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# Sputtering and D retention properties of RAFM steels under high-flux plasma exposure

D. Nishijima, R.P. Doerner, M.J. Baldwin

*Center for Energy Research, University of California - San Diego, La Jolla, CA, USA*

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2. **D retention properties of RAFM steels (preliminary)**
   - Comparison between CLF-1, Eurofer, and F82H

3. **LIBS thickness measurement of (Fe, Cr, Ni) mixed-material layers**
   - New technique developed for layers thinner than ablation rate
   - Simultaneous composition analysis

# Optical emission spectroscopy can provide element-resolved sputtered flux and sputtering yield.



- Line-of-sight integrated emission intensity of sputtered atoms, $I_a$, can be converted into sputtered atomic flux, $\Gamma_{spt}$, with S/XB defined by,

$$G_{spt} = 4p\frac{S}{XB}I_a$$

S: Ionization rate coefficient
X: Excitation rate coefficient
B: Branching ratio

(Note that *this relation is valid only when the geometrical loss flux from the observation volume is negligible.*)

Availability of S/XB for main elements in RAFM steel

| | **Fe I** | **Cr I** | **W I** |
|---|---|---|---|
| Experimental S/XB | N/A | N/A | Available |
| Theoretical S/XB | N/A | Available | Available |

# S/XB values of Fe I and Cr I lines were experimentally determined for the first time.



$$G_{spt} = 4p\frac{S}{XB}I_a \Rightarrow \frac{S}{XB} = \frac{G_{spt}}{4pI_a} = \frac{YG_i}{4pI_a}$$

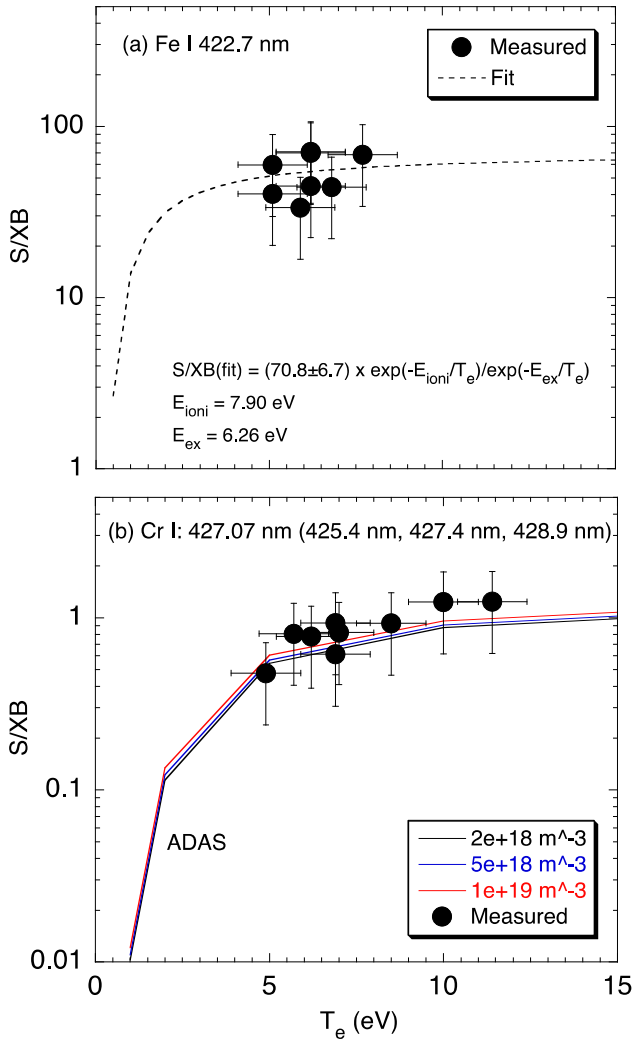
Y: Sputtering yield from mass loss measurement

$\Gamma_i$: Ion flux from single probe measurement

- Fe I 422.7 nm
  - -- No theoretical data is available
  - -- Fit to a simple function:

$$\frac{S}{XB} = b \times \exp\left(-\frac{E_{ioni}}{T_e}\right) / \exp\left(-\frac{E_{ex}}{T_e}\right)$$

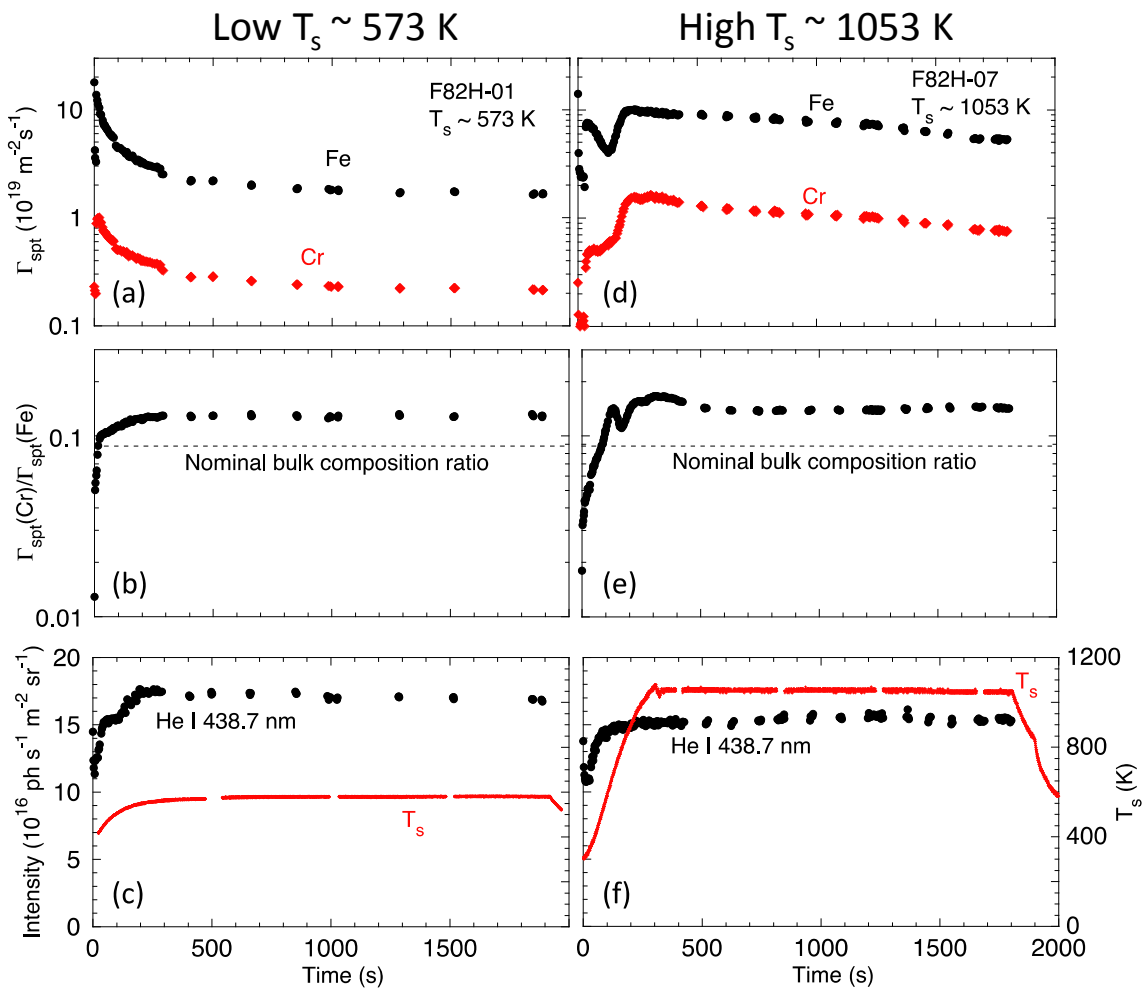
- Cr I 427.07 nm (425.4, 427.4, 428.9 nm)
  - -- A multiplet-averaged line
  - -- Good agreement with ADAS



# Reduction of sputtering yield of Fe and Cr due to He plasma exposure is clearly observed.



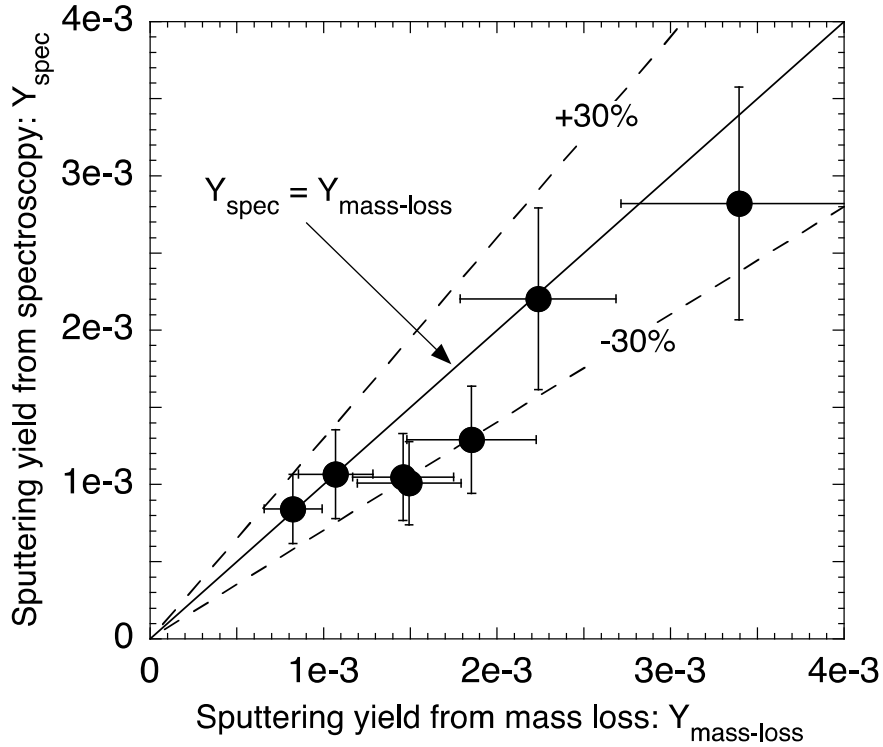
- He plasma exposure
  - $\Gamma_i$ ~ (2-4)x$10^{22}$ $m^{-2}s^{-1}$
  - $E_i$ ~ 80 eV
- No W I lines were observed.
  - W was not sputtered.
  - Preferential sputtering of Fe and Cr.
- Decay of $\Gamma_{spt}$(Fe) and $\Gamma_{spt}$(Cr) is faster at $T_s$ ~ 573 K than at $T_s$ ~ 1053 K.
- $\Gamma_{spt}$(Cr)/$\Gamma_{spt}$(Fe) is higher than the nominal bulk composition ratio (~0.088).
  - Diffusion of Cr is faster than that of Fe.



# Good agreement of sputtering yield between spectroscopy & mass loss verifies that our measured S/XB values are valid.



Comparison of the time- (fluence-) integrated F82H sputtering yields between the spectroscopic ($Y_{spec}$) and mass loss ($Y_{mass\text{-}loss}$) techniques.

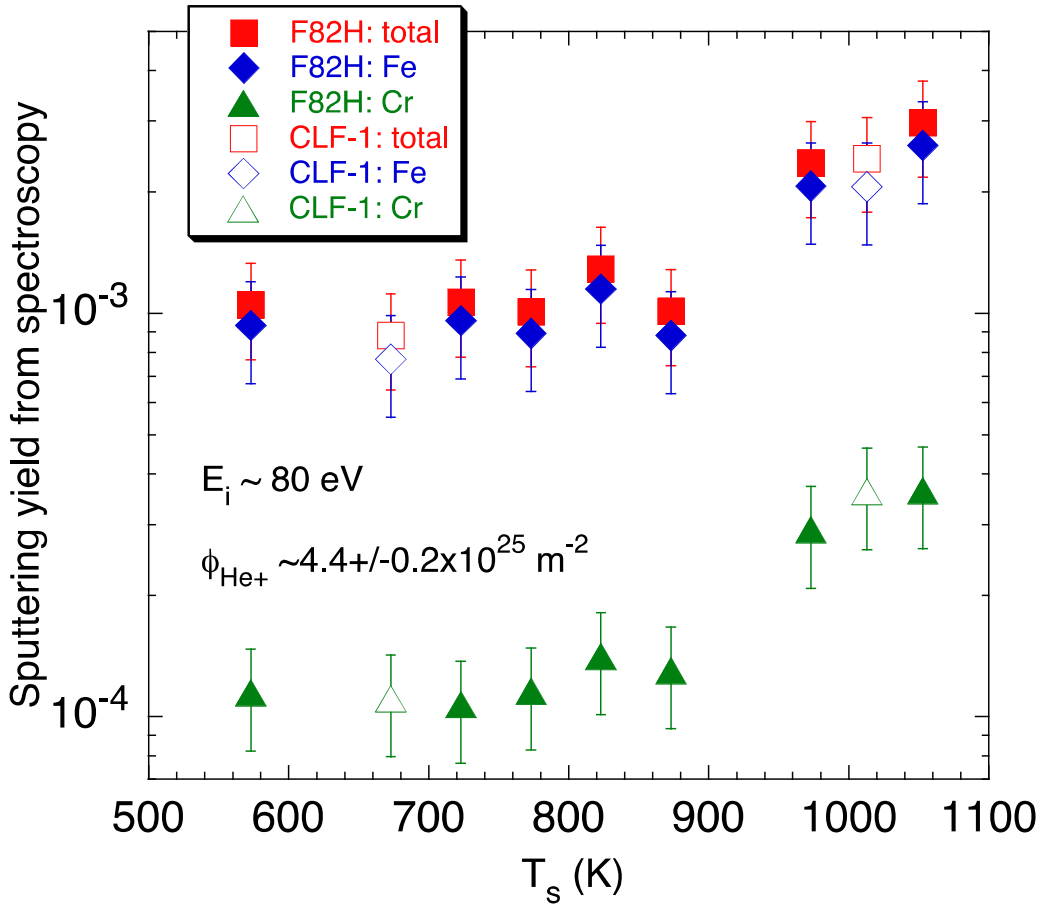



The number of sputtered atoms from mass loss is calculated with the effective atomic mass (~55.5 amu) evaluated from spectroscopy.

# Sputtering yield is found to be constant up to $T_s$ ~ 900 K, and to be enhanced at $T_s$ > 900 K.



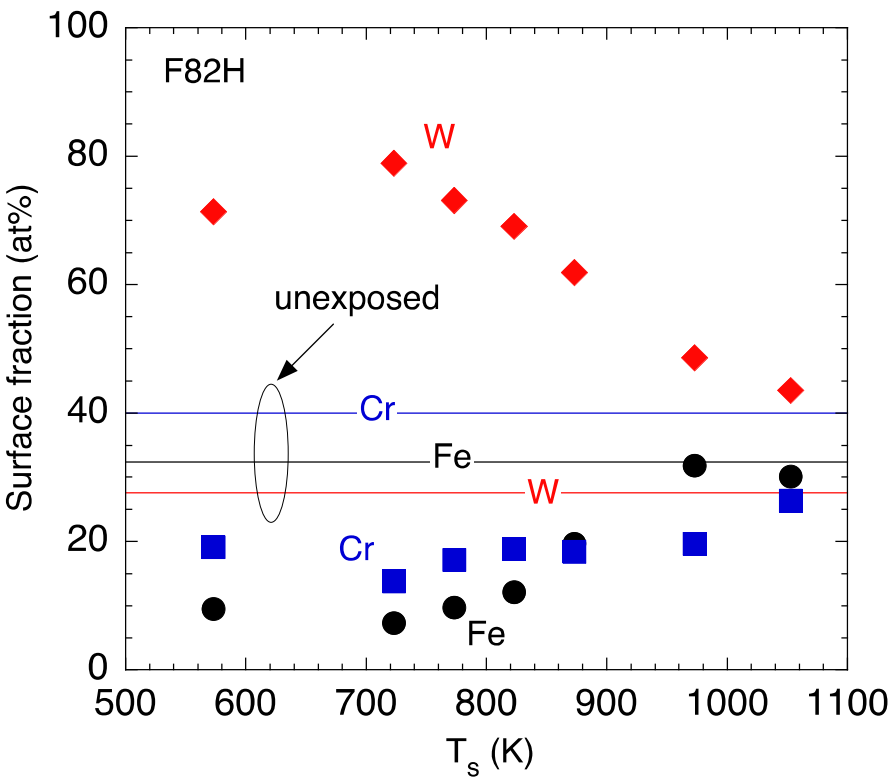
- He plasma exposure
  - $E_i$ ~ 80 eV
  - $\phi_{He+}$ ~ 4.4+/-0.2x$10^{25}$ $m^{-2}$
- Y(F82H) ≈ Y(CLF-1)
  - While the surface preparation is different between F82H (sanded) and CLF-1 (mirror-polished).



# Surface enrichment of W due to preferential sputtering of Fe and Cr is confirmed with AES analysis.

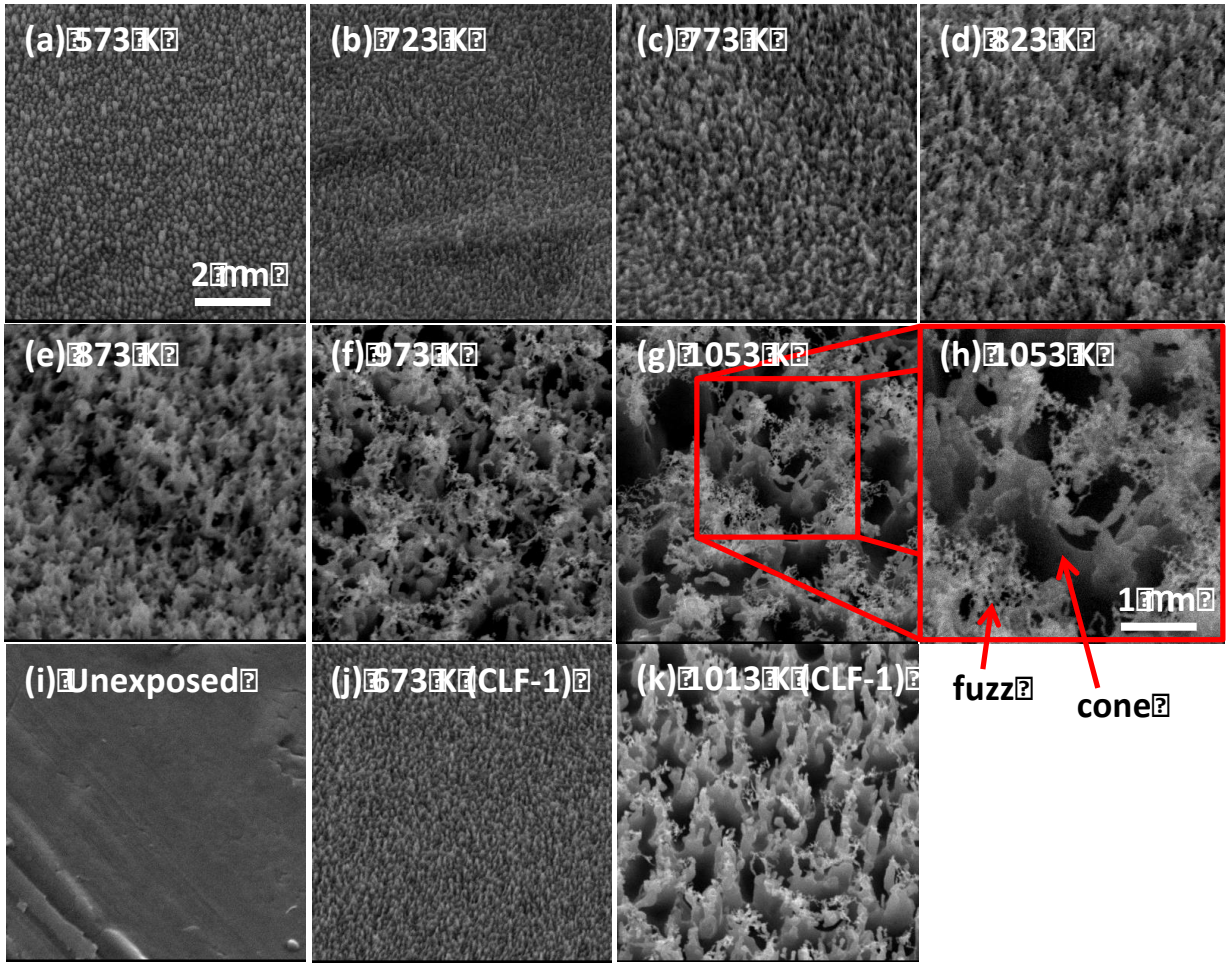


- Fe may be preferentially removed by sanding, thus the surface composition of the unexposed surface strongly deviates from the bulk composition.
- The enrichment of W as well as the depletion of Fe and Cr are clearly seen, especially, at $T_s$ < 900 K.
- The W enrichment is reduced at $T_s$ > 900 K due presumably to the enhanced diffusion of Fe and Cr.



# Cone-like structures become larger with increasing $T_s$, and W fuzz is formed on top of cones at $T_s \geq 973$ K.





# In future fusion devices with RAFM steel first walls, $T_s$ will need to be kept below 900 K from the sputtering point of view.



- S/XB values of Fe I 422.7 nm and Cr I 427.07 nm (425.4, 427.4, 428.9 nm) lines were experimentally determined for the first time.
  - Good agreement between $Y_{spec}$ and $Y_{mass\text{-}loss}$ confirms that the measured S/XB values are valid.
  - This enables us to study sputtering properties of RAFM steels in more detail with spectroscopy.

- High-flux He plasma exposure to RAFM steels at low $E_i$ ~ 80 eV leads to the reduction of the sputtering yield.
  - Preferential sputtering of Fe and Cr
    - Surface enrichment of W
  - Formation of sputter cone structures
    - W fuzz formation at $T_s \geq 973$ K on top

# 2. D retention properties of RAFM steels



Chemical composition (wt%) of 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 |

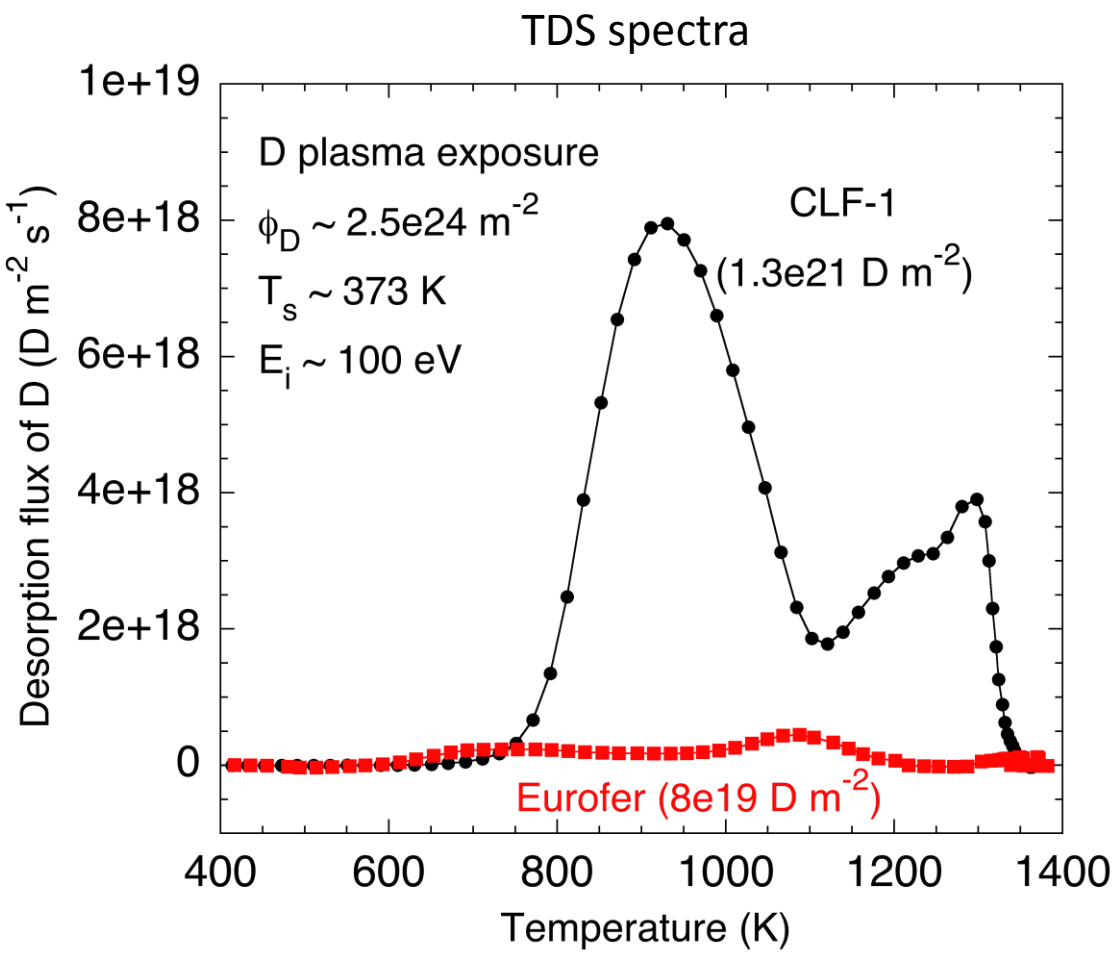
- Only elements with ≥ 0.1 wt%.
- P92 additionally contains Mo at 0.3-0.6 wt%.

- CLF-1, Eurofer, and F82H have been used in our experiments so far.
- But the number of these samples is very limited (no CLF-1 samples left).
- P92 may be used in future, since the composition of P92 is very similar to that of the RAFM steels.

# D retention in CLF-1, Eurofer, and F82H RAFM steels were investigated under D plasma exposure in PISCES-A.



- D plasma exposure
  - $\phi_D$ ~ 2.5e24 $m^{-2}$
  - $\Gamma_i$ ~ (2.5-3.5)e21 $m^{-2}s^{-1}$
  - $T_s$ ~ 373 K
  - $E_i$ ~ 100 eV
- Desorption flux of D is evaluated from both HD and $D_2$ signals.
- D retention in CLF-1 is much higher than that in Eurofer.
- D retention in F82H is much lower than CLF-1 and Eurofer, below or around the detection limit (~1e17 D $m^{-2}$).

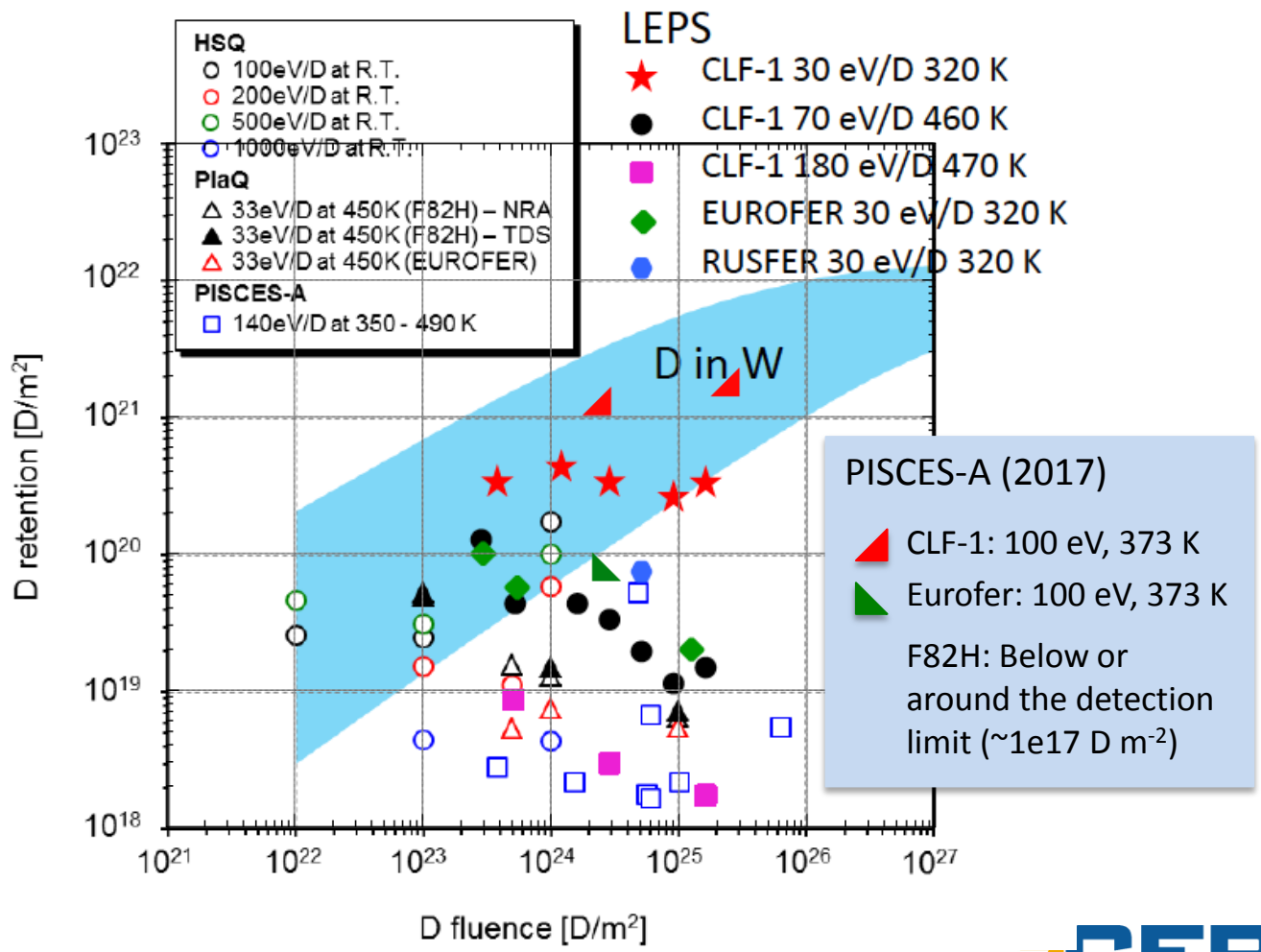


# PISCES-A (2017) data shows, Eurofer is consistent with others, while CLF-1 is higher than others.



- PISCES-A (2017) data does not decrease with increasing the fluence. (Need more data points, though.)
- Why does D retention decrease with increasing the fluence???
- Why is D retention in F82H very low in PISCES-A?

*Comparison of fluence dependence of D retention between W and RAFM steels (CLF-1, EUROFER and F82H)*



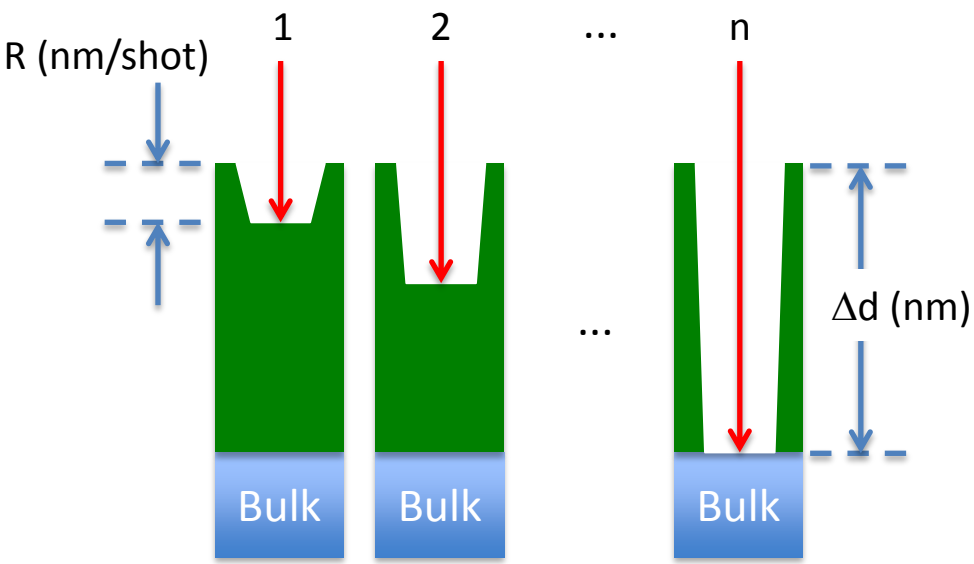

*Original graph from W. meeting & P. Wang, 16th PFMC (2017)*

# 3. LIBS thickness measurement of layers thinner than ablation rate



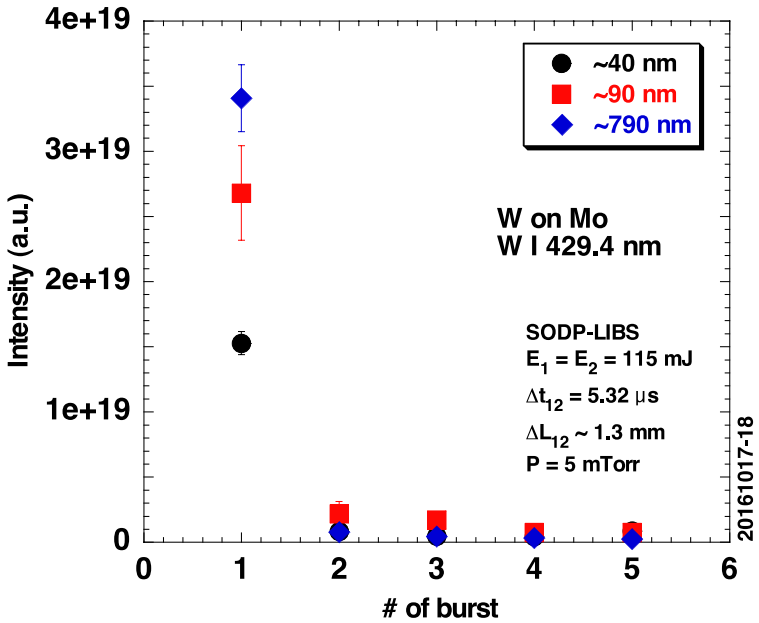
- For a layer much thicker than the ablation rate

$$\Delta d\ (nm) = R\ (nm/shot) \times n$$




- This standard method **cannot be applied for a layer thinner than the ablation rate**.

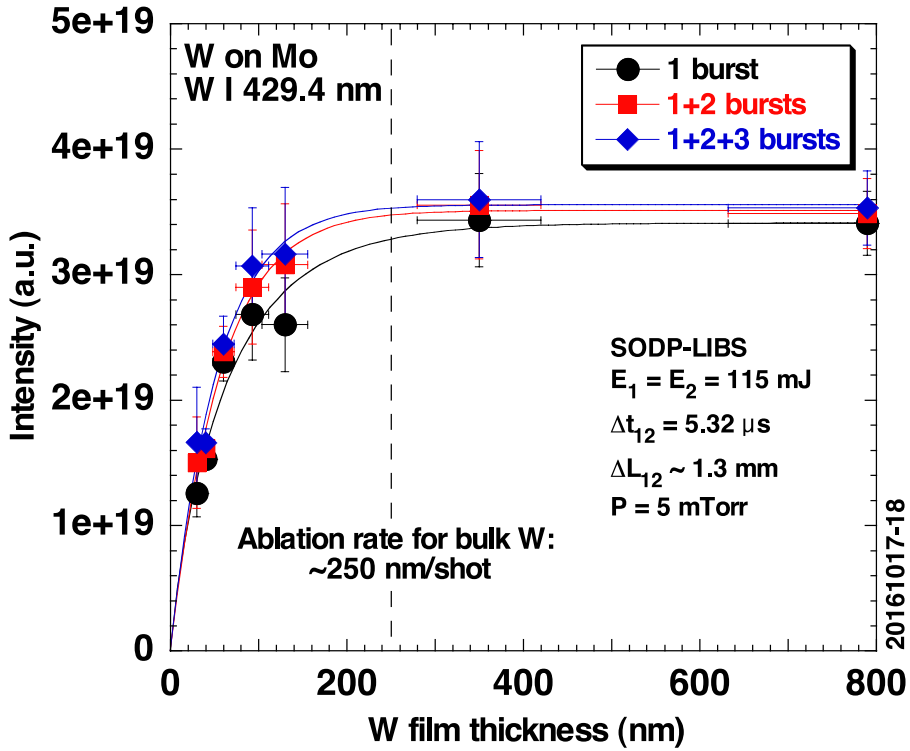
- Develop a new method to measure the thickness of a layer thinner than the ablation rate (less than a few hundred nm).
  - The signal intensity increases with an increase in the layer thickness.



# The new method utilizes the layer thickness dependence of the signal intensity as a calibration curve.



- The W I intensity depends strongly on the layer thickness up to around the ablation rate of bulk W.
- The film thickness can be obtained from the intensity below the ablation rate.
- Why does the intensity ($I_{12}$ and $I_{123}$) saturate above the ablation rate?




- Fit function and coefficients

$$y = a(1 - \exp(-bx^c)).$$

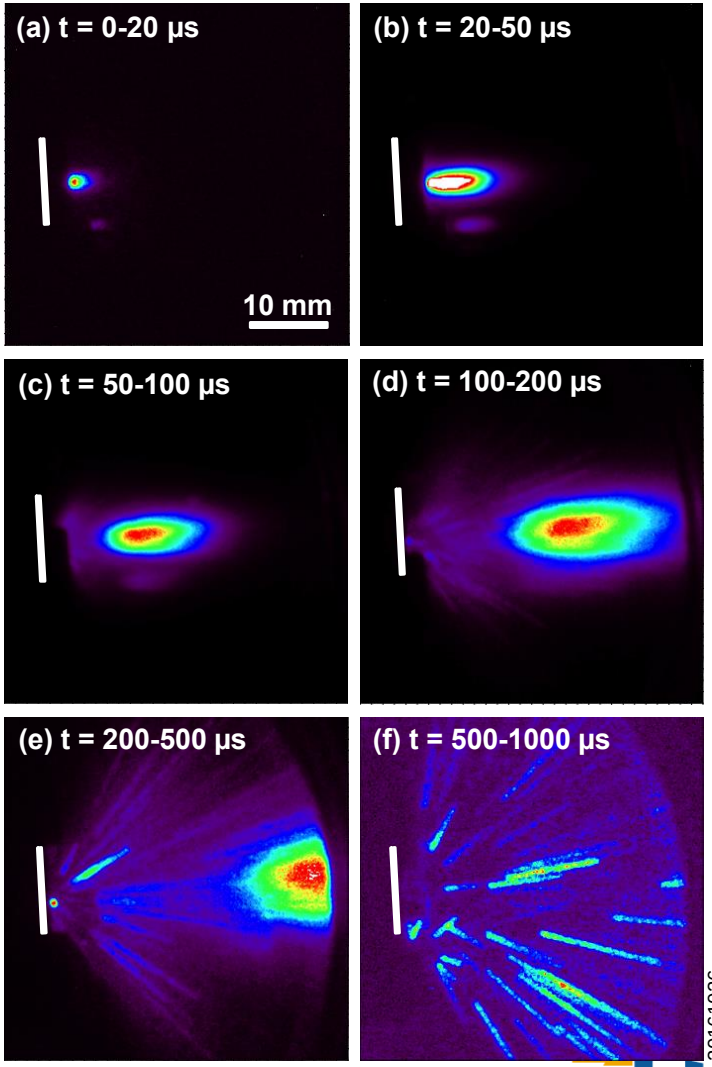
| | $a$ | $b$ | $c$ | $R$ |
|---|---|---|---|---|
| 1 burst | 3.4e19 ± 1.6e18 | 2.5e−2 ± 1.7e−2 | 0.88 ± 0.18 | 0.980 |
| 1 + 2 bursts | 3.5e19 ± 8.2e17 | 1.6e−2 ± 6.7e−3 | 1.02 ± 0.11 | 0.994 |
| 1 + 2 + 3 bursts | 3.6e19 ± 1.1e18 | 1.7e−2 ± 9.6e−3 | 1.02 ± 0.14 | 0.988 |

# The underlying W layer below the ablated layer is significantly ejected as dust.



- The temporal evolution of ablated W plasma at the 1st burst was observed with an ICCD camera.
- Adhesion of the W layer to the mirror-polished Mo substrate may be weak.
- This can lead to the significant dust ejection.
- There was no W layer left after the 1st burst, and thus the W I intensity became very weak from the 2nd burst.

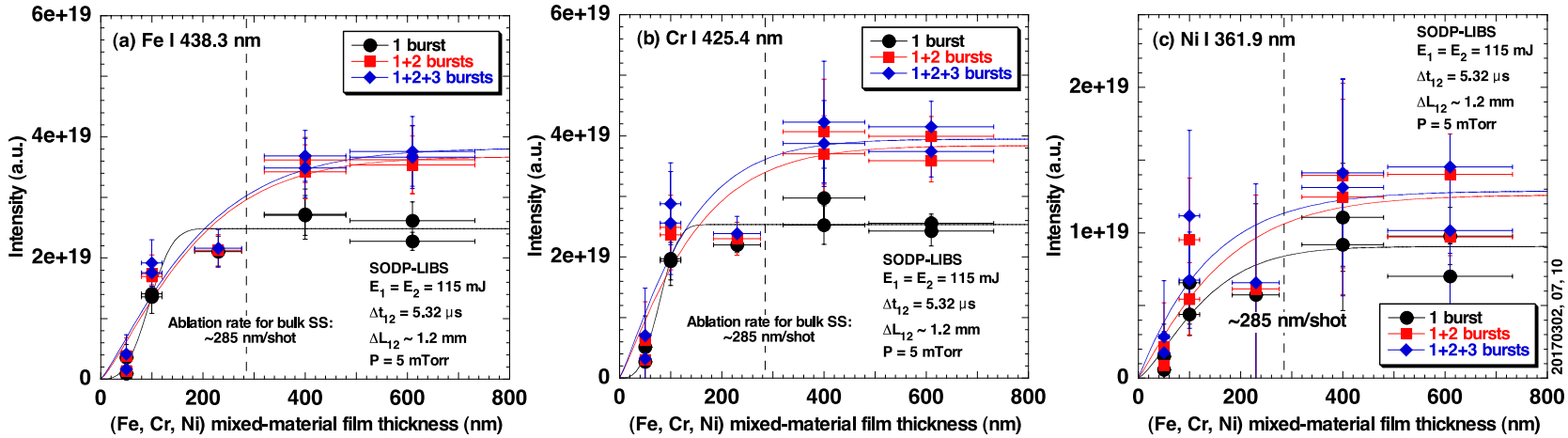
W layer ($\Delta$d ~ 790 nm) on Mo



# This new method also works with mixed-material layers.

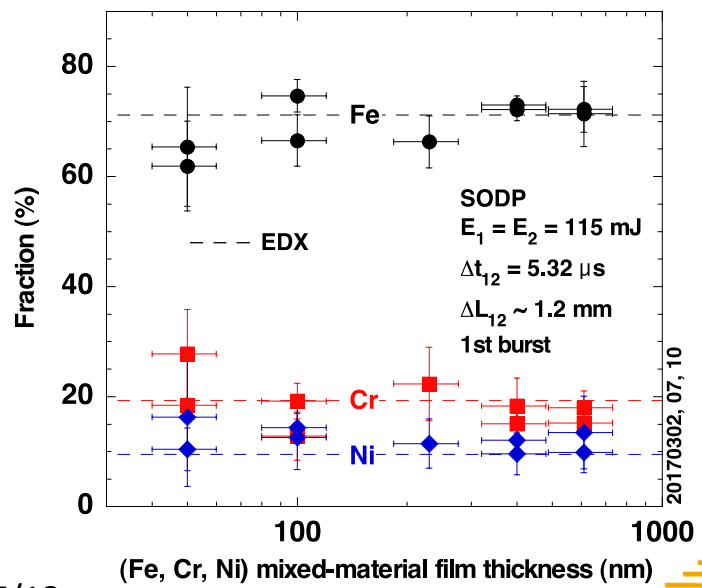


- (Fe, Cr, Ni) mixed-material layers




- The composition of the multi-element layers is simultaneously derived with the calibration-free (CF) LIBS technique.

- The composition from LIBS agrees with that from EDX analysis.



# Future work



- **D retention properties of RAFM steels**
  - Investigate the fluence and temperature dependence
  - Investigate effect of He mixture
  - Use commercially available P92 FM steel as a surrogate?

- **In-situ real-time LIBS surface analysis during plasma exposure**
  - Detect surface composition change due to preferential sputtering