Erosion and fuel retention of different RAFM steel grades

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Outline

◆ Sample preparation & experimental details
◆ Erosion of CLF-1 steels exposed to deuterium plasma
◆ Comparison of sputtering yield of different RAFM steels
◆ Comparison of sputtering yield of RAFM steels with different roughness
◆ Fuel retention in various steel grades
◆ Work plan
Linear experimental plasma system--LEPS

Parameters of LEPS plasma:
- Magnet field: 0.12-0.15 T
- Plasma beam diameter: 50 mm
- Ion flux: $2-8 \times 10^{21} \text{ m}^{-2}\text{s}^{-1}$
- Ion composition: mainly $D_3^+$
- Electron density: $10^{16}-10^{18} \text{ m}^{-3}$
- Floating potential: -15 V
- Working pressure: 0.5-1.0 Pa

LEPS deuterium plasma profile measured by LP
### RAFM steel materials

#### Table 1. Composition of various RAFM steel grades (in wt.%) (Fe balance)

<table>
<thead>
<tr>
<th>RAFM</th>
<th>C</th>
<th>Cr</th>
<th>W</th>
<th>Mn</th>
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</thead>
<tbody>
<tr>
<td>EUROFER97</td>
<td>0.09-0.12</td>
<td>8.5-9.5</td>
<td>1.0-1.2</td>
<td>0.2-0.6</td>
<td>0.15-0.25</td>
<td>0.10-0.14</td>
<td>0.015-0.045</td>
<td>0.004-0.005</td>
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</tr>
<tr>
<td>CLF-1</td>
<td>0.11</td>
<td>8.5</td>
<td>1.5</td>
<td>0.5</td>
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<td>0.1</td>
<td>0.02</td>
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<td>RUSFER</td>
<td>0.15</td>
<td>11.17</td>
<td>1.13-1.3</td>
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**CLF-1 steel from ASIPP (Aug, 2015)**

- CLF-1
- CLF-1-L
- 10×12×1mm
- transversal

**EUROFER from IPP (2015)**

- EUROFER
- 15×12×1mm

**CLAM, 2 Kg from FDS (May, 2017)**

- CLAM
- 15×12×1mm

**RUSFER 10 pic (Jan, 2017)**

- RUSFER
- 15×12×1mm
Experimental details - Analyses

**Structure**

SEM: (JSM-5601)
HRTEM: (FEI Tecnai)
Voltage: 200 kV
Resolution: 0.24 nm

**Composition**

GDOES: (Profiler-2)
Ar power: 30 W
Sputtering rate: 2 nm/s

RBS: (Peking University)
Probing beam: $^4\text{He}^+$
Energy: 3 MeV

XPS: (Phi-5702)
Source: Al kα
Pass energy: 29.4 eV

**Deuterium retention**

TDS:
Base pressure: $5 \times 10^{-6}$ Pa
Heating range: RT-1200 K
Measuring mass: 4(D$_2$) & 3(HD)
Heating ramp: 15 K/min

NRA: (IPP Garching)
Probing beam: $^3\text{He}^+$
Energy: 0.7-4.5 MeV
Simulation: NRADC
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Surface roughness increases with increasing incident ion energy and fluence, no roughness saturation was observed at a fluence up to $10^{25}$ D/m$^2$. 

![Graph showing surface roughness of CLF-1 steels after deuterium exposure.](image-url)

![Graph showing surface line profile of CLF-1 exposed to 180 eV/D deuterium plasma.](image-url)
Morphology changes - Fluence dependence

Deuterium plasma exposure: energy: CLF-1, 150 eV/D, temperature: 450 K
Morphology changes - Energy dependence

Deuterium plasma exposure: CLF-1, fluence: $7.2 \times 10^{24} \text{ D/m}^2$, temperature: 440-450 K
Sputtering yield

Fluence dependence of sputtering yield of CLF-1 steel

Energy dependence of sputtering yield of CLF-1 and EUROFER steels

Sputter yield was determined by mass loss.

Clear decreases of yield of CLF-1 steels with increasing of incident fluence, no clear saturation of yield at fluence up to $10^{25}$ D/m$^2$. Sputtering yield of CLF-1 and EUROPER steels is lower than pure Fe.

 Preferential sputtering changes the composition, leads to the enrichment of tungsten and reduces the total sputter yield.
Surface tungsten enrichment

W enrichment was observed by RBS measurements but not good agreement with data from F82H steel exposed to comparable incident fluence, could be:

Measurement difference or various initial W concentration in bulk (1.5 and 2.0 wt% W in CLF-1 and F82H bulk, respectively)

Surface tungsten enrichment
In the grey region, the Fe and Cr EDX signals increase from background level to their maximum level and this transition region is about 20 nm thick.

The Pt EDX line partially overlaps with the W line such that a small fraction of W in a large background of Pt (stemming from the protection layer) cannot be safely distinguished in the EDX line scans.
Surface tungsten enrichment

CLF-1  Deuterium plasma exposure: $150\text{eV, }7.2 \times 10^{24} \text{ D/m}^2$
A pure platinum layer were deposited on CLF-1 as a protecting layer

◆ There is no obvious uneven sputtering region between the crystalline bulk and the Pt protecting layer.
◆ Few nm damage zone exists and mixed with pure Pt layer
The Pt EDX line partially overlaps with the W line such that a small fraction of W in a large background of Pt cannot be safely distinguished in the EDX line scans.
Surface tungsten enrichment

CLF-1 sample, Deuterium plasma exposure: 150eV, $7.2 \times 10^{24}$ D/m$^2$

An amorphous carbon layer was deposited on sample surface before TEM analyses.

About 5 to 10 nm region damaged by plasma exposure was observed by TEM, which partially overlaps with the amorphous carbon at the mixed interface.
Surface tungsten enrichment

- Amorphous carbon layer was deposited on CLF-1 surface to avoid the influence of Pt on composition depth profile measurement, however, W enrichment was found in whole carbon layer region and much thicker than as expected.
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# Sputtering yields of various RAFM steels

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Sputtering yields of various RAFM steels

Initial W concentration of various RAFM steels: $W_{CLF-1} = W_{CLF-1-L} > W_{EUROFER}$

Sputtering yield of various RAFM steels: $Y_{CLF-1} > Y_{CLF-1-L} > Y_{EUROFER}$

Other factor influence of sputtering yield? (Grain orientation, roughness)
Morphology changes

Deuterium plasma exposure (180 eV/D, 450K, $7.2 \times 10^{24}$ D/m$^2$)
Sputtering yields of various RAFM steels

Initial W concentration of various RAFM steels: $W_{CLF-1}$ = $W_{CLF-1-L}$ > $W_{EUROFER}$

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Sputtering yields of various RAFM steels

Deuterium plasma exposure at: 180eV, 7E24 D/m2

Ra=20 nm  Ra=50 nm  Ra=100 nm  Ra=400 nm
Sputtering yields of various RAFM steels

Deuterium plasma exposure at: 70eV, 7E24 D/m²

Ra=20 nm  Ra=50 nm  Ra=100 nm  Ra=400 nm
Sputtering yields of various RAFM steels

Deuterium plasma exposure at: 180eV, 7E24 D/m2

Ra=20 nm  Ra=50 nm  Ra=100 nm  Ra=400 nm
Sputtering yields of various RAFM steels

RAFM steel samples with different initial rough show comparable sputtering yield.

More accurate measurement based on spectroscopy is needed.
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Deuterium retention in CLF-1 decreases with increasing fluence except the samples exposed to 30 eV/D deuterium plasma, in which D retention keeps as a constant (about $3 \times 10^{20} \text{ D/m}^2$)
Fuel retention of various RAFM steels

**Deuterium depth profiles of CLF-1 and EUROFER97 measured by TDS**

Deuterium retention at 30 eV/D is one order of magnitude higher than EUROFER, NRA show a high D retention region at near surface region (surface to 2μm)

**Deuterium release from CLF-1, CLAM, EUROFER97 and RUSFER measured by TDS**

Deuterium plasma exposure:
180 eV/D 440-450 K
Fuel retention of various RAFM steels

Deuterium release of CLF-1 and EUROFER97 measured by TDS

Deuterium depth profiles of CLF-1 and EUROFER97 measured by TDS

CLF-1, CLF-1-L and EUROFER samples were exposed in one batch (30 eV/D 320 K)
CLF-1 & CLF-1-L: deuterium release at 450 K
EUROFER: deuterium release at 420 K

Deuterium retention at 30 eV/D is one order of magnitude higher than EUROFER, NRA show a high D retention region at near surface region (surface to 2μm)
Fuel retention of various RAFM steels

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

- Deuterium retention after LEPS plasma exposure measured by TDS
  - D retention in RAFM steels is lower than in W
  - D retention in RAFM steel decreases with increasing incident fluence
  - D retention in CLF-1 steel at 30 eV/D is higher than other RAFM steels

W. Jacob, IAEA steel CRP meeting
Work plan

• Erosion of RAFM steel samples extends to higher temperature, to study the surface W enrichment with RBS and TEM
• Compare sputtering yield more precisely using other methods
• Study the fuel retention in steel samples after 3.5 MeV iron ions damaging
• Extend the fuel retention and composition depth profile up to several tens nm region using GDOES
Thank you for your attention!