Anna V. Golubeva

NRC 'Kurchatov Institute', Moscow, Russia

Progress in the study of deuterium interaction with low-activated ferrite-martensitic steels

Persons involved:

A.V. Golubeva, B.I. Khripunov, A.S. Anikin, A.A. Mednikov, D. Kozlov, N.P. Bobyr, N.N. Kolobylina – *NRC “Kurchatov institute” Moscow, Russia*

D. Kovalenko, N. Klimov – *TRINITI, Troitsk, Russia*

V. Efimov, Yu.M. Gasparyan,– *MEPhI, Moscow, Russia*

V.M. Chernov – A.A. Bochvar’s institue, *Moscow, Russia*

D. Terentiev - *SCK•CEN, Belgium*

N. Ashikawa - *The Graduate University for Advanced Studies & National Institute for Fusion Science, Gifu, Japan*

Q. Huang - *Institute of Nuclear Energy Safety Technology, Hefei, China*

Materials:

1. Rusfer (Prof. V.M. Chernov)
2. Eurofer (Dr. Dmitry Terentiev)
3. CLAM (Prof Qunying HUANG, Hefei)
4. F82H (Prof. Naoko Ashikawa)

Topics:

1. ELMs-like D plasma irradiation
2. Selective sputtering study

Composition of RAFMS, wt. %

γ-phase stab., Impr. Long-term creep

Strong carbide former, HT strength

γ-phase stab., Ni repl., ↓ crit. cooling rate for martecite formation

Strong carbide former, stab. γ-phase, Mo replacement

Martencite formation

Corrosion resistance

Grain refinement, red. ↓-phase

Steel	Smelting Nr	Ni	C	N	Si	Ti	V	Cr	Mn	Fe	Ta	W
Rusfer	ДШ41056	0.03	0.16	0.07	0.4	0.05	0.4	**12**	0.6	BASIS	0.15	1.09
Eurofer	83699	0.007	0.15	0.017			0.19	**8.99**	0.44		0.014	1.1
CLAM	1506		0.12	0.0026	0.025	0.21	0.19	**9.06**	0.42			1.52
F82H	?		0.1				0.2	**8**	0.45		0.06	2.0

Samples preparation

Samples 10x10 mm, 1-2-3 mm thick

1. Polishing

F82H in Japan, CLAM in China,

Rusfer & Eurofer in Russia, at grinding and polishing machine Multiprep (Allied).

a) Disks with silicon carbide shaped
b) Diamond films with size of abrasive particles from 35 to 9 μm. At polishing the samples are wetted with water.
c) Polishing textile with the application of diamond suspensions based on glycol with a decrease in crystal size from 6 microns to 0.04 microns.

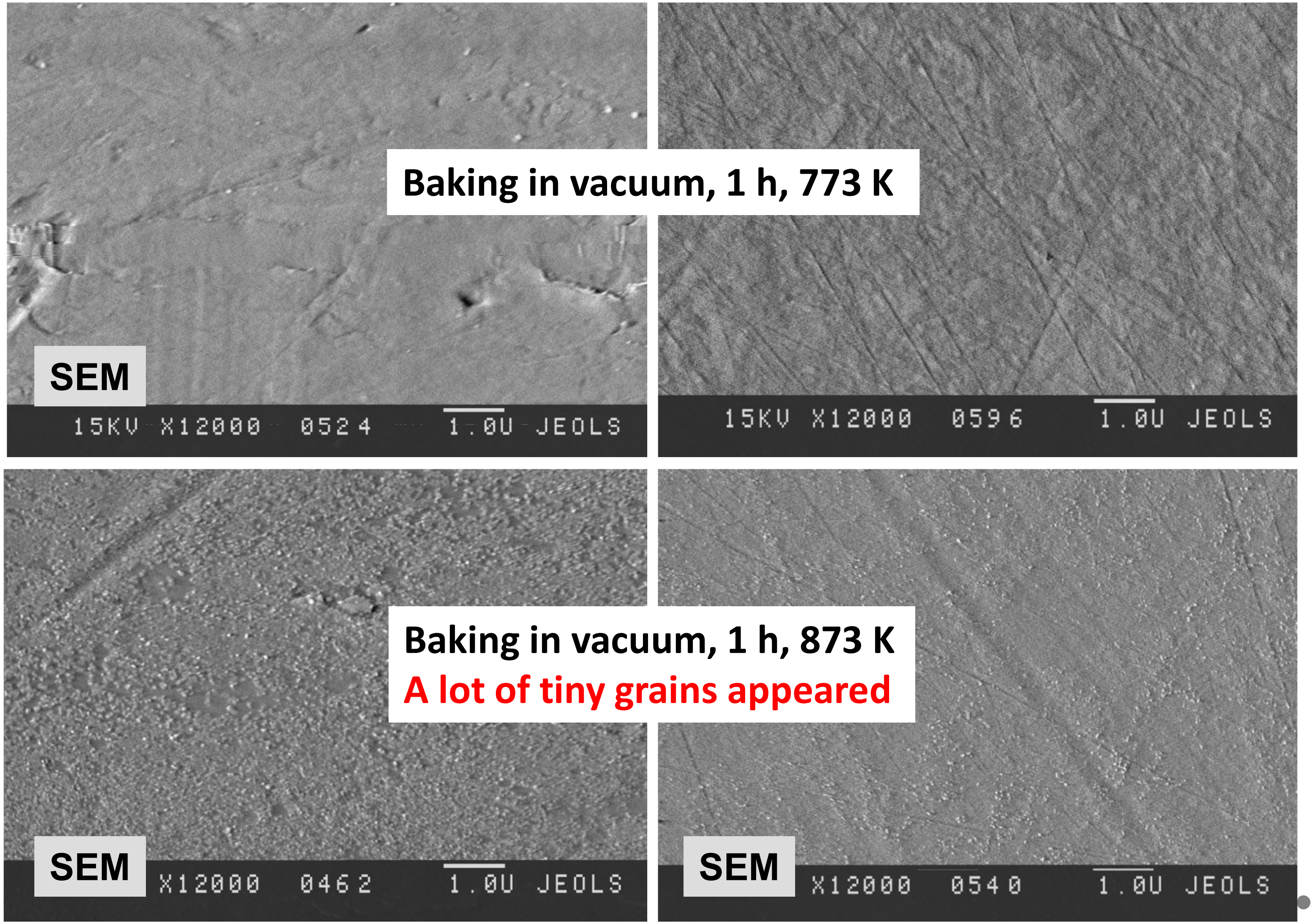
2. Cleaning in ultrasonic bath

a) alkaline liquid solution for cleaning metals (5%) + distilled water - 15 minutes
b) isopropanol-15 minutes
c) ethanol – 15 minutes

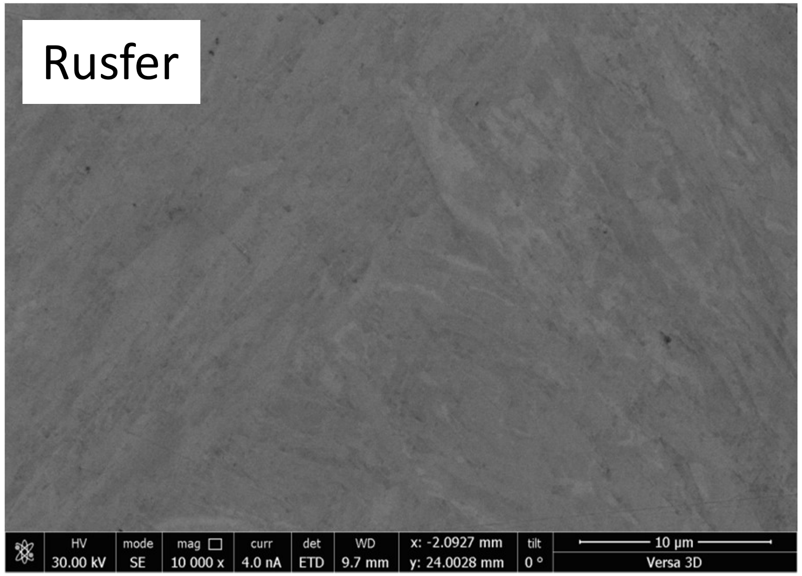
3. Heating in vacuum at 450 C (773 K) for 2 hours

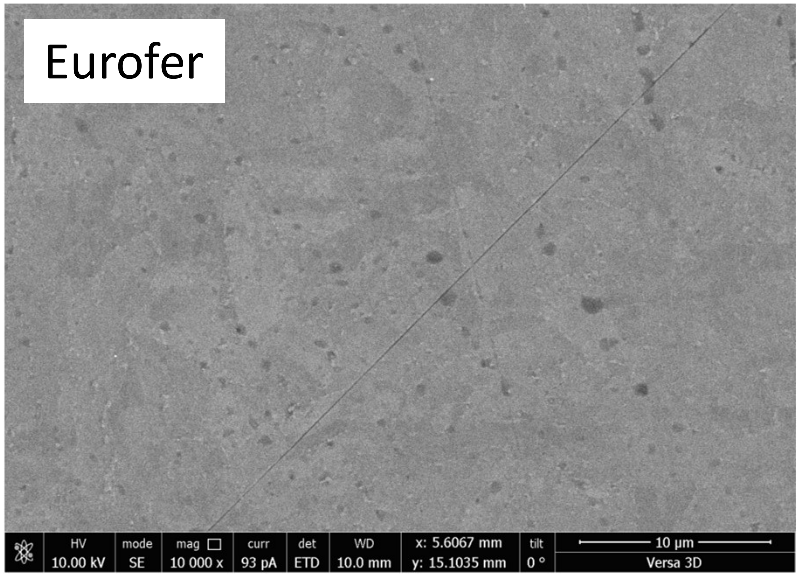
Structure changes of materials at annealing

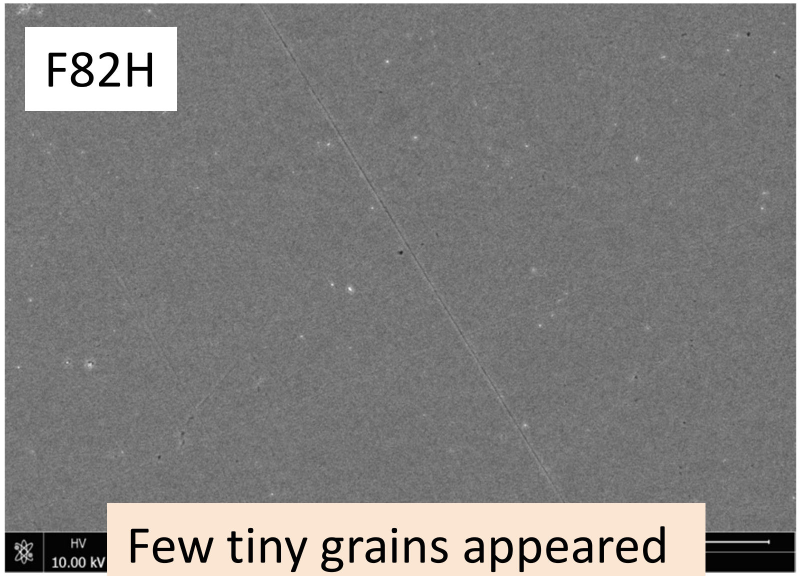
Rusfer (working limit 973 -1023 K) | Eurofer



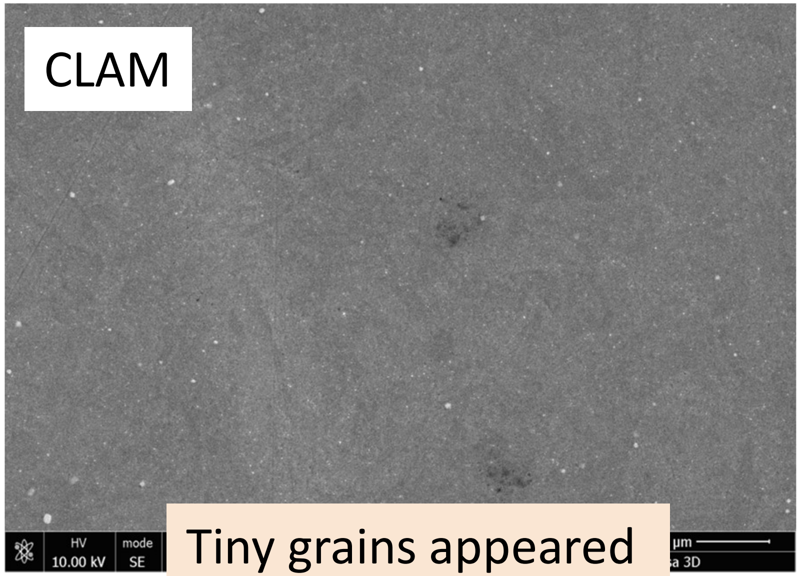
Microscopy







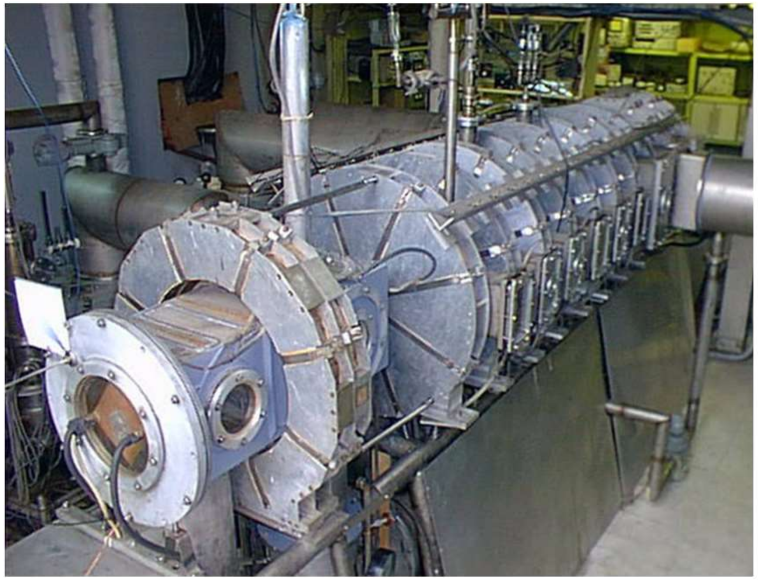
Few tiny grains appeared

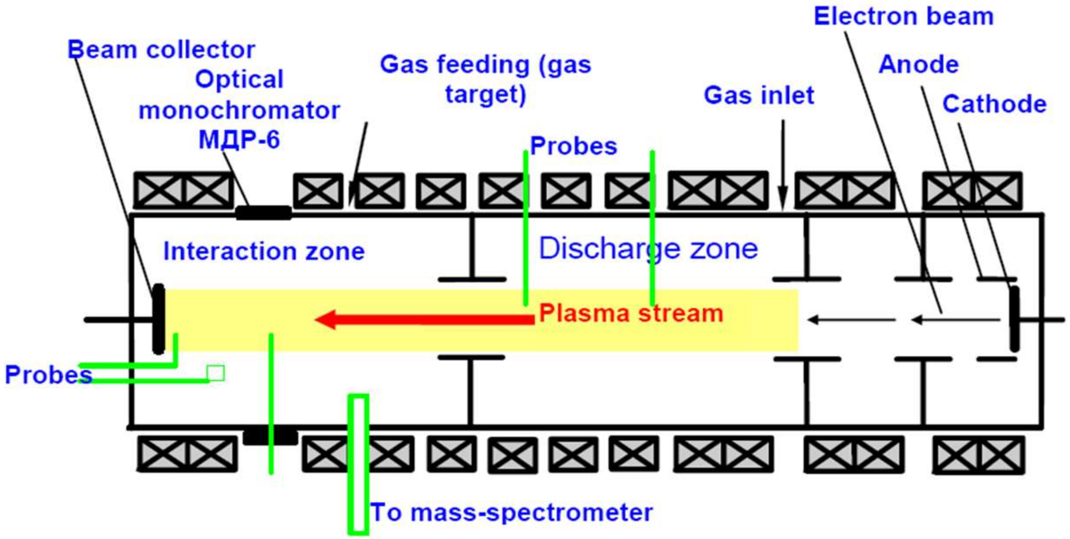


Tiny grains appeared

Selective sputtering study

LENTA *(linear plasma simulator for high flux plasma irradiation)*



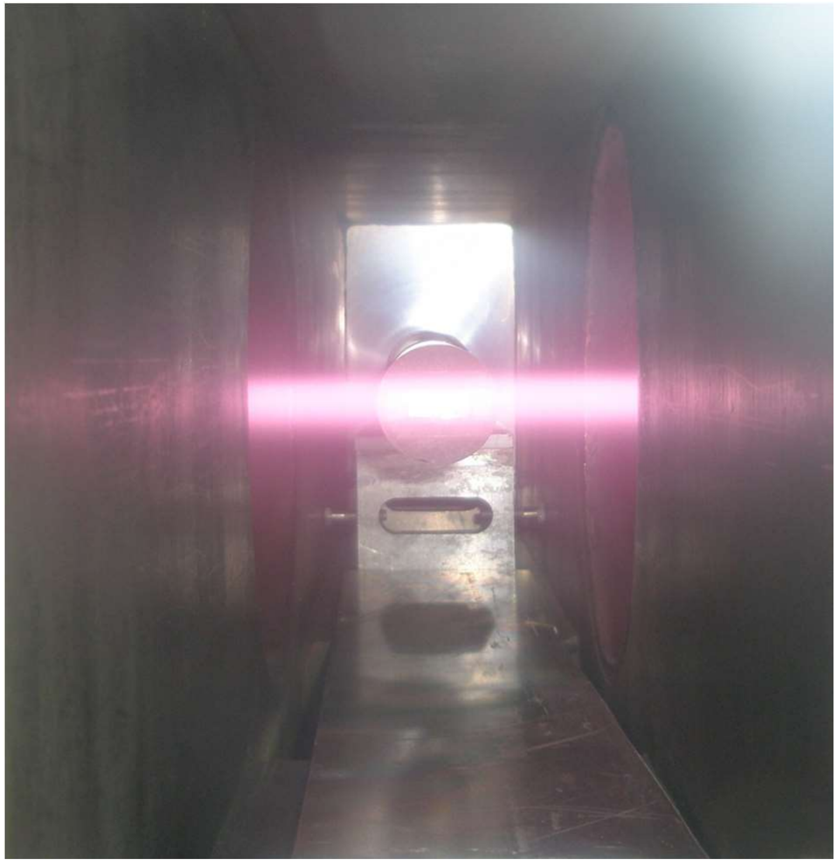


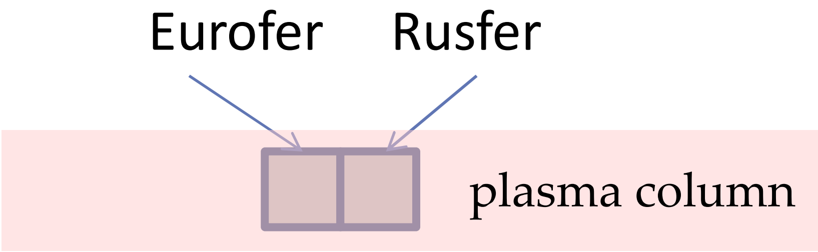
- Steady-state linear plasmas
- Electron beam is used to generate plasma in crossed E ⊥ B fields

Axial magnetic field	**0,2 T**	**Plasma density**	**$10^{11}\div5\times10^{13}$ cm^{-3}**
Injector power	**15 kW**	**Electron temperature**	**0.5÷30 eV**
Plasma density	**$10^{11}\div5\times10^{13}$ cm^{-3}**	**Plasma flux**	**$10^{17}\div10^{19}$ ion/c × cm^{-2}**
Electron temperature	**0.5÷30 eV**	**Plasma fluence**	**$10^{17}\div10^{19}$ ion/c × cm^{-2}**
Plasma composition		**Ion energy**	**10-500 eV**
D_2^+ - 70÷30%, D^+ - 30÷90%, D_3^+, O^+		**Sample temperature**	**RT-1800K**

Selective sputtering: conditions of experiment

Samples orientation: parallel to the ion beam

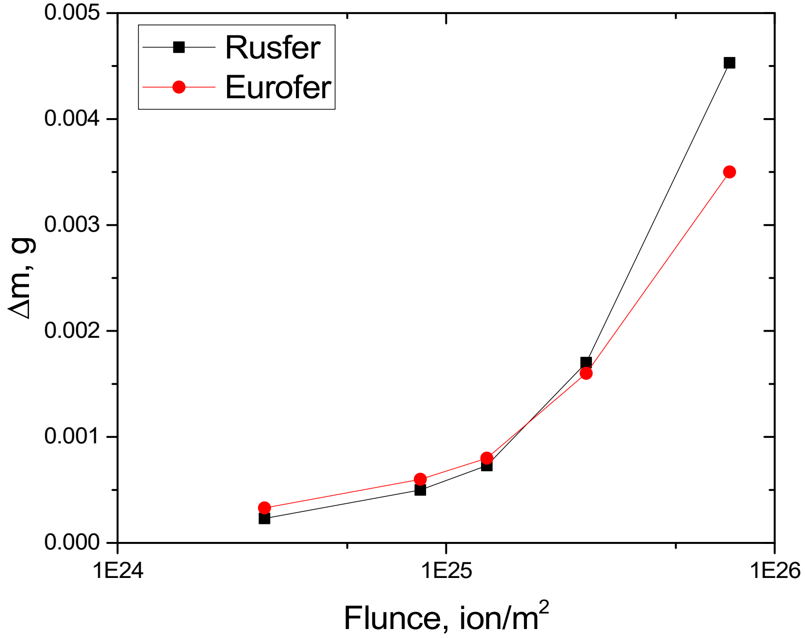


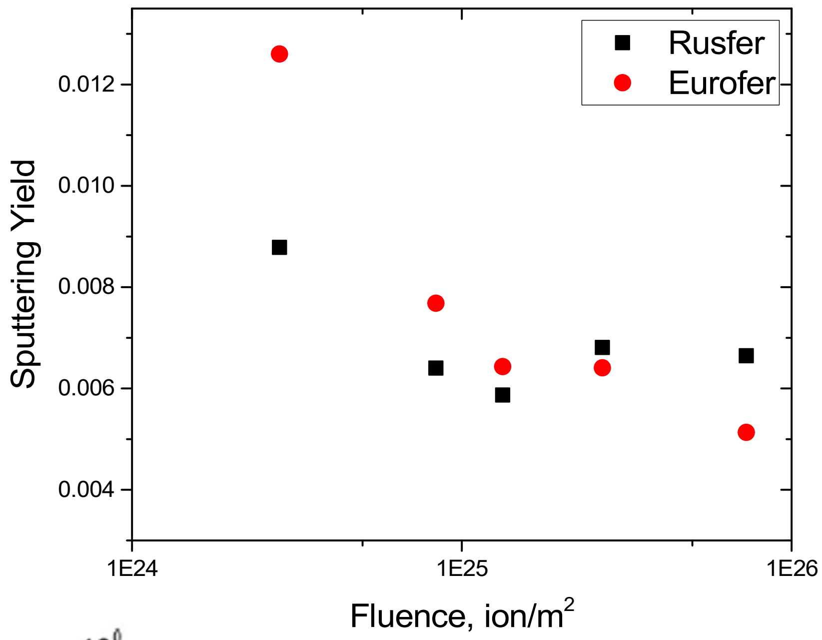


1 mm thick samples

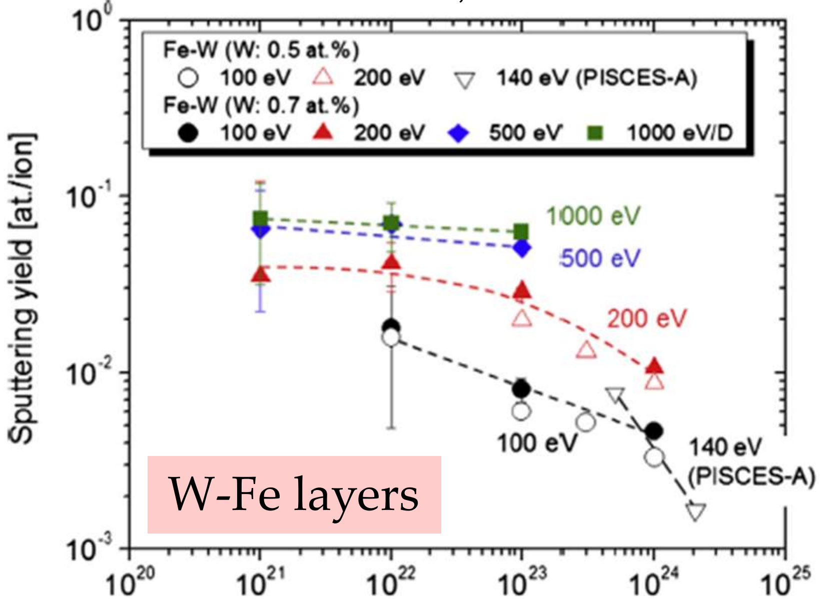
Ion energy: 100 eV
Flux: $3 \cdot 10^{21}$ D/m^{2}s
Fluences:
$3 \cdot 10^{24}$, $8 \cdot 10^{24}$, 10^{25}, $3 \cdot 10^{25}$, $7 \cdot 10^{25}$ ion/m^2
Plasma composition – mainly D^+, $D_2^+ < 10$ %, $O^+ < 1$%
Temperature: 380→443 K at the end of all irradiations

Weight loss



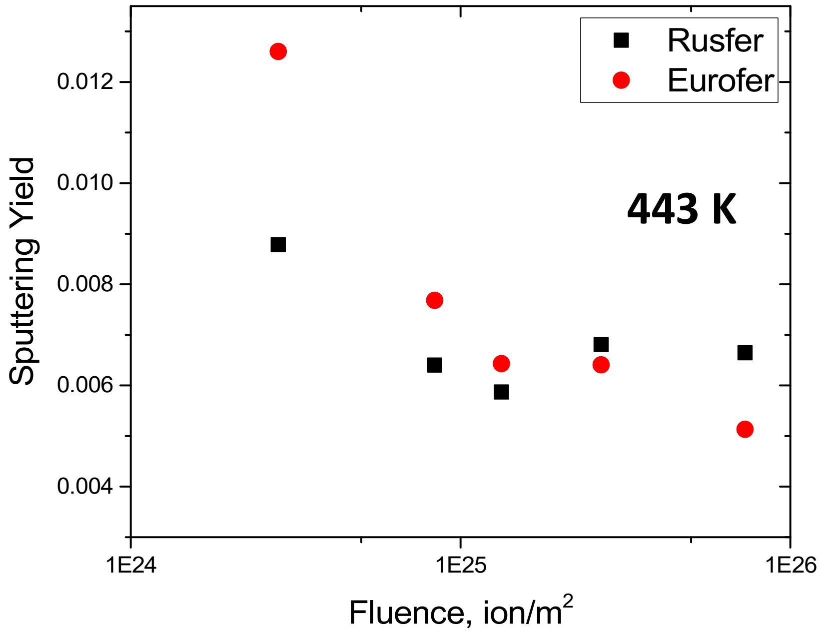


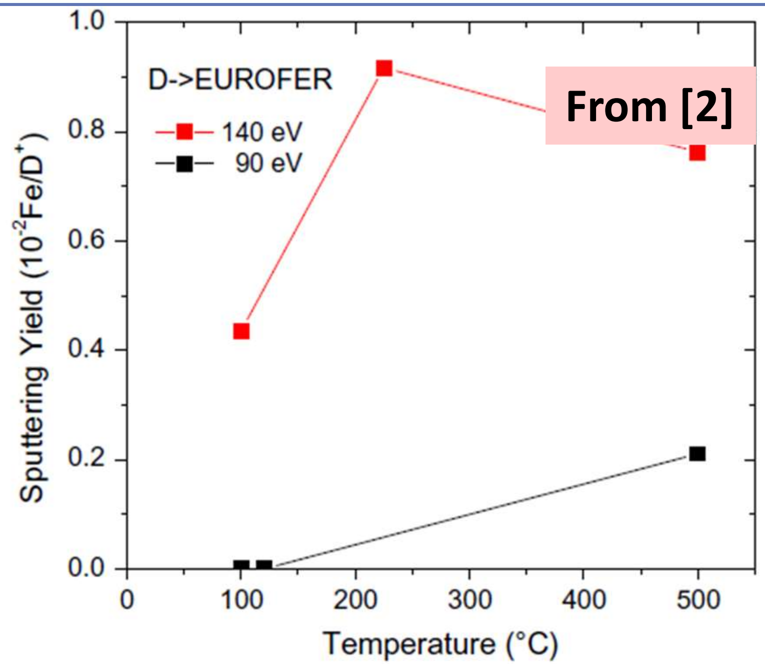
1. **Very close weight losses**
2. **Sputtering yield decreases with fluence**



[K. Sugiyama et al. JNM 463 (2015) 272]

Sputtering Yield





O concentration in plasma is < 1 %, but unknown

Sputtering coefficients, E=100 eV

D^+ R (Fe, RT)=0.00035 [1]

R (Fe, 170)=0.0035 [2]

O^+ R (Fe, RT)=0.4 [SRIM]

R(W, RT)=0.026 [SRIM]

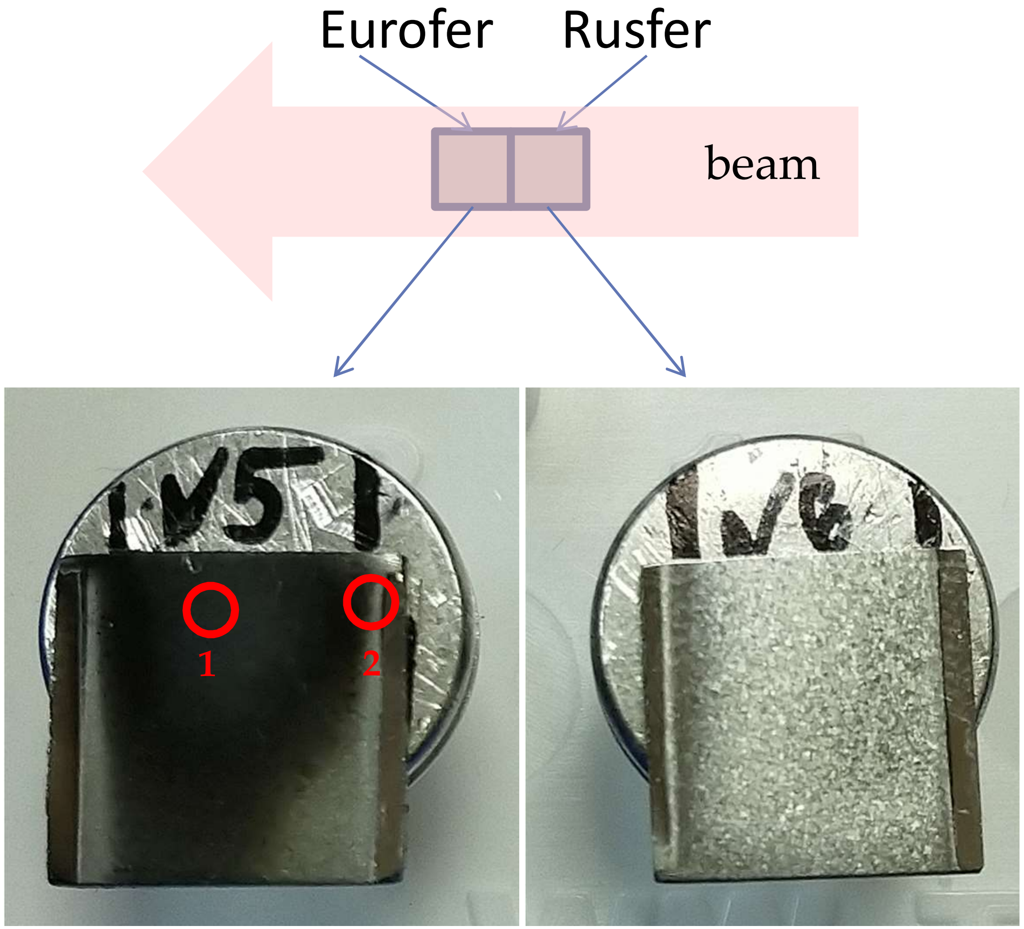
R (Fe, 170 C) =?

Very rough estimation gives, that < 0.75 % of O^+ (even 0.075 % if temperature dependence is the same as supposed for D Sputtering yield)can give about 40% of sputtering

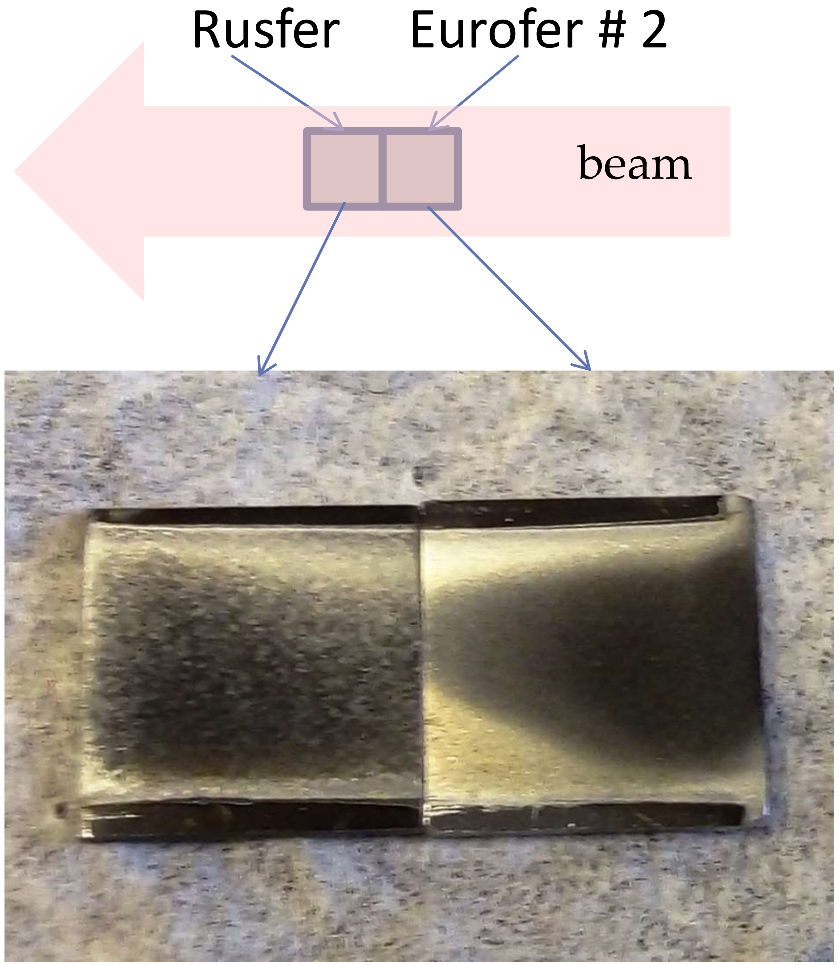
[1] Eckstein, Garcia-Rosales, Roth, Ottenberg, Sputtering data, IPP, 1993
[2] Roth, Sugiyama, Alimov, Hoeschen et al., JNM 454 (2014) 1–6

Selective sputtering - surface

10^{25} ion/m^{-2}

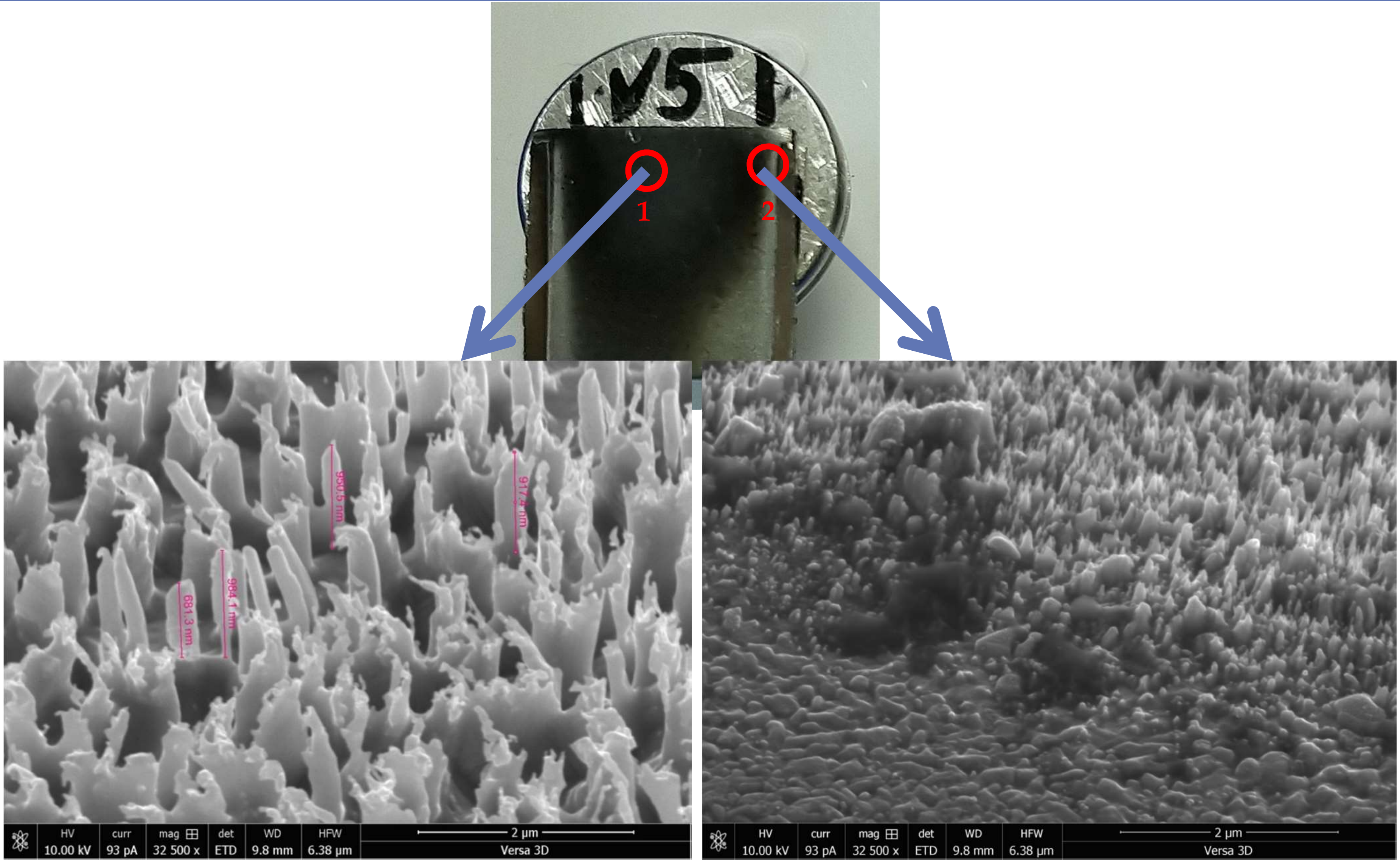


$7.3 \cdot 10^{25}$ ion/m^{-2}



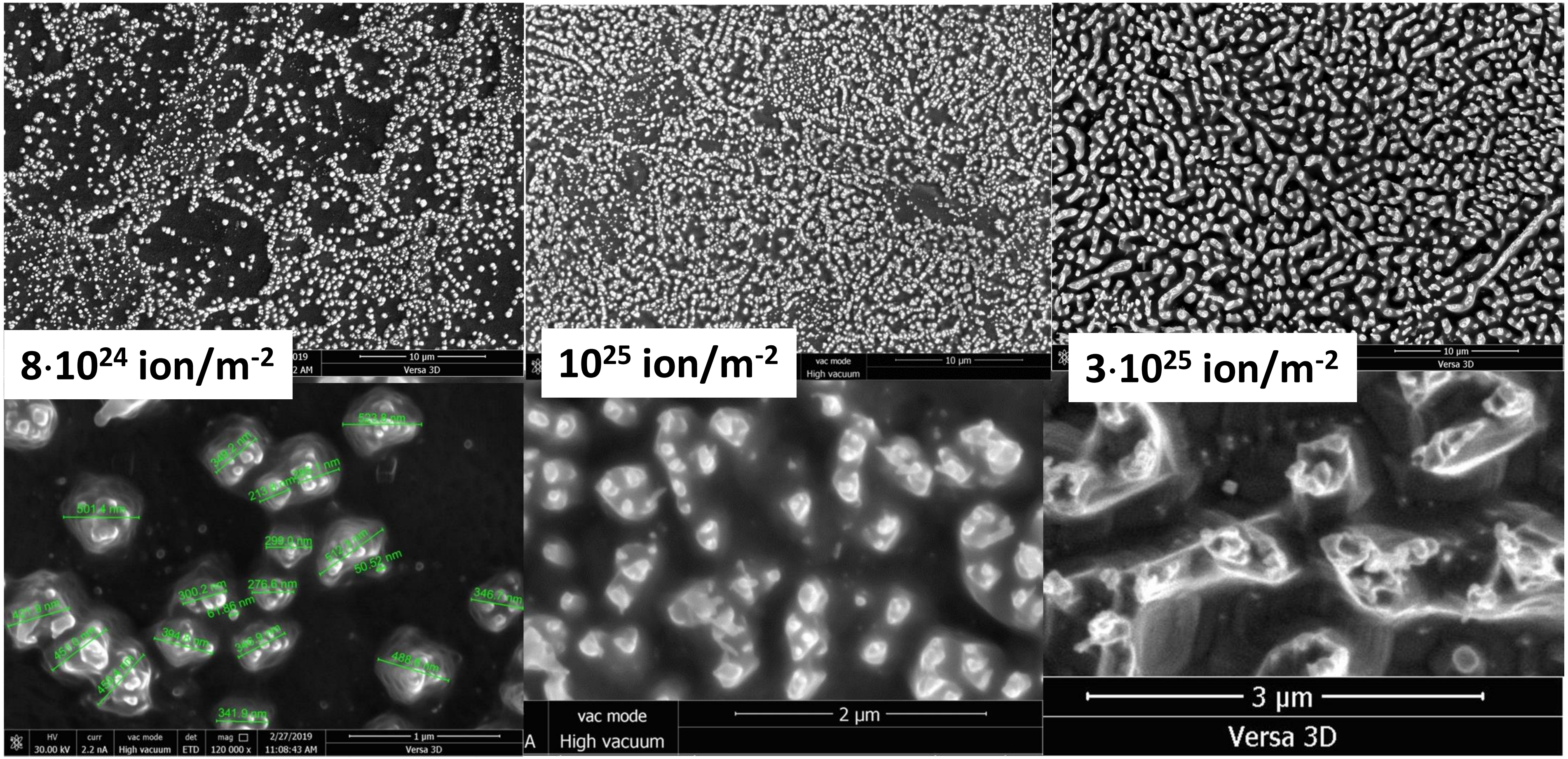
- **Sputtered area ≠ sample area (magnetic samples)**

$3\cdot10^{25}$ ion/m^{-2}, Eurofer

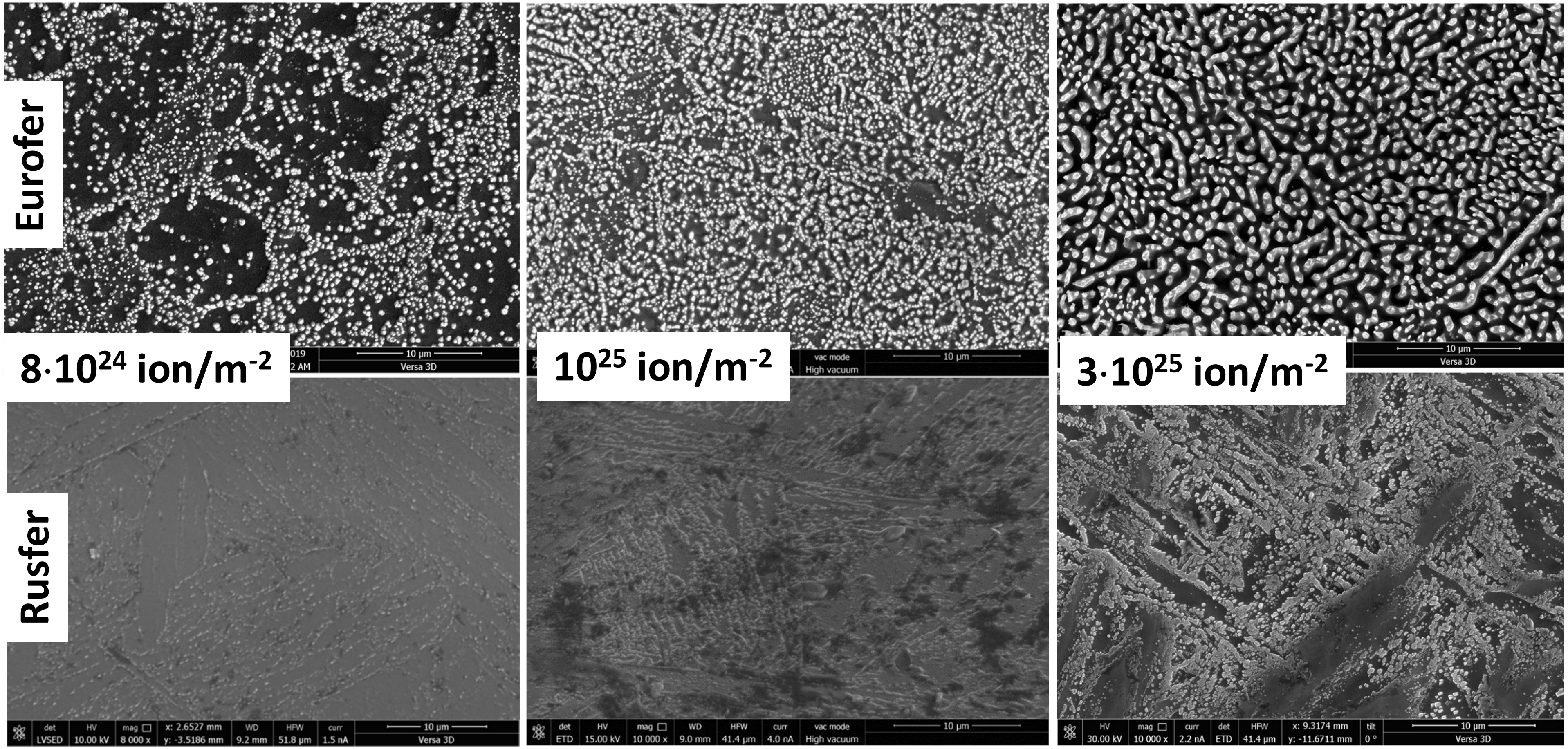


Possible reason for non-homogeneity– magnetic material

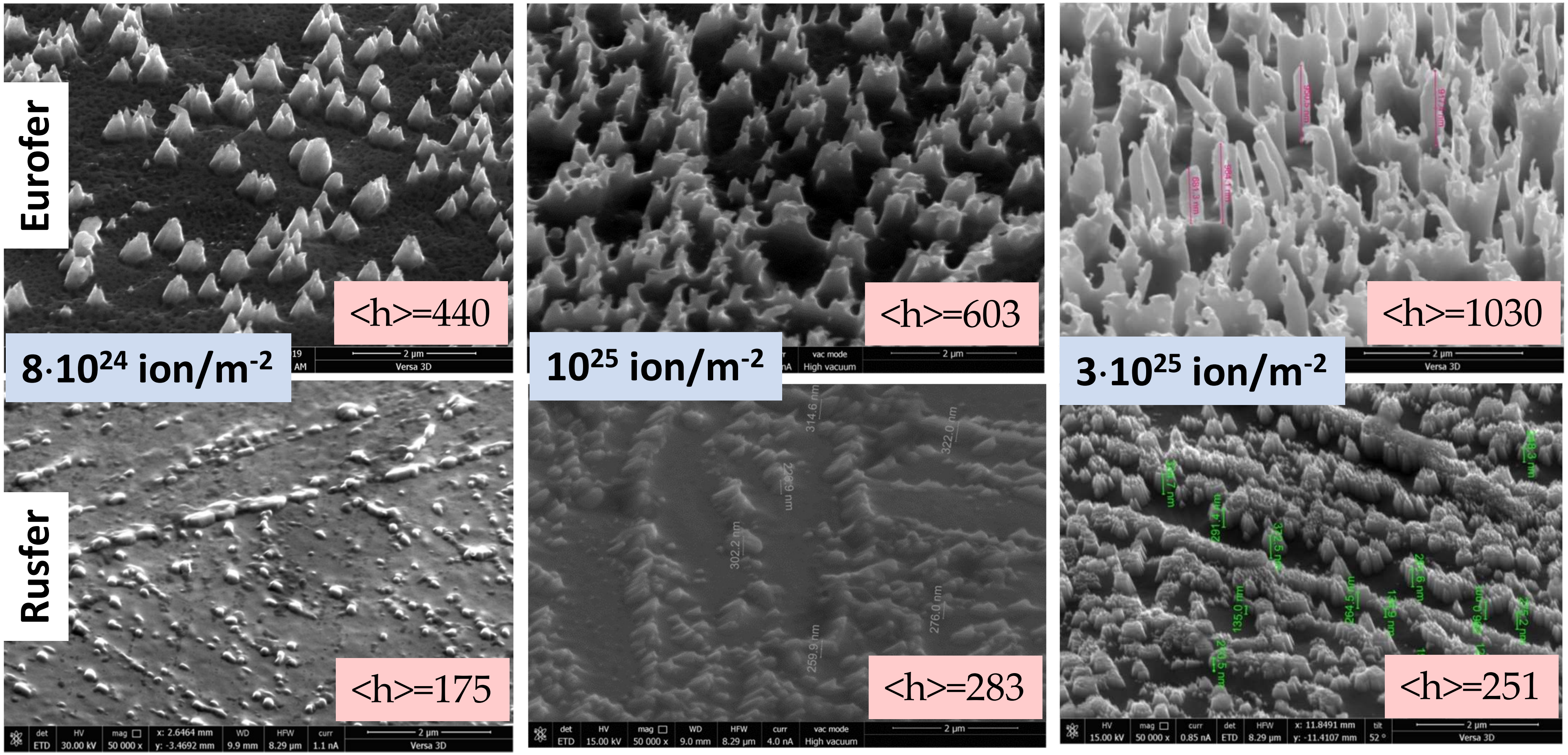
Eurofer Relief, 0°



Relief, 0°

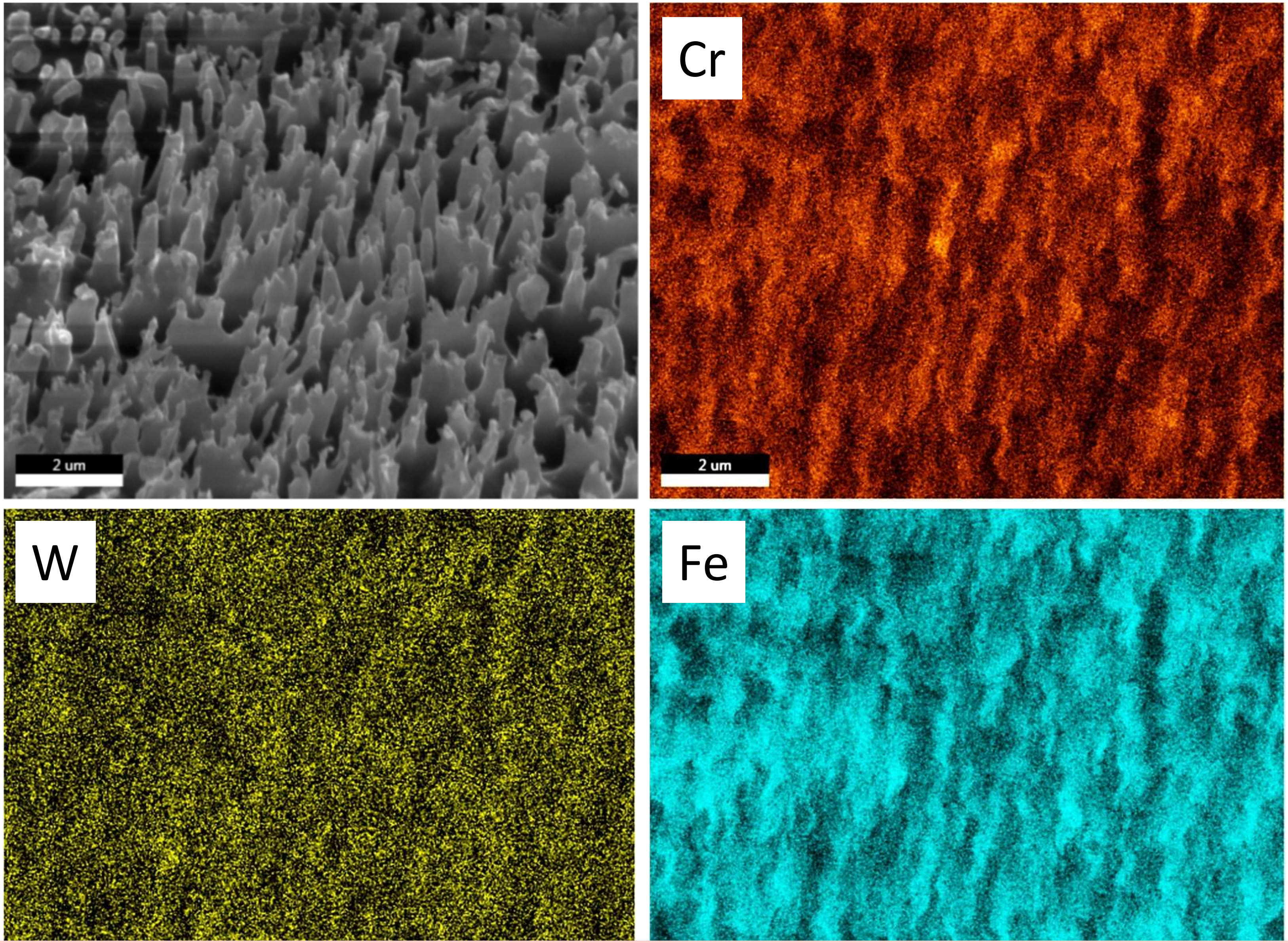

Eurofer
Rusfer
$8 \cdot 10^{24}$ ion/m^{-2}
10^{25} ion/m^{-2}
$3 \cdot 10^{25}$ ion/m^{-2}
10 µm
Versa 3D
High vacuum
det
LVSED
HV
10.00 kV
mag
8 000 x
x: 2.6527 mm
y: -3.5186 mm
WD
9.2 mm
HFW
51.8 µm
curr
1.5 nA
ETD
15.00 kV
10 000 x
9.0 mm
41.4 µm
4.0 nA
vac mode
30.00 kV
2.2 nA
x: 9.3174 mm
y: -11.6711 mm
tilt
0 °

Relief, 52°



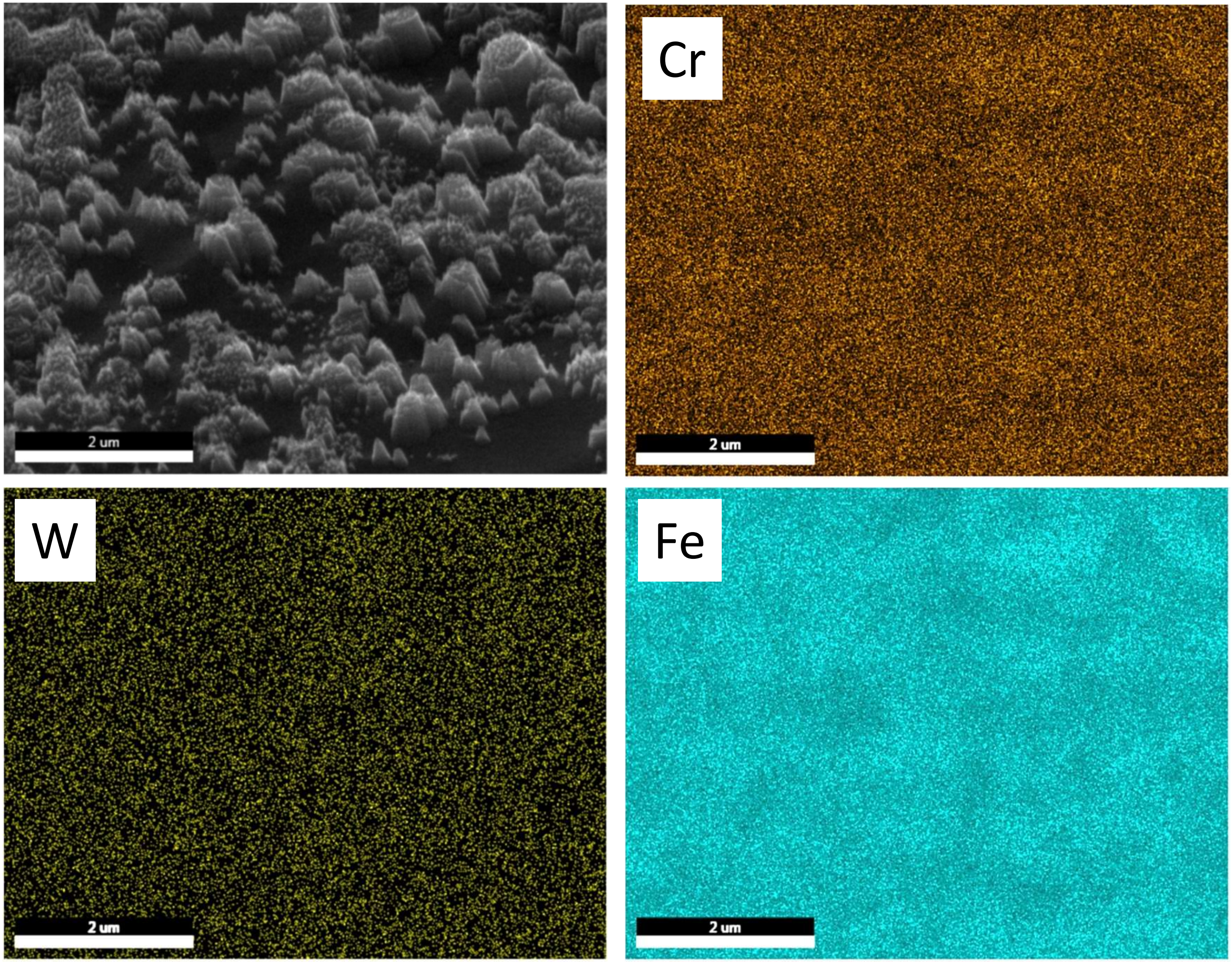
Eurofer
Rusfer
$8\cdot10^{24}$ ion/m^{-2}
10^{25} ion/m^{-2}
$3\cdot10^{25}$ ion/m^{-2}
<h>=440
<h>=603
<h>=1030
<h>=175
<h>=283
<h>=251

$3\cdot10^{25}$ ion/m^{-2}, Eurofer, elements mapping by EDS



Ridges are enriched with W. Concentraions of W & Cr in ridges is decreases

$3{\cdot}10^{25}$ ion/m^2, Rusfer, elements mapping by EDS



Main result:

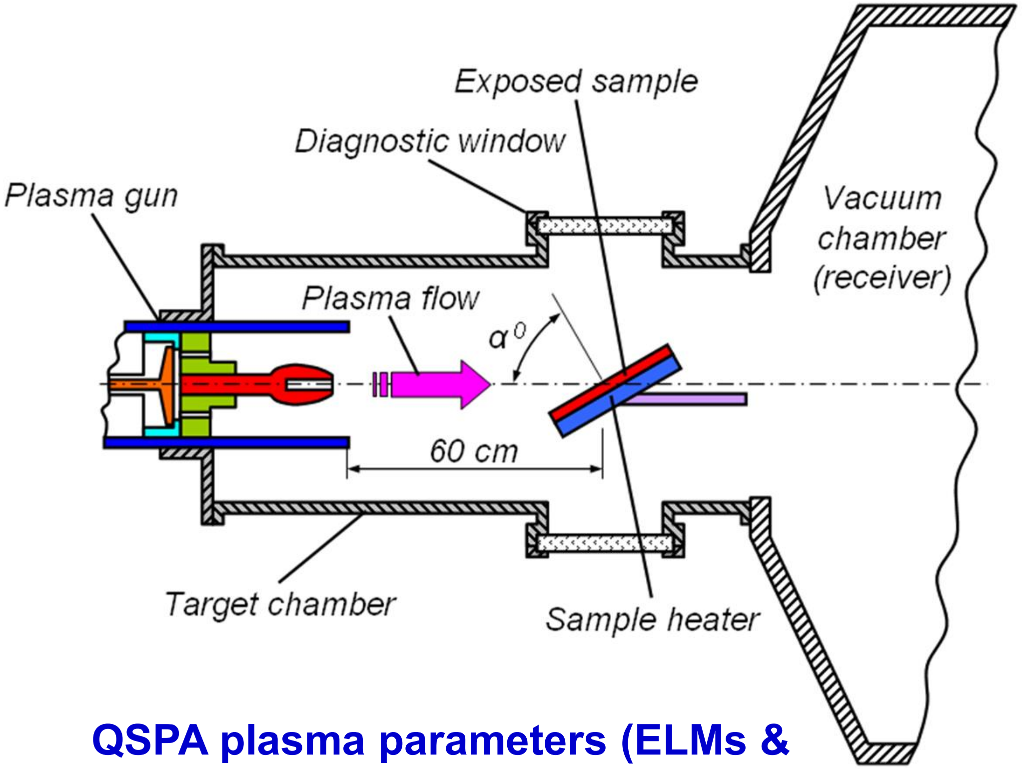
- Surface of Eurofer changes quicker than of Rusfer, combs are several times higher at Eurofer surface at the same fluence
- In combs concentration of Fe & Cr is lower and concentration of W is higher than between combs

Plans:

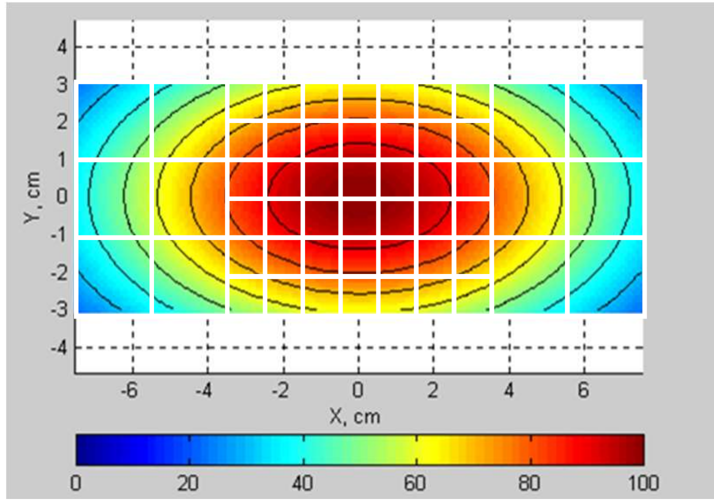
1. Sputtering of CLAM and F82H at the same conditions (this year)
2. Temperature dependence for 4 RAFMS (2020-2021)

ELMs-like D plasma irradiation

QSPA facility, TRINITI, Troitsk



Absorbed energy density distribution,%



Load uncertainty 10-15%

QSPA plasma parameters (ELMs & disruptions):

- Heat load 0.5 ÷ 5 MJ/m^2
  - 0.5÷1.5 MJ/m^2 in ELMs experiments
  - ~2.3 MJ/m^2 in disruption experiments
- Pulse duration 0.1 ÷ 0.6 ms
- Plasma stream diameter 6 cm
- Ion impact energy 0.1 ÷ 1.0 keV
- Electron temperature < 10 eV
- Plasma density $10^{22} \div 10^{23}$ m^{-3}

QSPA facility, TRINITI, Troitsk

Pulsed D plasma irradiation for ELMs simulation

2 types of loads were chosen:

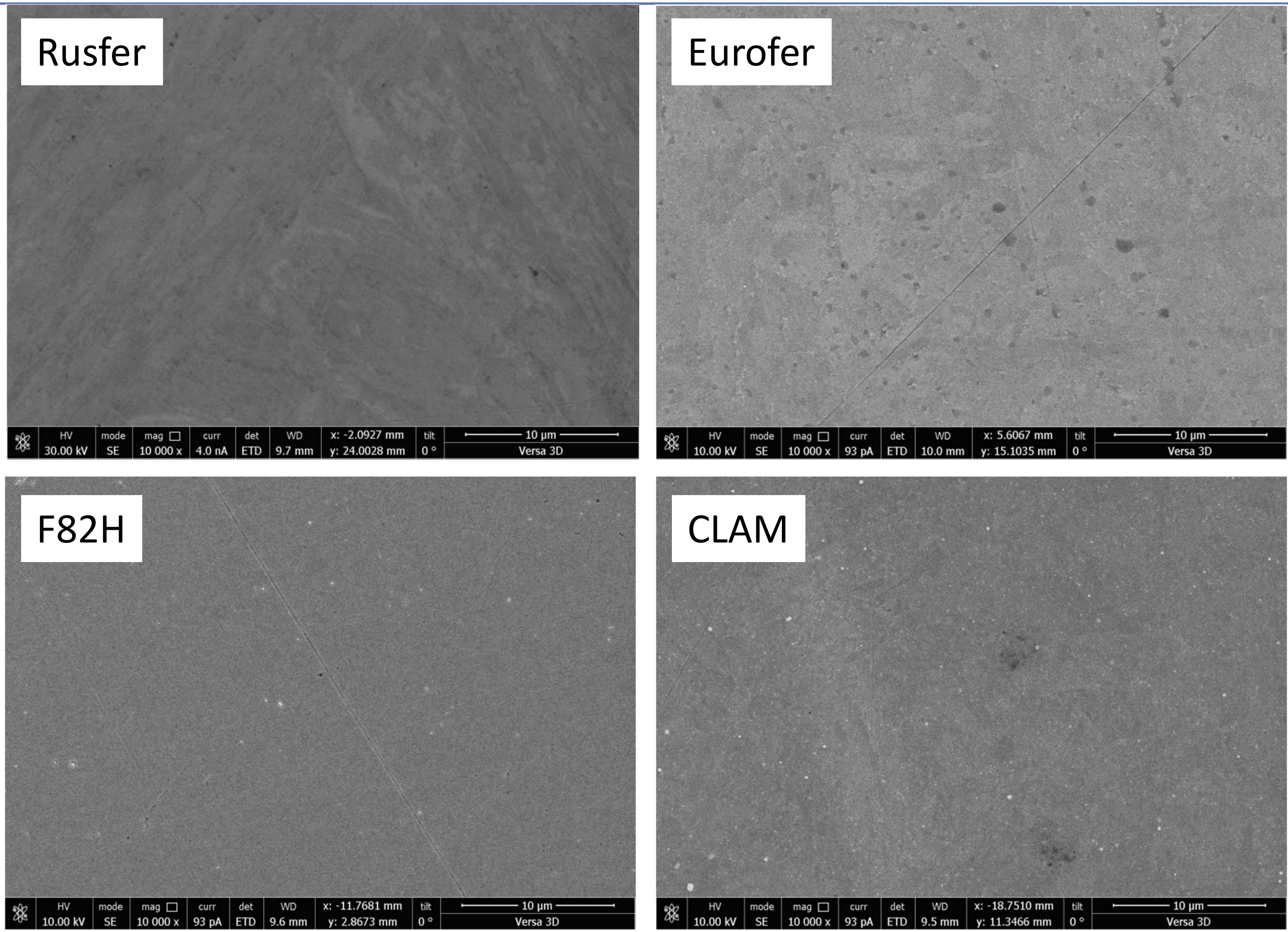
“below melting” – 0.3 MJ/m^2, 1 ms
“above melting” – 0.6 MJ/m^2, 1 ms
Flux at one pulse of 1 ms - 10^{23}-10^{24} D/m^2
Number of pulses – 1, 5, 10, 20, 50

Irradiation in sets of 4 samples – Rusfer (2mm thick), Eurofer (2 mm), F82H (1 mm), CLAM (3 mm) simultaneously

Questions to answer:

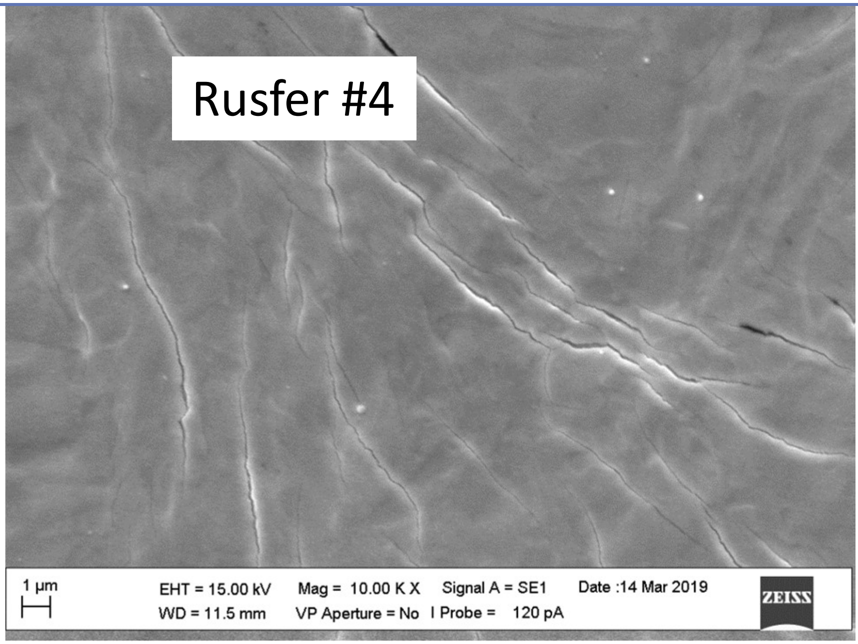
1. How RAFMS sustain ELM-like loads?
2. What amount of D will be retained at ELM-like loads?
3. How ELM-like D plasma damage influences further D retention at interaction with gas and D plasma irradiation?

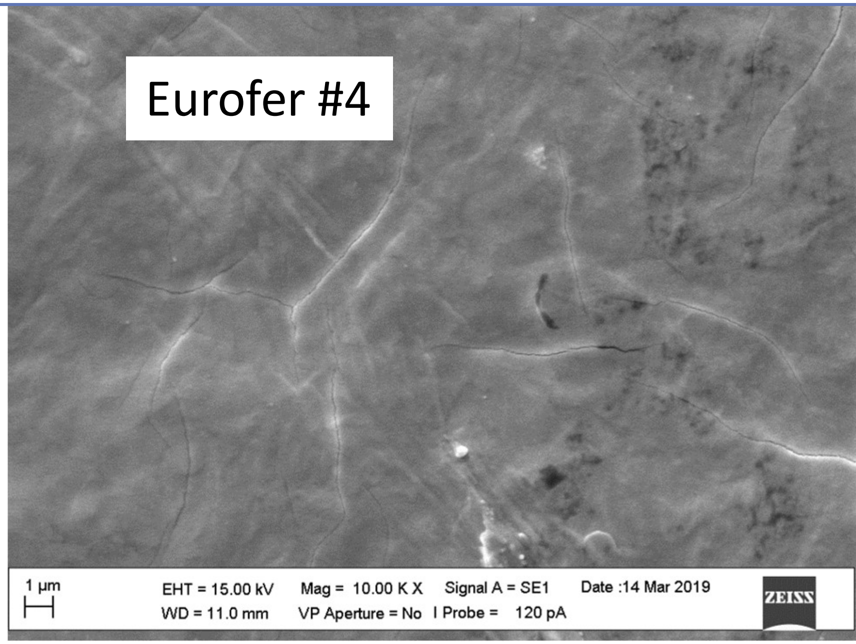
Before irradiation

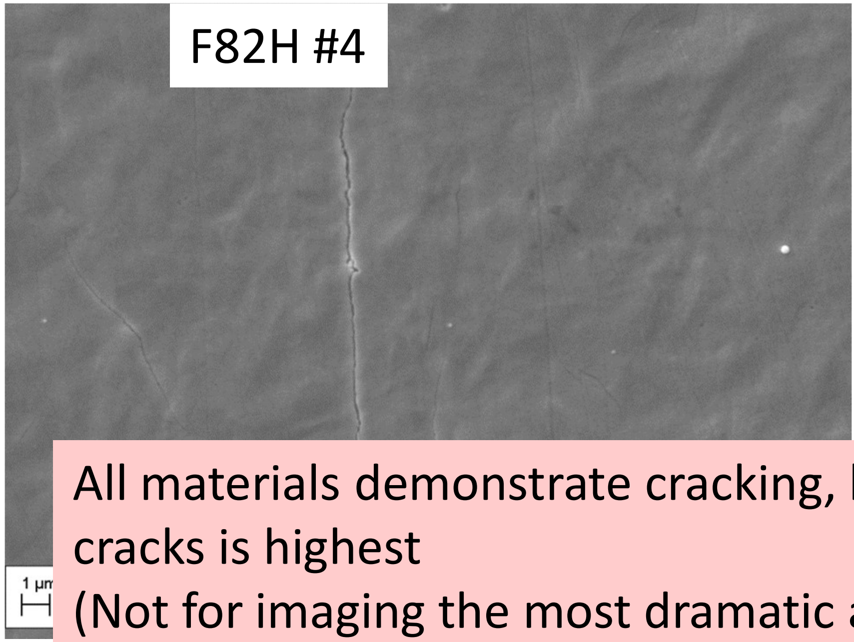


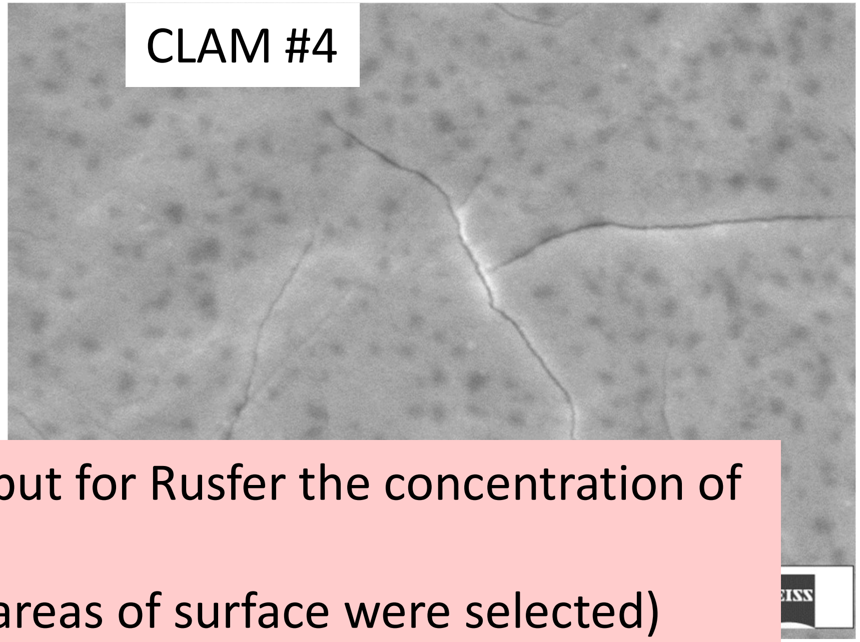
Before melting, 1 pulse – almost no changes

Before melting, 5 pulses



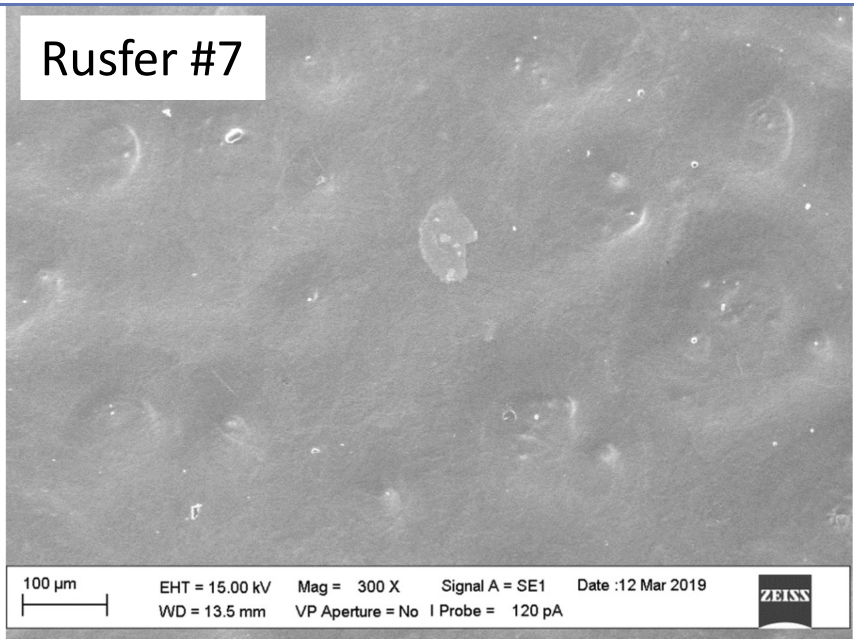


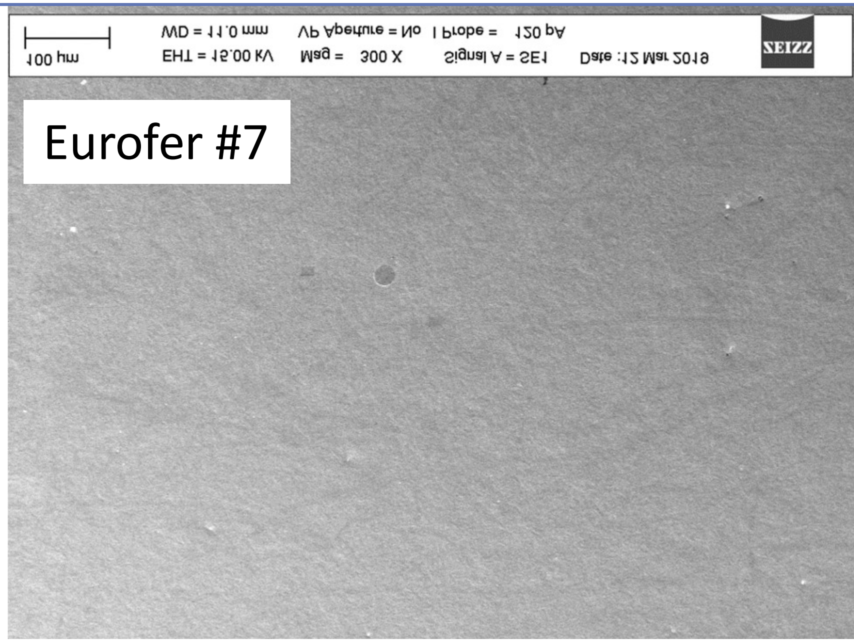


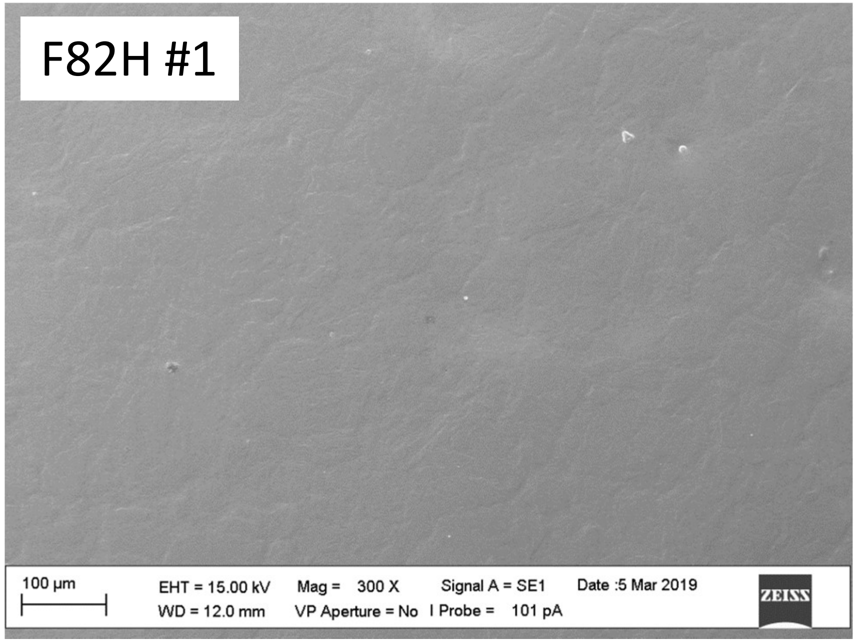


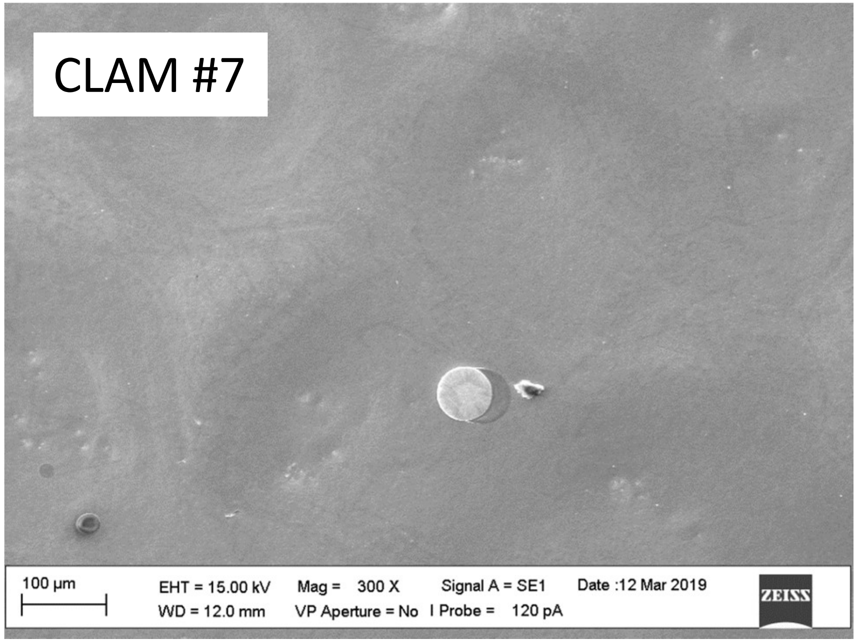
All materials demonstrate cracking, but for Rusfer the concentration of cracks is highest
(Not for imaging the most dramatic areas of surface were selected)

Above melting, 1 pulse

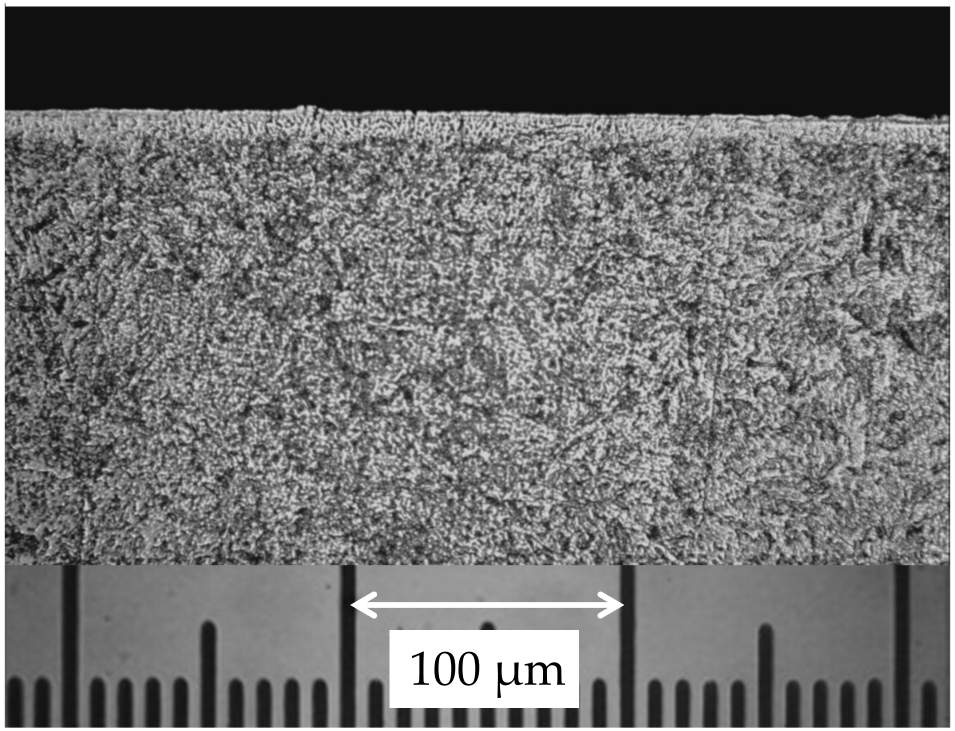


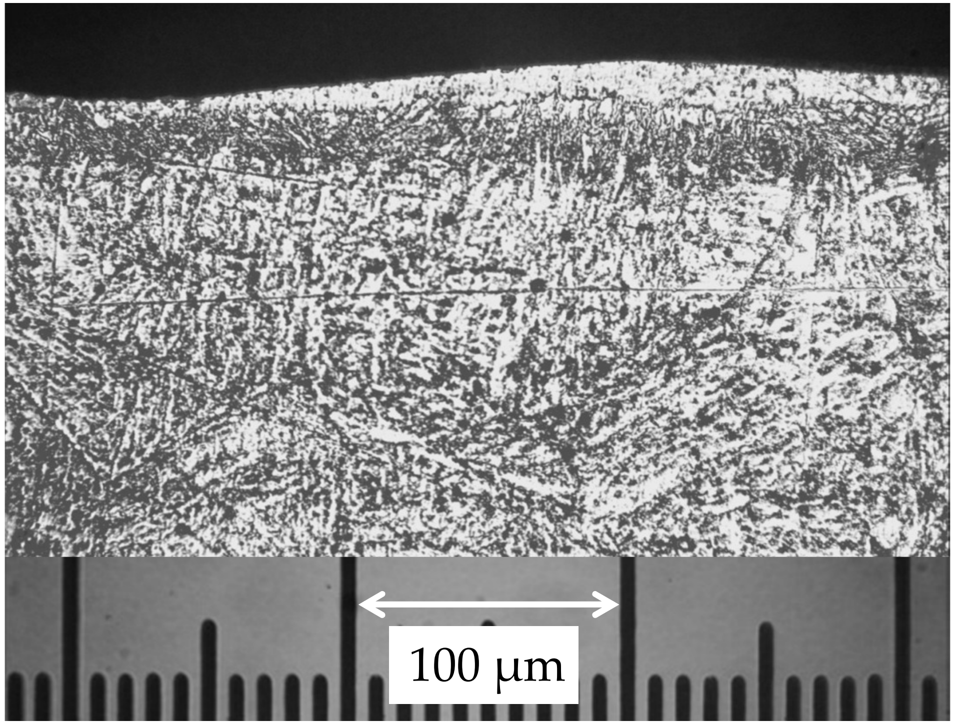




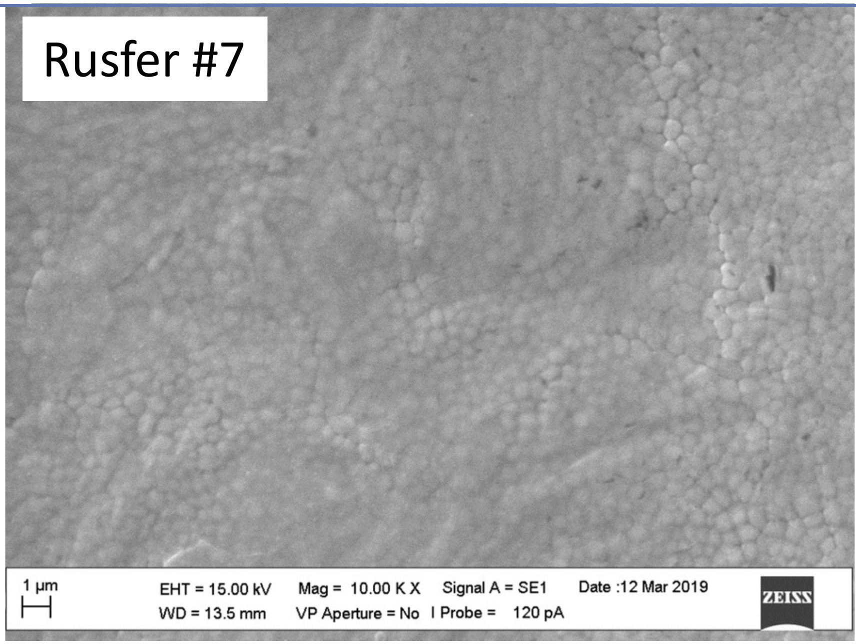


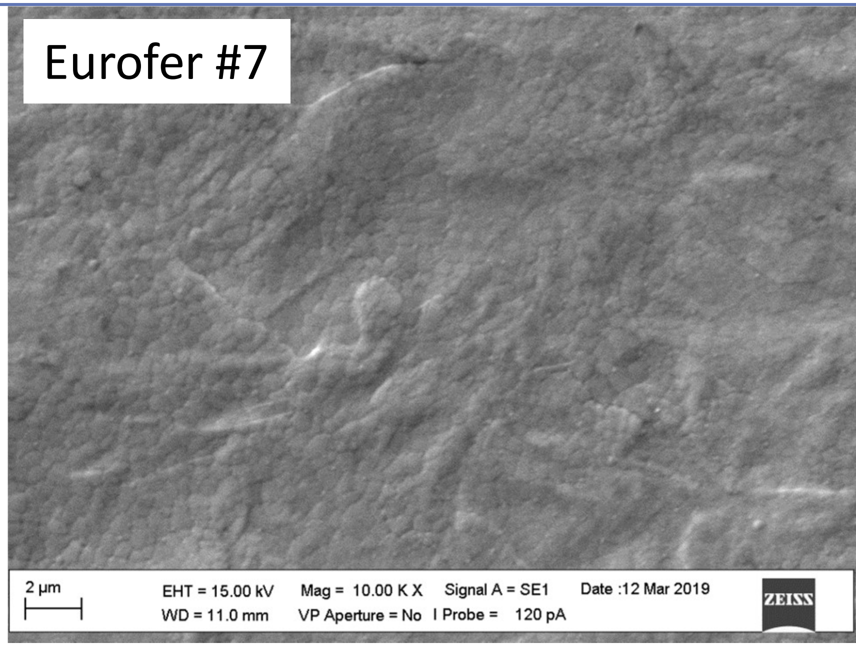
Above melting, 1 pulse

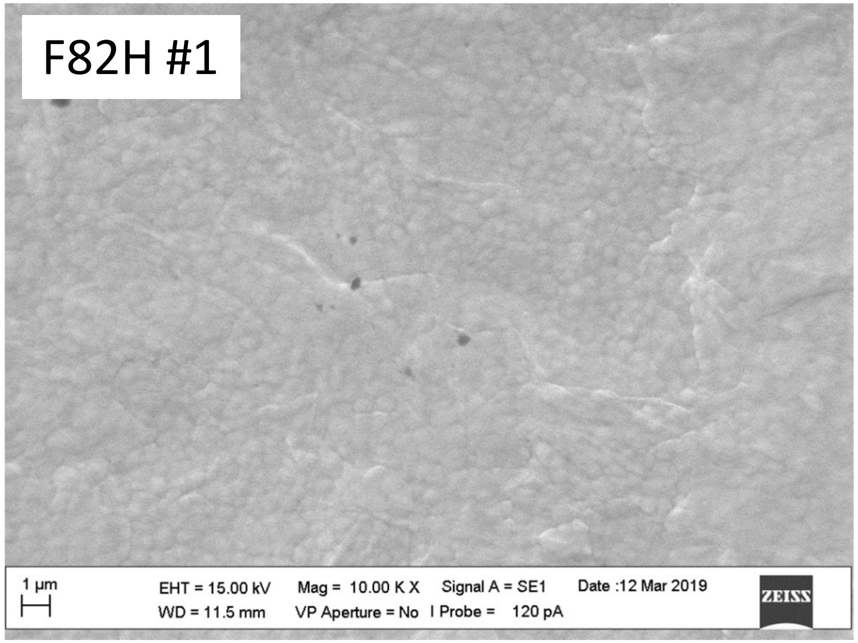


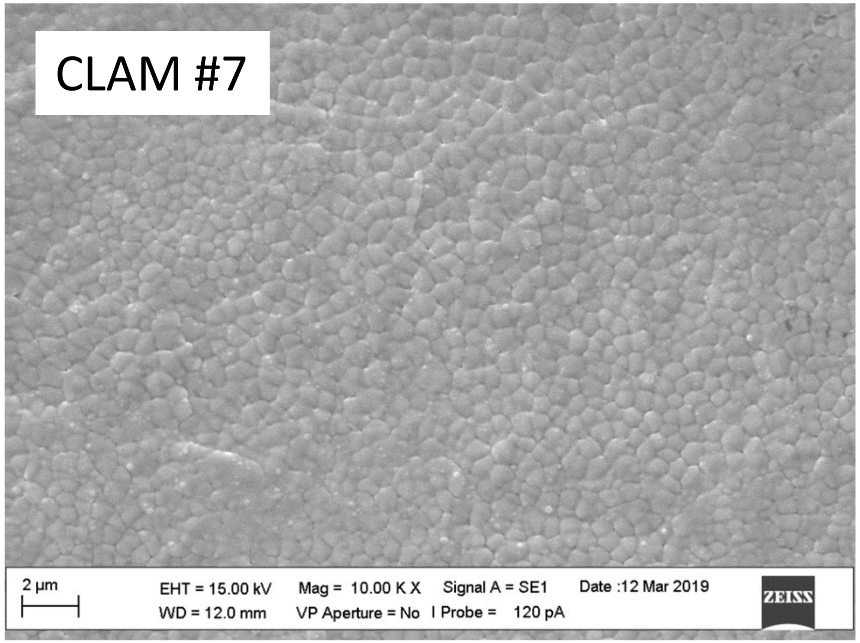


Above melting, 1 pulse

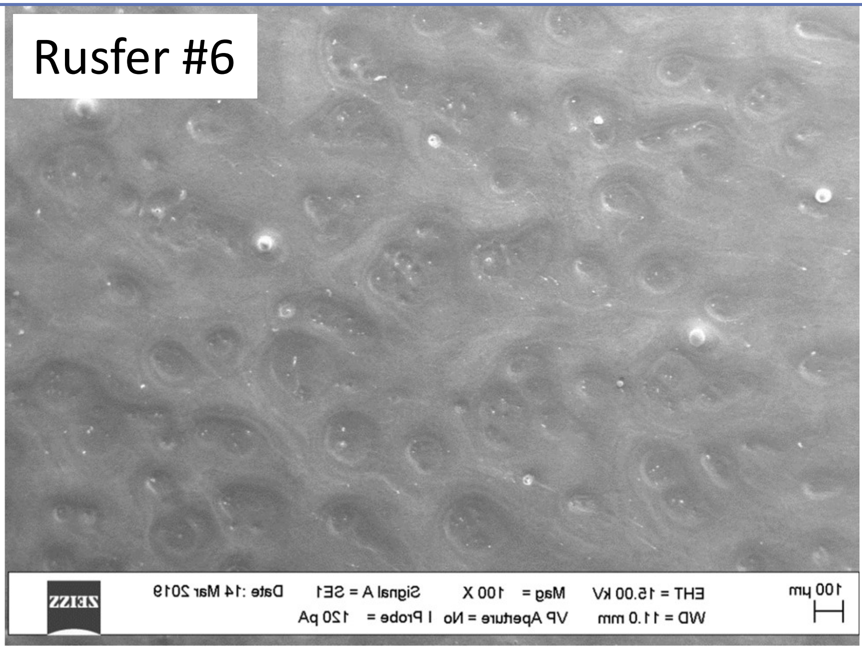


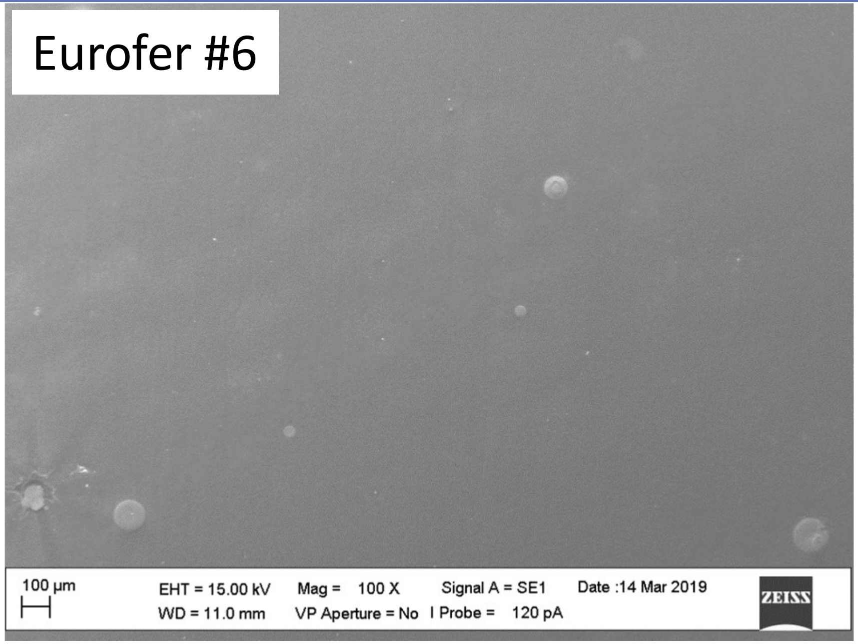


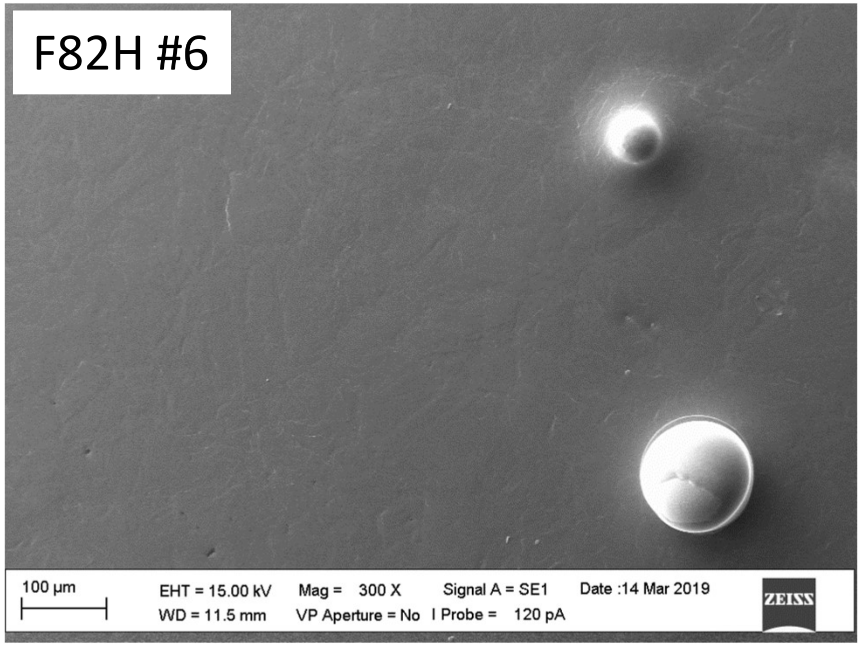


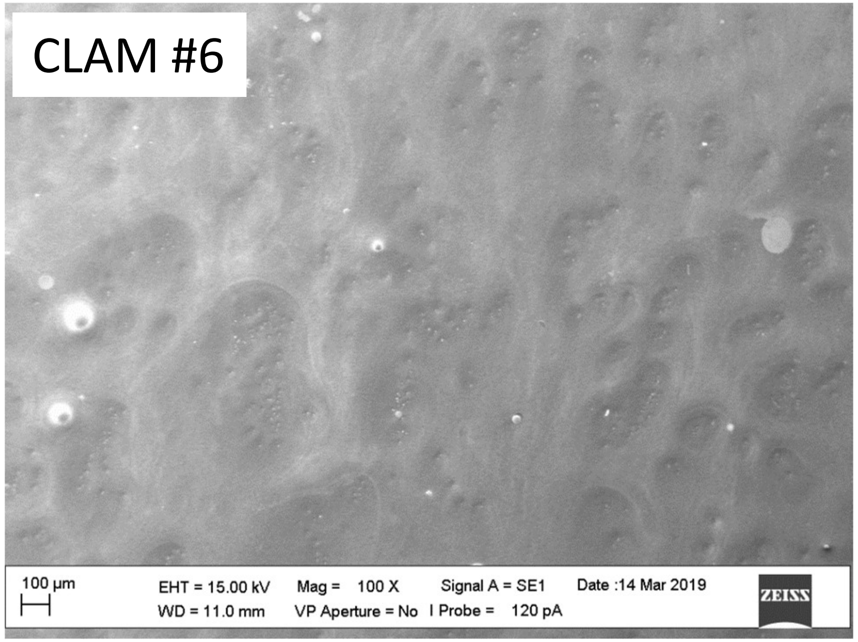


Above melting, 5 pulses

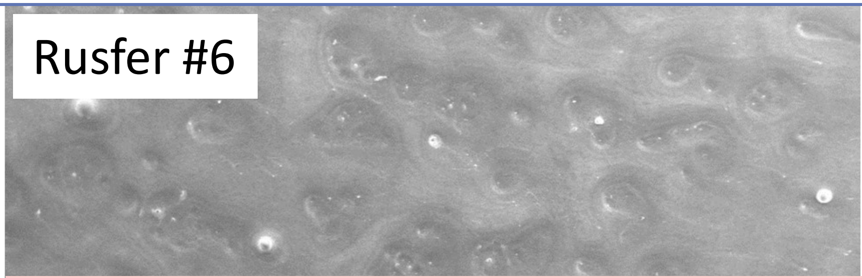


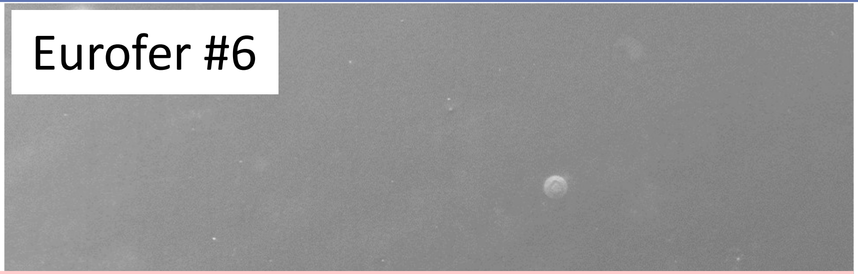






Above melting, 5 pulses

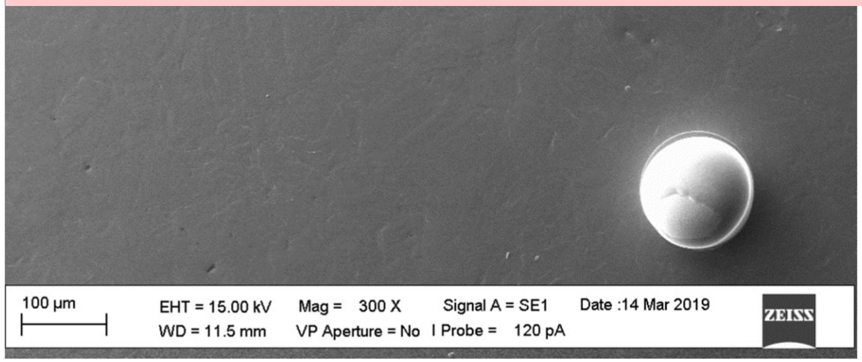


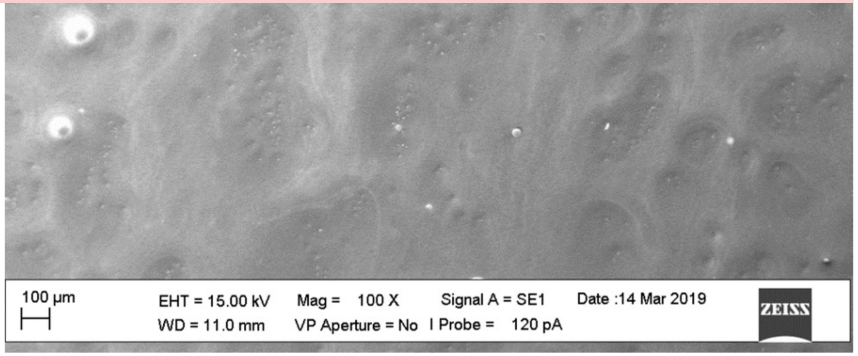


All materials were melted !

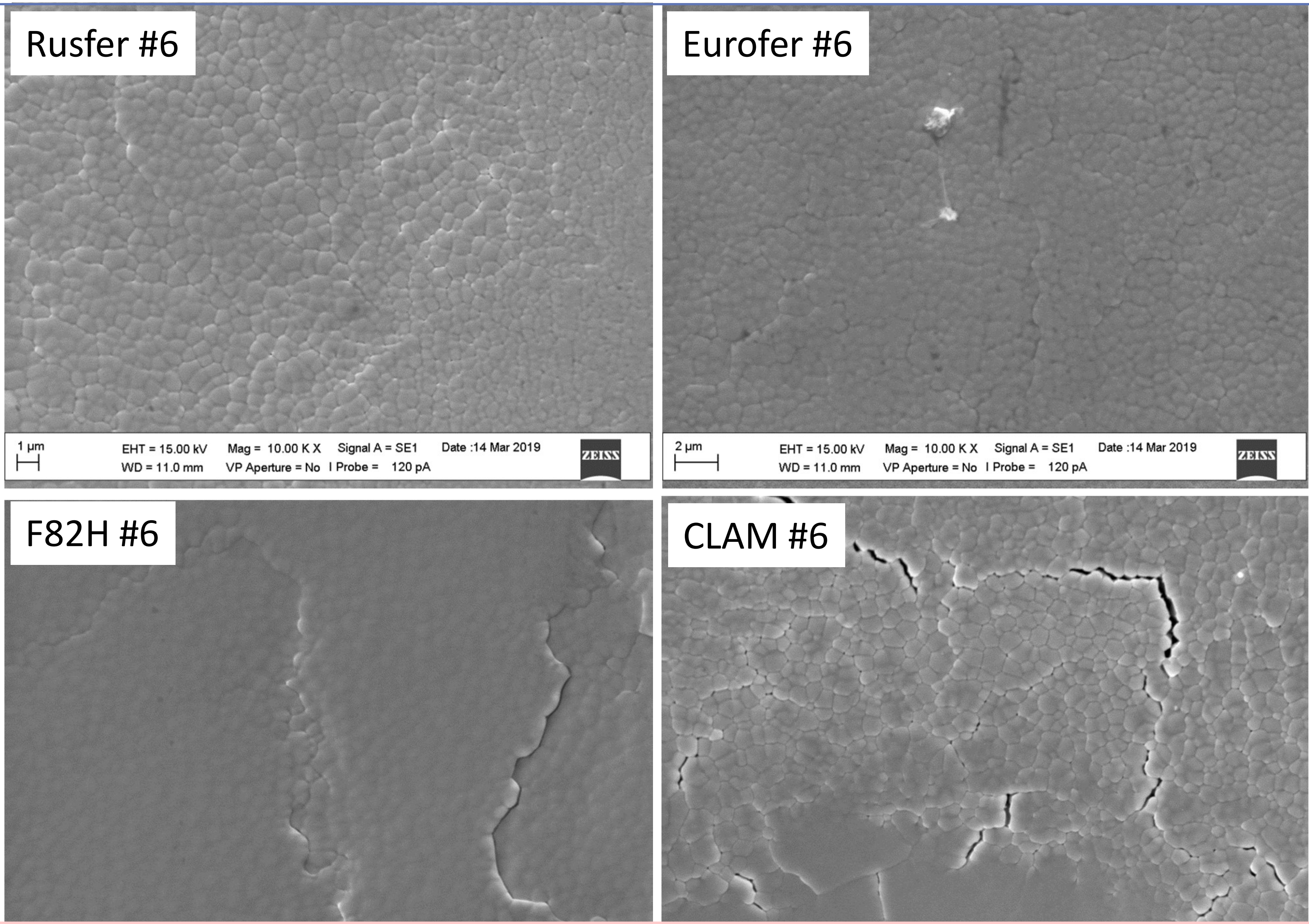
For use as PFMS those materials better that do not move at melting.

Similar situation was described with austenitic steels
[Madarame et al., FED 9-1989-207, Madarame et al., FED 15 I1-1991-75]
It was found that impurities – S, O led to wavy surface (from some %),
While Ca, Al and Ti suppress waving.
These impurity contents affect the temperature coefficient of surface tension in liquid iron.
Probably, other impurities also have similar effects.





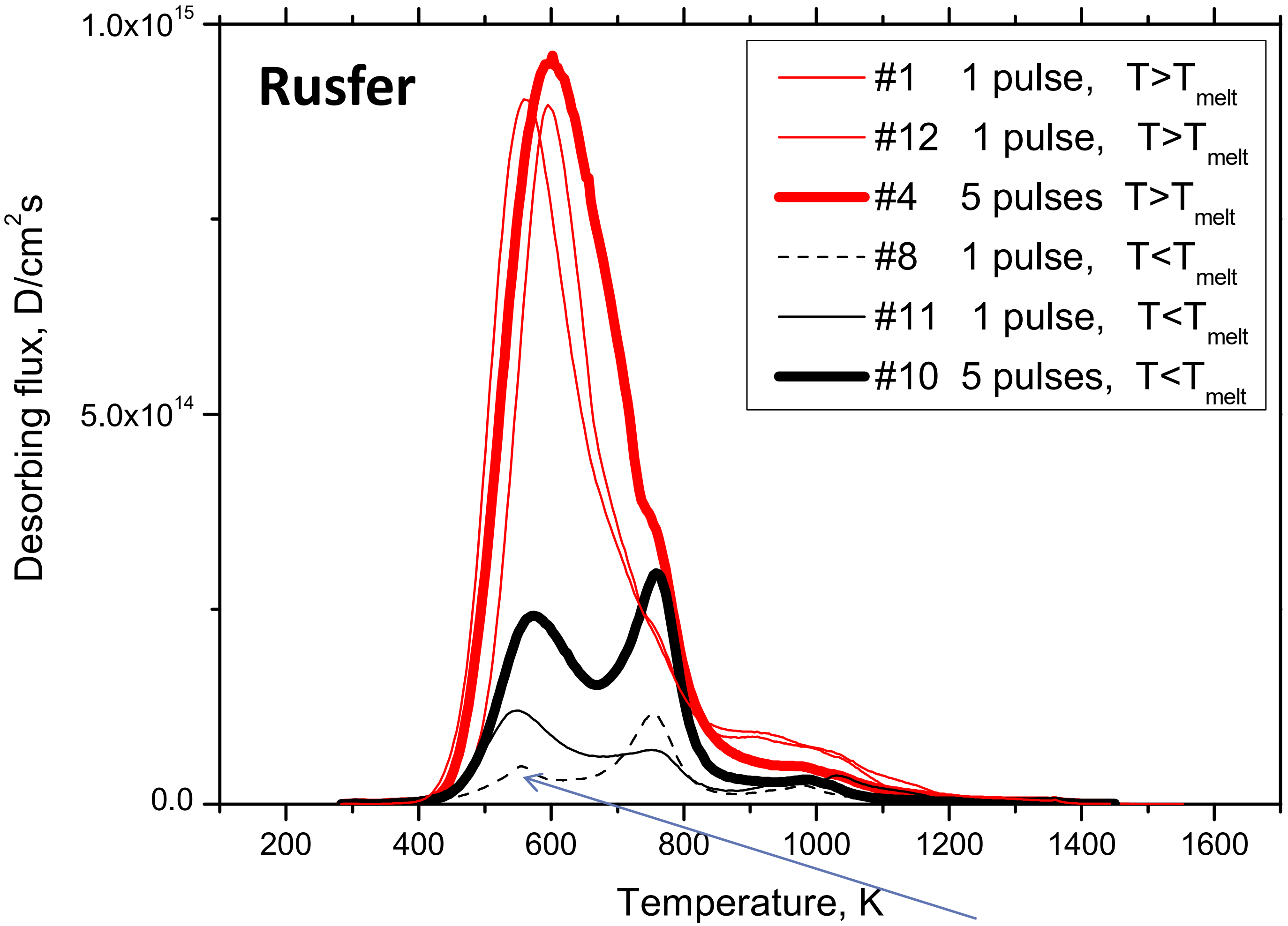
Above melting, 5 pulses



crystallites <1µm are formed at all surfaces. Cracking I most pronounced for CLAM

D retention after pulsed D plasma loads

TDS, 2 K/s



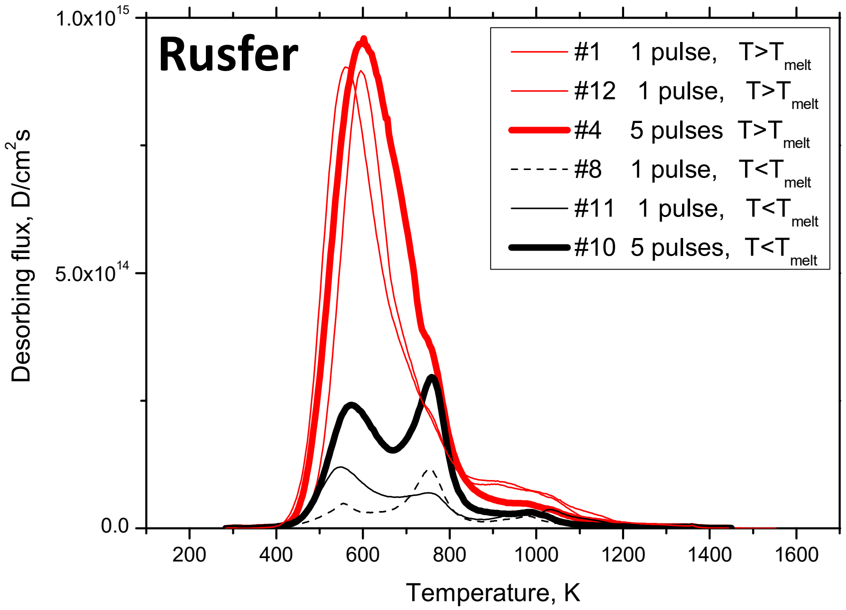
- Retention increases with number of pulses
- If surface melts, retention significantly increases

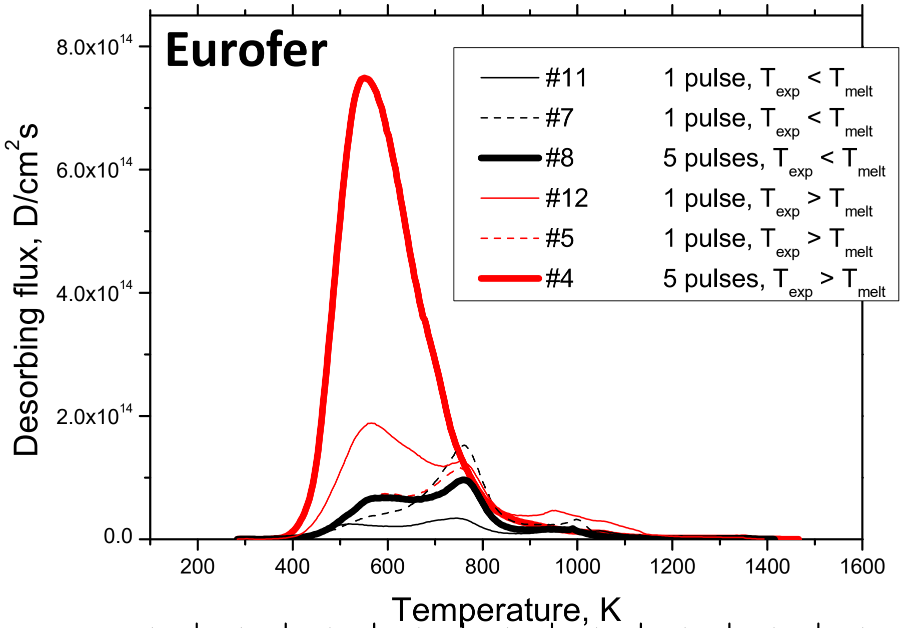
If samples were extracted to atmosphere after plasma pulsed loads with warm surface → Cr oxidation occurs, forming barrier for D coming out of the sample.

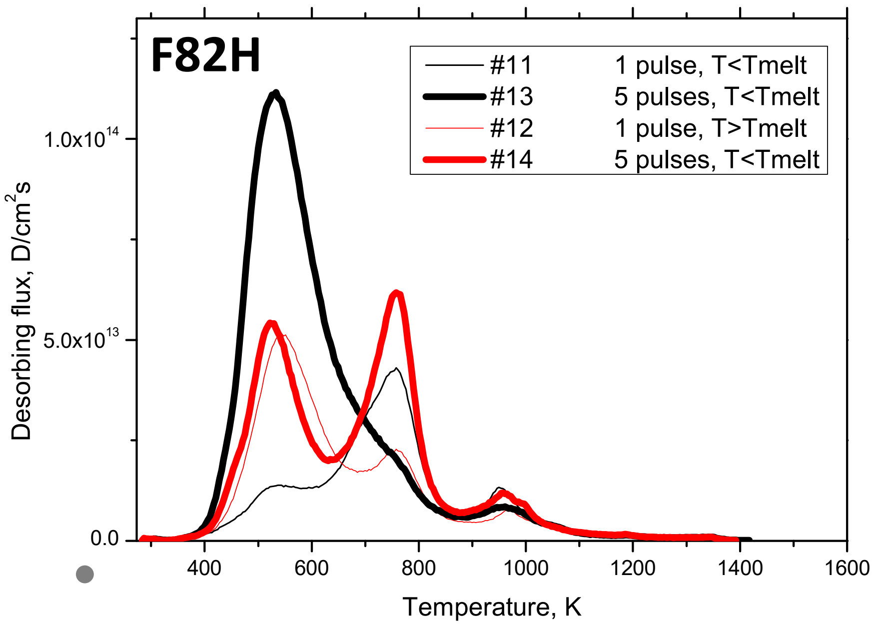
Otherwise D comes out from “week” site (dashed line) and data will be too dependent on time of delay between irradiation and TDS, which we do in different towns, Troitsk and Moscow.

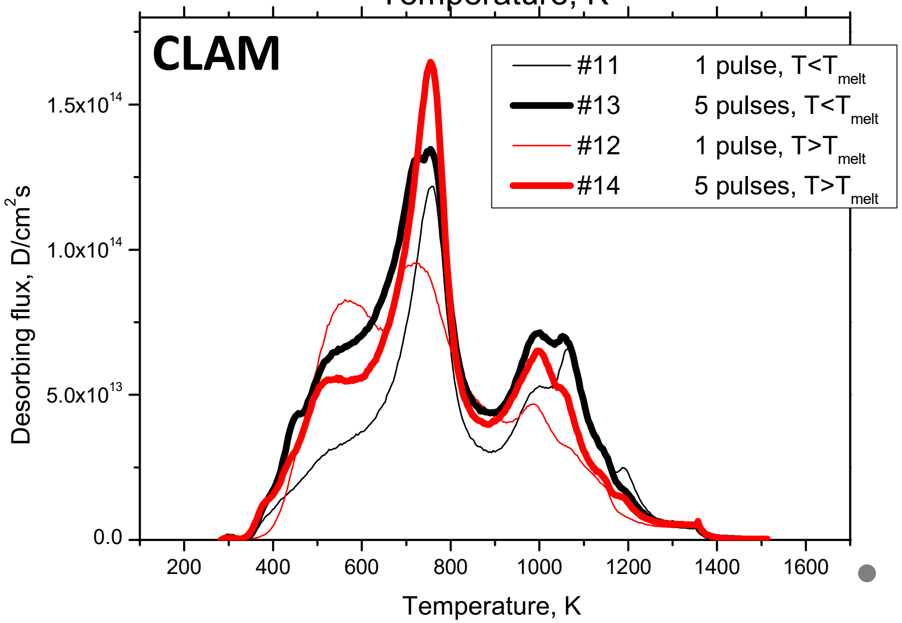
D retention after pulsed D plasma loads

TDS, 2 K/s

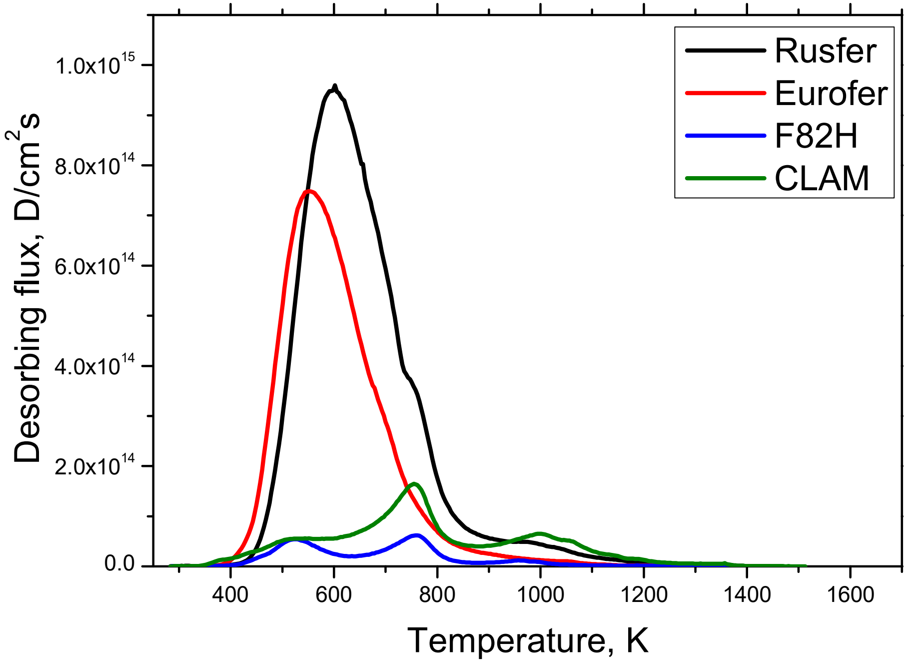




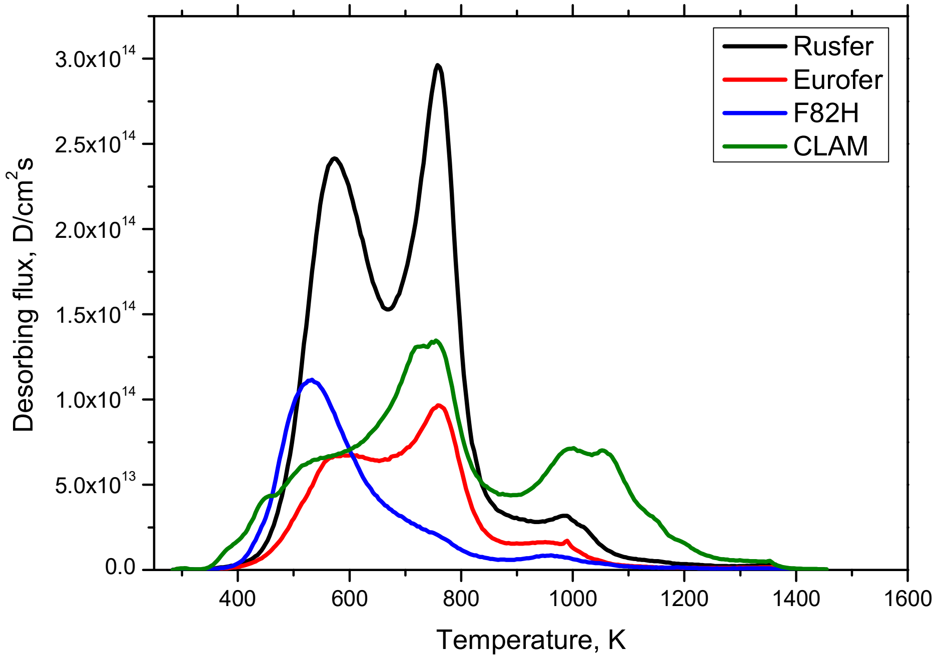




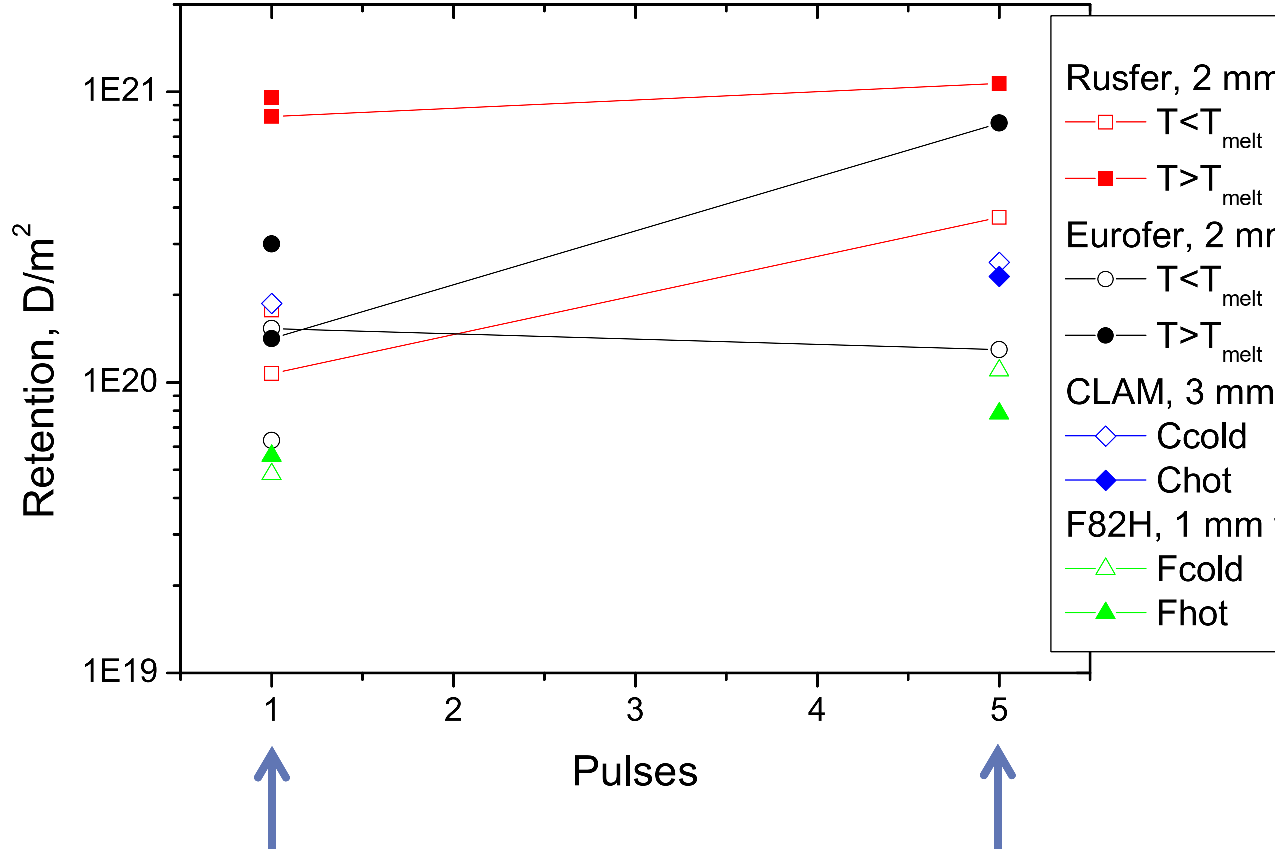
5 pulses above melting



5 pulses below melting



Total retention level



10^{23}-10^{24} D/m²

5·10^{23}-5·10^{24} D/cm²

NOTE different thickness

0.1%÷1 of irradiating ions are retained

[Y. Martynova et al, Nucl. Mater.&Energy 12-2017-648]

40 eV D, 1e26 D/m², 450 K →RAFMS

~ 1e20 D/m² is retained

ELMs-like plasma loads - main results:

1. At D plasma loading 0.3 MJ/m², 1 ms, 5 pulses RAFMS surfaces are flat, covered with crystallines of a size ≤ 1 μm. Cracks can be found at surfaces of all materials. The concentration of cracks is highest at Rusfer surface.
2. After D plasma loading 0.6 MJ/m², 1 ms, 1 and 5 pulses surfaces of Eurofer and F82H are flat wile surfaces of Rusfer and CLAM are waved. Surfaces of all RAFMS consist of crystallines of a size ≤ 1 μm. The most pronounced cracking was found at CLAM sample.
3. Retention in Rusfer>retention in Eurofer & CLAM> Retention in F82H. BUT **note different thicknesses**, Rusfer & Eurofer – 2 mm, CLAM – 3 mm, F82H – 1mm
4. The maximum level of retention 1e21 D/m² was reached in Rusfer at plasma loading above melting (0.6 MJ/m², 1 ms)
5. At pulsed D plasma irradiation retention in Rusfer and Eurofer is significantly higher if surface was melted.

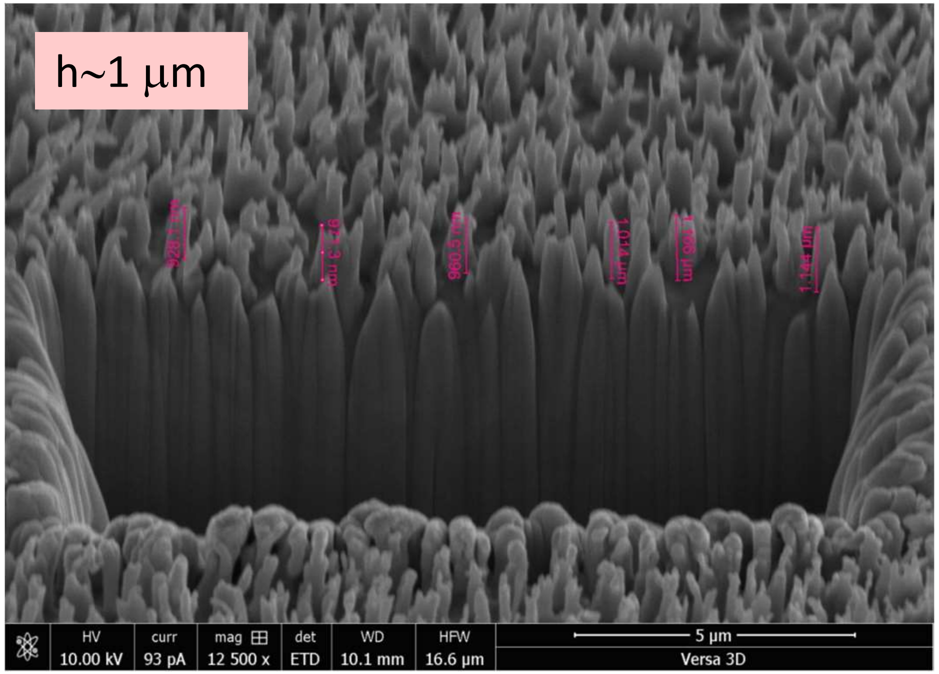
ELMs-like plasma loads - plans:

1. Irradiation of all RAFMS with 10, 20, 50 pulses below and above melting. Microscopy & retention (2019).
2. Investigation of influence of ELMs-like loads on further D retention at different conditions of exposure in gas and plasma irradiation (2019-2020).

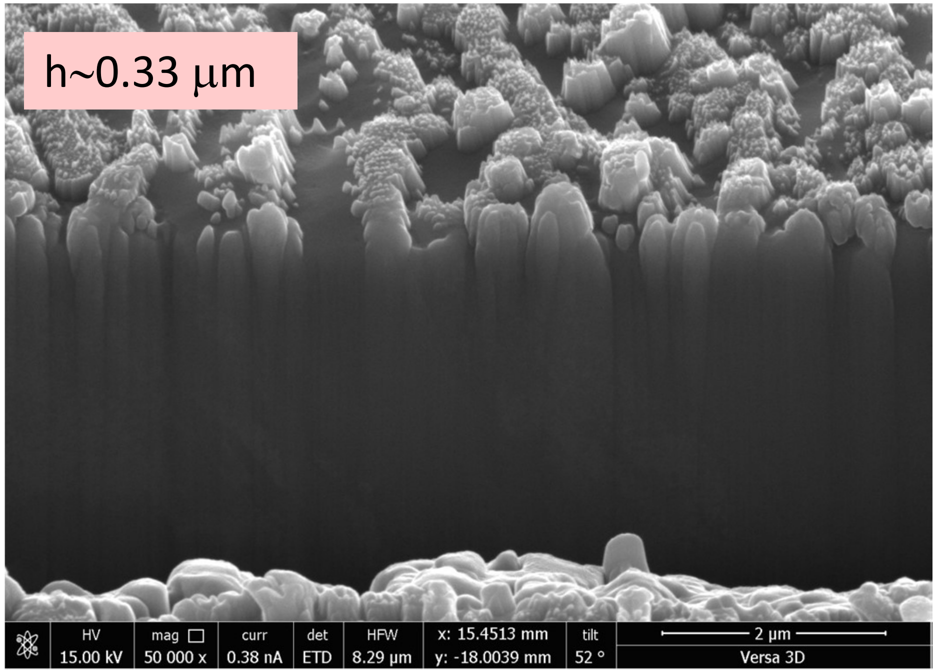
Thank you for your attention!

$3{\cdot}10^{25}$ ion/m^{-2}, FIB cut

Eurofer



Rusfer



Note different scales