mathrm{sqrt}(\zeta I_0/2s\mu))/(\textit{density of filled traps}))$$



Ret_TDS = Ret_surf + Ret_bulk

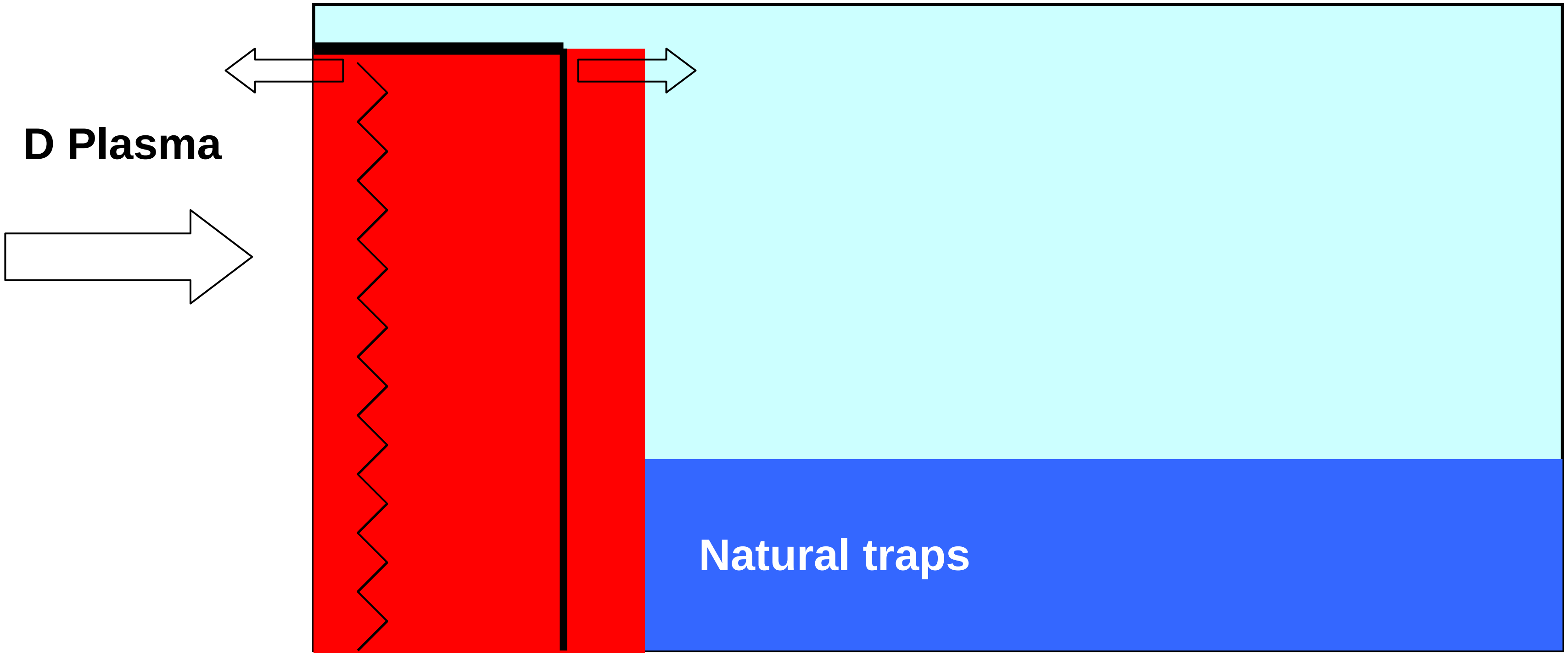
Sputtering changes the front propagation of D

Radiation damage up to ~ 1 dpa, RT



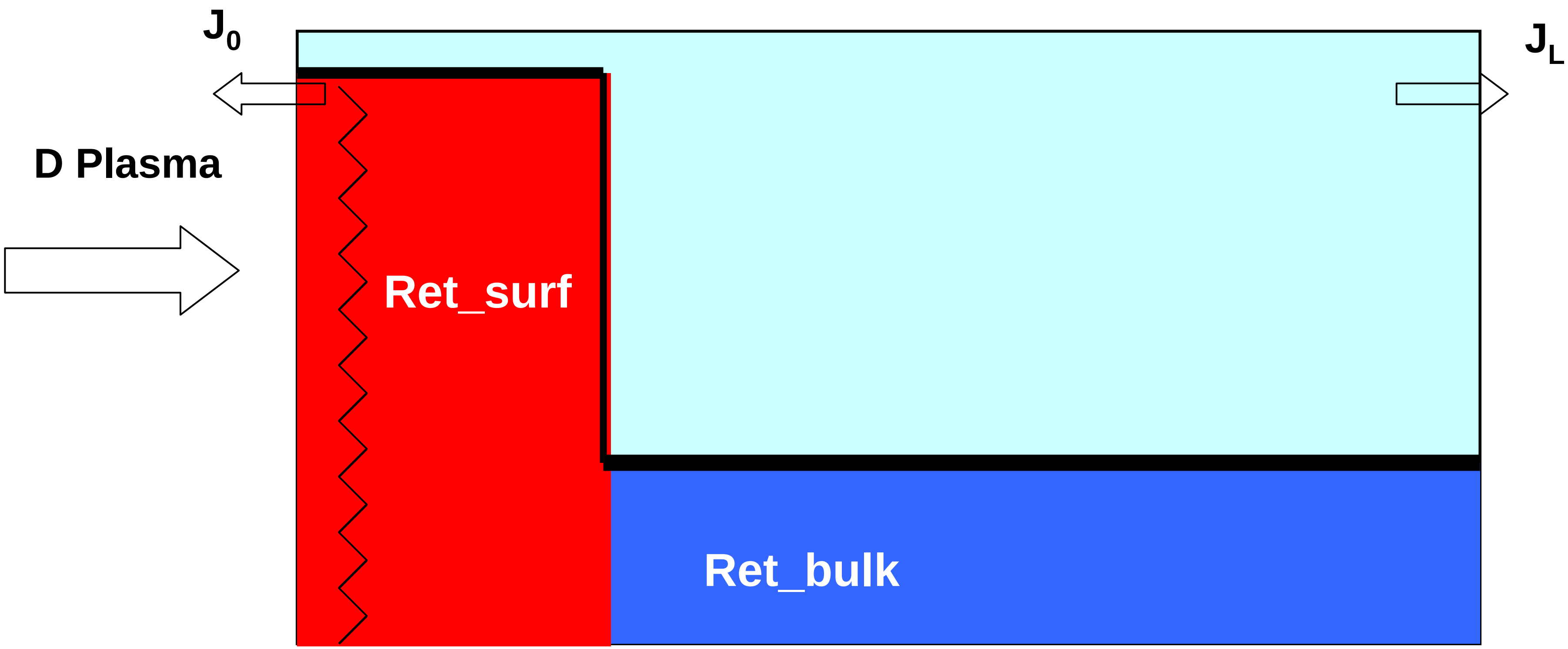
Sputtering changes the front propagation of D

Radiation damage up to ~ 1 dpa, RT



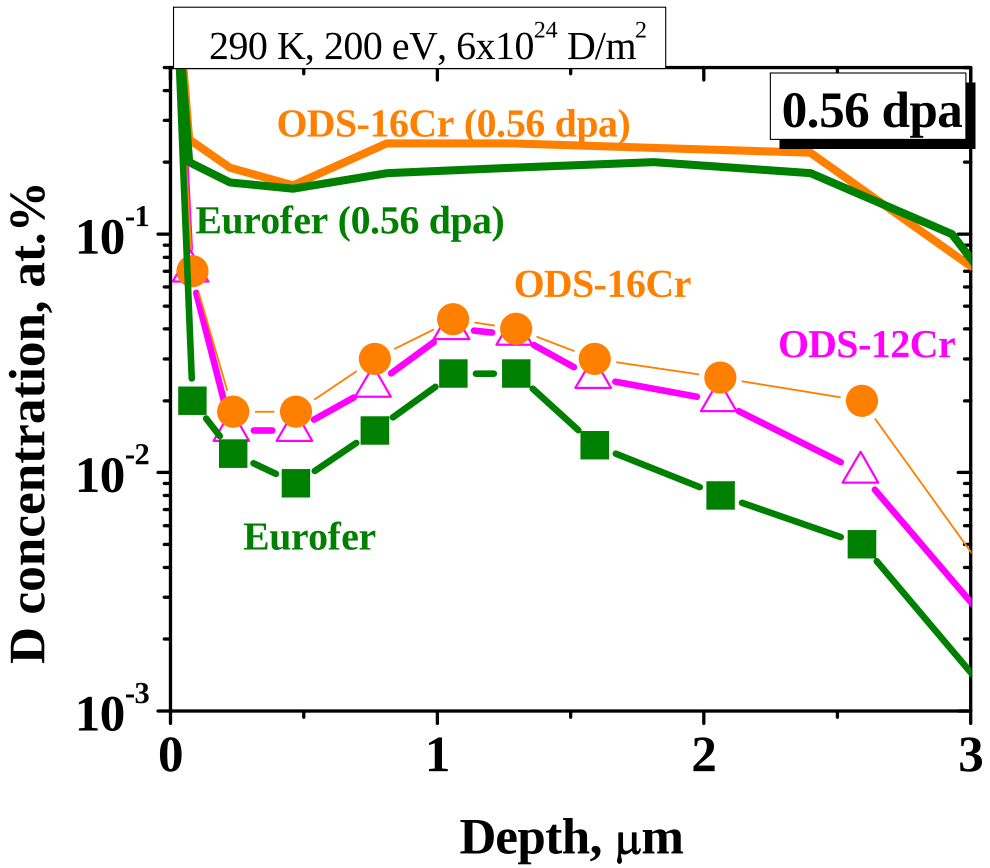
Sputtering changes the front propagation of D

Radiation damage up to ~ 1 dpa, RT



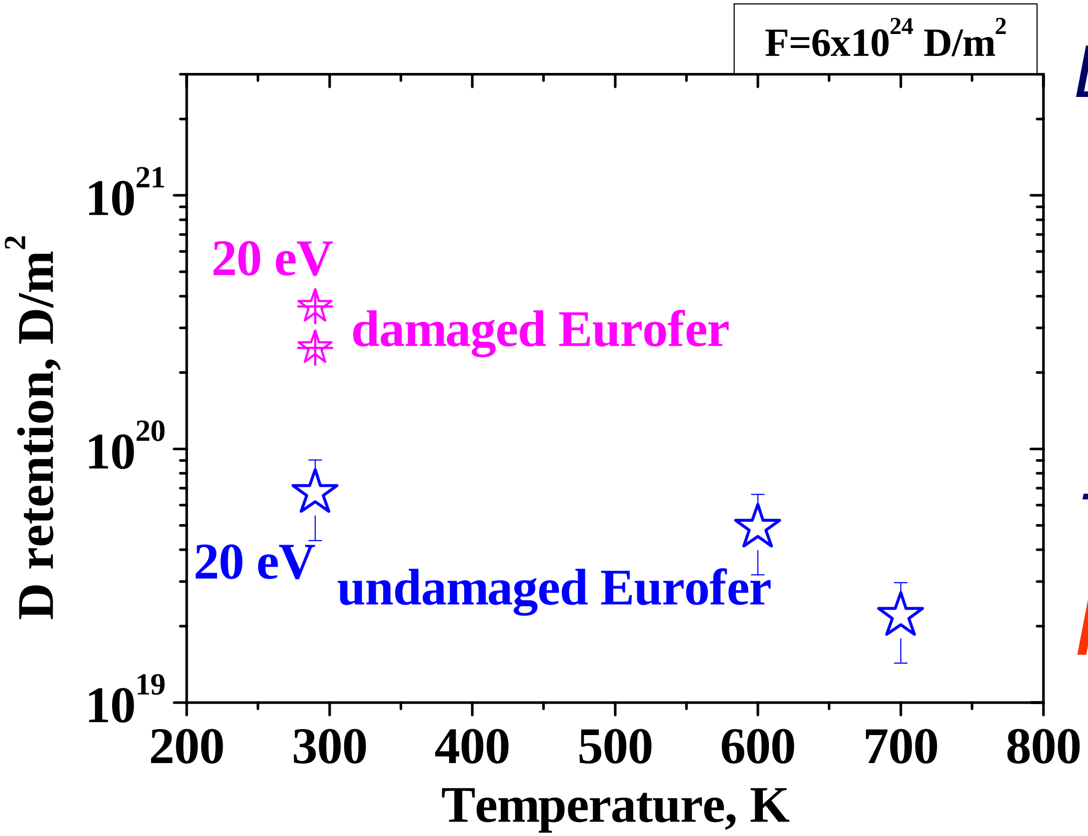
Ret_TDS = Ret_surf + Ret_bulk

The D retention in damaged Eurofer & ODS



❖ **The D concentration at radiation-induced damage in ODS and Eurofer is similar**

Does formation of ‘fuzz’ influence the D retention?

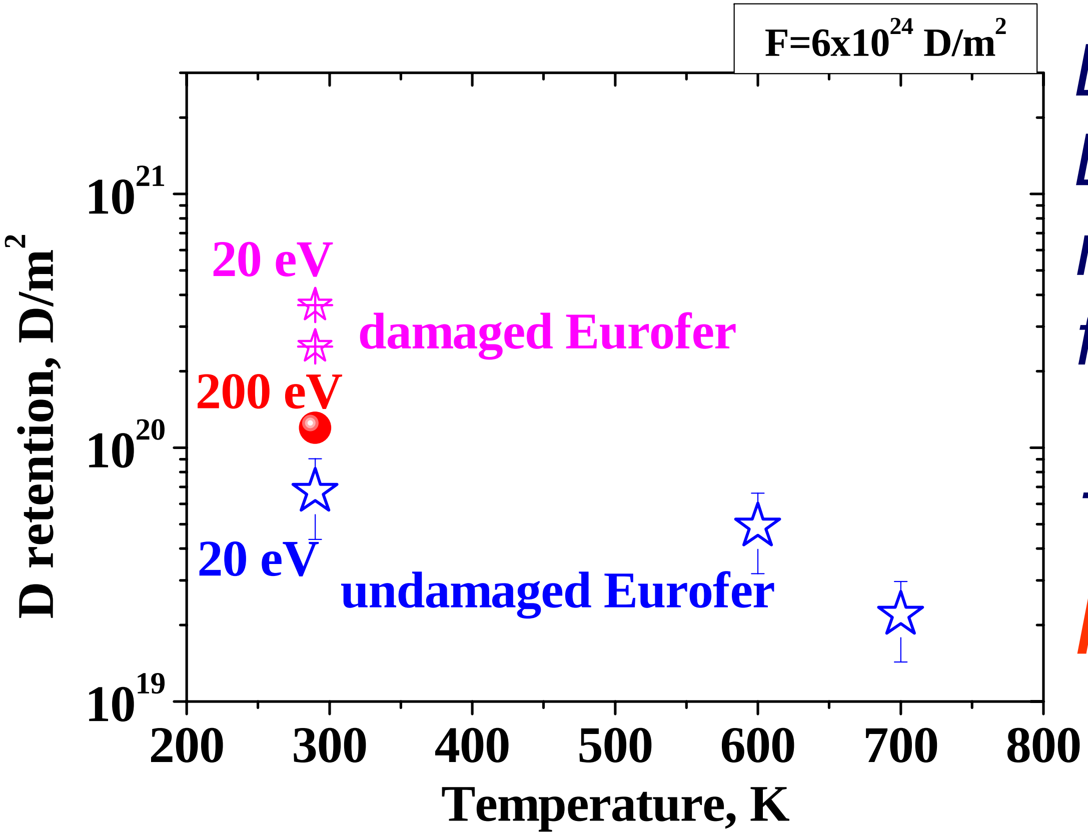


Damaged Eurofer:

Total D retention = Ret_surf + Ret_bulk

❖ **Radiation-induced defects up to 3 mkm result in an increase of the total D retention. The D retention near the plasma-facing surface contributes in the total retention**

Does formation of ‘fuzz’ influence the D retention?

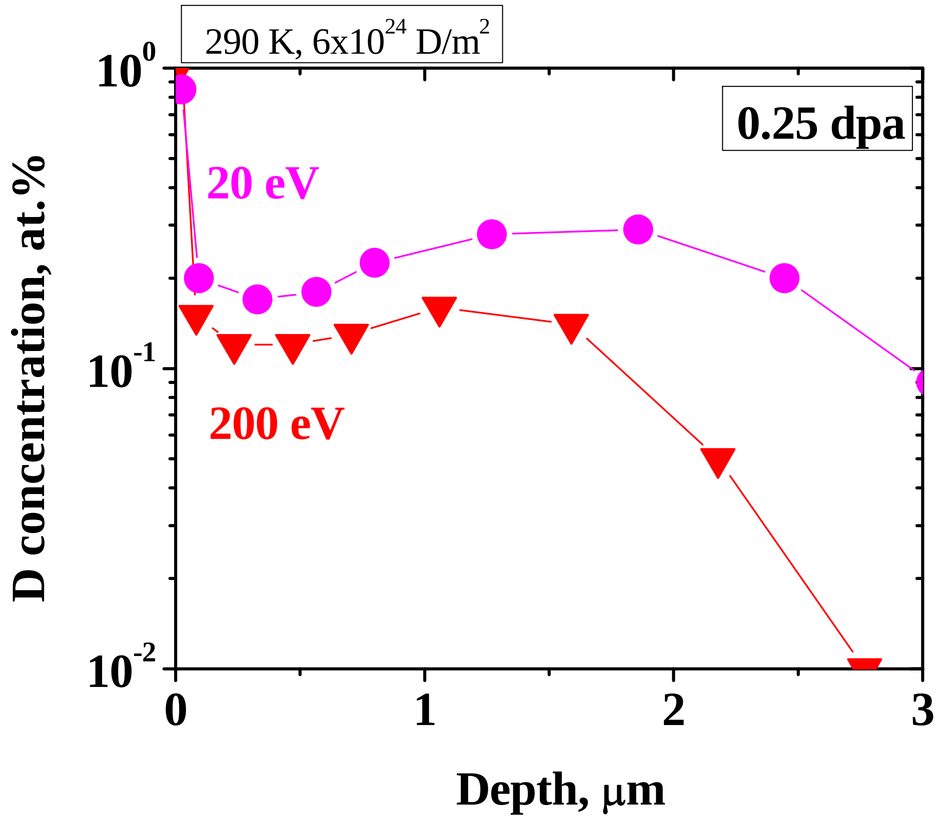


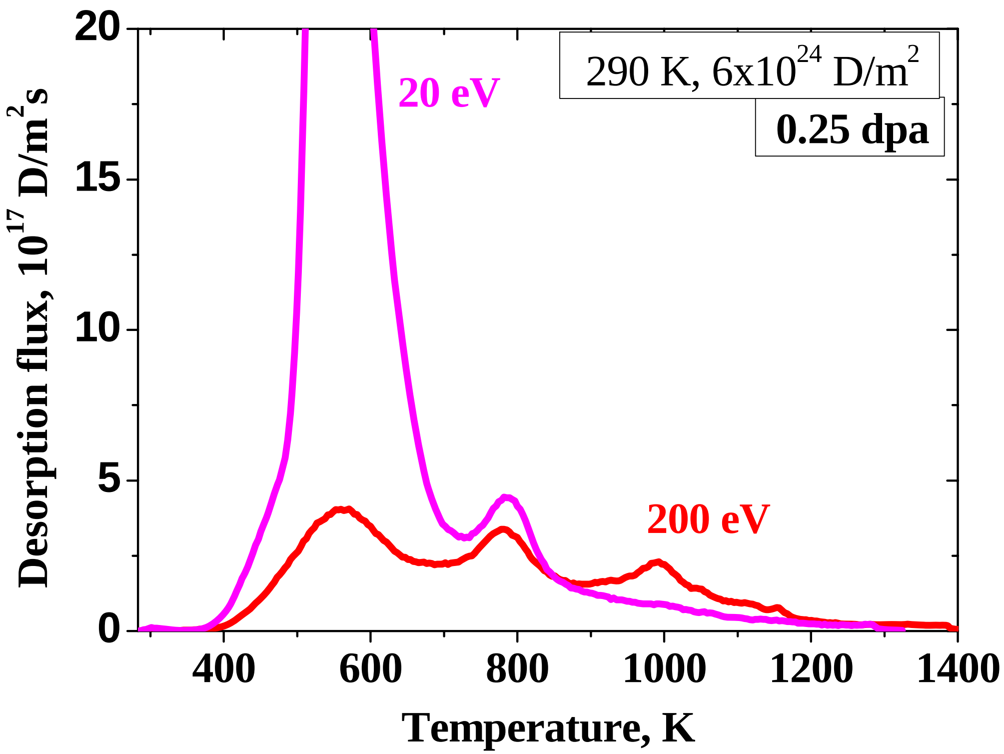
Damaged Eurofer: Decrease of the D retention with formation of ‘fuzz’

Total D retention = Ret_surf + Ret_bulk

- **Formation of ‘fuzz’ decreases the D retention in pre-damaged W because the surface retention contributes in the total retention**

Does formation of 'fuzz' influence the D retention?

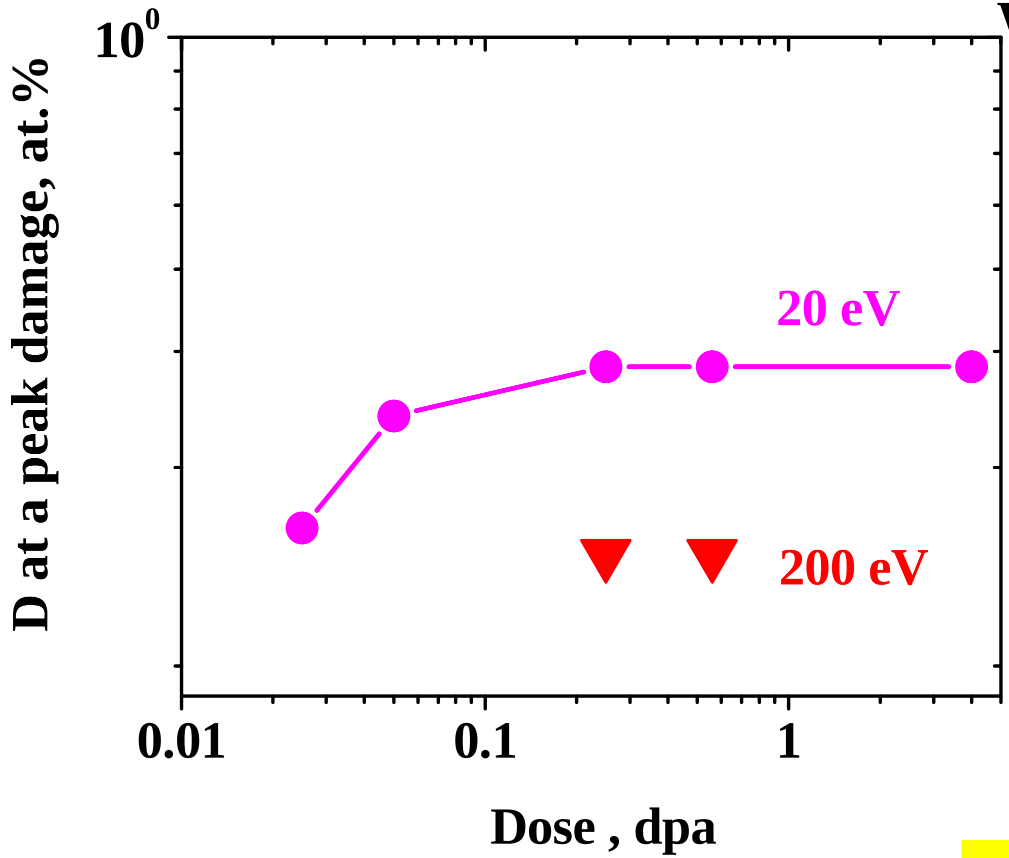




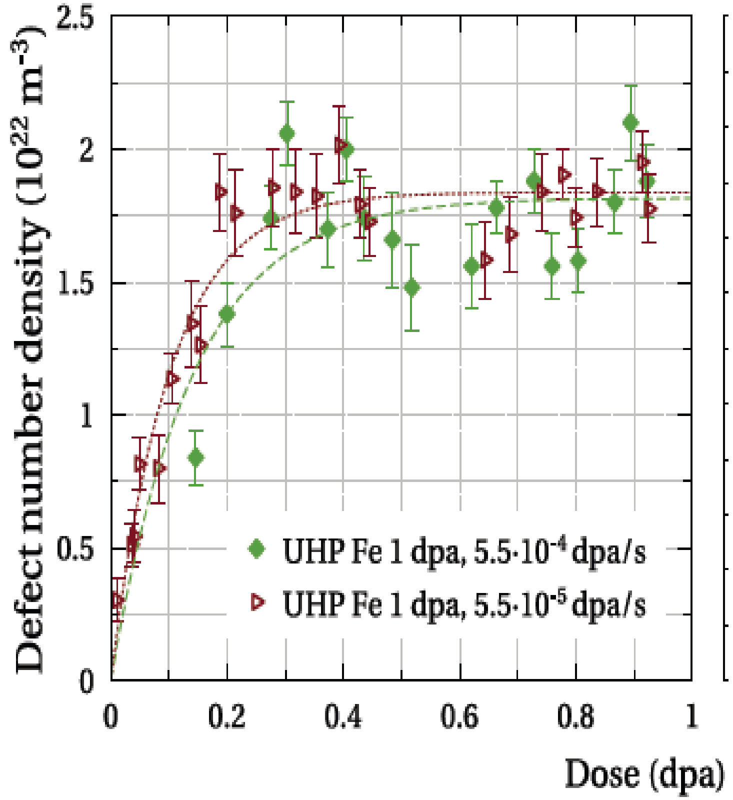
❖ **Formation of 'fuzz' decreases the D retention in pre-damaged W because the surface retention contributes in the total retention**

Total D retention = Ret_surf + Ret_bulk

The D retention in damaged Eurofer & ODS



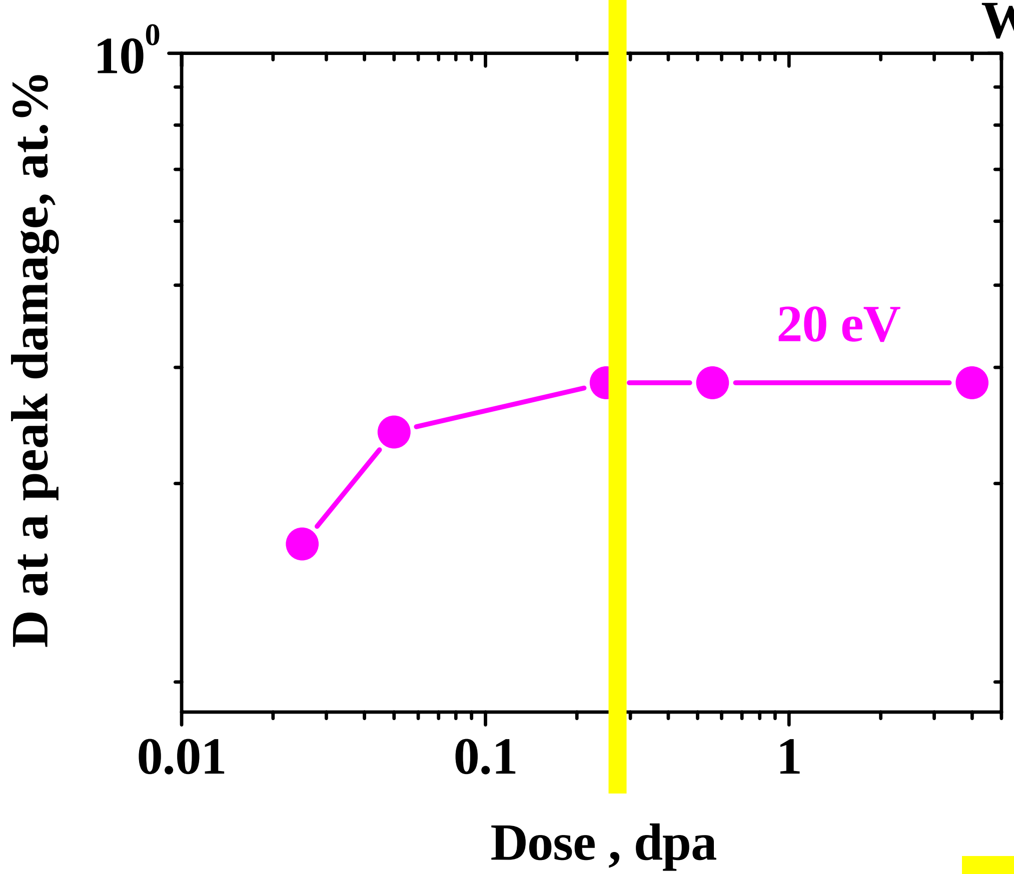
EVOLUTION OF VISIBLE DEFECTS WITH DOSE



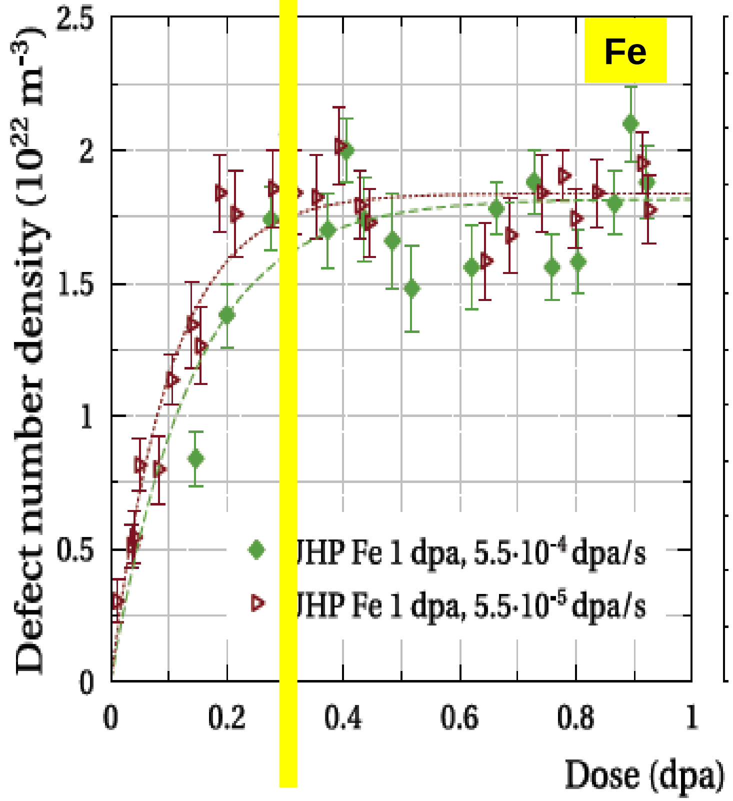
Anna Prokhodtseva et al, ICFRM-16, October 2013, Beijing

- ❖ **The D concentration at radiation-induced damage is saturated at ~0.25 dpa**

The D retention in damaged Eurofer & ODS



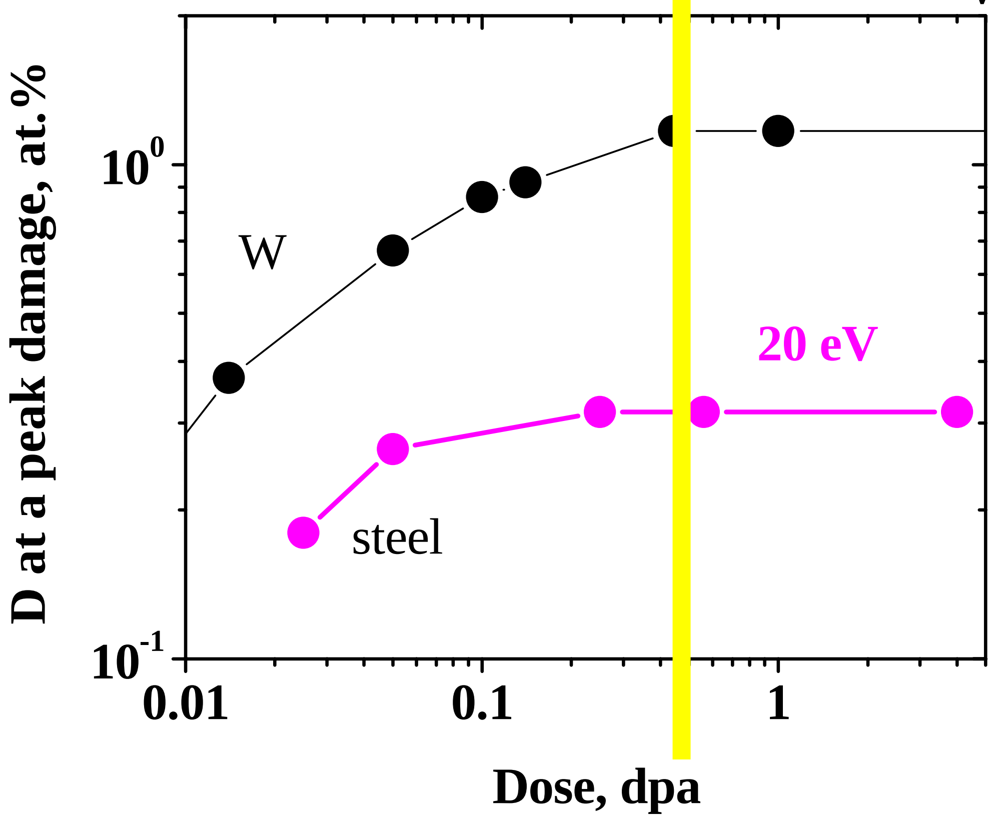
EVOLUTION OF VISIBLE DEFECTS WITH DOSE

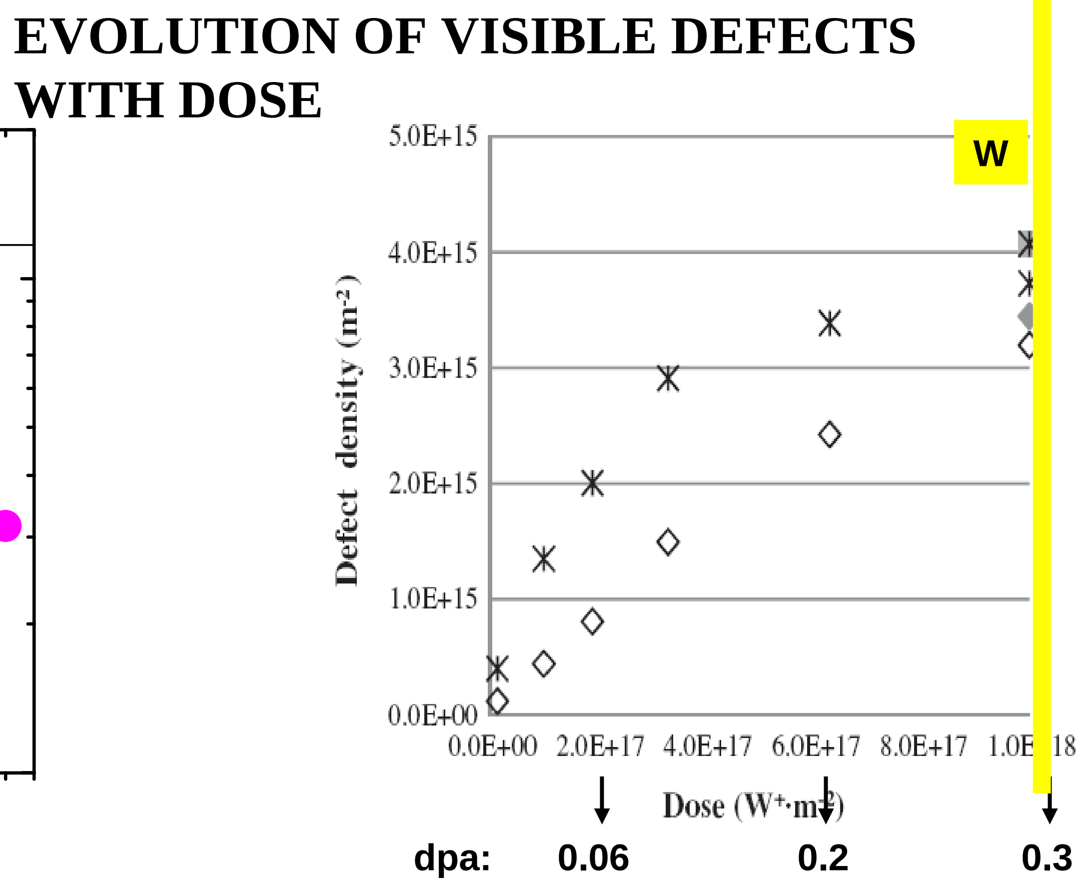


Anna Prokhodtseva et al, ICFRM-16, October 2013, Beijing

❖ **The D concentration at radiation-induced damage is saturated at ~0.25 dpa**

The D retention in damaged steel vs W

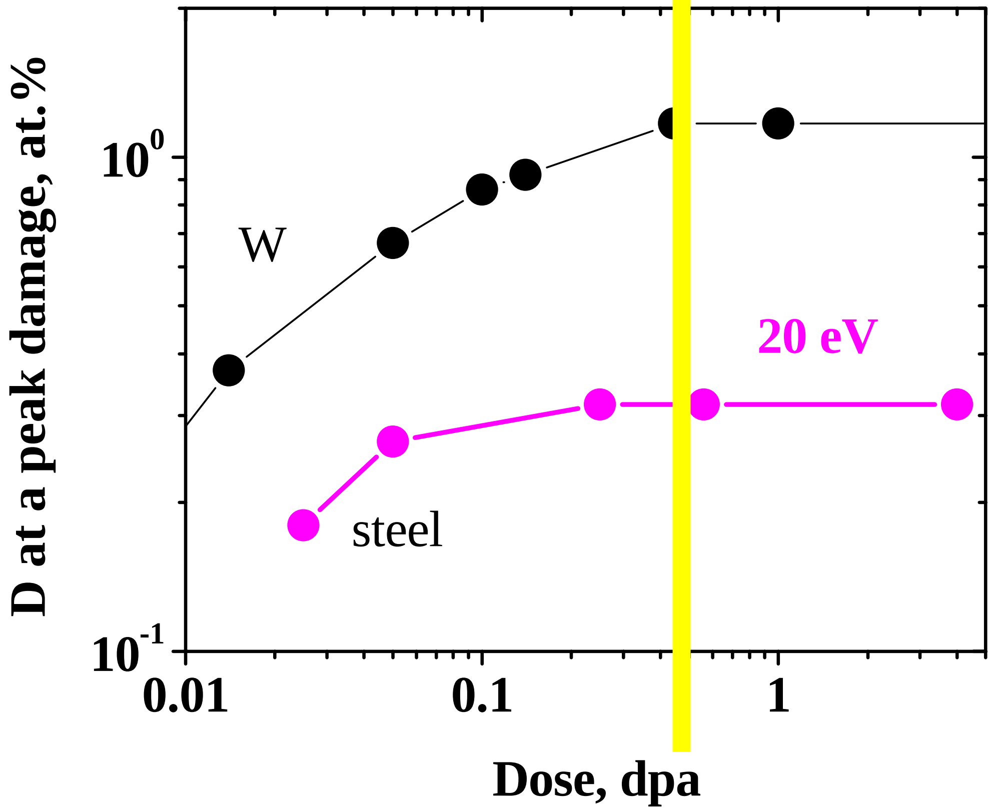




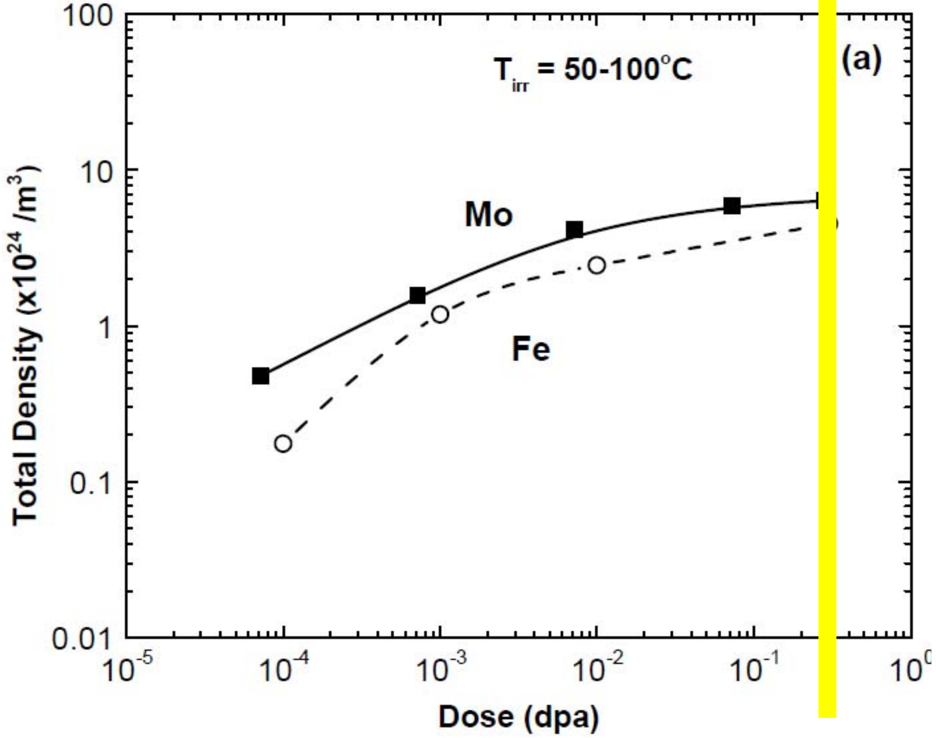
X. Yi et al, ICFRM-16, Phyl. Mag., 2013

❖ **The D concentration at radiation-induced damage is saturated at ~0.25 dpa for steel and at ~0.5 dpa for W**

The D retention in damaged steel vs W



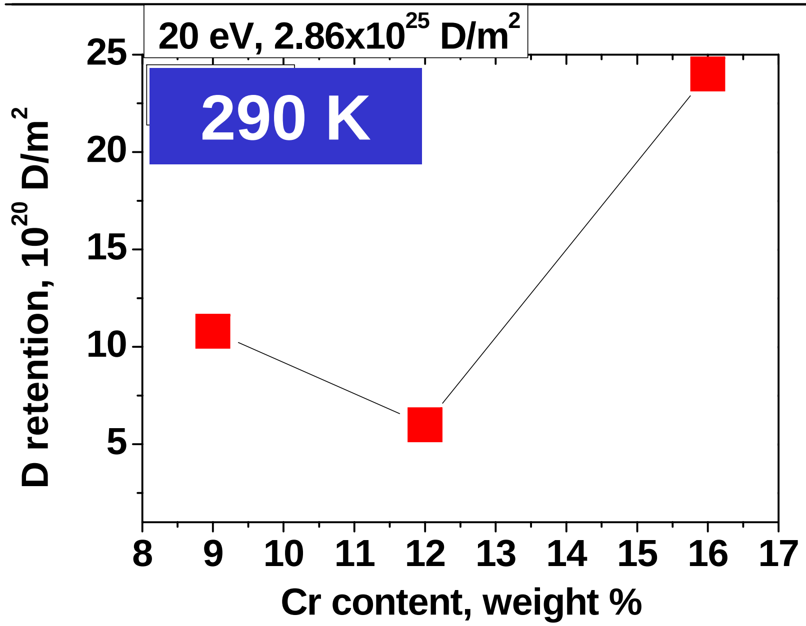
EVOLUTION OF VACANCY CLUSTERS WITH DOSE

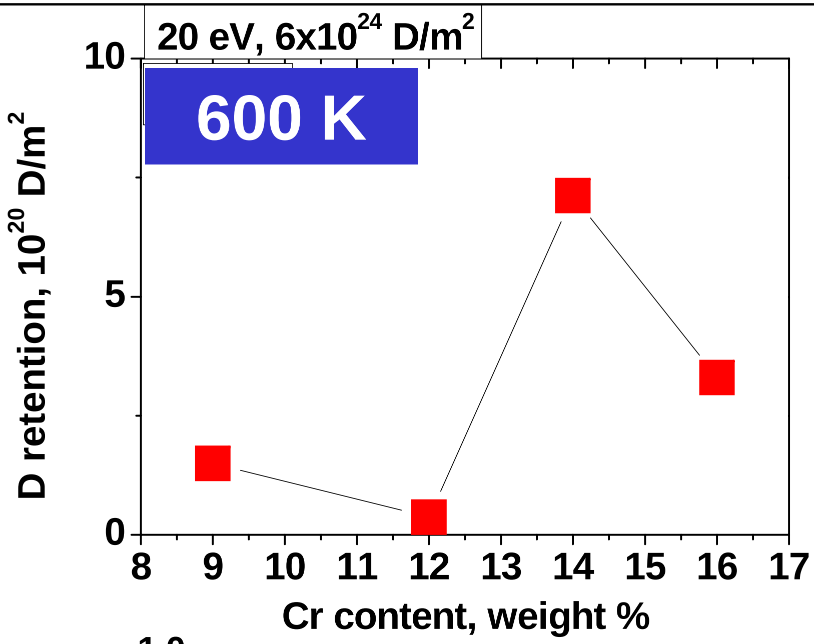


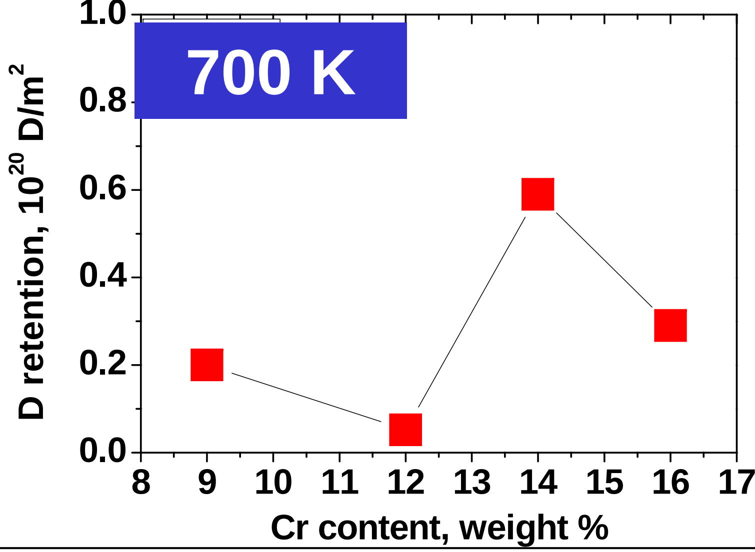
Eldrup et al, JNM, 2008: n-irradiated Mo and Fe

- **The D concentration at radiation-induced damage is saturated at ~0.25 dpa for steel and at ~0.5 dpa for W**

Effect of Cr concentration in ODS

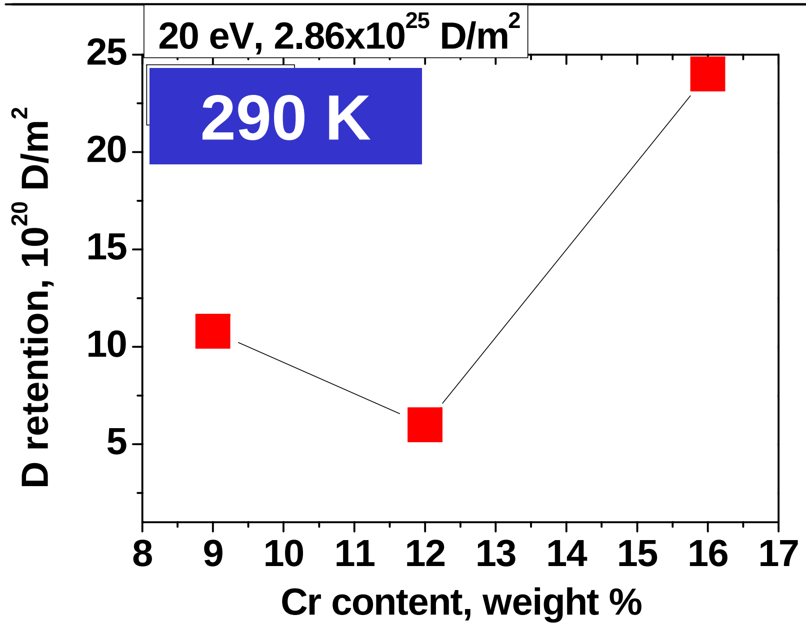


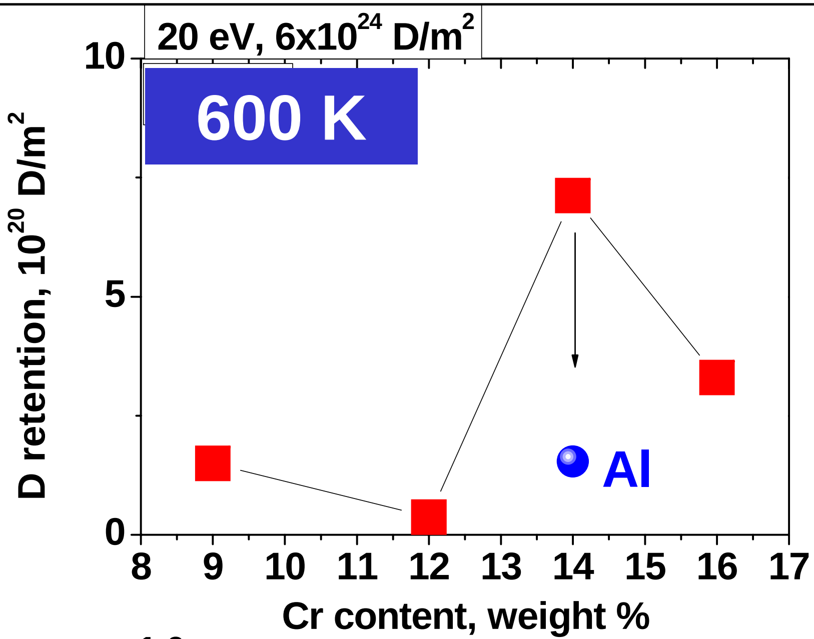


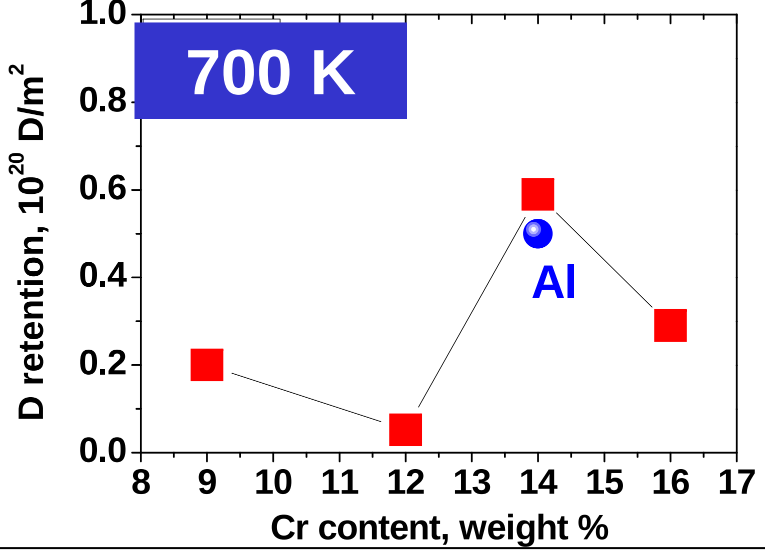


- ODS-12Cr has the minimum of the D retention

Replacement of Ti on Al in ODS-14Cr

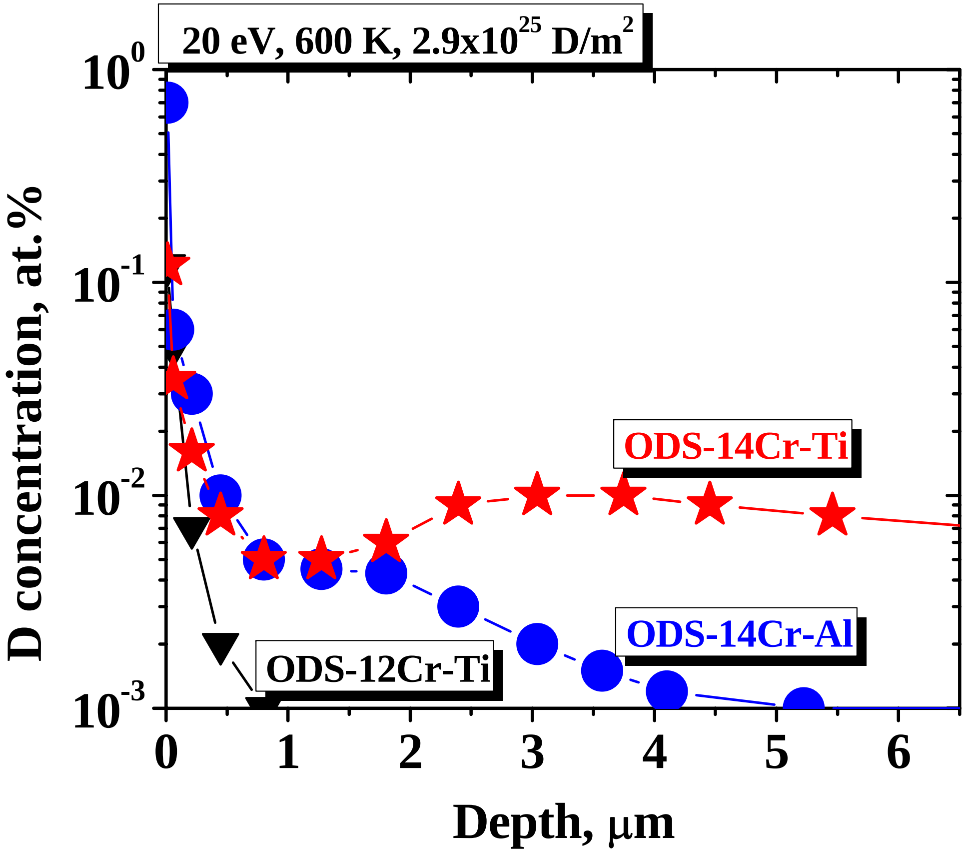


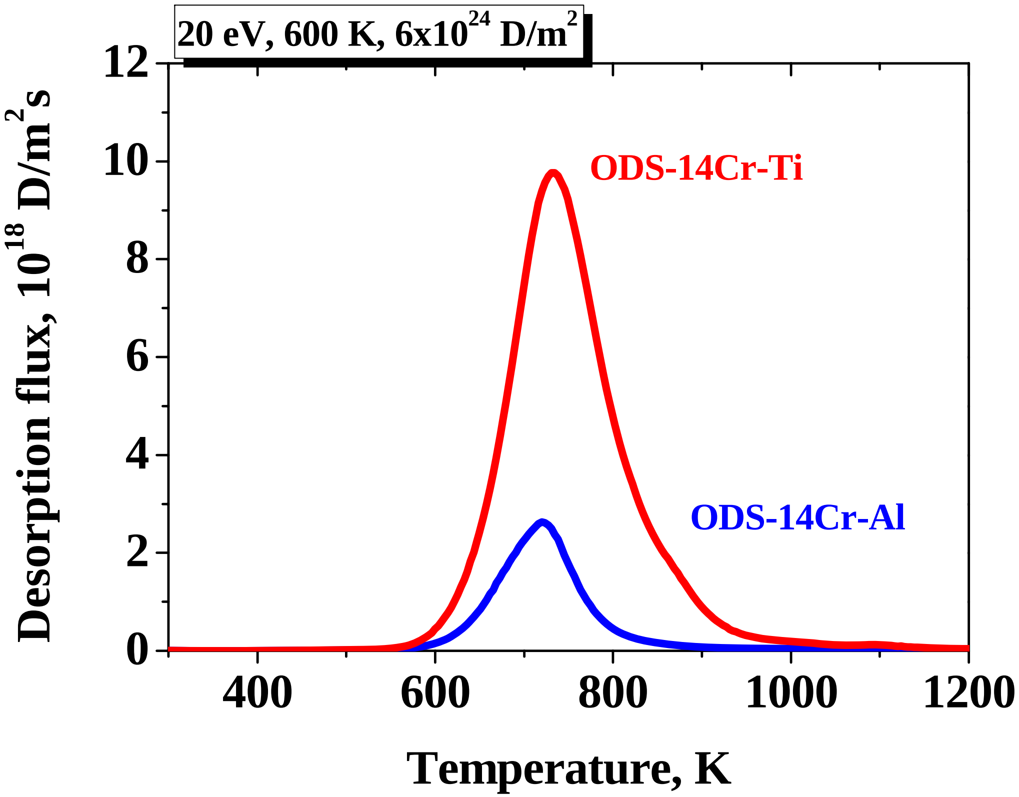




❖ **Replacement of Ti on Al decreases the D retention**

Replacement of Ti on Al in ODS-14Cr





❖ **Replacement of Ti on Al decreases the D retention because of a reduction of the diffusion into the bulk**

Conclusions (undamaged steel)

- **The formation of ‘fuzz’-like structure enriched in W or Ta was observed at incident D energy of 200 eV for both Eurofer and ODS steels**
- **The ‘fuzz’ growth enhances with increasing of the temperature**
- **Sputtering yield = f (ion energy, temperature)**
- **The formation of ‘fuzz’ decreases the D retention near the surface and can reduce the total D retention**

Conclusions (undamaged steel)

- ❖ **The D retention in ODS is higher than in Eurofer**
- ❖ **The D inventory depends on Cr concentration: ODS-12Cr has minimum in the D retention**
- ❖ **Using Al instead of Ti in ODS reduces the D retention**

Conclusions (damaged steel)

- **Pre-irradiation with W ions results in the same D concentration in both ODS and Eurofer**

- **The D concentration does not depend on the Cr concentration in pre-damaged steel**

- **The D concentration at radiation-induced defects saturates at 0.25 dpa**

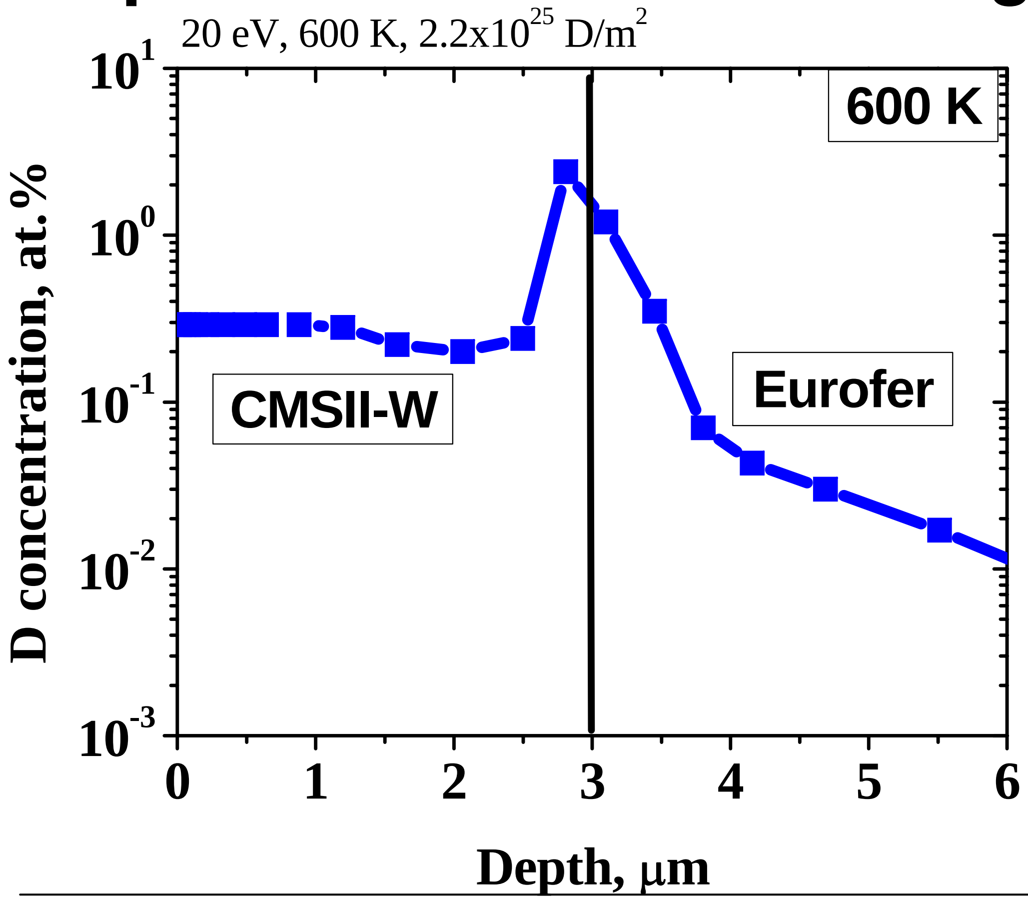
- **No effect of pre-damaging at RT on the D retention after the D plasma exposure at T > 500 K**

Conclusions

- **Bare steel cannot be used as plasma-facing material due to strong sputtering by D ions => protection with W coating:**

Conclusions

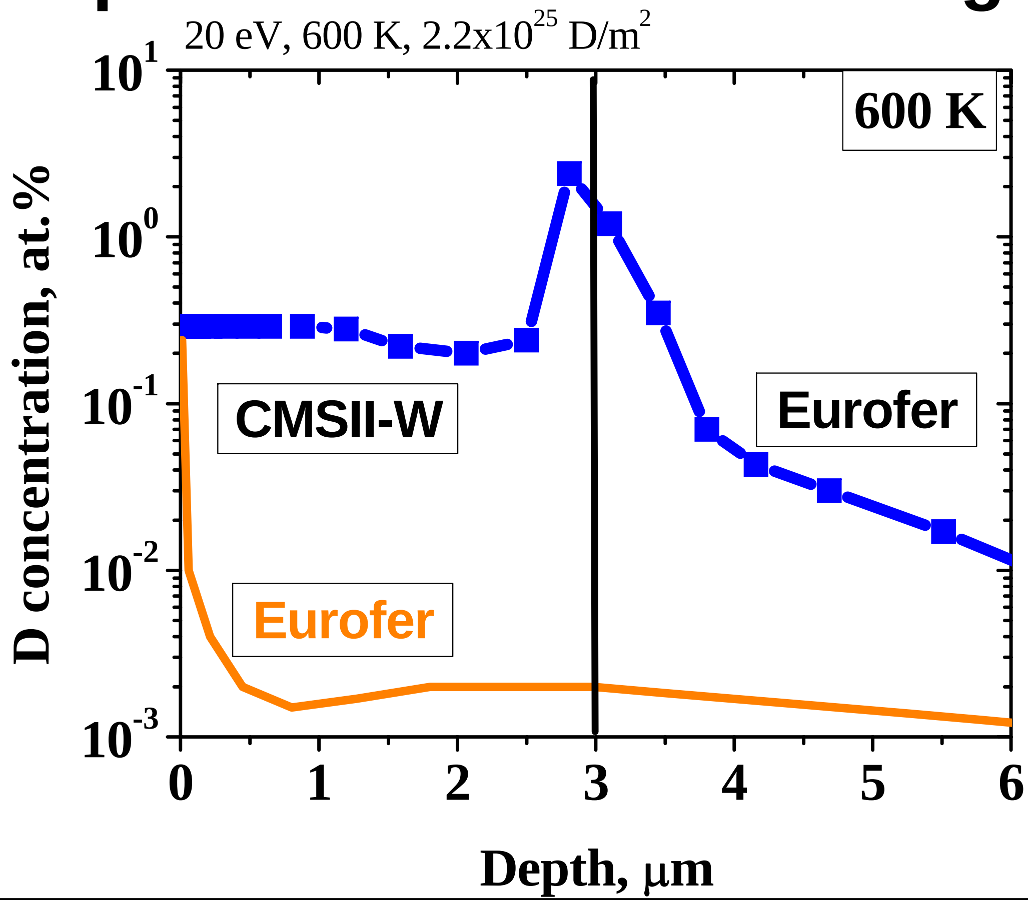
❖ **Bare steel cannot be used as plasma-facing material due to strong sputtering by D ions => protection with W coating:**



❖ **D retention in interface between W coating and steel can be a concern**

Conclusions

❖ **Bare steel cannot be used as plasma-facing material due to strong sputtering by D ions => protection with W coating:**



❖ **D retention in interface between W coating and steel can be a concern**

❖ **D retention is higher in W/Eurofer compared to Eurofer**

Future plan

- ❖ **Fusion relevant behavior at 500-700 appm He needs to be addressed: double beam experiment at MEPhI (D+He) is planning at 2016**

Materials composition

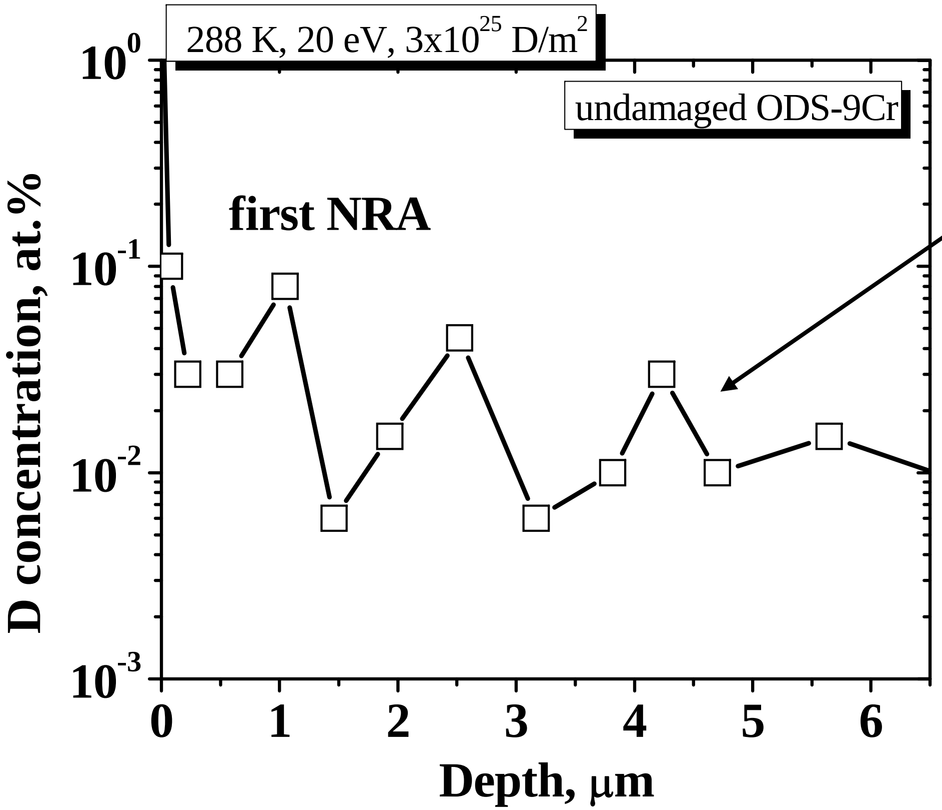
Sample	Compositions
9Cr-ODS (2)	Fe-9Cr-1.5W-0.1C-0.15Si-0.5Ti-0.35Y2O3
12Cr-ODS	Fe-12Cr-2W-0.15Si-0.5Ti-0.35Y2O3
14Cr-ODS(Ti)	Fe-14Cr-2W-0.15Si-0.5Ti-0.35Y2O3
14Cr-ODS(Al)	Fe-14Cr-2W-0.15Si-4.0Al-0.35Y2O3
16Cr-ODS	Fe-16Cr-2W-0.15Si-0.5Ti-0.35Y2O3

Processing parameters (MA+HIP+Forging):
HIP:100MPa；1150 ℃，3h;
Forge: initial temperature 1150-1170 ℃, final temperature 970℃, forging ratio 3:1;
Y2Ti2O7 (EDS, TEM): several nm, Y2TiO5
Large dispersoid: Ti0, TiN
Weight percent

	C	Mn	Si	P	S	Cr	Ni	W	N	Cu	B	Co	Nb	Ta	Ti	V	Mo	Zr	Ce
Eurofer	0.12	0.49	0.5	0.005	0.003	8.9	0.005	1.15	0.03	0.0037	0.001	0.005	0.2	0. 14	0.01	0,2	0,0012		
Rusfer	0,16	0,6	0,4	0,01	0,006	12	0,03	2	0,04		0,006		0,01	0,15	0,05	0,25	0,01	0,05	0,05

Does formation of 'fuzz' influence the D retention?

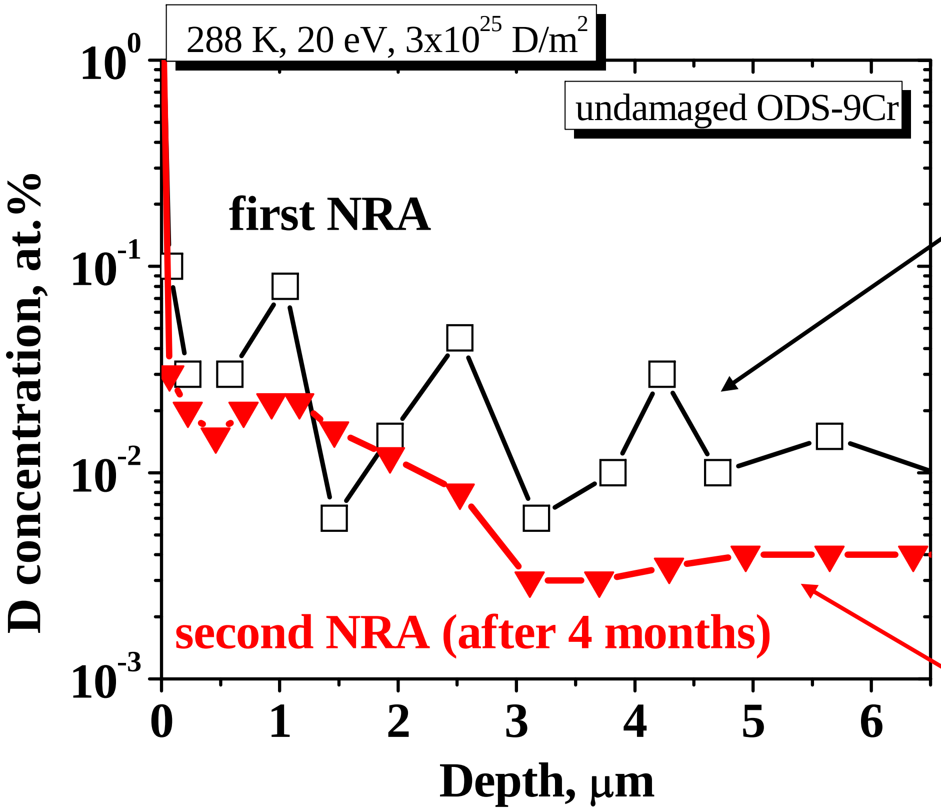
NRA: D depth profile measurement



- ❖ **Weakly bounded D**
- ❖ **Never was observed in W or Mo**

Does formation of ‘fuzz’ influence the D retention?

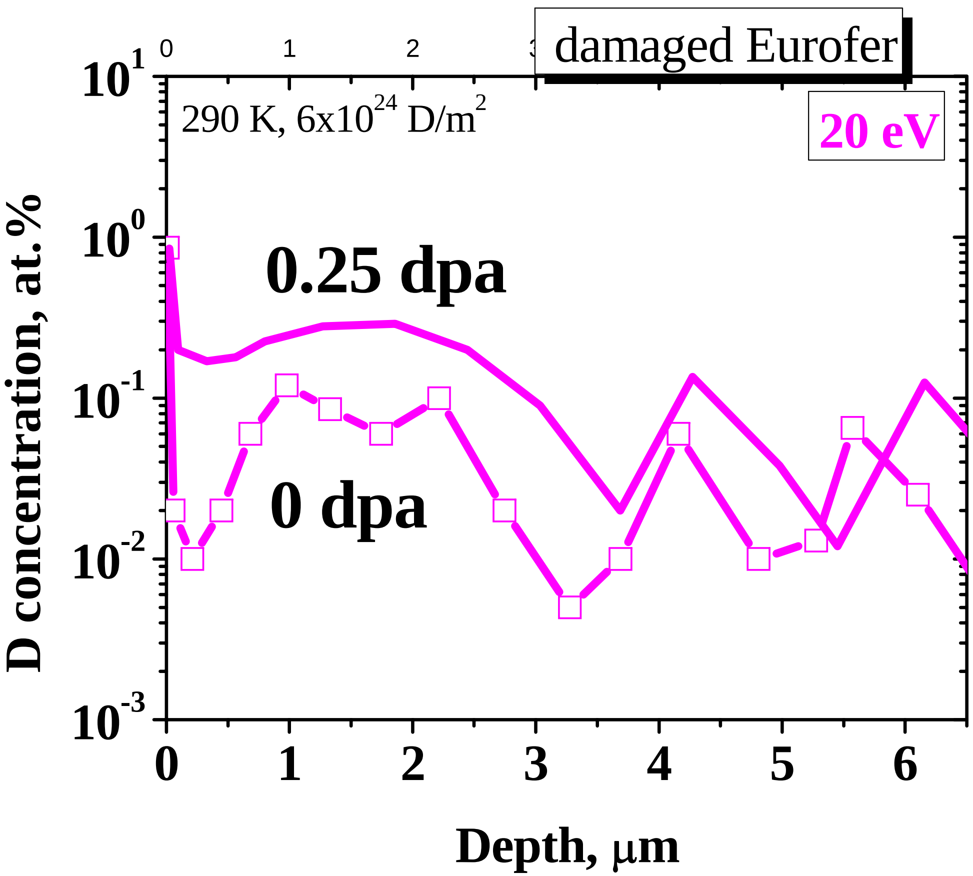
NRA: D depth profile measurement

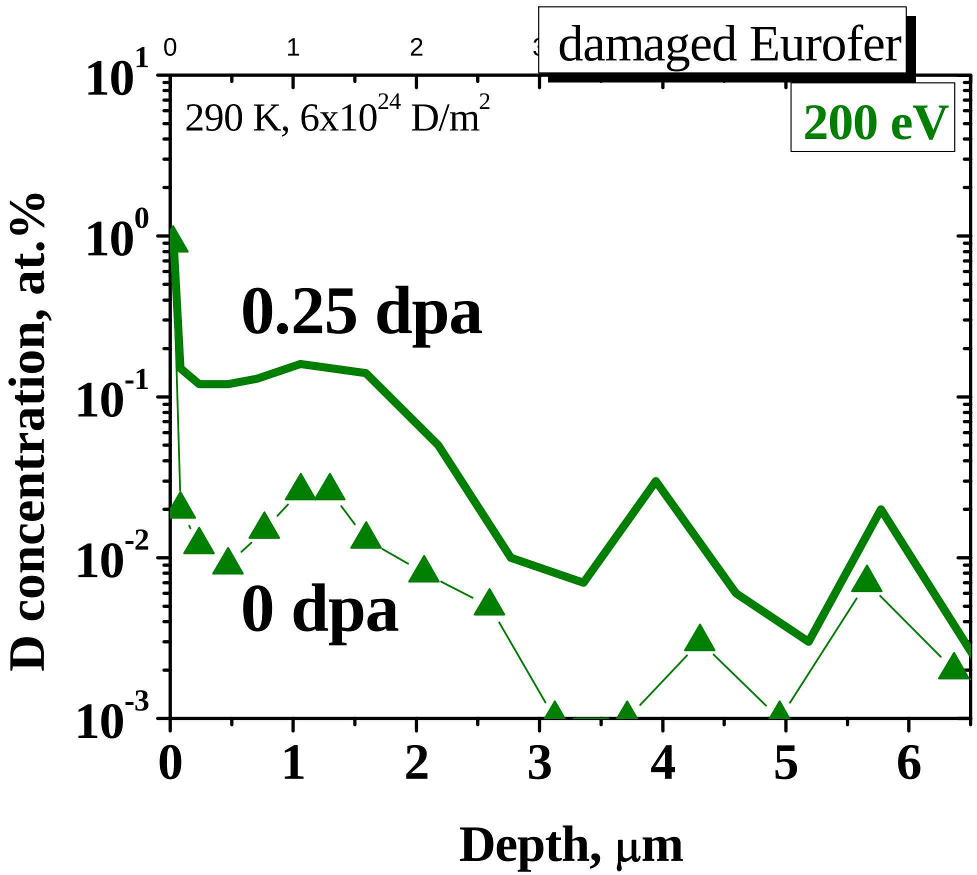


- **Weakly bounded D**
- **Never was observed in W or Mo**

Weakly bounded D was desorbed: TDS after several months underestimates the D retention

Does formation of ‘fuzz’ influence the D retention?

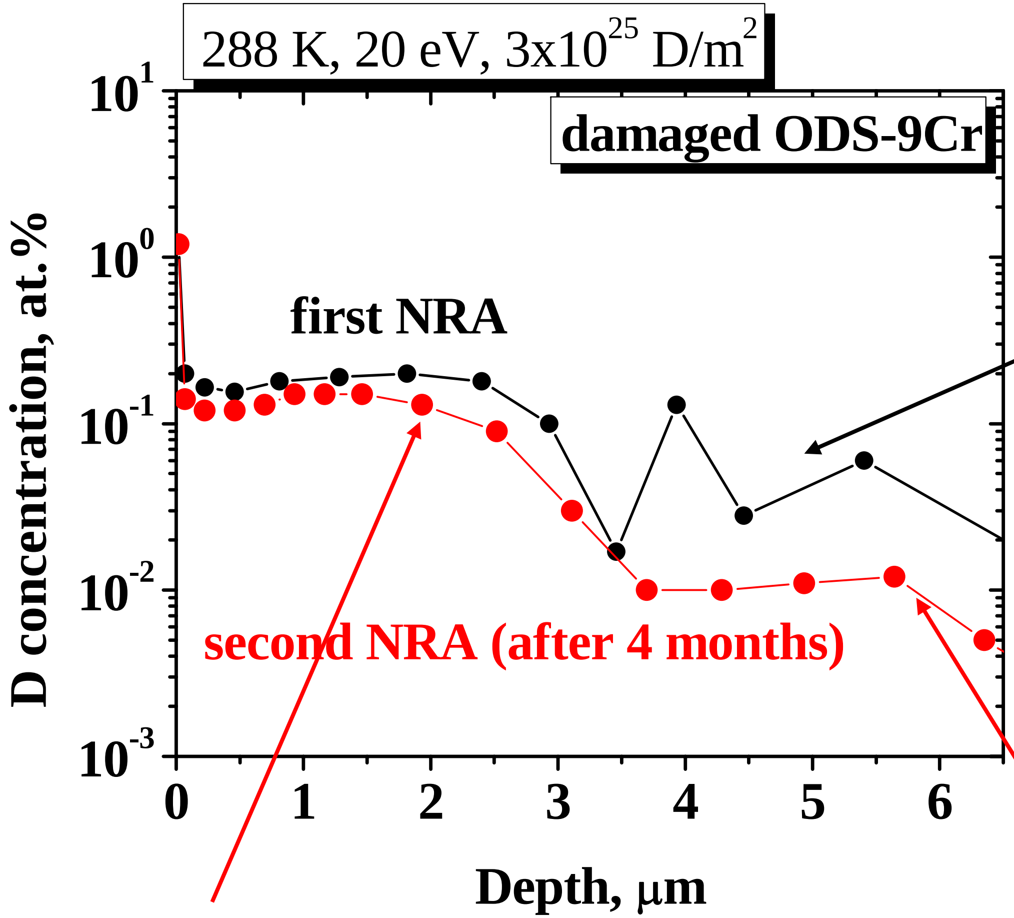




- **Irradiation with W ions increases the D retention in damaged zone up to 3 mkm.**

Total D retention = Ret_surf + Ret_bulk

Desorption of weakly bounded D with time



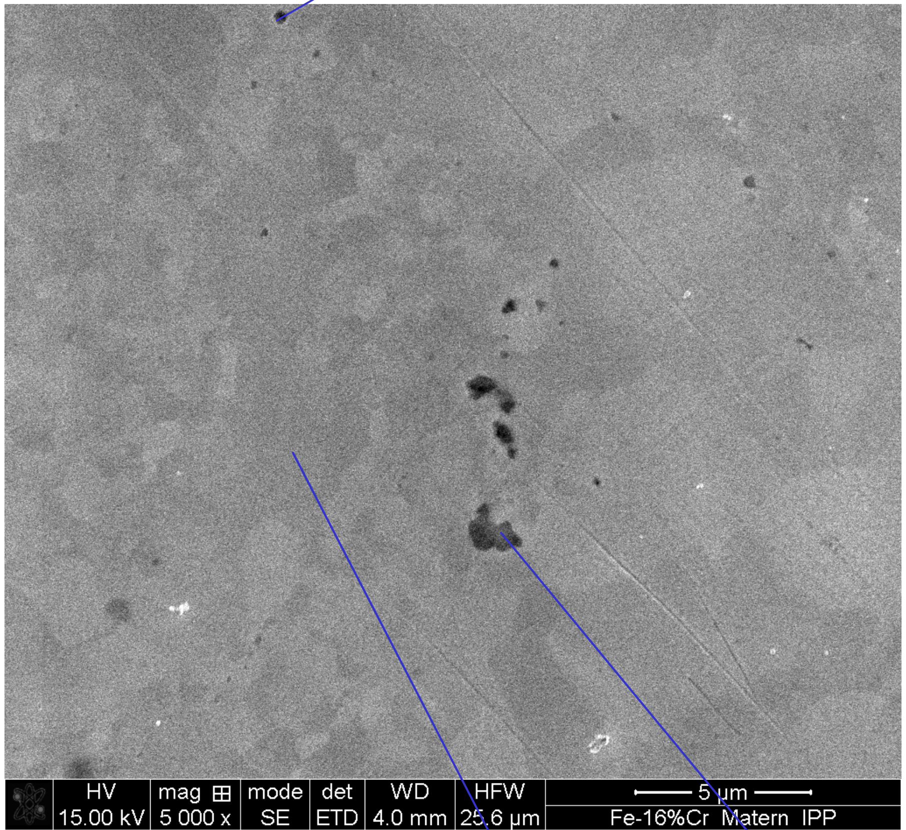
❖ **Weakly bounded D**

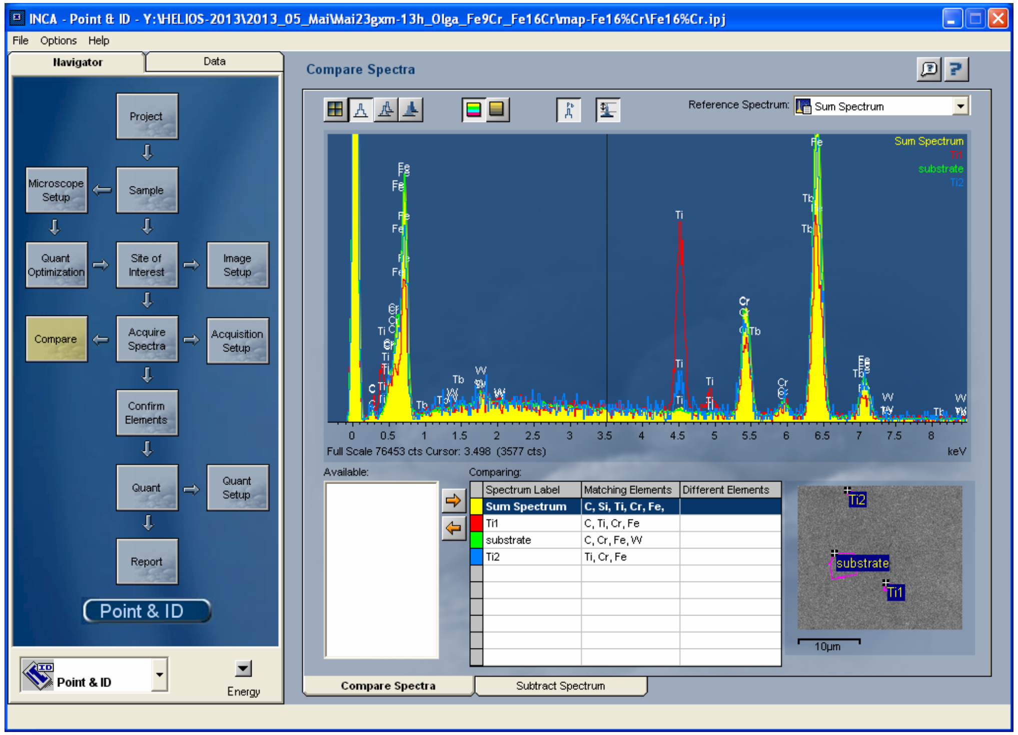
Weakly bounded D was desorbed

Negligible change in the D retention in the damaged zone after several months

D retention as a function of tem

Ti-Cr

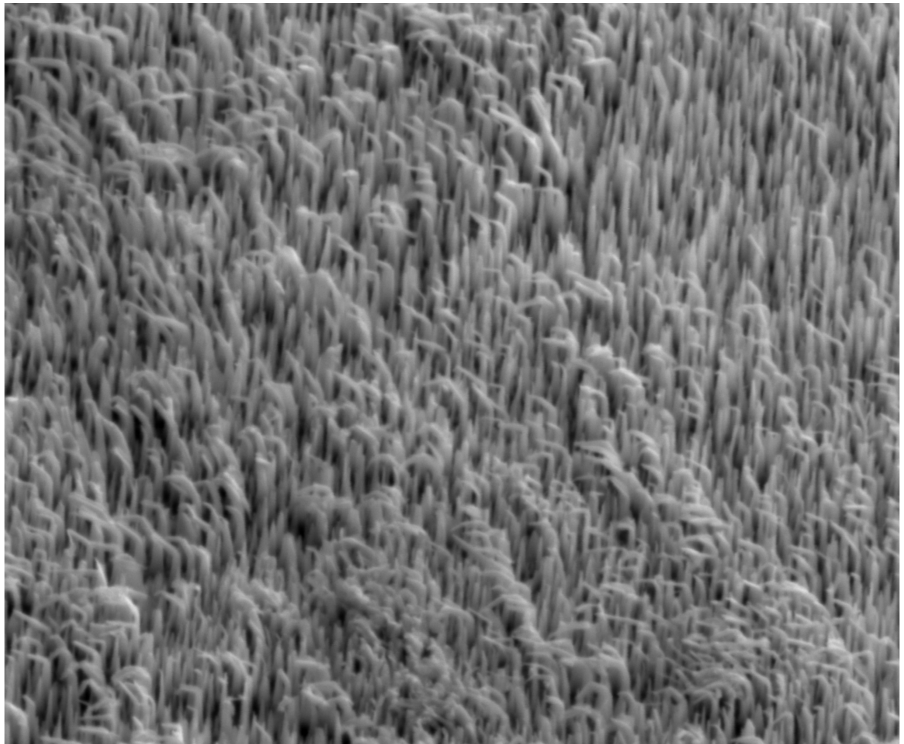


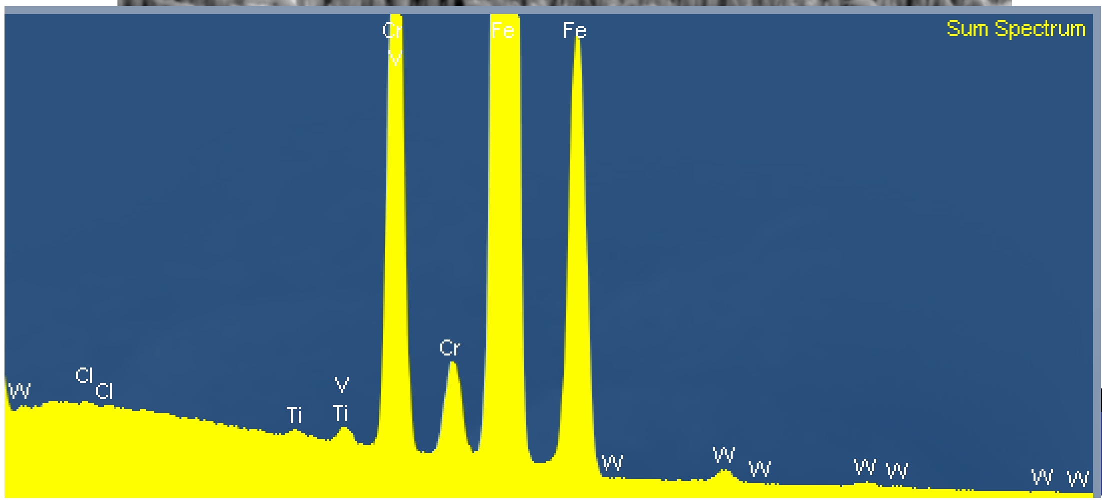


Substrate: C,Cr, W, Fe

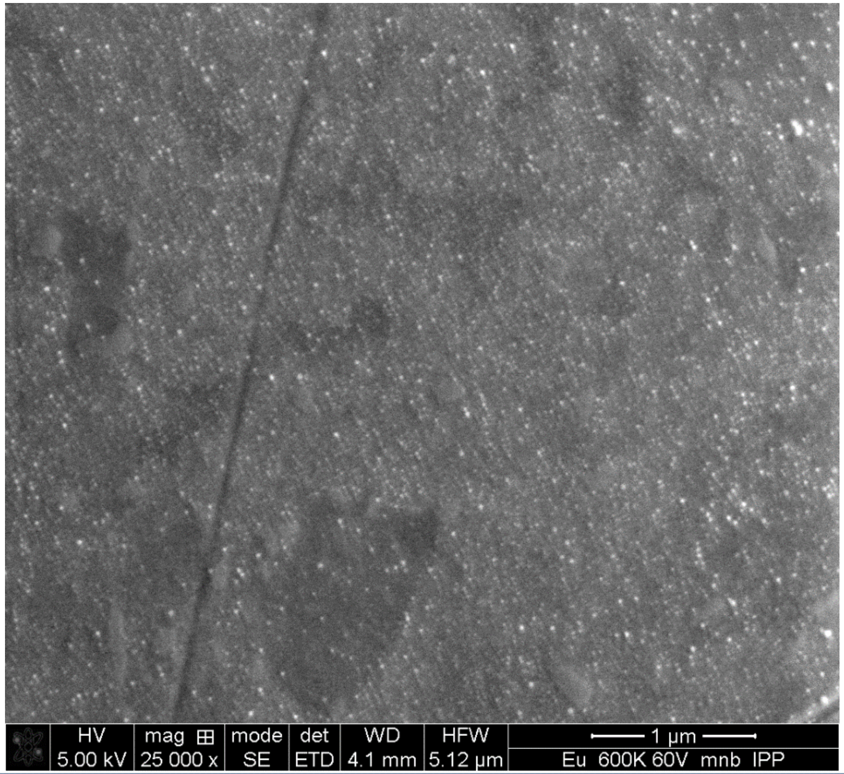
Ti-Cr + small C

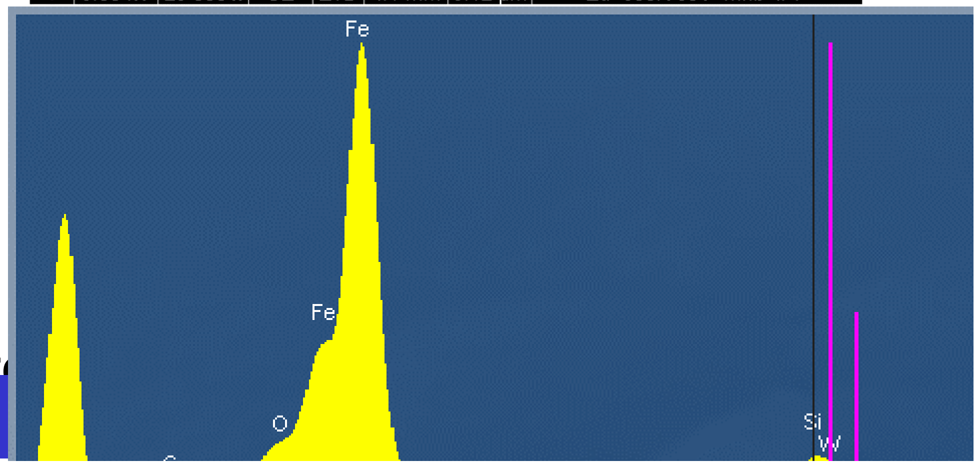
Eurofer, 600 K, 600 V



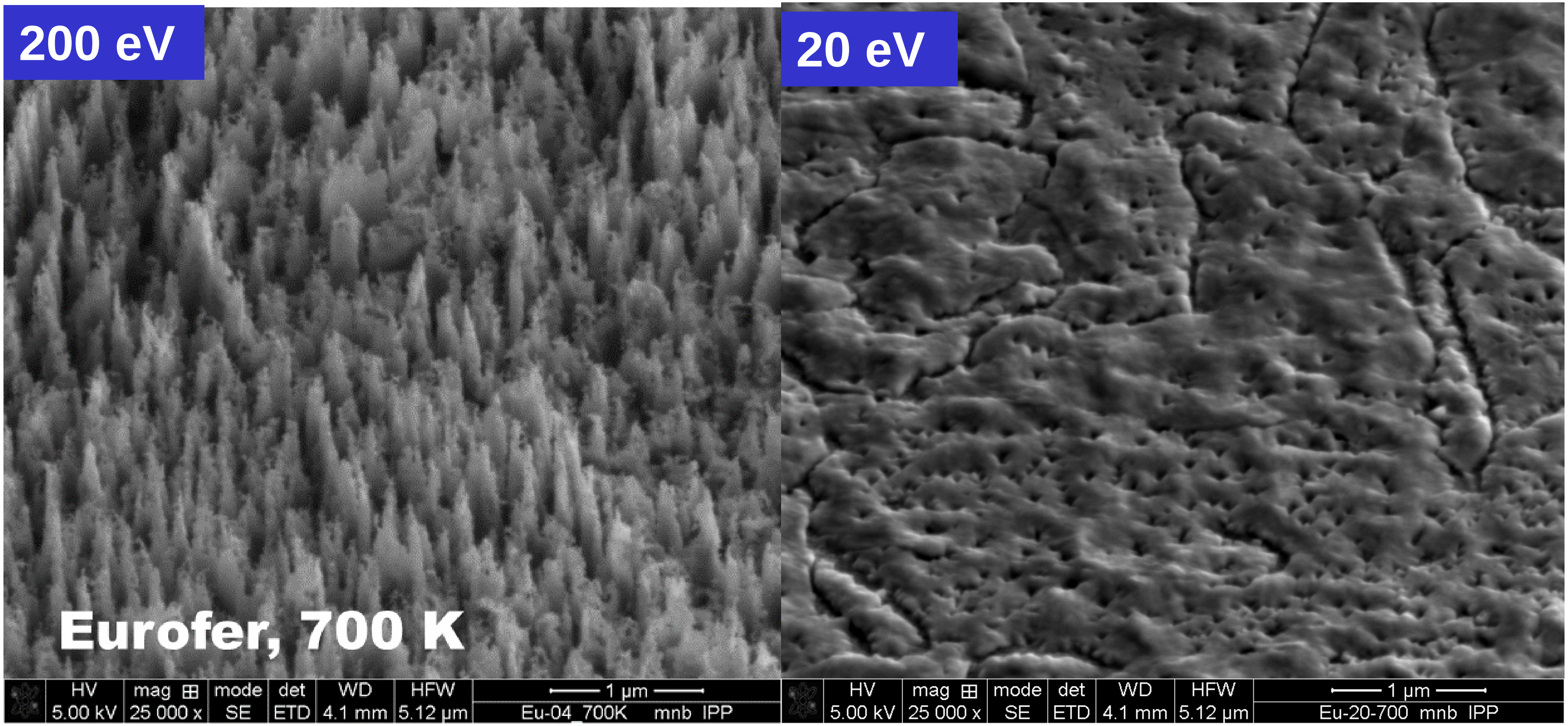


Eurofer, 600 K, 60 V





Surface morphology: energy effect



Even subthreshold D energy of 20 eV can cause sputtering, many pinholes are observed at elevated temperatures