

# Phase formation of Erbia coatings on EUROFER 97, phase stability and deuterium permeability

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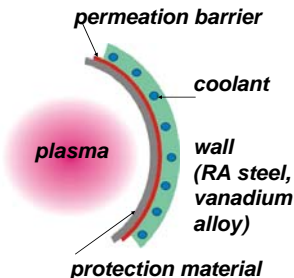
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## Motivation

Tritium permeation from plasma into and through structural materials of future fusion devices has to be avoided:

- radioactive tritium ends up in the coolant and may leave the reactor vessel with contamination of environment
- hydrogen and its isotope leads to embrittlement of material, especially of steel (structural material)
- loss of fuel due to hydrogen permeation

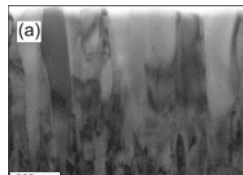


Application of a thin ceramic layer as permeation barrier between plasma facing material and structural material

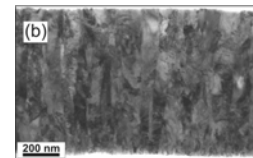
- high permeation reduction ( $10^2$ - $10^3$ ) with thin ( $\approx 1 \mu\text{m}$ ) oxide coatings ( $\alpha$ - $\text{Al}_2\text{O}_3$ ,  $\text{Er}_2\text{O}_3$ ) [1-3]
- $\text{Er}_2\text{O}_3$  shows **phase transformation** under ion irradiation [4]: cubic C-phase  $\Rightarrow$  monoclinic B-phase
- Influence on permeation barrier properties ?

[1] Levchuk et al., Phys. Scr. T108 (2004) 119.  
 [2] Levchuk et al., J. Nucl. Mater. 367 (2007) 1033.  
 [3] Chikada et al., Fusion Eng. Des. 84 (2009) 590.  
 [4] Tang et al., J. Appl. Phys. 99 (2006) 063514.

## Microstructure (STEM)

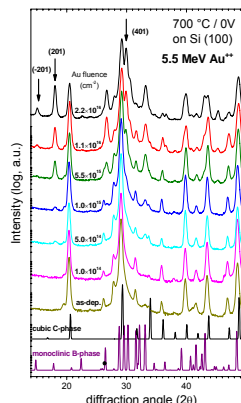


C-phase  $\text{Er}_2\text{O}_3$  on Eurofer (600 °C / 0 V)



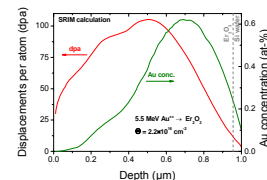
B-phase  $\text{Er}_2\text{O}_3$  on Eurofer (400 °C / 250 V)

## Irradiation Effects



**Irradiation of  $\text{Er}_2\text{O}_3$  films on Si wafer with 5.5 MeV  $\text{Au}^{++}$  at RT**

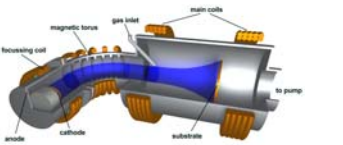
- starting with almost pure C-phase (700 °C / 0 V), transformation into B-phase starts at a fluence of  $5 \times 10^{14} \text{ Au}^{++}/\text{cm}^2$
- no influence of ion irradiation observed by XRD when starting with the B-phase
- $\Rightarrow$  **B-phase is stable against further phase transformation!**



## Experimental

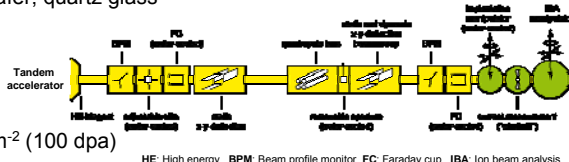
### $\text{Er}_2\text{O}_3$ thin films by filtered cathodic arc

- pure Er cathode,  $5 \times 10^{-2}$  Pa oxygen
- $\approx 1 \mu\text{m}$  thickness (15 min deposition)
- RT – 700 °C, 0 – 450 V bias
- substrates: Eurofer, Si wafer, quartz glass



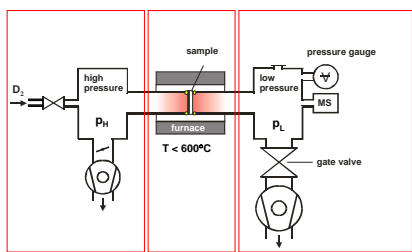
### 5.5 MeV $\text{Au}^{++}$ irradiation

- RT (with water cooling)
- flux:  $\approx 1$ - $2 \times 10^{12} \text{ cm}^{-2}\text{s}^{-1}$
- fluence: max.  $2.2 \times 10^{16} \text{ cm}^{-2}$  (100 dpa)



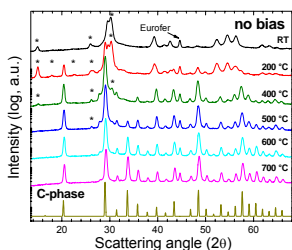
### Permeation apparatus

- QMS sensitivity range:  $10^{-7}$  to  $10^{-2} \text{ mol/m}^2\text{s}$  (deuterium flux)
- liquid nitrogen trap for input gas
- temperatures range: up to 600 °C
- hydrogen driving pressures range:  $10^{-3}$  to  $10^3 \text{ mbar}$

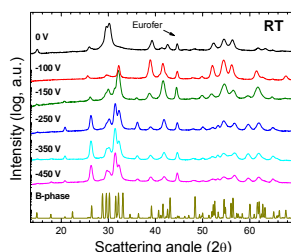


## XRD

XRD diffractograms of  $\text{Er}_2\text{O}_3$  thin films on Eurofer deposited at 0 V bias and varying temperature. Below 600 °C mono-clinic B-phase diffraction peaks occur (\*)



XRD diffractograms of  $\text{Er}_2\text{O}_3$  thin films on Eurofer deposited at RT and varying sample bias

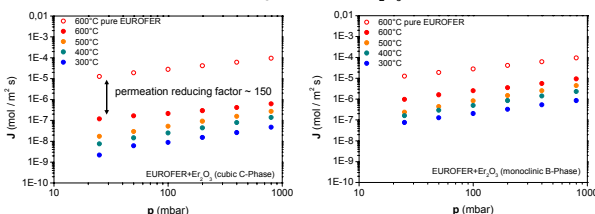


### XRD at 3° incidence angle

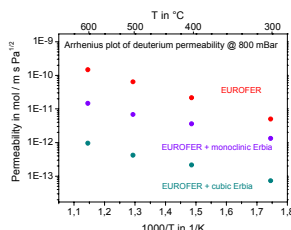
- pure cubic C-phase for  $T \geq 600 \text{ °C}$  / 0 V
- pure monoclinic B-phase for  $-150$  –  $-450 \text{ V}$  bias and  $T \leq 400 \text{ °C}$
- strong B-phase texture
- Missing peaks can be detected by tilting the sample (0-2θ scan)

## Permeation Results

Gas driven deuterium permeation through Eurofer and  $\text{Er}_2\text{O}_3$  thin films on Eurofer substrate



Deuterium permeation flux  $J$  as a function of driving pressure



### Gas driven deuterium permeation

- Eurofer  $\rightarrow$  diffusion limited regime
- thin layer of Erbia leads to surface limited regime
- higher permeation reduction for the cubic phase
- permeation reducing factor  $\sim 150$

## Acknowledgement

Joachim Dörner and Michael Fußeder are acknowledged for performing the Au irradiation experiments.