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Effect of He on D retention in various RAFM steels

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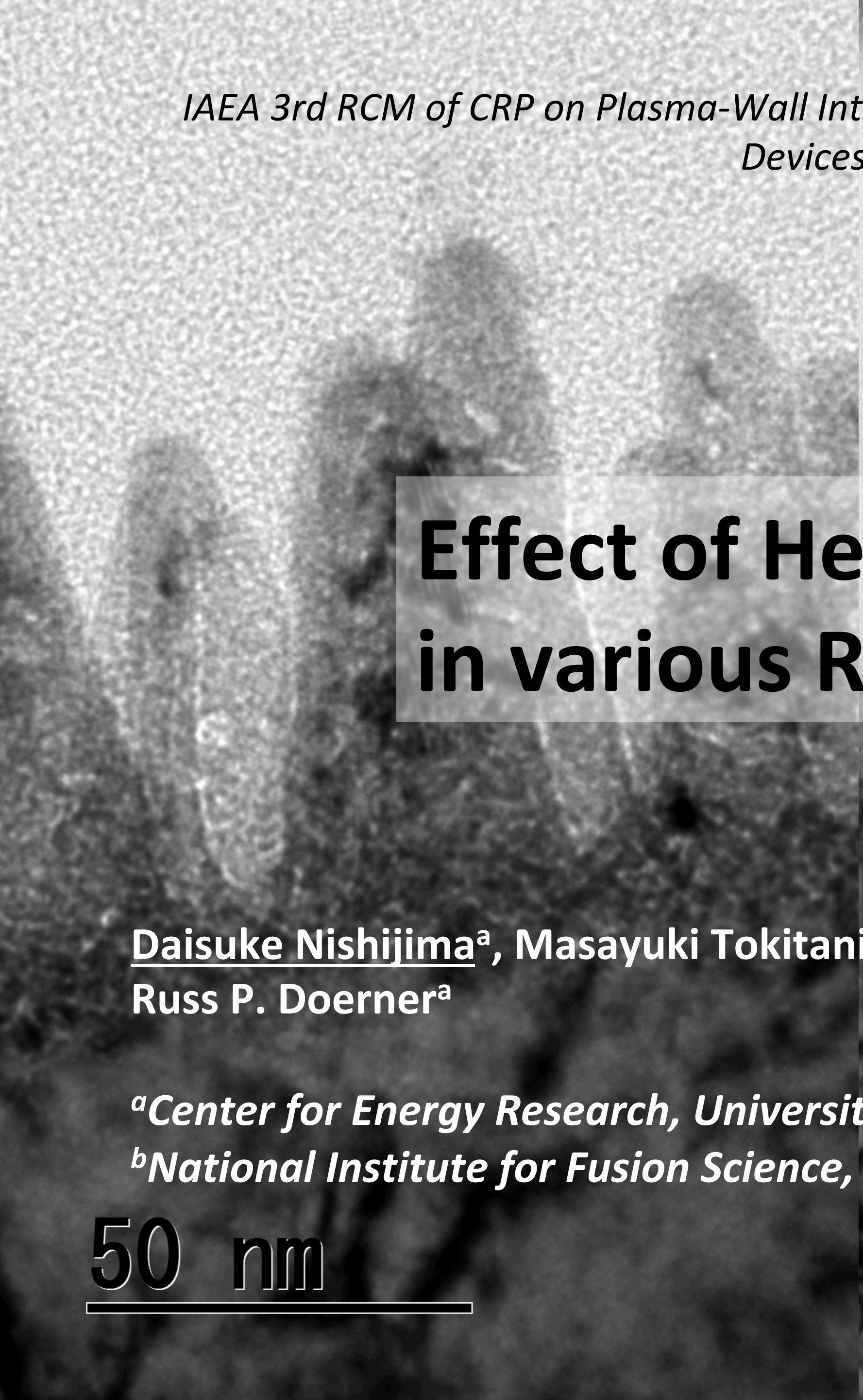

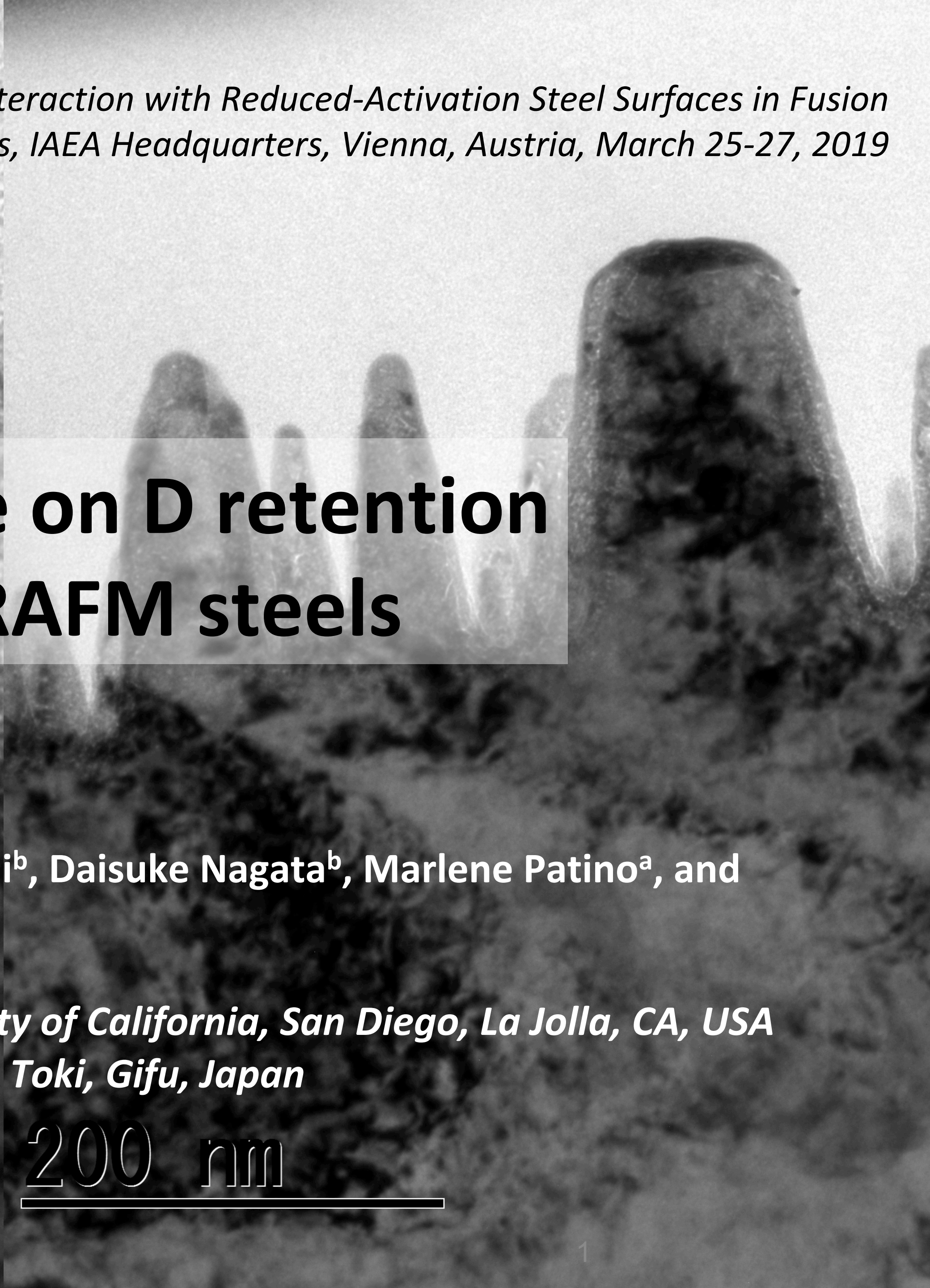

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- **Material dependence of D retention caused by pure D plasma exposure**
  - Strong material dependence
- **Fluence dependence of D retention caused by pure D plasma exposure**
  - Counter-intuitive fluence dependence
- **He effect on D retention**
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- **Analysis of TDS spectra**
  - Low and high temperature D desorption components
- **He retention**
  - Weak material dependence
- **Microstructures in the near-surface region**
  - Formation of high-density He bubble layer
- **Surface morphology and composition**
  - Formation of cones and W surface enrichment

Various RAFM steel samples were examined. Thank you for providing us with the samples.

PISCES

	Sample size (mm)	Exposed area (mm)	Thickness (mm)	Surface
CLF-1	12x10	10x8	1.0	Mirror-polished
Eurofer	15x12	12x9.2	0.65	Mirror-polished
F82H	ϕ25	ϕ22	1.9	Mirror-polished
Rusfer	10x8.5	7.5x6.5	0.57	As-received (not polished)

- Outgassed at T = 500 C (773 K) for 1 hour before plasma exposure

- Chemical composition (wt%) of the RAFM steels as well as commercially available P92 FM steel

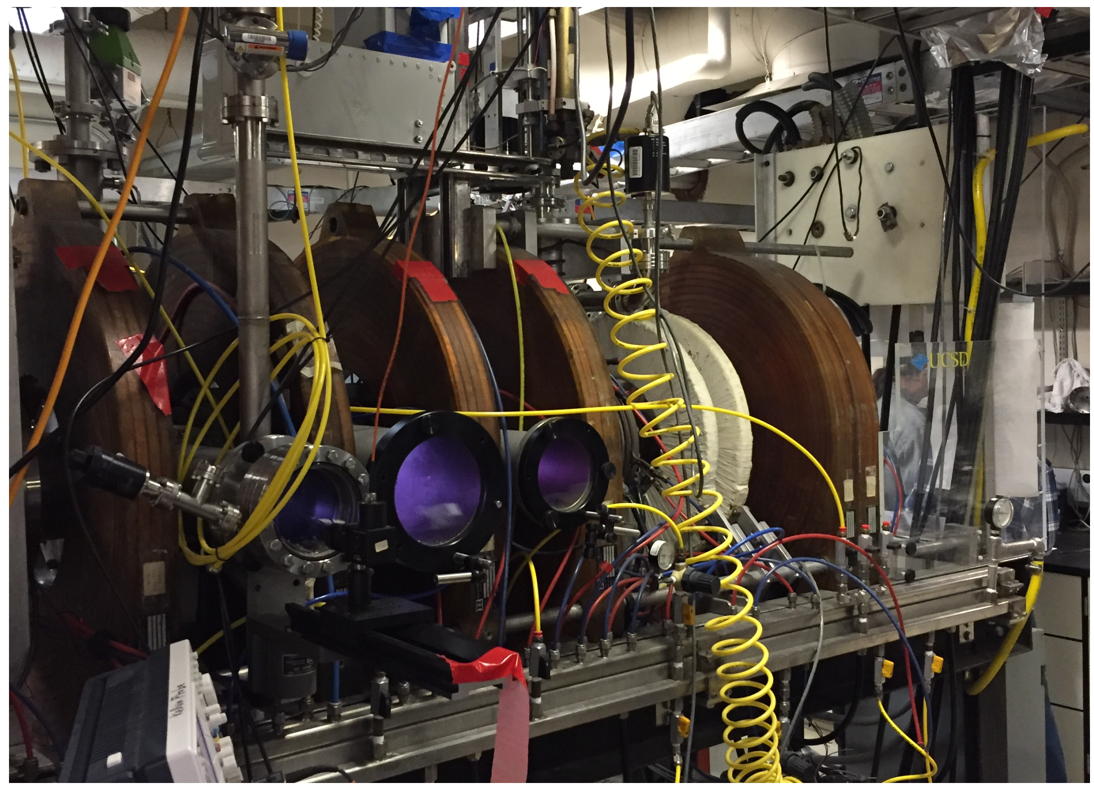
	Cr	W	V	Ta	C	Mn	Si	Ni
CLF-1	8.5	1.5	0.25	0.1	0.1	0.5	-	-
Eurofer	9	1.1	0.2	0.1	0.1	0.4	-	-
F82H	8	2	0.2	-	0.1	0.2	0.1	-
Rusfer	11	1.1	0.25	0.1	0.15	0.7	0.3	-
P92	8.5-9.5	1.5-2	0.15-0.25	-	0.07-0.13	0.3-0.6	≤ 0.5	≤ 0.4

Fe: Balance

Plasma exposures to the RAFM steels were conducted in PISCES-A at UCSD.

- Three types of plasma exposure were made:
  1. Pure D plasma exposure
  2. Sequential pure He and pure D plasma exposure
  3. Simultaneous D and He mixed plasma exposure

- Plasma conditions:
  - ϕ_D ~ 1e25 m^{-2} (~2e23-4e25 m^{-2} for ϕ_D scan)
  - ϕ_{He} ~ 1e24 m^{-2}, 0.5e24 m^{-2}
  - c_{He+} ~ 10%, 5%
  - T_s ~ 100 C (373 K), 250 C (523 K)
  - E_i ~ 100 eV
  - Γ_i ~ 1.5-2e21 $m^{-2}s^{-1}$
  - ✓ The plasma parameters were measured with a reciprocating single Langmuir probe system.
  - ✓ c_{He+} was measured with a spectroscopic technique.
  - ✓ E_i was controlled with target biasing.



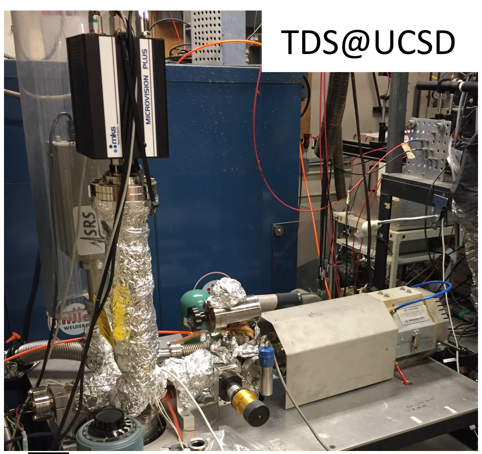
Before and after plasma exposure, RAFM steel samples were analyzed with several techniques.

- Thermal desorption spectroscopy (**TDS**): D (as well as He) retention
  - Both HD and D_2 signals were taken into account.
  - D_2 and He signals were resolved with a high resolution RGA.
  - TDS temperature: ~ 300 – 1130 K
  - 0.25 K/s ramping rate
- Focused ion beam (**FIB**)-scanning electron microscopy (**SEM**): Sample preparation for TEM and surface observation
- Transmission electron microscopy (**TEM**) w/ Energy dispersive X-ray spectroscopy (**EDX**): Surface and internal microstructure/composition
- Sputter-Auger electron spectroscopy (**AES**): Depth profile of near-surface composition
- **Microbalance**: sample mass loss

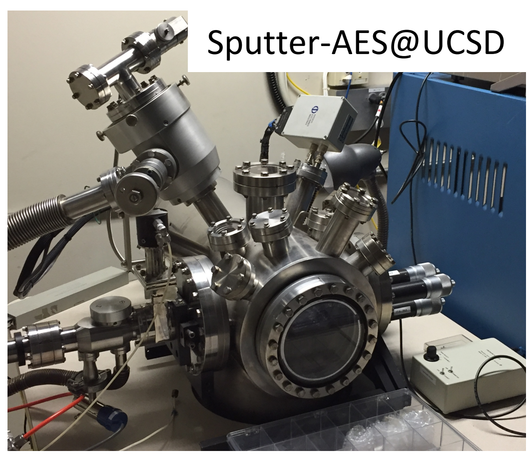
FIB-SEM (HITACHI NB5000) @NIFS

TEM (JEOL JEM-2800)@NIFS

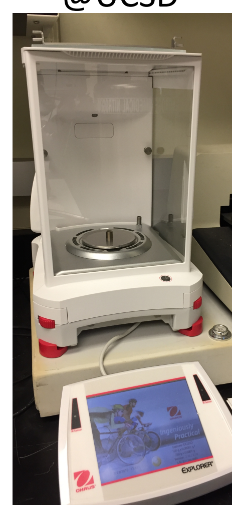
TDS@UCSD



Sputter-AES@UCSD



Microbalance @UCSD



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D retention caused by pure D plasma exposure depends strongly on the type of RAFM steels.

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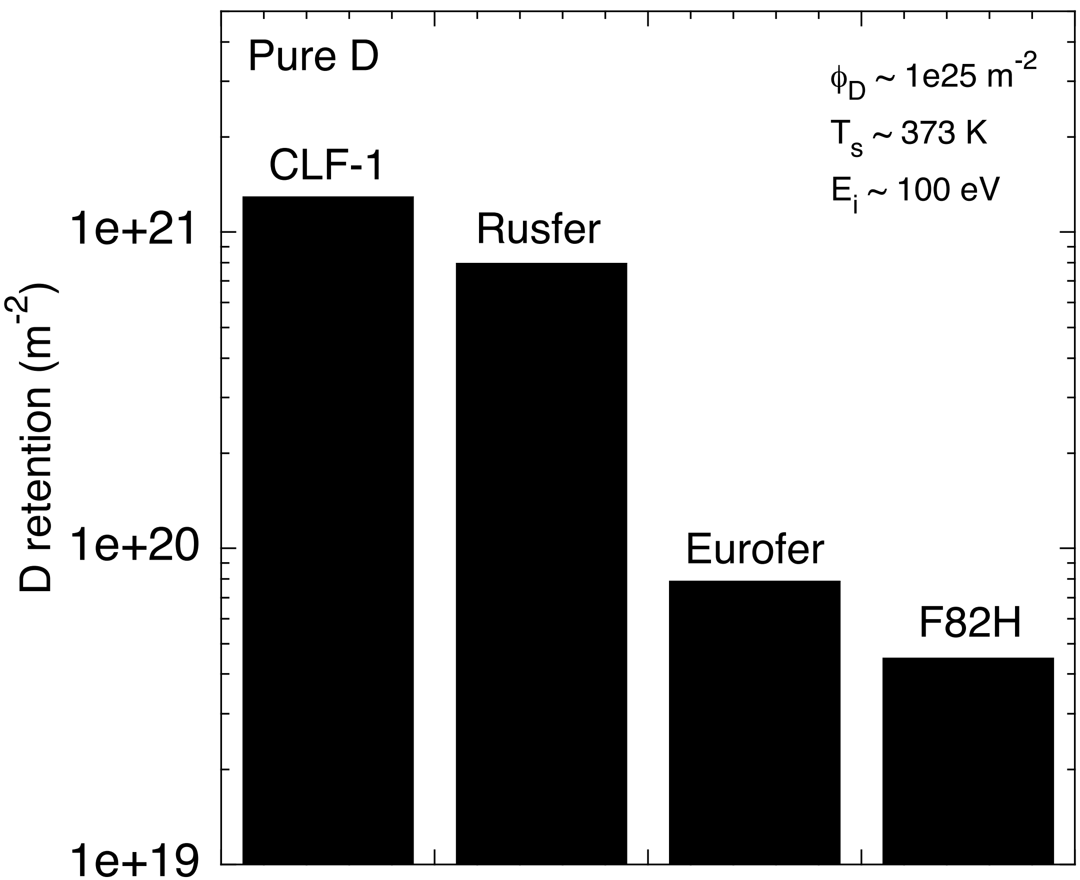
- **Up to ~30 x difference** between CLF-1 and F82H even with the same plasma exposure condition as well as the similar nominal composition.



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A higher Cr content in the near-surface region may lead to the higher D retention. Formation of Cr-D or Cr-O-D?

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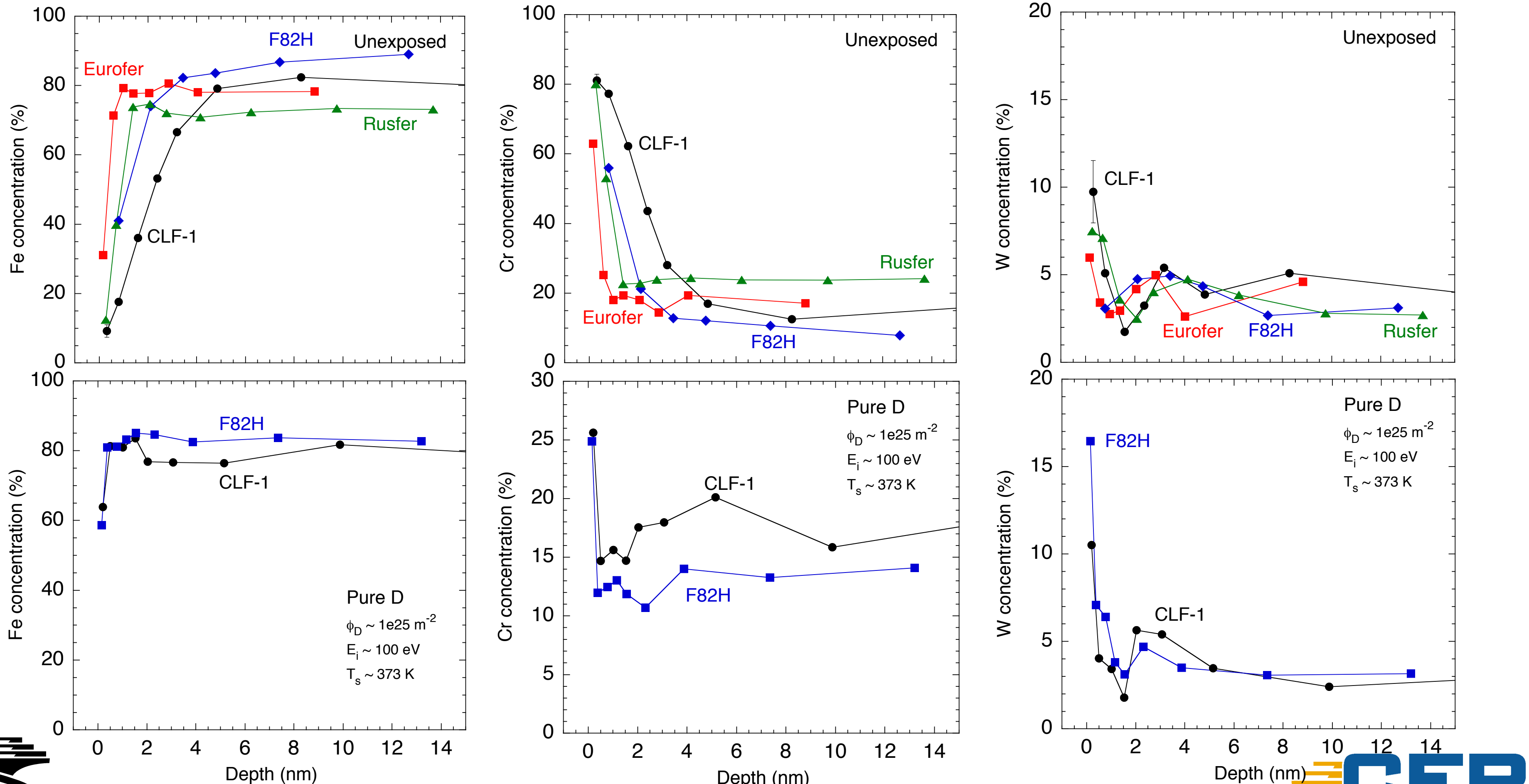
- Sputter-AES analysis of unexposed surfaces shows "less Fe and more Cr" for CLF-1 and Rusfer, having a higher D retention caused by pure D plasma exposure.
- More Cr than Fe diffuse to the surface during outgassing at 500 C before plasma exposure.
- After pure D plasma exposure, near-surface Cr concentration for CLF-1 is still higher than that for F82H.



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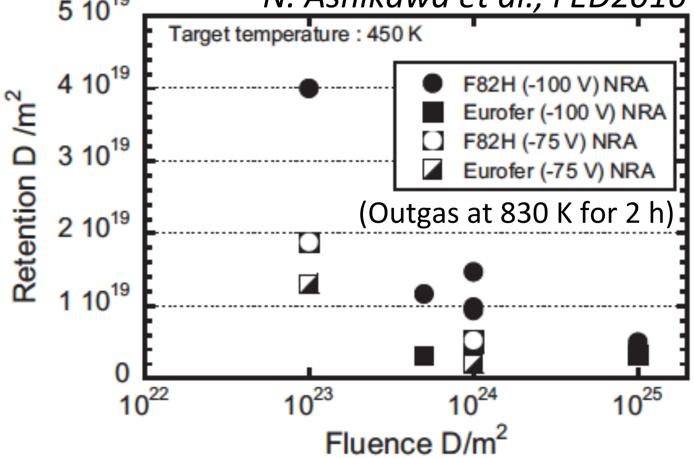
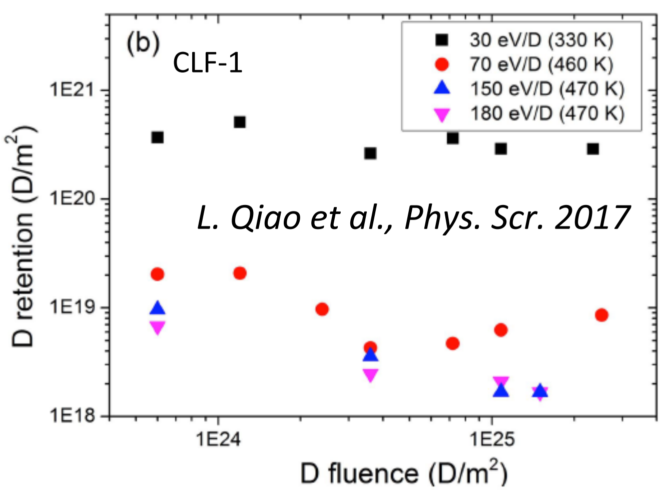
The counter-intuitive fluence dependence of D retention is observed also in PISCES-A.

- Two previously published studies reported a decrease in D retention with increasing the fluence.

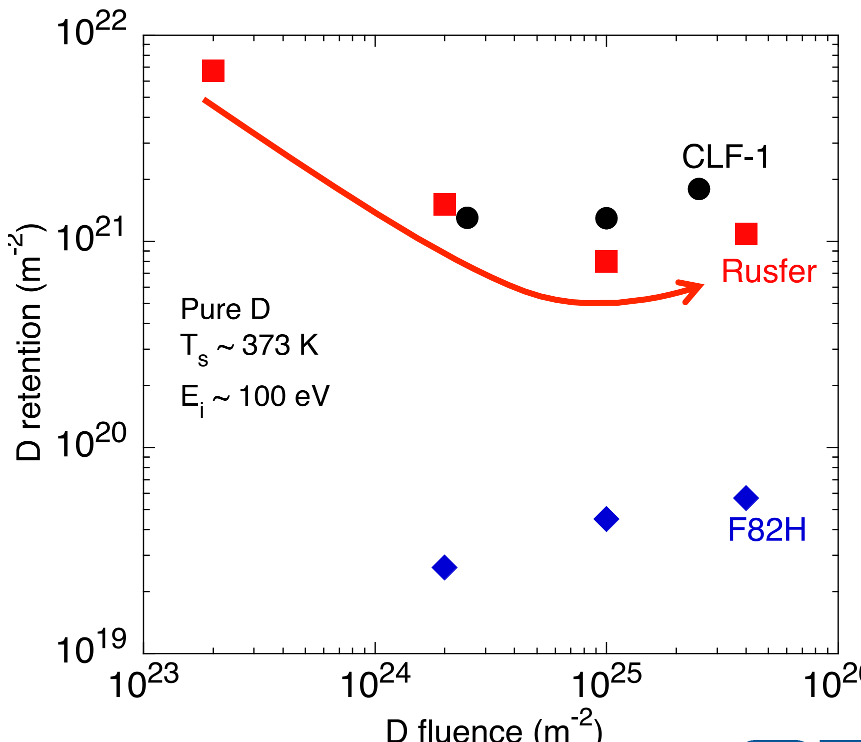
N. Ashikawa et al., FED2016





(Outgas at 873 K for 1 h)

- The D retention decreases with increasing the D fluence at the lower fluence range.
- It seems that the D retention slightly increases with increasing the D fluence at the higher fluence range.



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The counter-intuitive fluence dependence can also be explained by the Cr content in the near-surface region.

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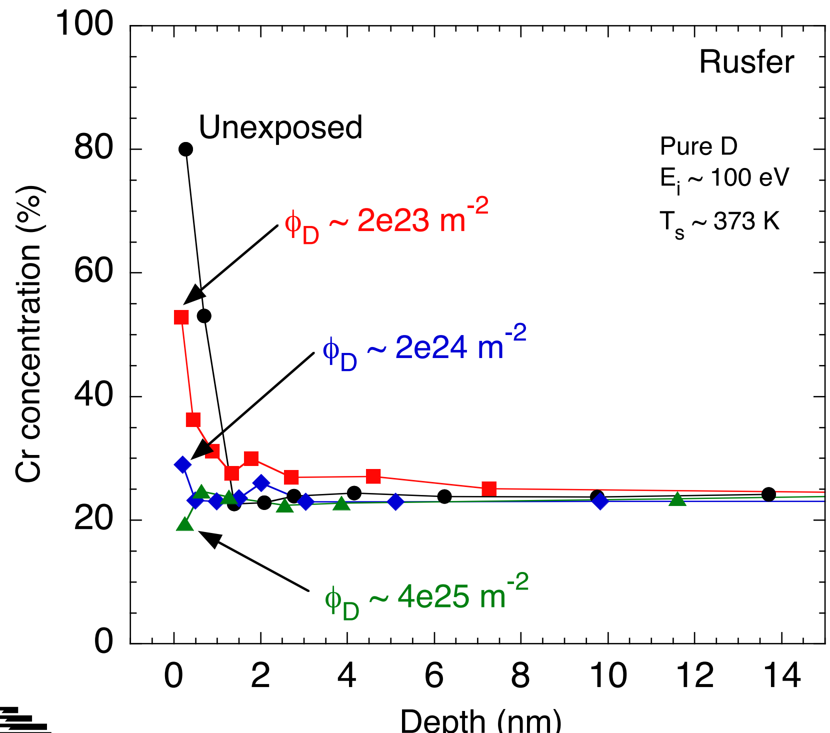
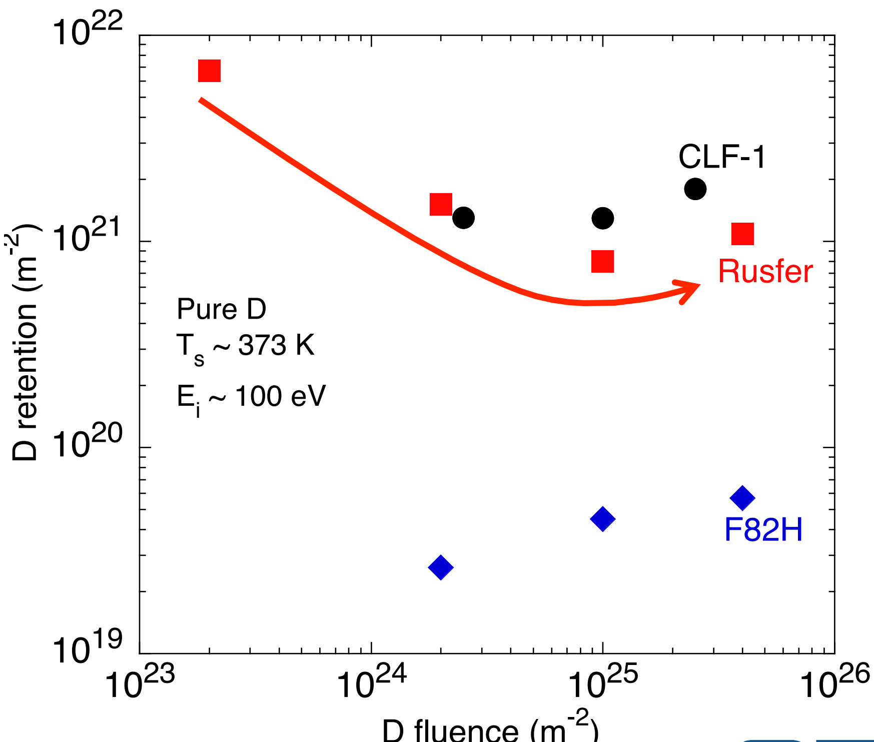
- **A Cr-rich surface layer** is formed as a result of the Cr diffusion to the surface during outgassing at 500 C before plasma exposure.
- **As the D fluence increases** during plasma exposure, **the Cr-rich surface layer is progressively sputtered**, and then **the Cr concentration decreases**. Correspondingly, **the D retention decreases**.
- At a certain D fluence, where the Cr-rich surface layer is removed, the reduction of D retention stops, and the D retention increases with increasing the D fluence.





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Two contradicting results have been reported on He effect on D retention in RAFM steels.

PISCES

- D retention slightly decreases with He admixture.

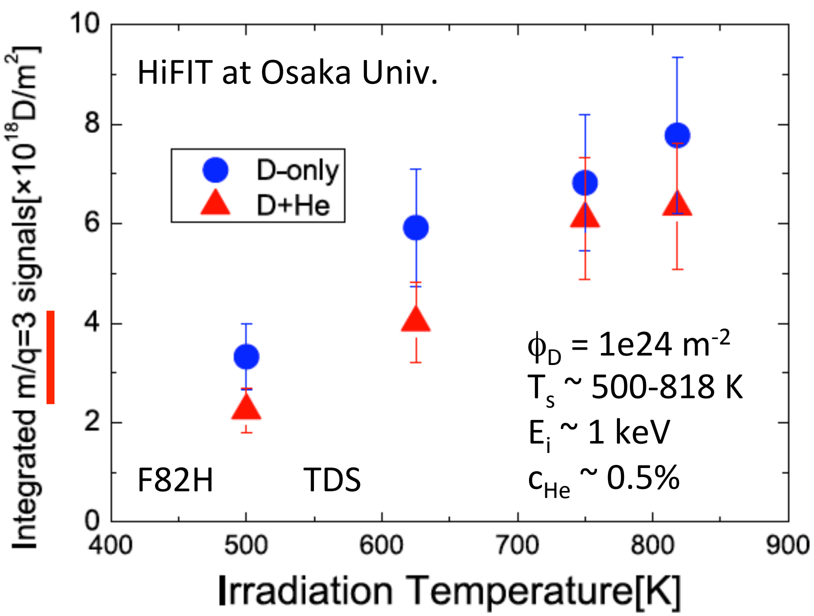

Figure 3. Integrated $m/q = 3$ signals from figure 2 as function of irradiation temperature which clearly shows a systematic decrease in retention due to He.

K. Yakushiji et al., Phys. Scr. T167 (2016) 014067

(Outgas at 1000 K for 1 h)

- D retention increases with He admixture by a factor of > 2.

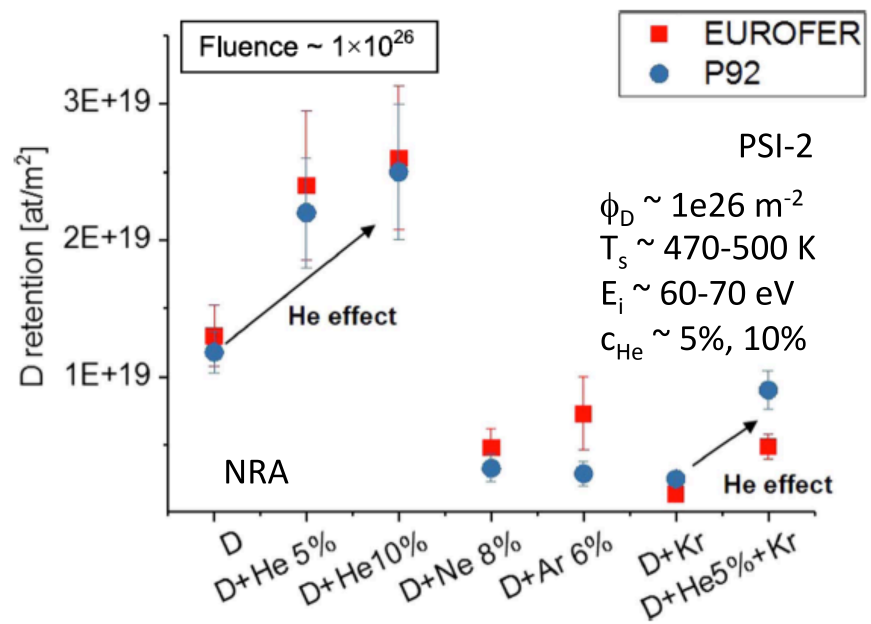

Figure 7. Integral D retention up to 8 μm with respect to plasma composition, measured by NRA using a 4.5 MeV ^{3}He beam.

M. Rasinski et al., Phys. Scr. T170 (2017) 014036

(Outgas at 723 K for 1 h)

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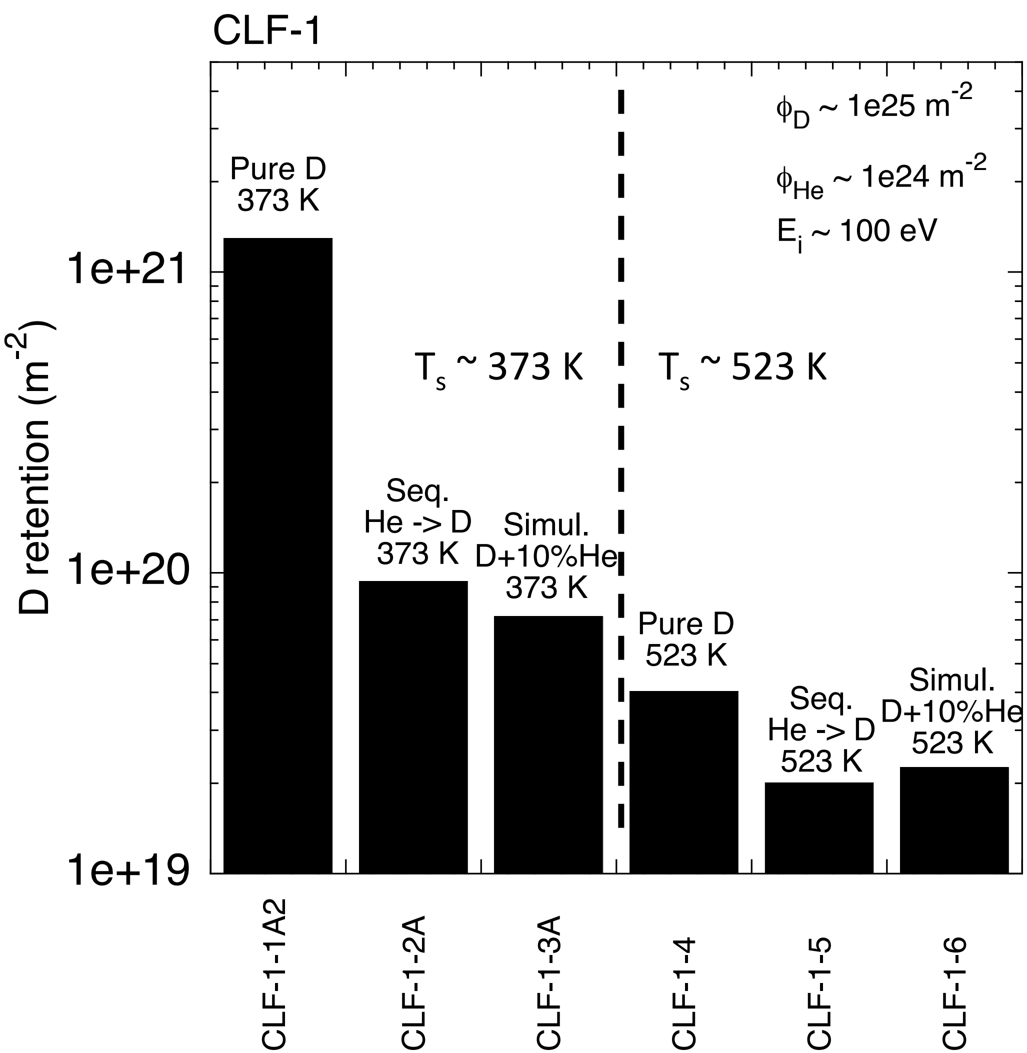
CLF-1: D retention is reduced with He by a factor of ~10 at T_s ~ 373 K and ~2 at T_s ~ 523 K.

- Addition of He, regardless of sequential or simultaneous exposure, leads to a reduction of D retention.

- T_s dependence:
  D retention, especially for the pure D case, significantly decreases with increasing T_s from 373 K to 523 K.



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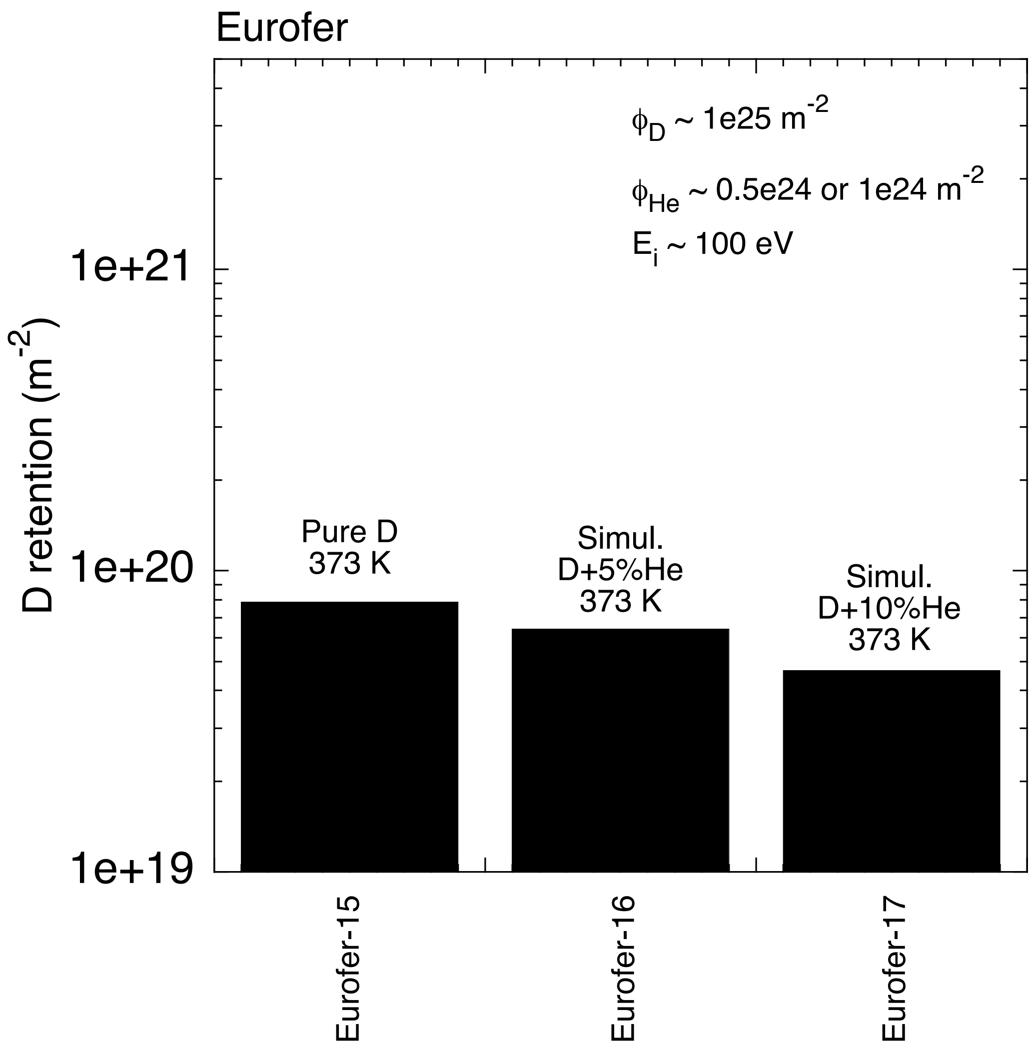
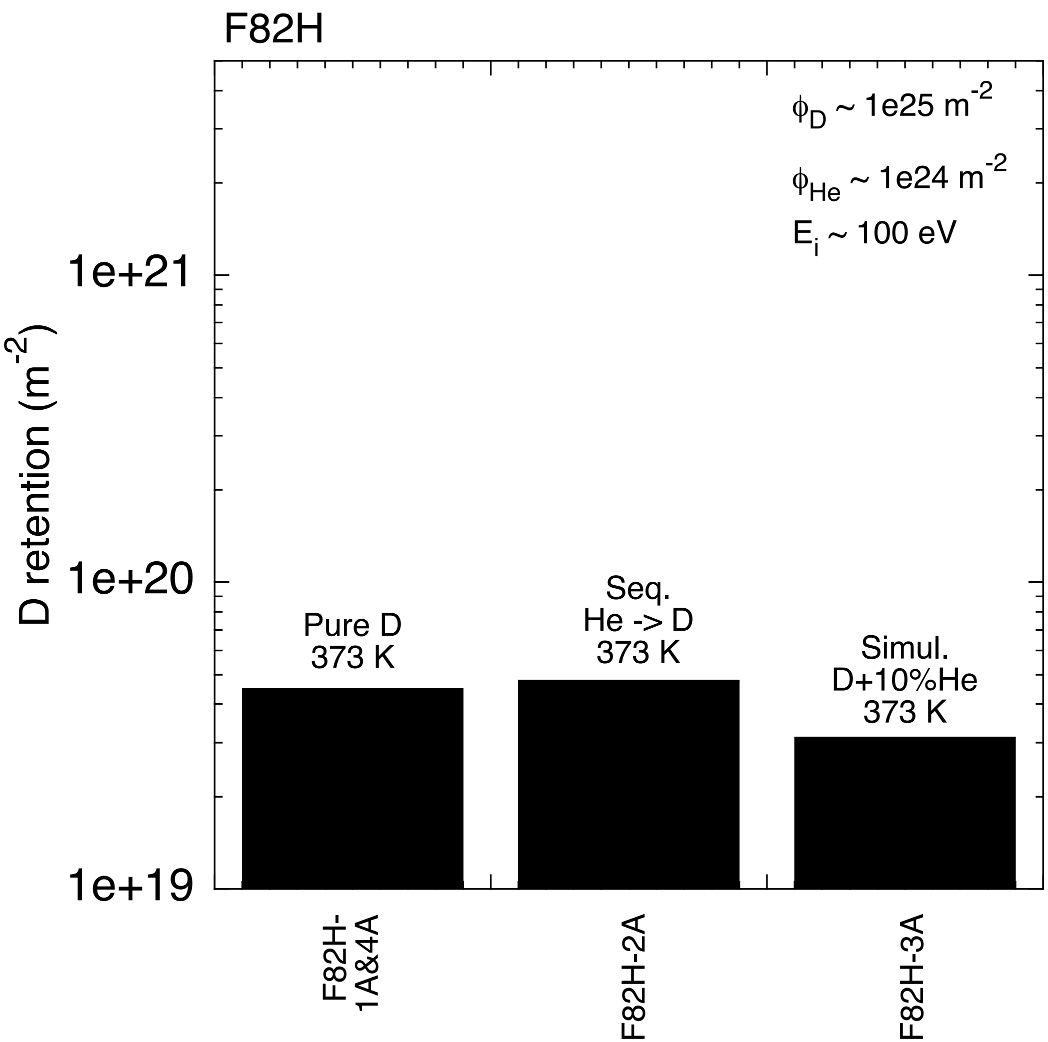
Eurofer: D retention decreases with admixture of He, but the reduction factor is only ~2 with 10% He.

- D retention decreases with increasing the fraction of He
- No increase in D retention was observed with admixture of He, contradicting Rasinski et al., (2017).



F82H: Only a slight decrease in D retention was observed with admixture of He.

- It seems to be consistent with Yakushiji et al. (2016), which also shows a slight decrease in D retention with He mixture. Discussed further later.



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Rusfer: D retention is reduced with He by a factor of ~2 for seq. exposure and ~6 for simul. exposure.

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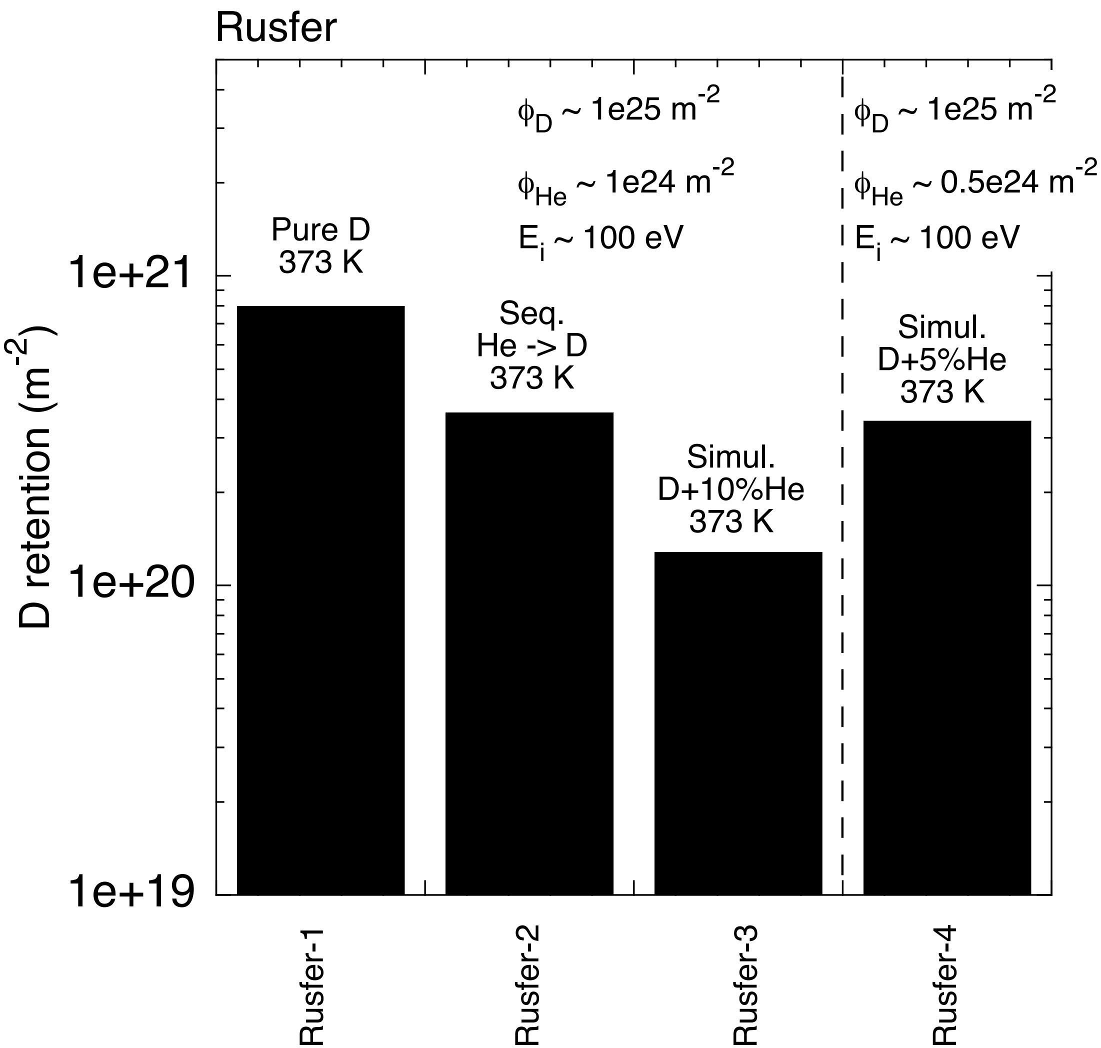
- The reduction of D retention is smaller compared to CLF-1, especially for the sequential exposure case.
- In the simultaneous exposures, D retention decreases with increasing the fraction of He (5% → 10%), which is consistent with Eurofer.



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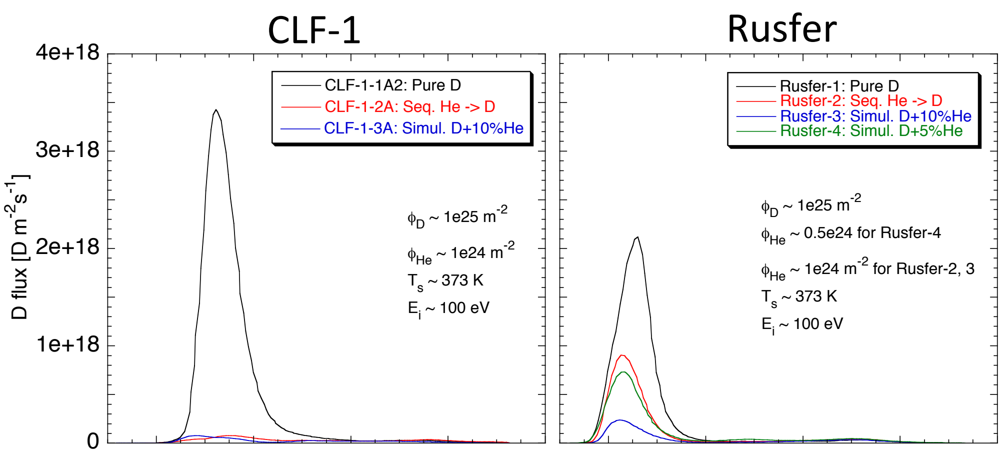
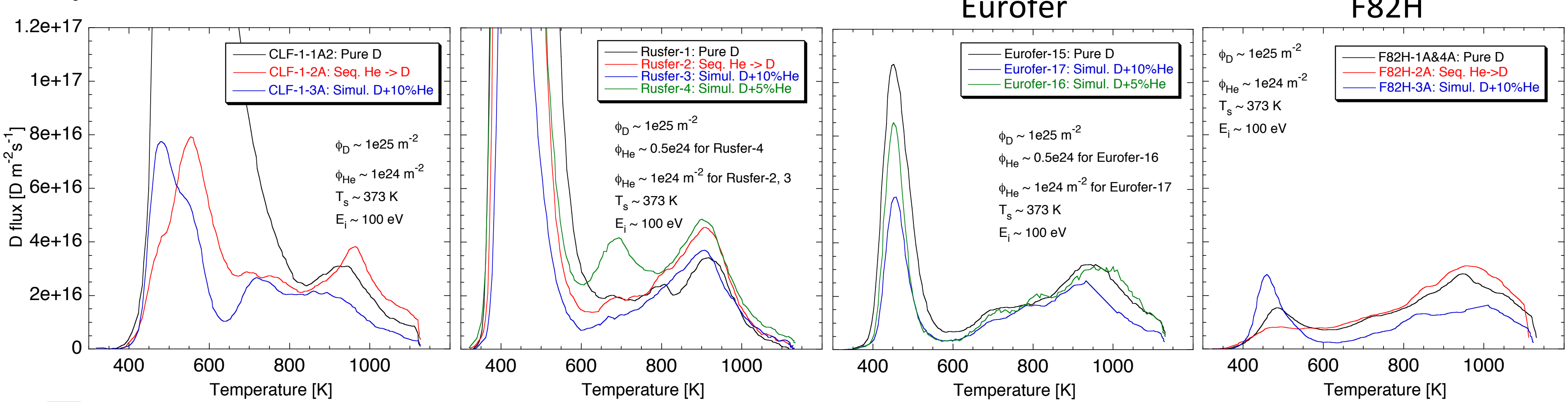
Low temperature D desorption is more sensitive to admixture of He and the type of RAFM steels.

- D desorption can be separated into low (< 600 K) and high (> 600 K) temperature components.
- Low temperature D desorption is more sensitive to
  (1) admixture of He and
  (2) the type of RAFM steels,
  compared to high temperature desorption.

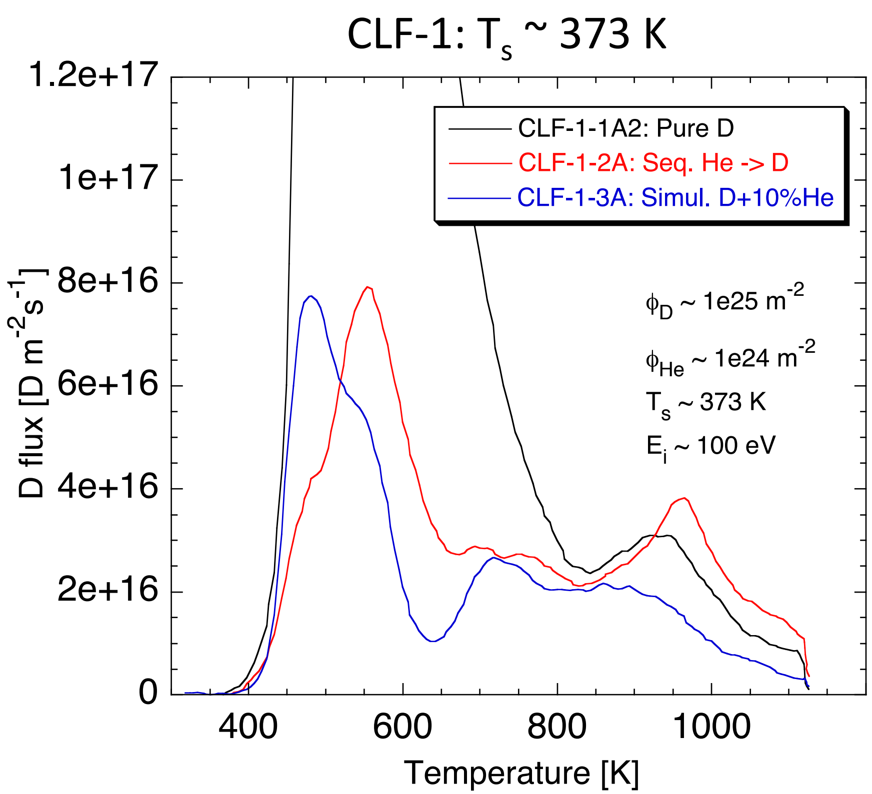


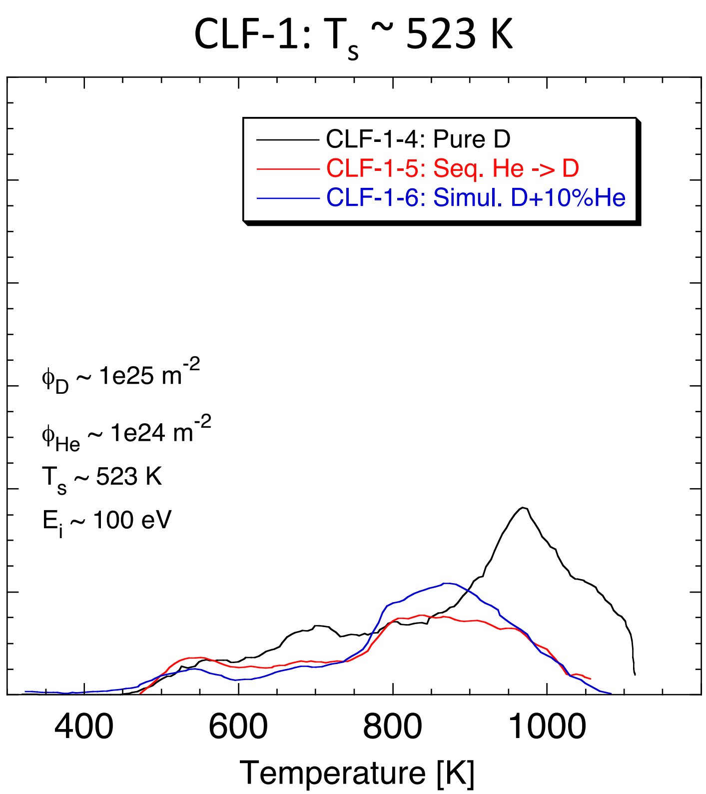


In addition, low temperature D desorption is more sensitive to the sample temperature.

PISCES

- Low temperature D desorption nearly disappears at a higher T_s ~ 523 K.





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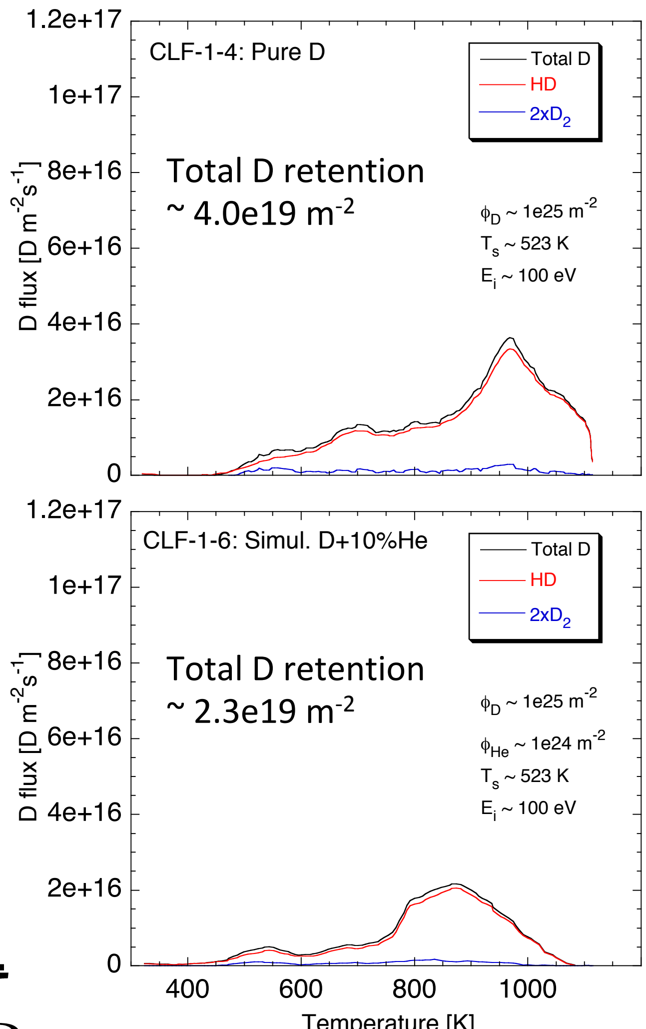
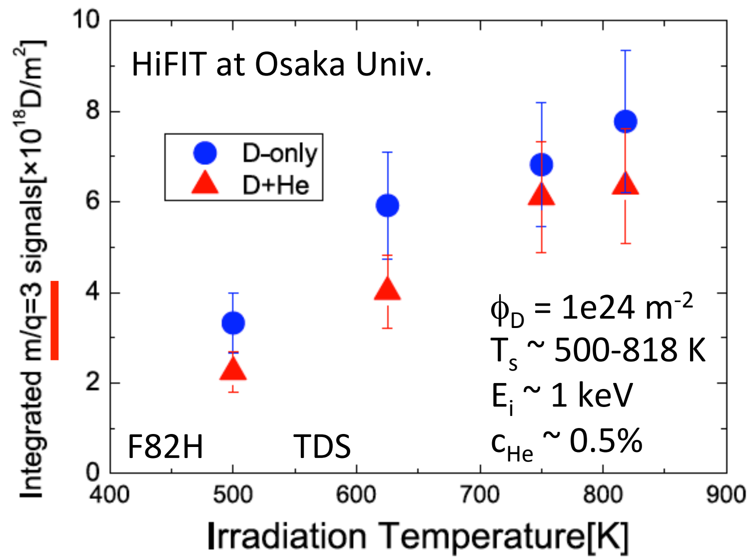
Our result at higher T_s ~ 523 K seems to be consistent with Yakushiji et al., at T_s ~ 500-818 K.

PISCES

- D retention is dominated by HD release (~90%) at a higher T_s ~ 523 K



- D retention obtained only from HD signal, by Yakushiji et al., should be close to total D retention because of the high T_s ~ 500-818 K.
- Reduction of D retention with He is consistent.
- Note that D retention decreases with T_s from 373 to 523 K in PISCES-A, while it increases with T_s from 500 to 818 K.



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K. Yakushiji et al., Phys. Scr. T167 (2016) 014067

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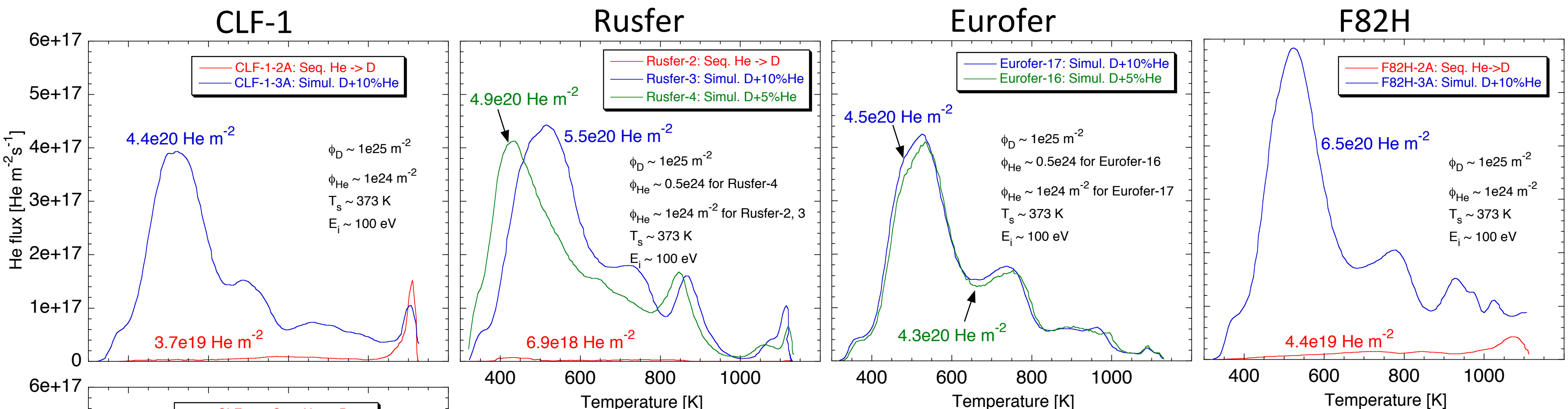
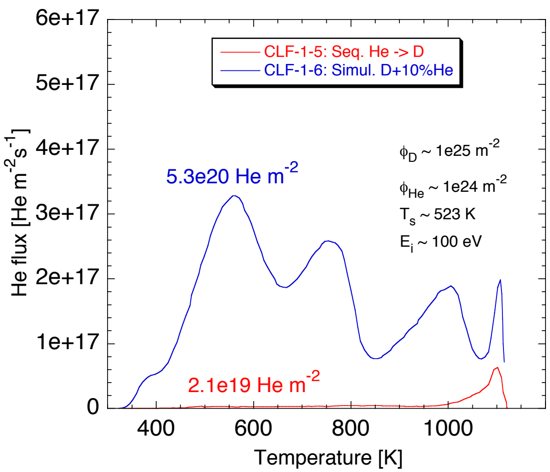
The material dependence of He retention in RAFM steels is weak unlike D retention.





- He retention for sequential exposure (He → D) is more than 10x lower than that for simultaneous D+He exposure, while the incident He fluence is the same for both cases.
  - Discussed later.
- He retention saturates already at ϕ_{He} ~ 0.5e24 m^{-2}.
- The T_s dependence of He retention is weak.

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TEM observations reveal that high-density He bubble layer is formed in the near-surface region only for a sample exposed to simultaneous D+He mixed plasma.

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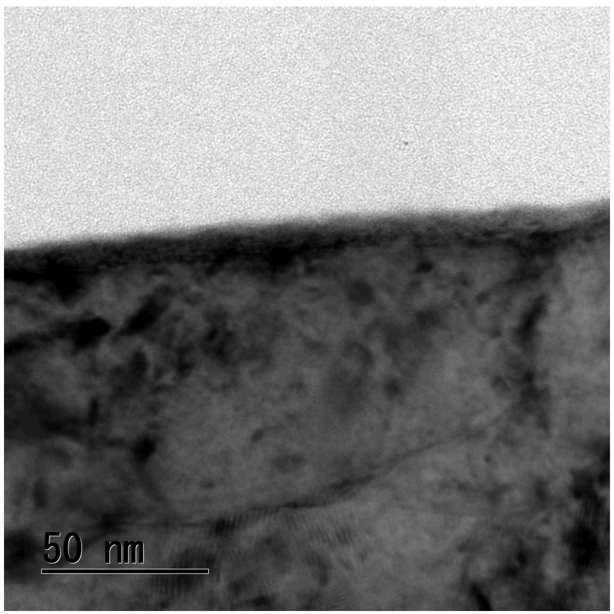
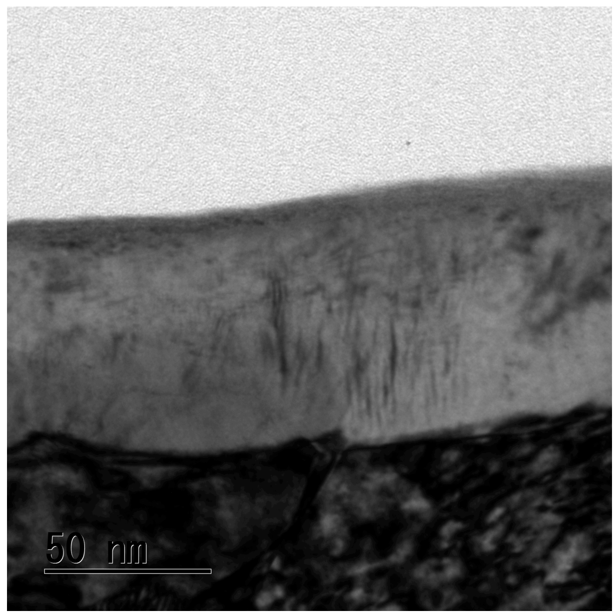
- A significant surface morphology change (cone structures) is seen only for a sample exposed to simultaneous D+He mixed plasma.
- He bubbles are also seen inside the cones.

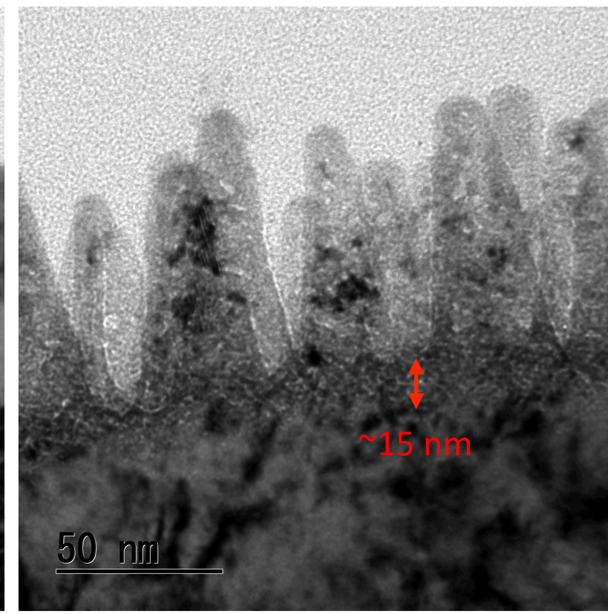
CLF-1-1B: Pure D, 373 K



CLF-1-2B: Seq. He->D, 373 K



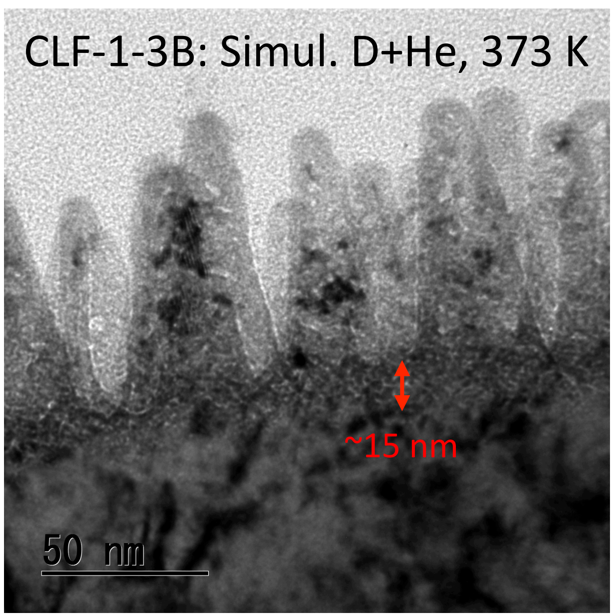
CLF-1-3B: Simul. D+He, 373 K

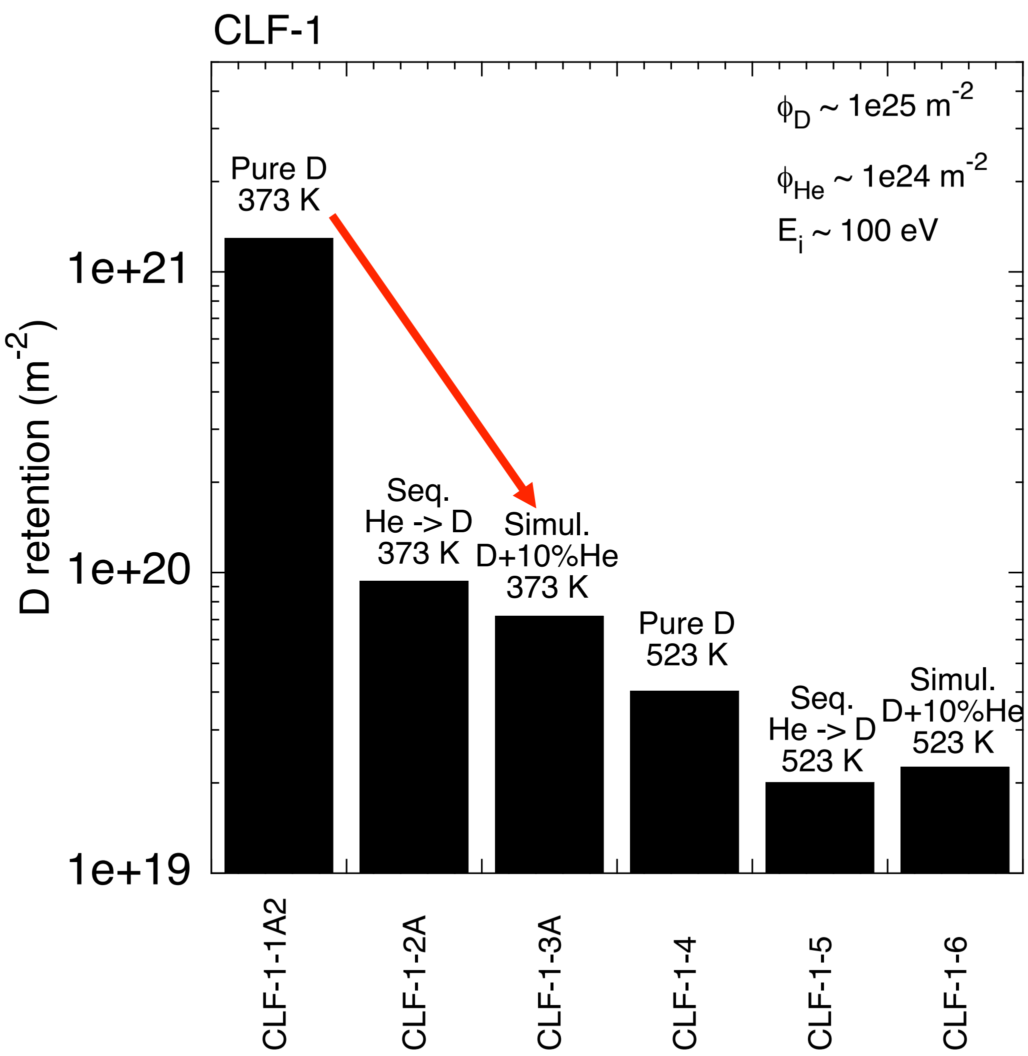


High-density He bubbles are considered to reduce D retention in the same way as for W.

PISCES

- High-density He bubbles can decrease the diffusion of D into the bulk and/or can increase the diffusion to the surface.
- Note that Rasinski et al., (2017) also observed He bubbles, but D retention increased with He admixture.





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Why is D retention reduced without high-density He bubbles for the sequential (He ->D) exposure case?

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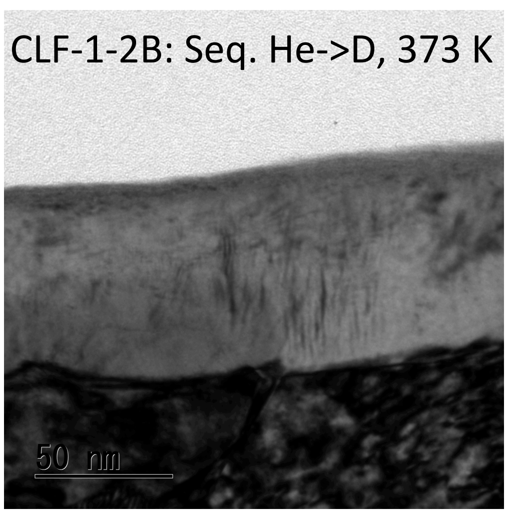
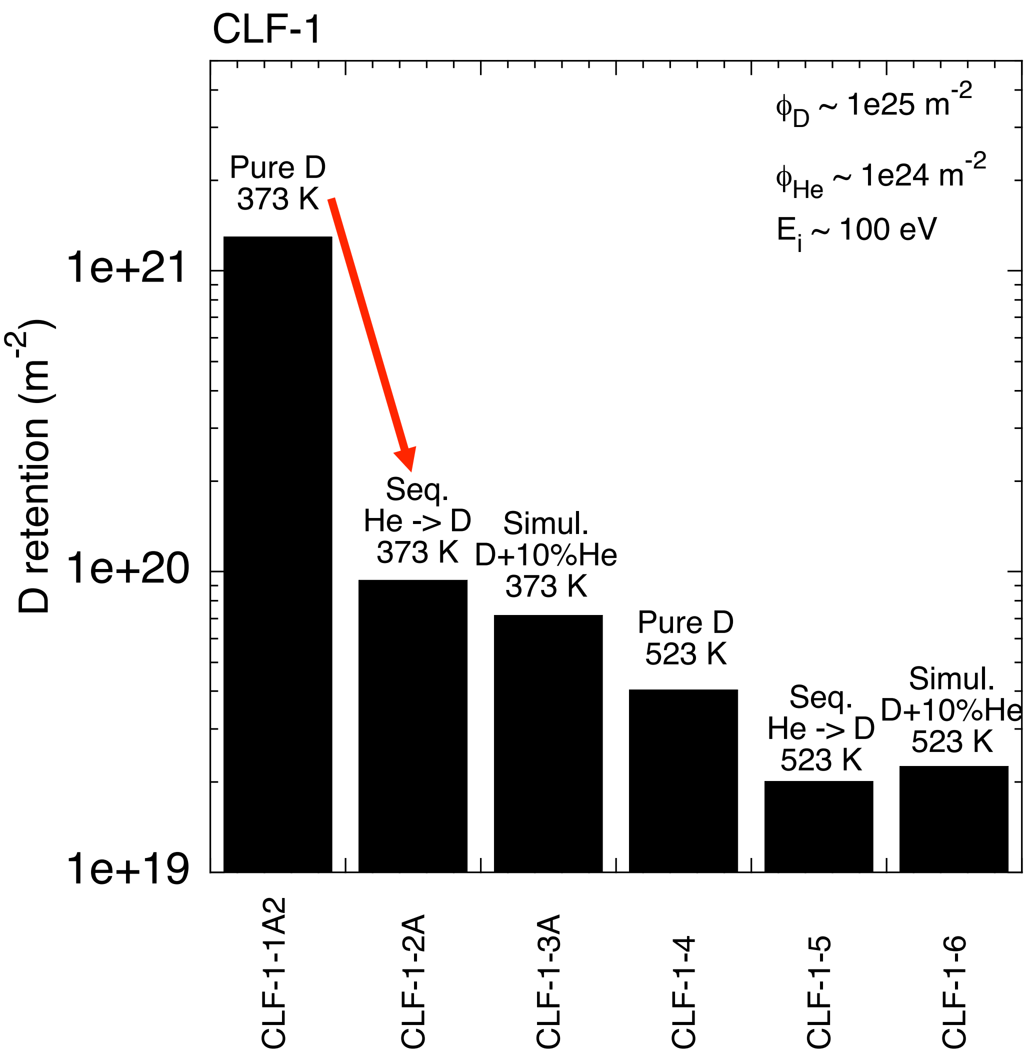
- A He bubble layer (~15 nm) should be formed during pure He plasma phase.
- But the layer can be sputtered during pure D plasma phase.
  - Erosion layer thickness during pure D phase is calculated to be ~70 nm from mass loss in pure D plasma exposure (CLF-1-1).
  - Consistent with the low He retention
- The He bubble layer does not exist during the entire pure D phase.
- But the D retention is reduced.





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The reduction of D retention for seq. (He -> D) case may be caused mainly by the lower Cr concentration.

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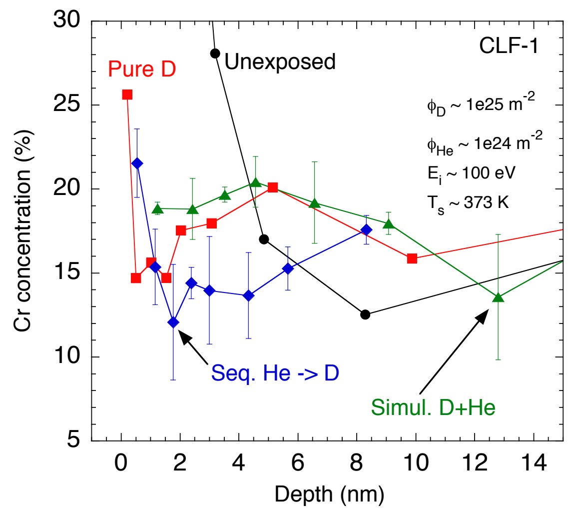
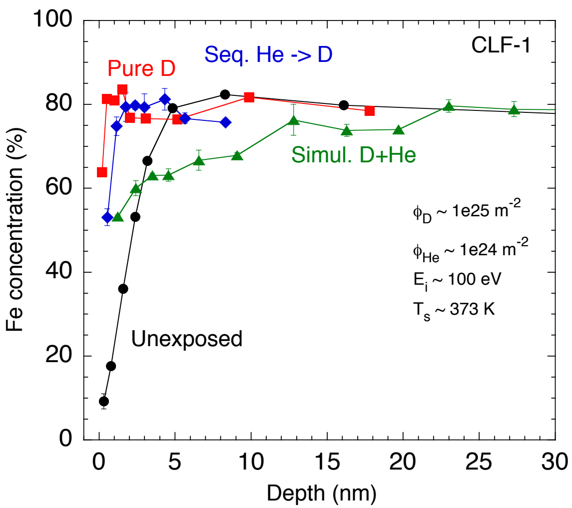
1. Before plasma exposure, Cr is dominant in the surface (< 5 nm) due to outgassing at 500 C.
2. During the pure He plasma exposure phase, Fe and Cr atoms in the near-surface are preferentially sputtered.
   → W surface enrichment

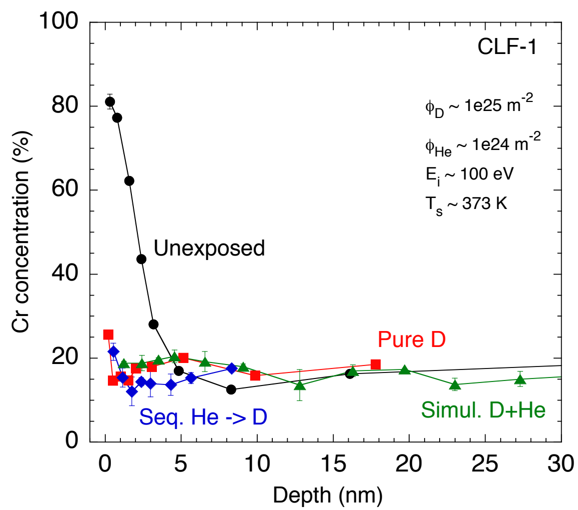


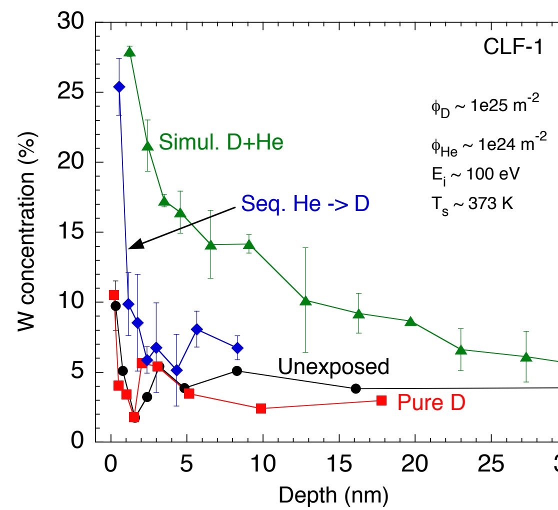
3. The Cr concentration for seq. He -> D case is lower than that for pure D case.
   → Reduction of D retention

Note that the Cr concentration for simul. D+He case is comparable to that for pure D case.

- ➢ Reduction of D retention caused by He bubbles







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- Fluence dependence of D retention caused by pure D plasma exposure
  - Counter-intuitive fluence dependence
- He effect on D retention
  - Reduction of D retention
- Analysis of TDS spectra
  - Low and high temperature D desorption components
- He retention
  - Weak material dependence
- Microstructures in the near-surface region
  - Formation of high-density He bubble layer
- **Surface morphology and composition**
  - Formation of cones and W surface enrichment

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Larger structures (up to ~200 nm) exist on the surface of all the cases, in addition to smaller cones on CLF-1-3B.

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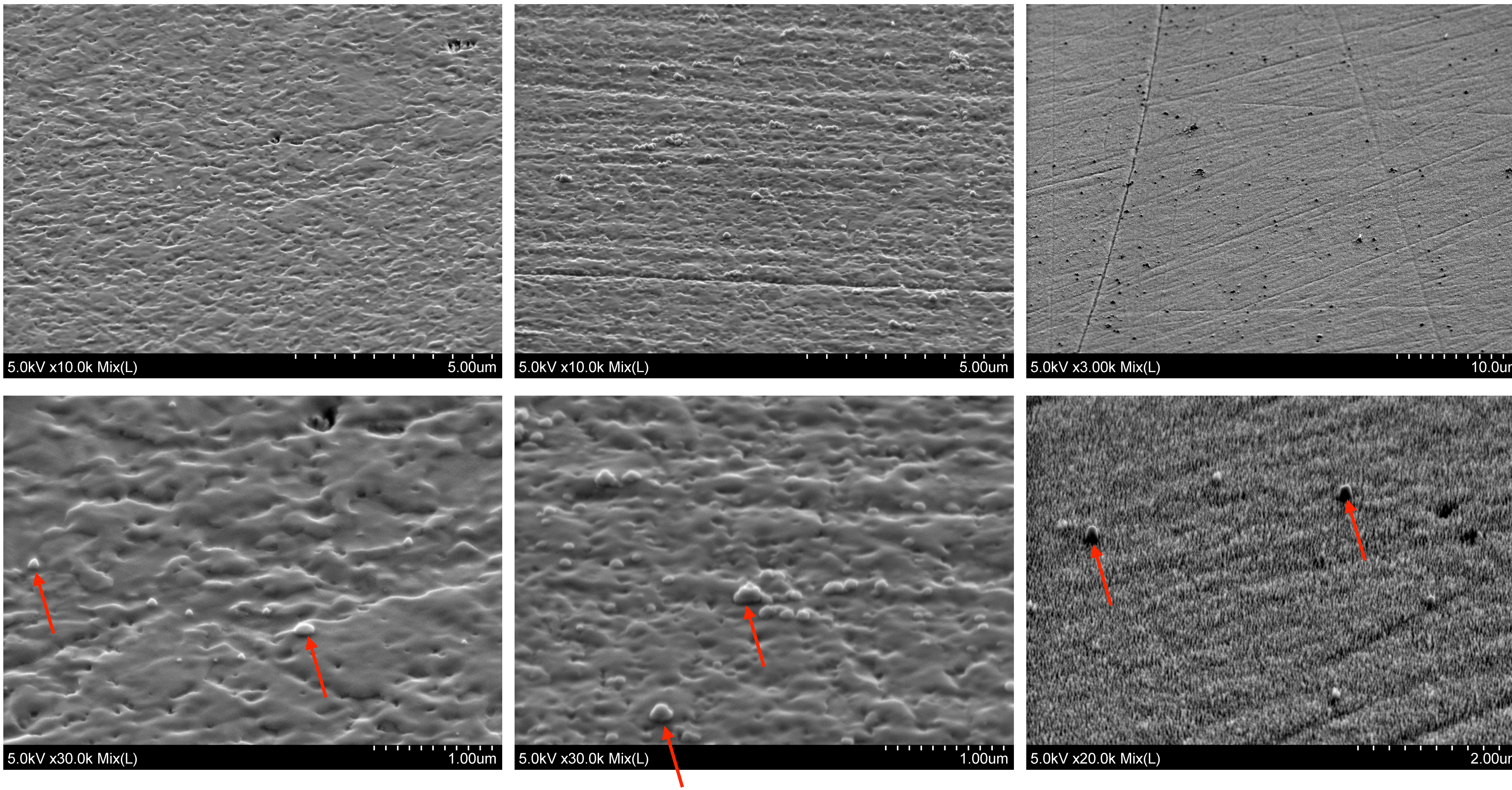
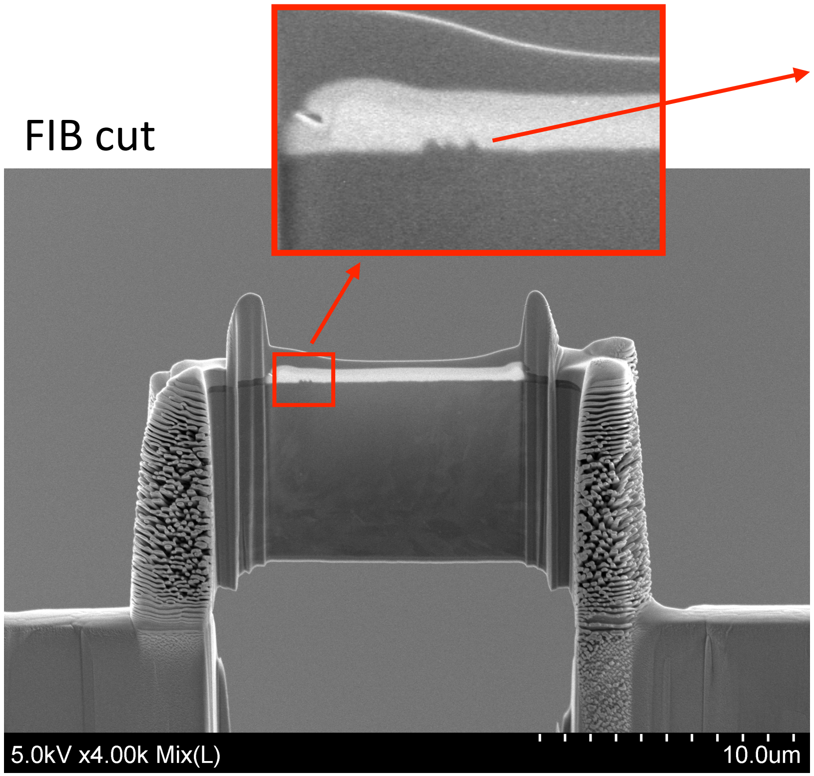
CLF-1-1B: Pure D, 373 K | CLF-1-2B: Seq. He->D, 373 K | CLF-1-3B: Simul. D+He, 373 K

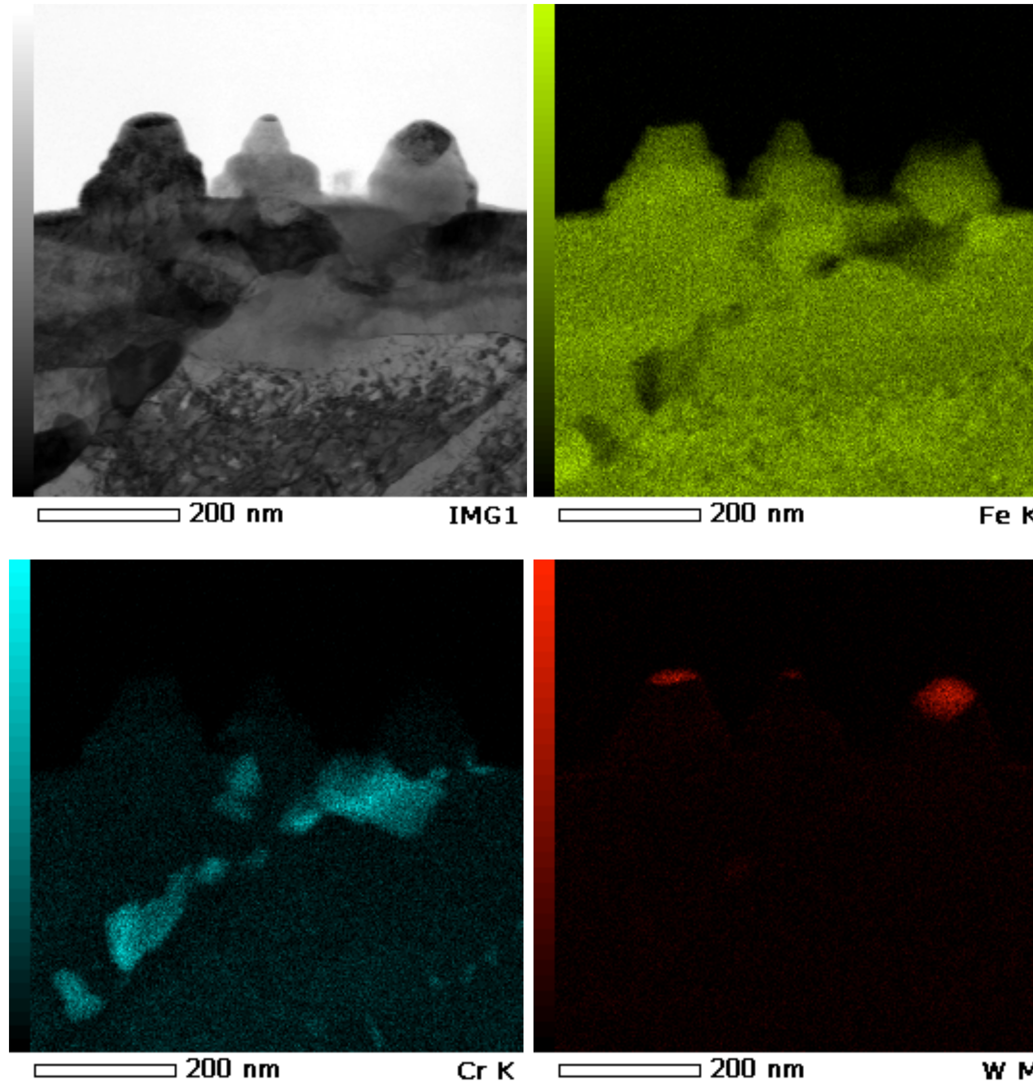


TEM/EDX observations reveal that they are larger cone structures with W on the tip.

CLF-1-2B: Seq. He->D, 373 K

FIB cut

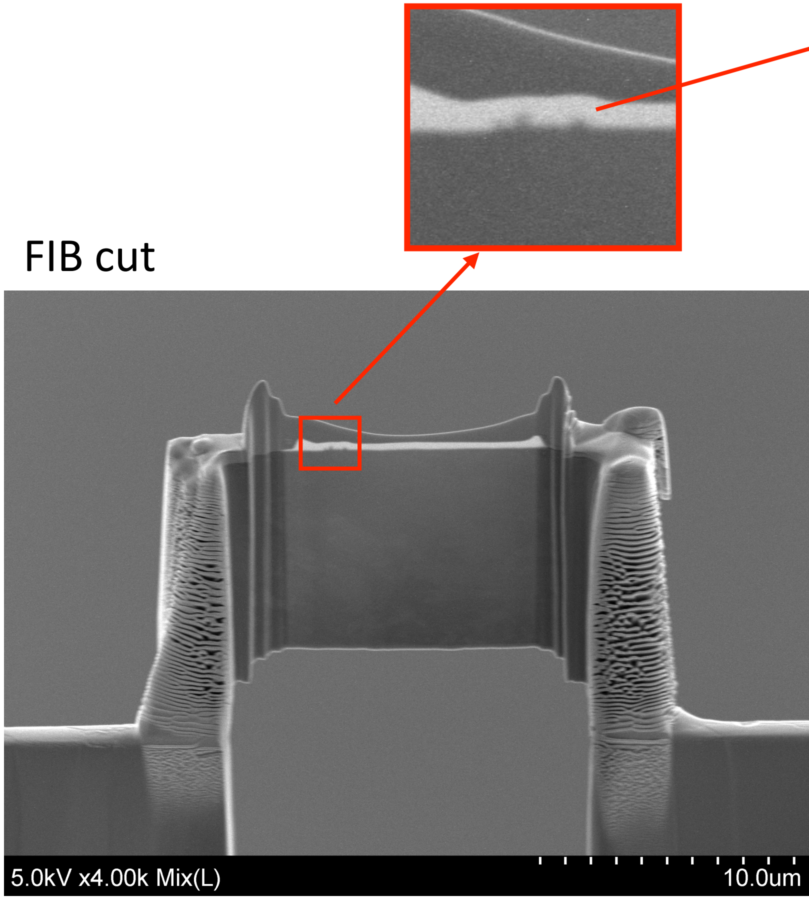


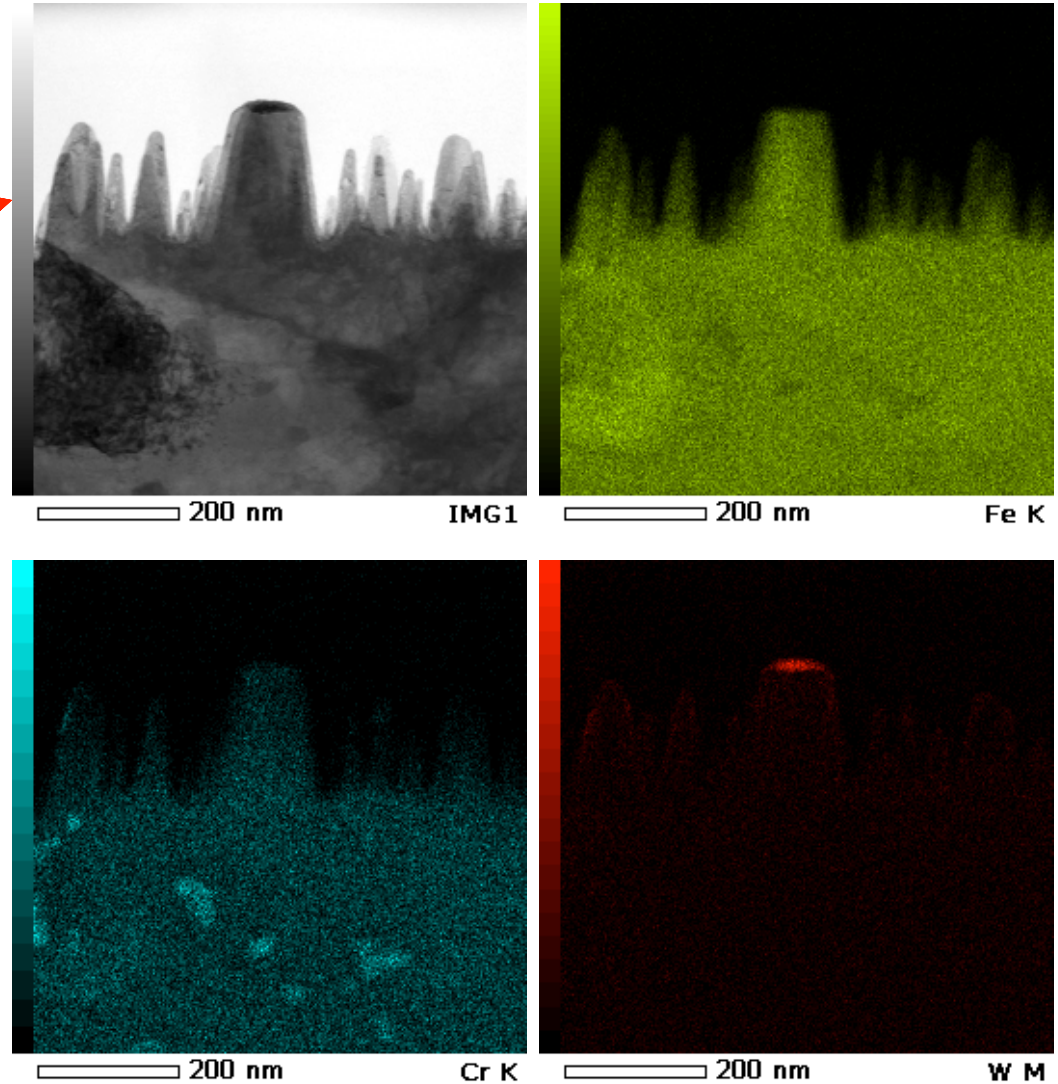


TEM/EDX observations reveal that they are larger cone structures with W on the tip.

CLF-1-3B: Simul. D+He, 373 K

FIB cut





Both small & large cones exist, depending maybe on the size & amount of W clusters. Small cones are dominant.

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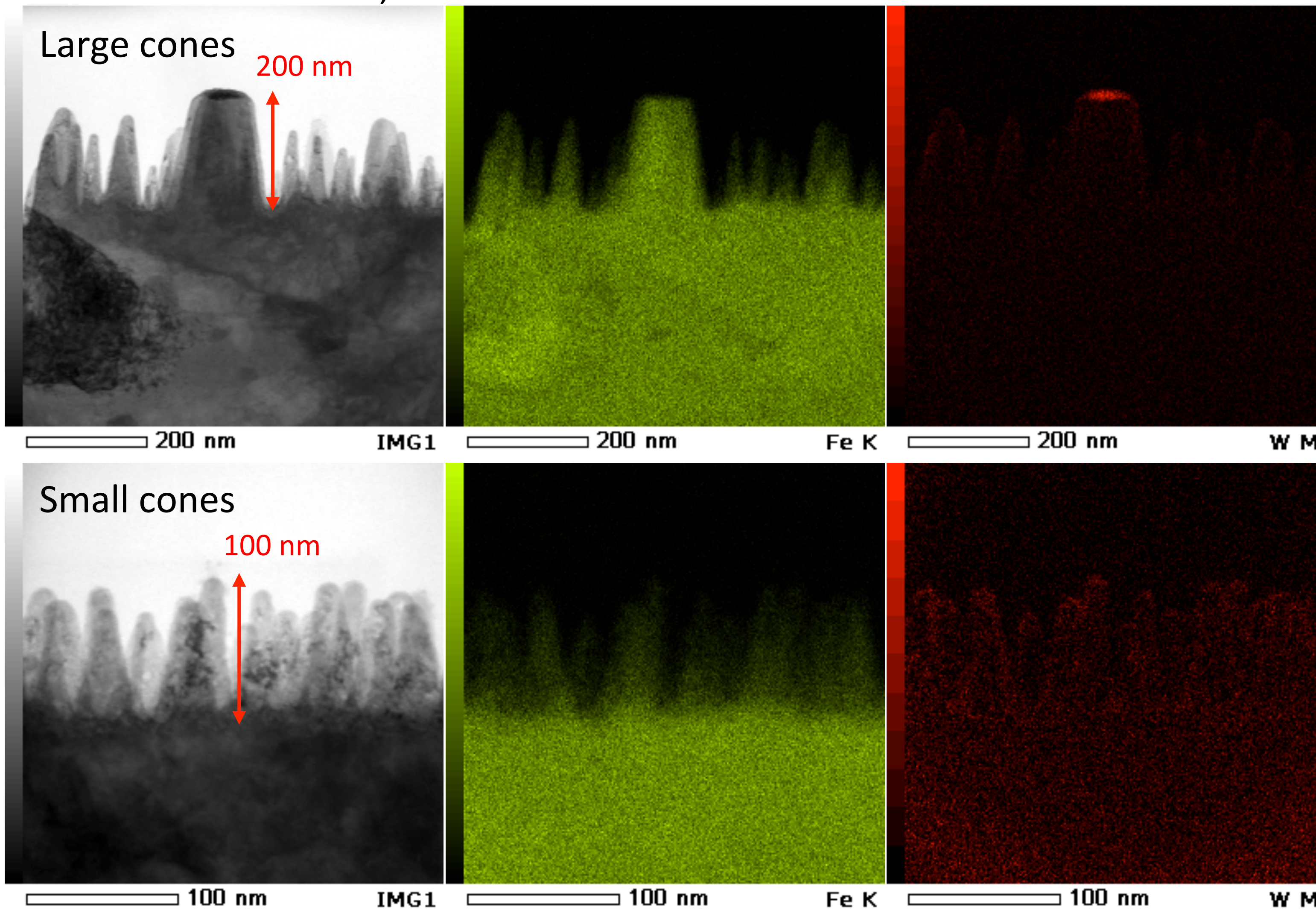
CLF-1-3B: Simul. D+He, 373 K

The cone height is roughly consistent with the erosion layer thickness of ~160 nm, estimated from mass loss (assuming a flat surface)



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Sputter-AES analysis shows the W surface enrichment, particularly, after He-containing plasma exposure.

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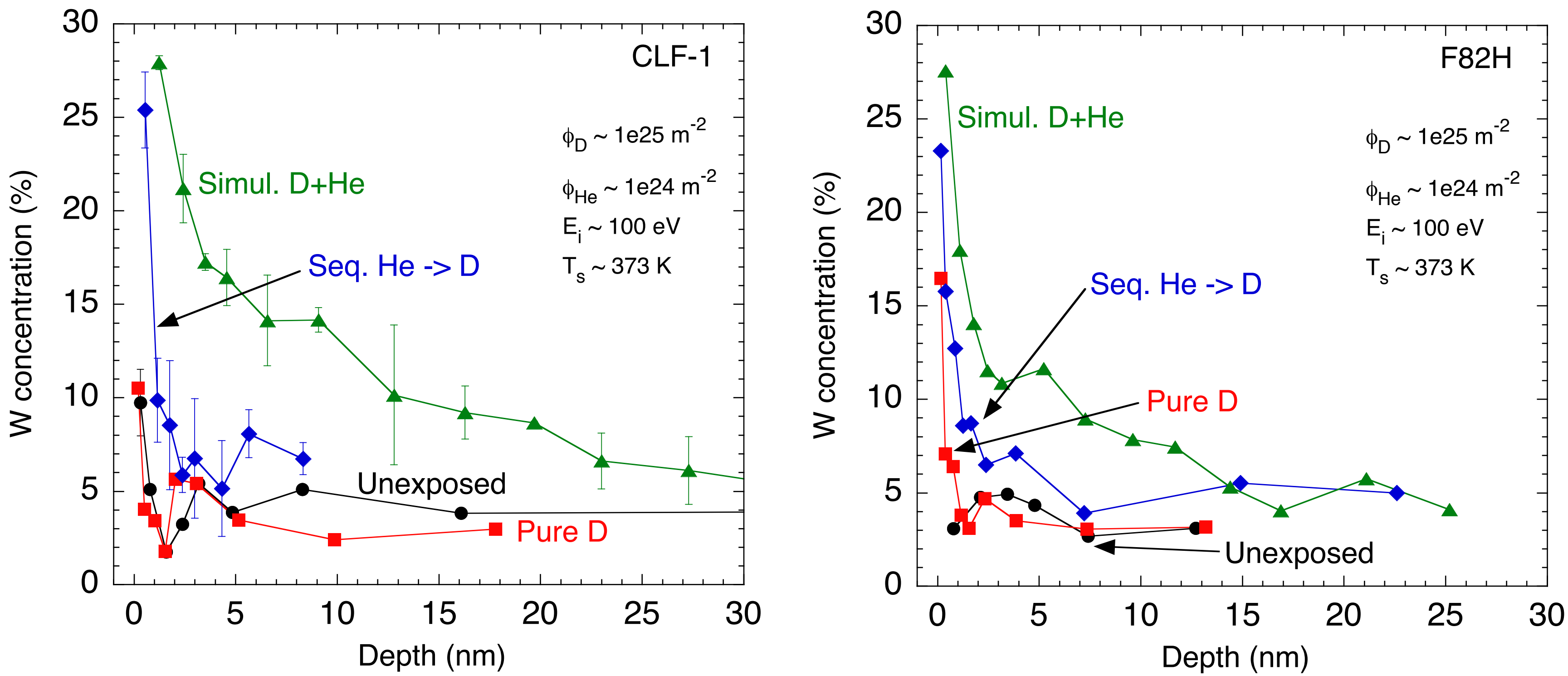
- W surface enrichment after pure D plasma exposure is less than after He-containing plasma exposure, which is consistent with less sputtering for pure D.
- The depth profile of the W concentration clearly extends to a deeper region for the simultaneous D+He mixed plasma exposure case.



*The escape depth of electrons (depth resolution of AES) is < 5nm.

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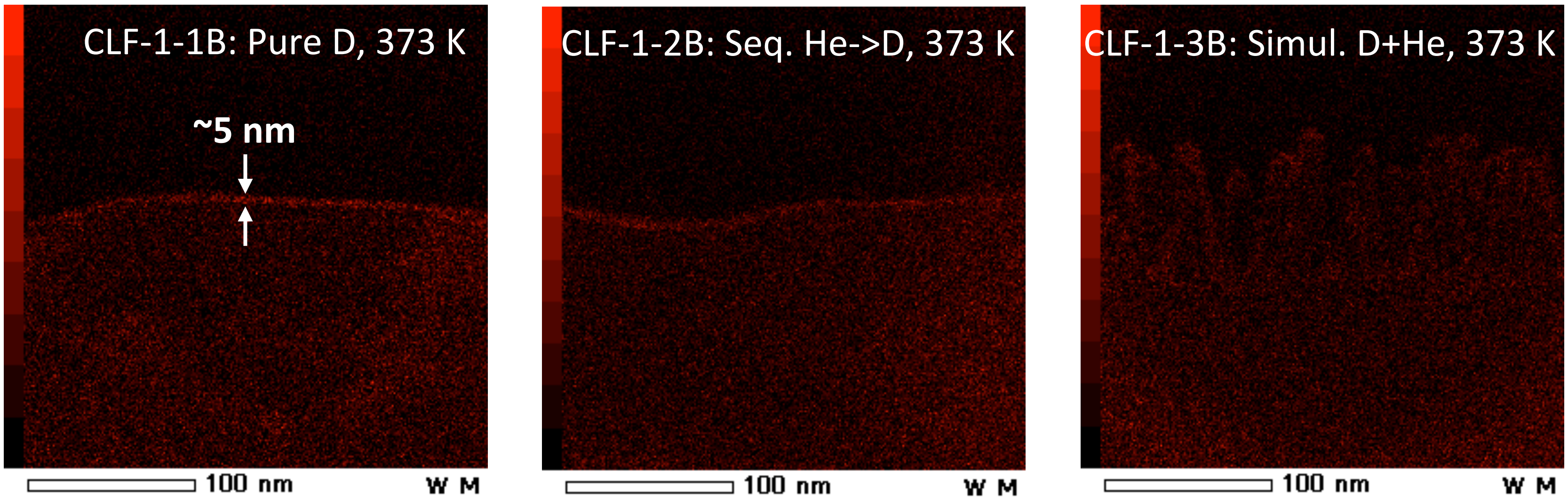
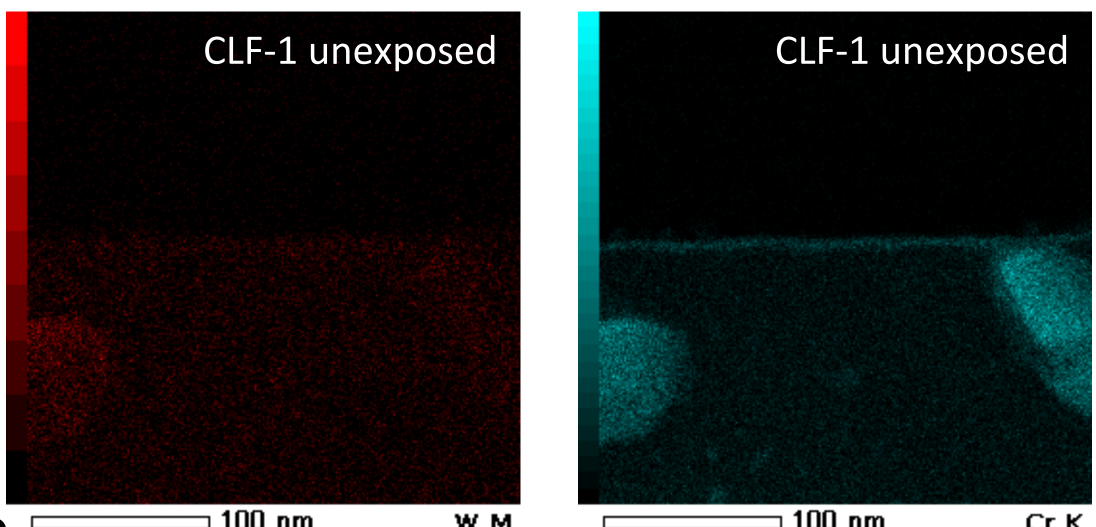
TEM/EDX analysis also observes a W-rich surface layer after plasma exposure.

- EDX 2D mapping of W in the near-surface region





- The higher W concentration in a deeper region measured with sputter-AES is caused by the cones.
- A Cr-rich surface layer is clearly observed for an unexposed surface.

Conclusion: Cr content and He bubbles in the near-surface significantly influence D retention in the RAFM steels.

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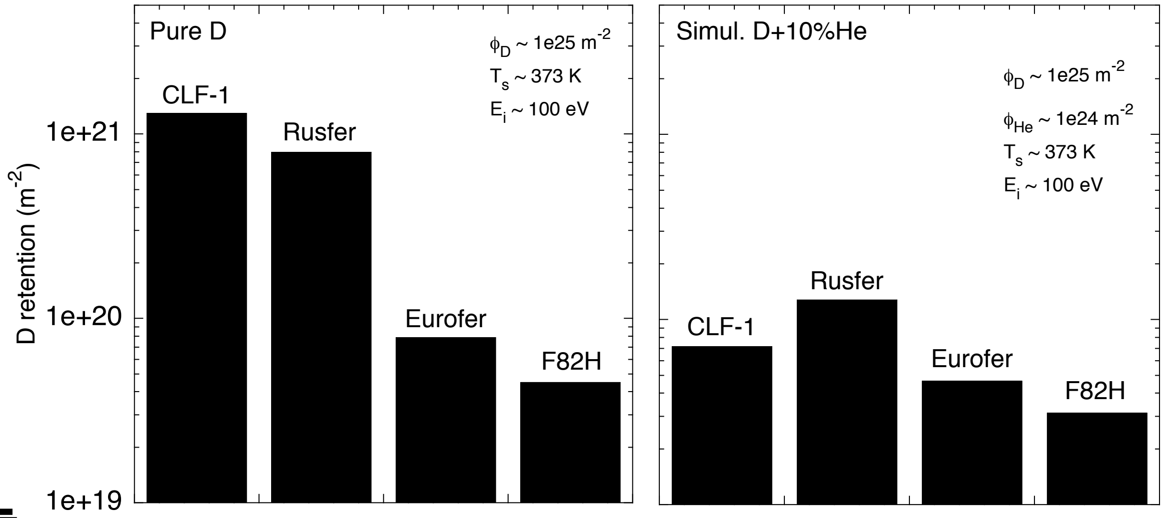
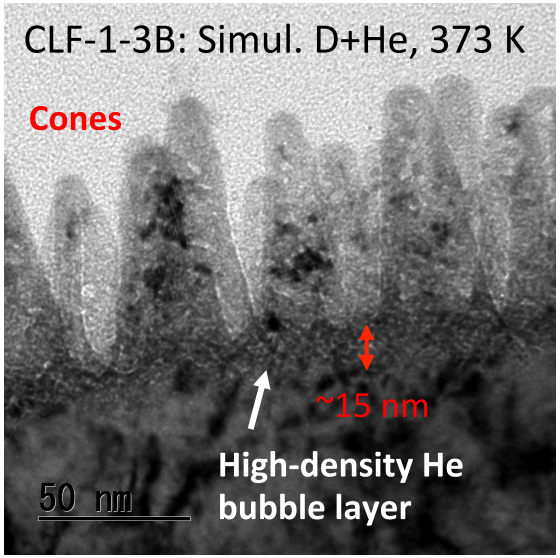
- **A higher Cr concentration in the near-surface region leads to a higher D retention.**
- **The D retention is reduced with a high-density He bubble layer formed in the near-surface region.**
- Future work:
  - Impurity (Ne, Ar, and N_2) effect on D retention
  - In-situ LIBS analysis of surface composition evolution “during” plasma exposure





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Additional slides

Low temperature desorption is dominated by D_2, while high temperature one is dominated by HD.

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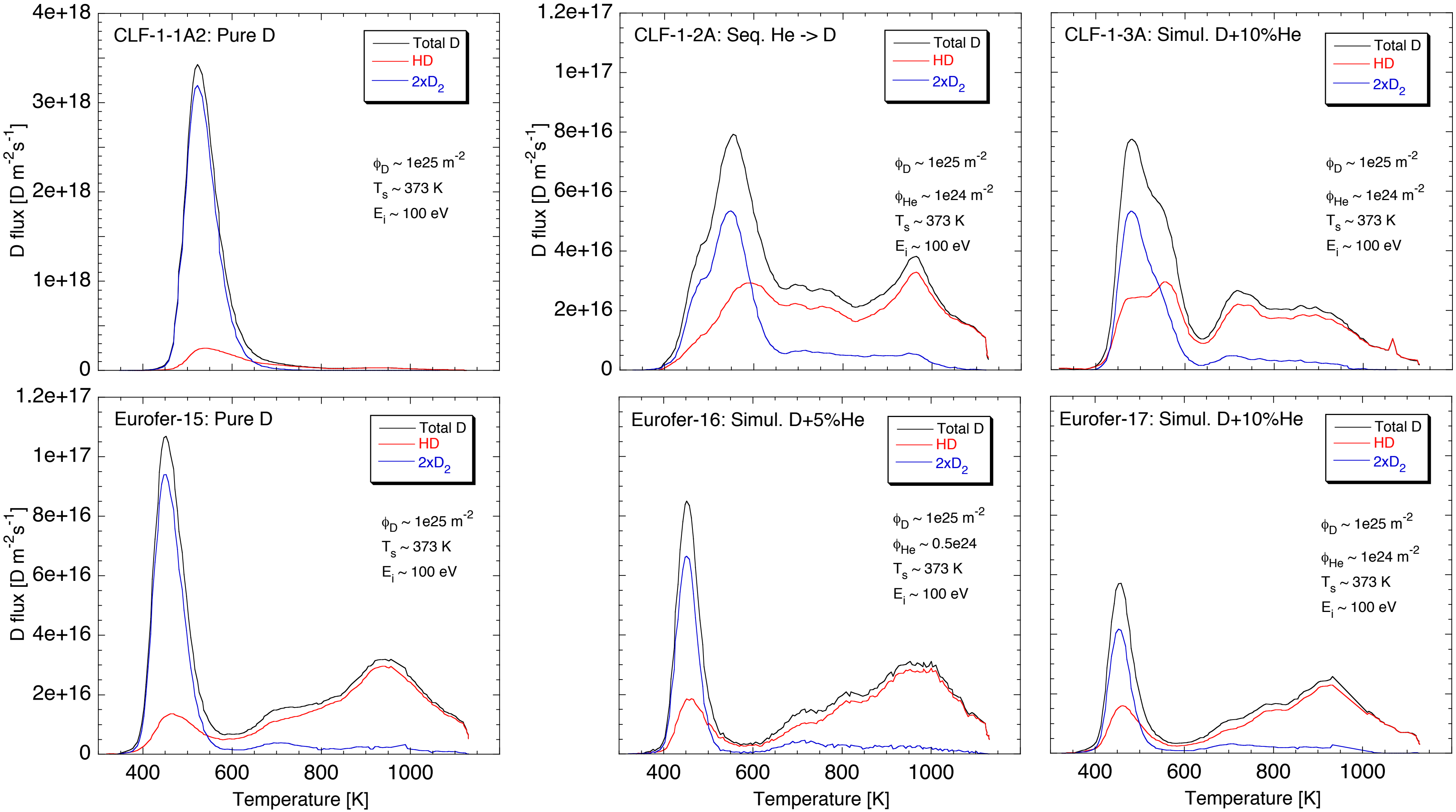

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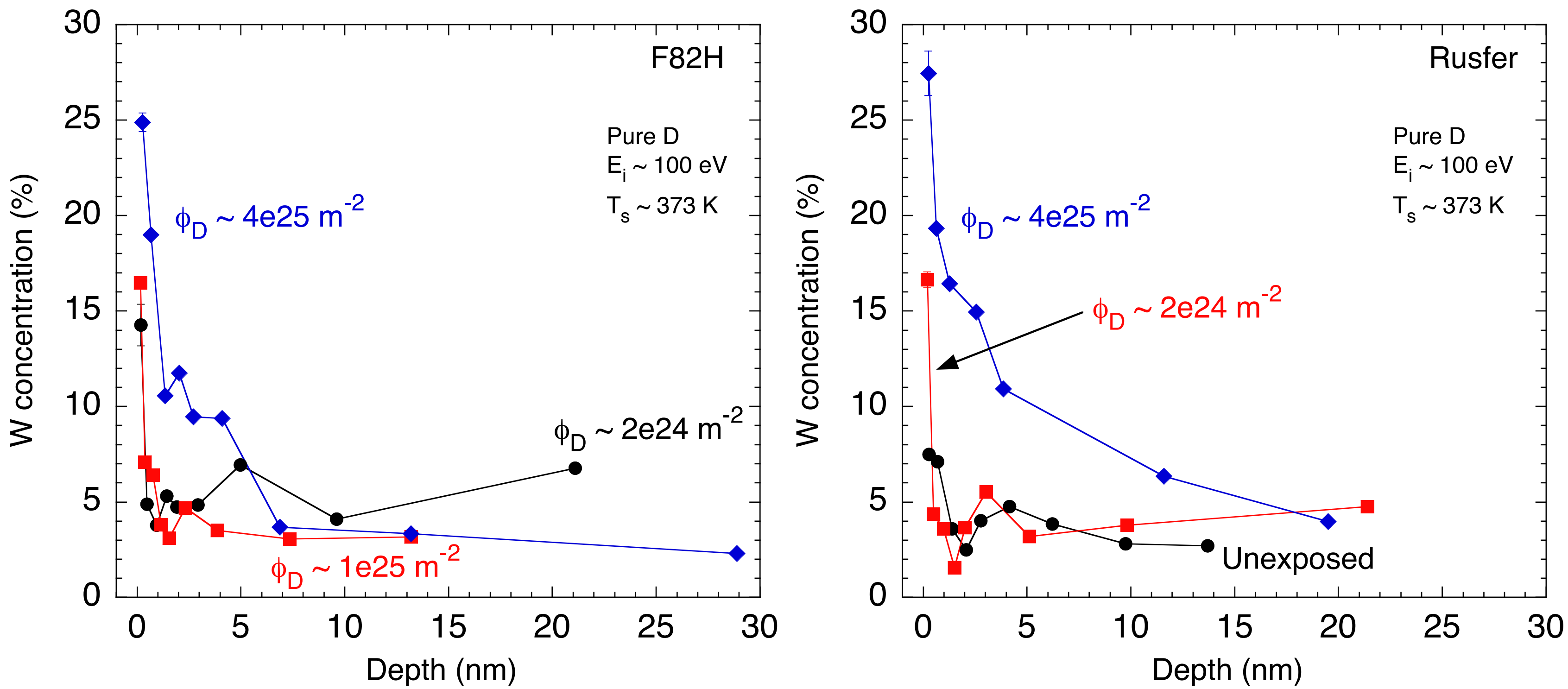
The W surface enrichment is enhanced with increasing the fluence due to more preferential sputtering.

PISCES



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After simul. D+He mixed plasma exposure, CLF-1 shows higher W and lower Fe concentrations than F82H.

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- Seq. He → D plasma exposure led to the similar composition between CLF-1 and F82H.