

The key enabling role of ductile amorphous oxide coatings for GIV and fusion nuclear power plants.

B. Paladino^{1,2}, M. Cabrioli^{1,2}, A. Stinchelli^{1,2}, S. Bassini³, M. Utili³, M. Angiolini³, M. Tarantino³, P. Agostini³,
F. Di Fonzo¹.

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³ENEA FSN Department, C.R. Brasimone, Camugnano (Bo), Italy

Why next generation nuclear systems need coatings and the key enabling role of ductile amorphous oxide coatings for GIV and fusion nuclear power plants.

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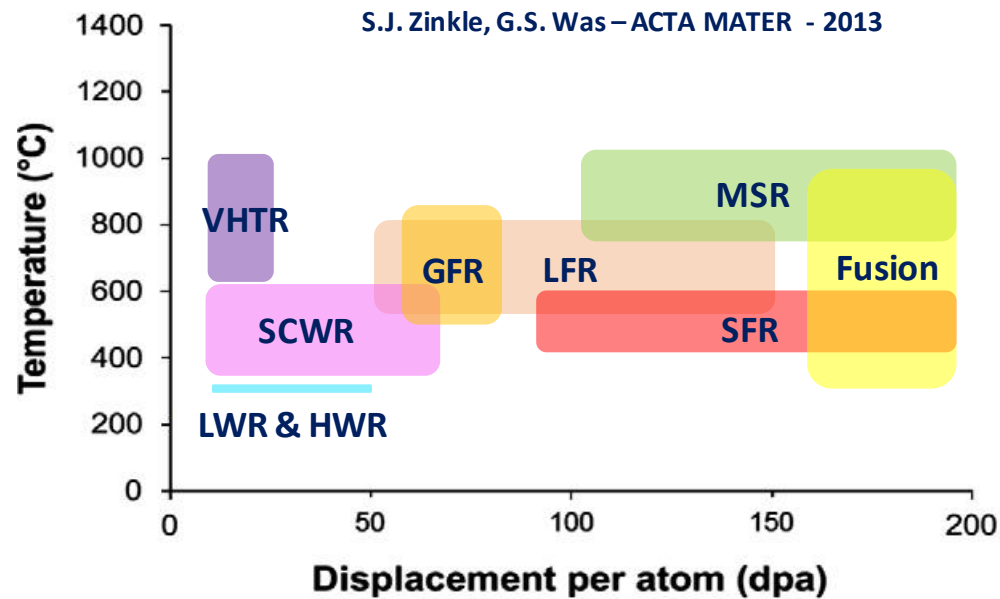
¹Center for Nano Science and Technology@PoliMi, Istituto Italiano di Tecnologia, Milano, Italy

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³ENEA FSN Department, C.R. Brasimone, Camugnano (Bo), Italy

➤ **Next generation nuclear systems benefits:**

- **increasing efficiency** (higher cycle temperature)
- **reducing waste generation** (burning effect)
- **enhancing safety** (passive systems, new coolants, etc.)
- **promoting non-proliferation** (closed fuel cycle)



FISSION REACTORS:

LWR: Light Water Reactor
HWR: Heavy Water Reactor

~~GII and GIII
 present reactors~~

SCWR: Supercritical Water Reactor
VHTR: Very High T Reactor
GFR: Gas-cooled Fast Reactor
LFR: Lead-cooled Fast Reactor
SFR: Sodium-cooled Fast Reactor
MSR: Molten Salts Reactor

Gen IV (GIV)
 reactors

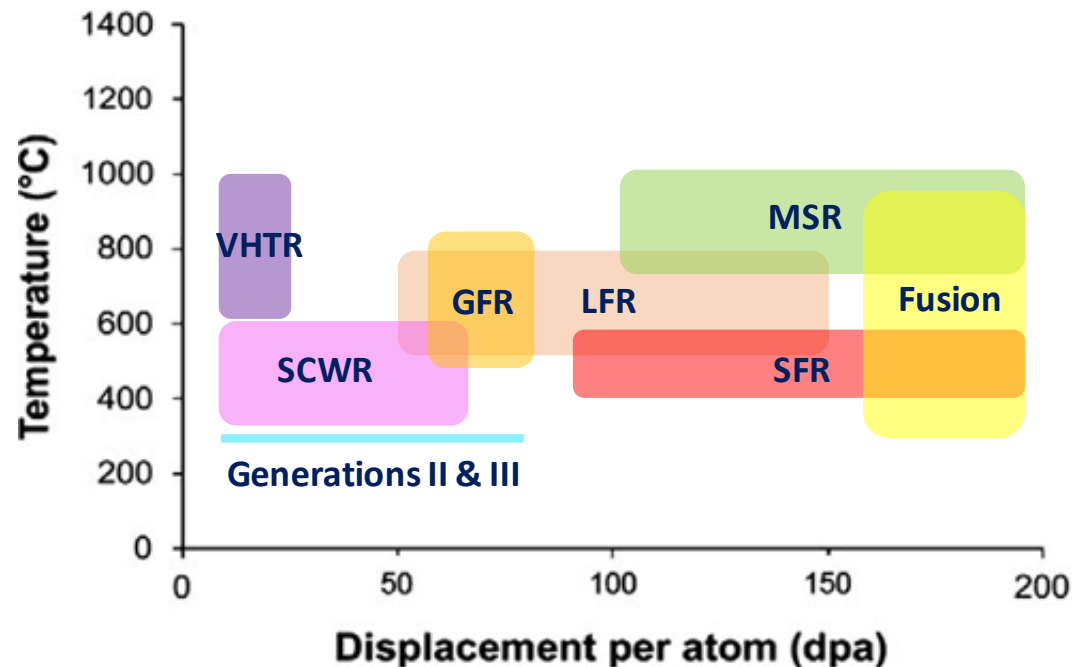
FUSION REACTORS

Future generation nuclear systems aim at:

- Increase efficiency
- Reduce waste generation
- Enhance safety
- Promote non-proliferation

Ultimate goal for LFRs:

- 800 °C
- 150 dpa



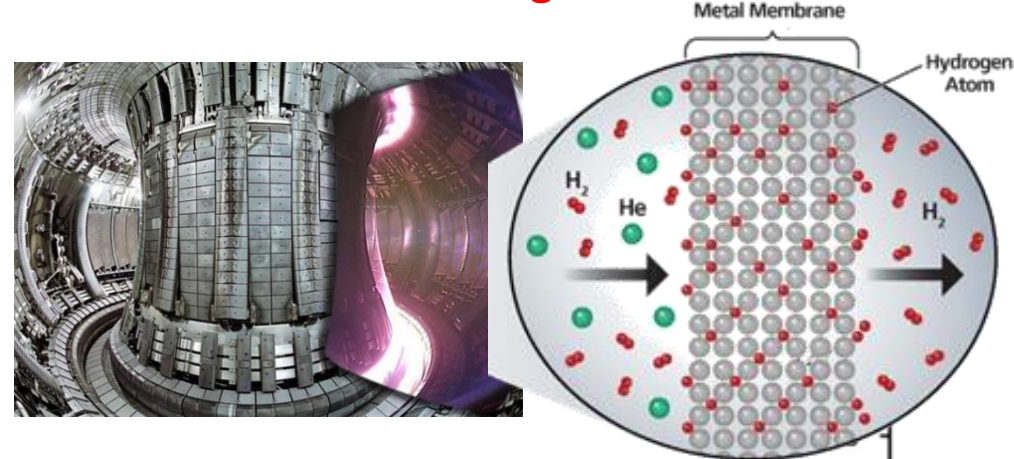
Advantages:

- safety
- transmutation of minor actinides / fuel breeding

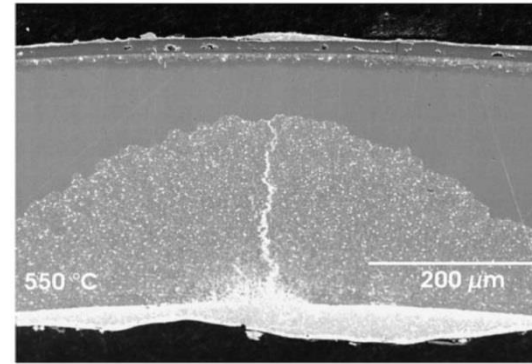
Major issues:

- corrosion
- radiation damage
- Tritium permeation

Tritium management

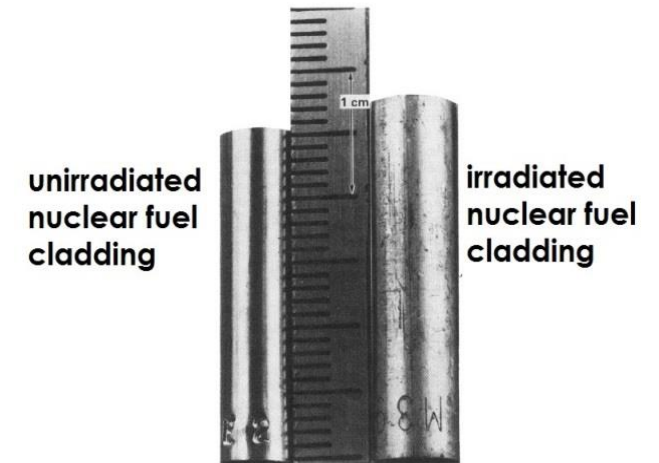


Corrosion



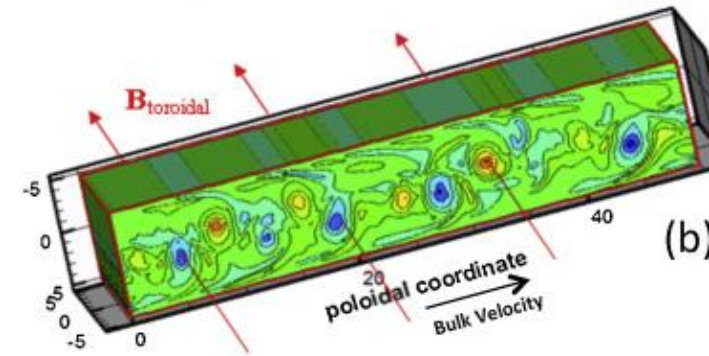
G. Mueller et al. – J Nucl Mater – 2004

Radiation damage

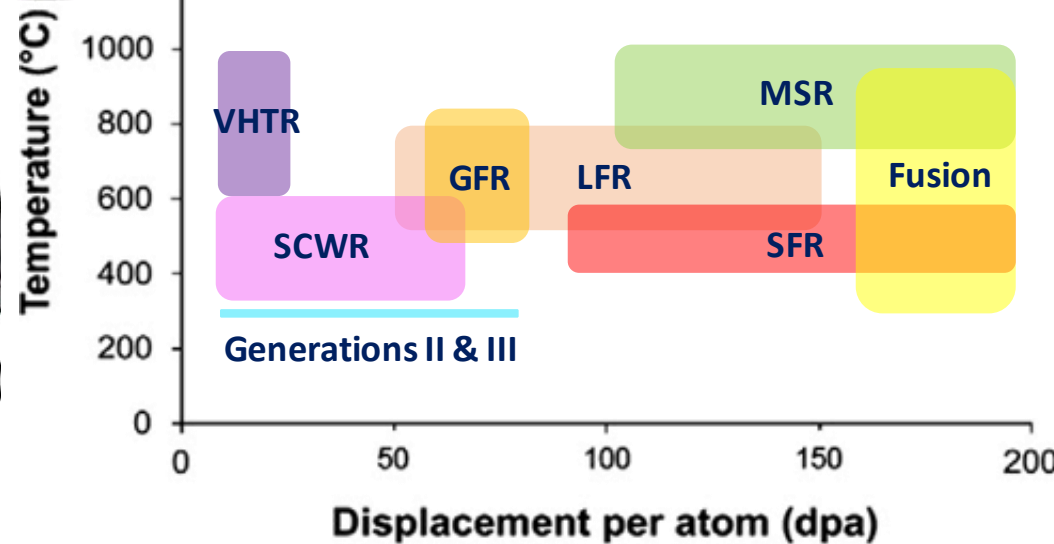


J.L. Straalsund – Westinghouse Hanford

Magnetohydrodynamics, MHD



M. Abdou et al. Fus Eng.&Des 2015



S.J. Zinkle and G.S. Was – Acta Materialia - 2013

**MAJOR BOTTLENECKS
 FOR ALL SYSTEMS**

↓
**NEED FOR
 COATINGS**

Tritium permeation (radiological hazard, loss of fuel)

Tritium production rates of different fission reactors (*) :

Reactor type	Total tritium source term (TBq.GWe ⁻¹ .year ⁻¹)	Tritium transferred into coolant (TBq.GWe ⁻¹ .year ⁻¹)
PWR	567	37
BWR	520	much lower than PWR (no boric acid)
GCR (MAGNOX)	705	very small due to Mg cladding
PHWR (CANDU)	54170	53650 (up to 90000)
SFR	3034	1000

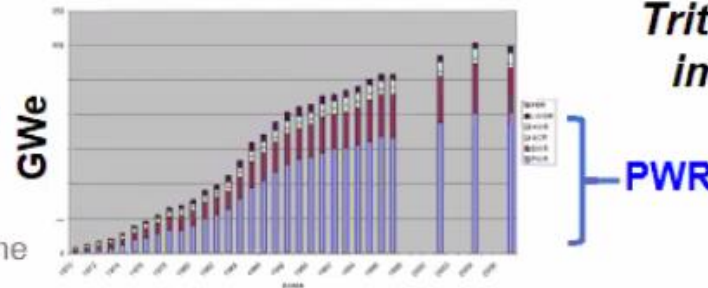
- in PWR tritium produced in fuel pins keeps occluded (affinity with Zr, limited permeation in ZrO₂)
- in SFR, stainless steel claddings (high T) is totally permeable to tritium

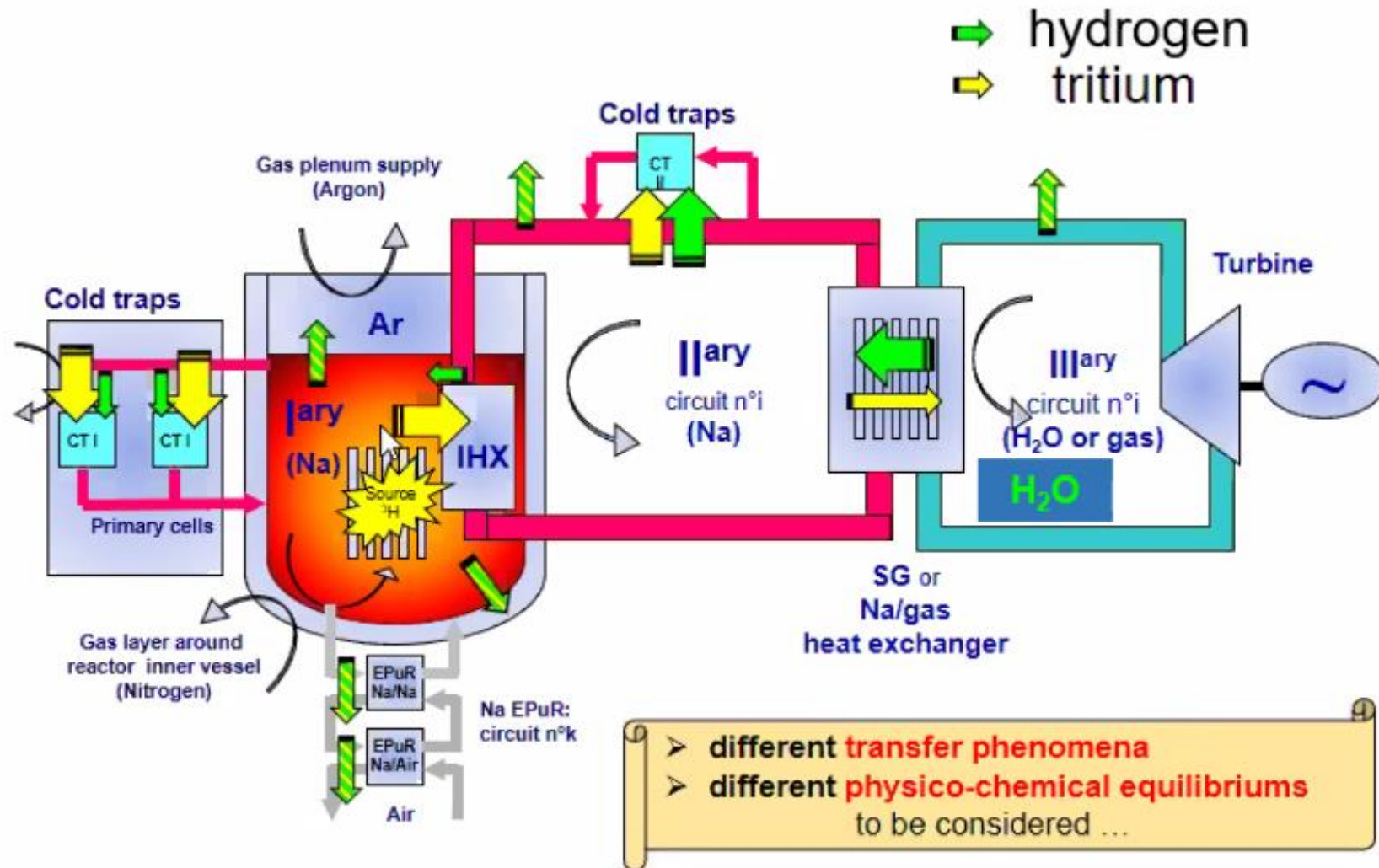


Tritium management is an important issue in SFR

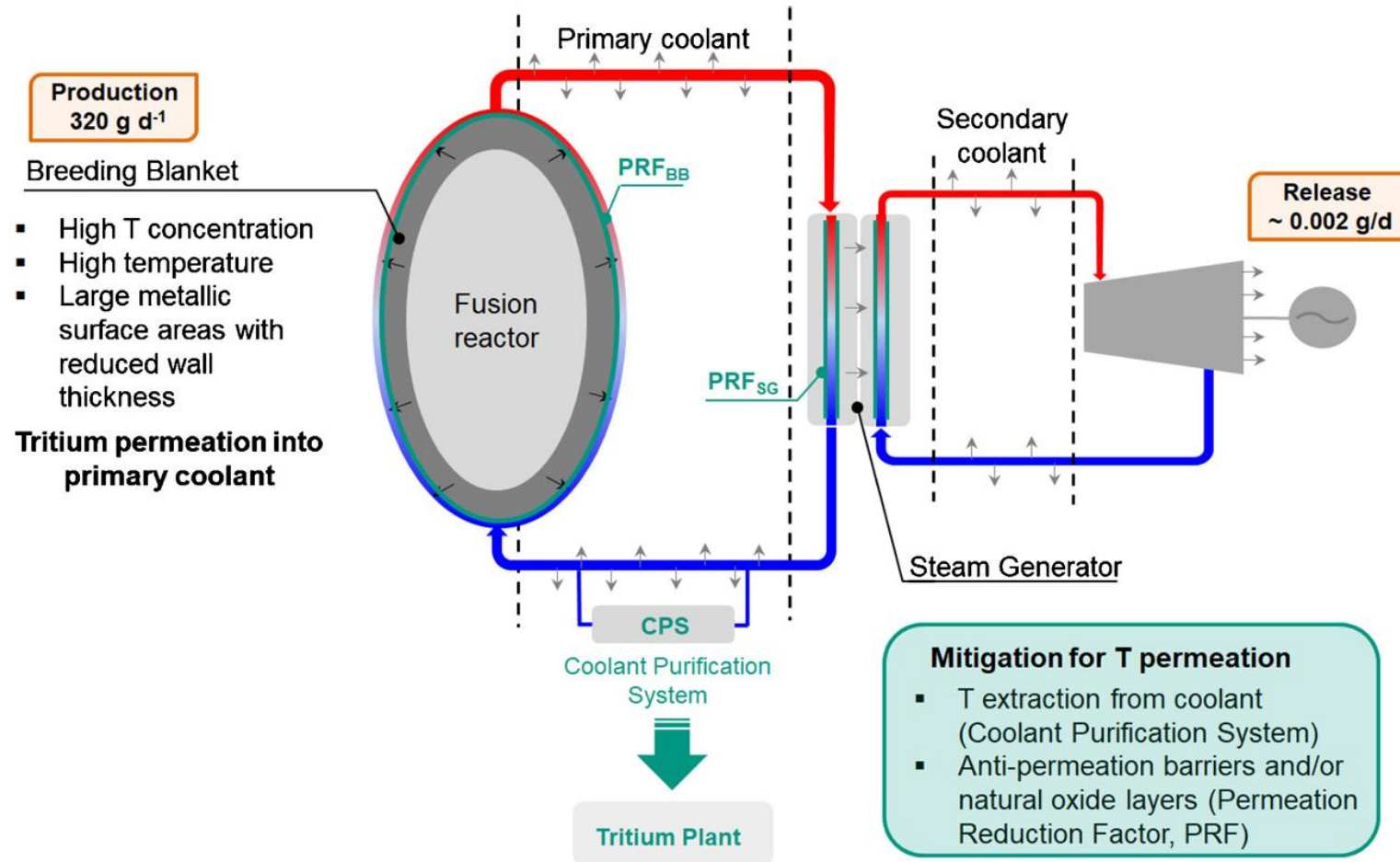
(*) average data from IRSN report and ASN report (« Livre blanc du tritium »)

Since 2000, **PWR cover ≈ 65%** of worldwide **electrical power production**





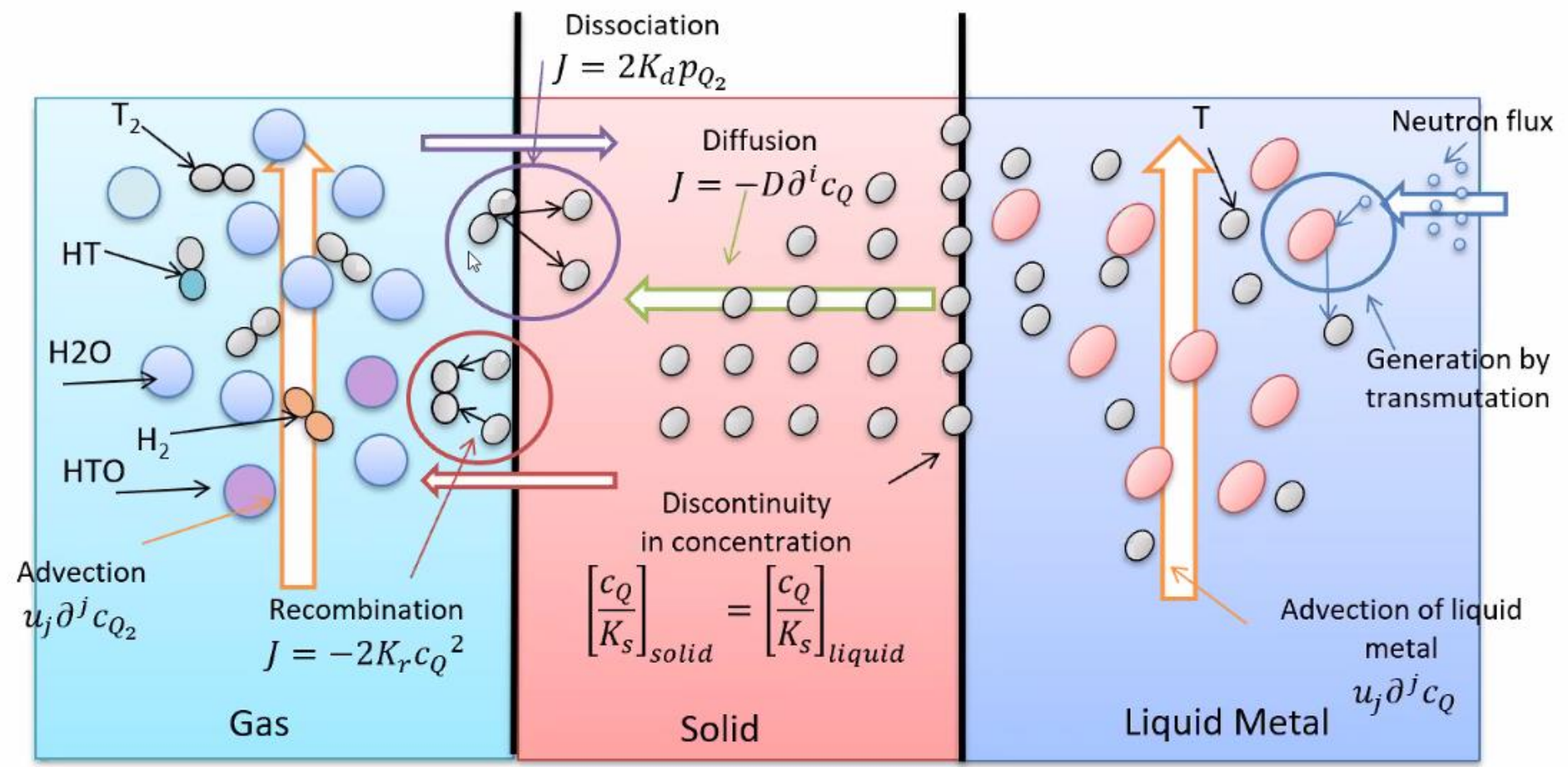
Tritium in fusion: mitigation strategies are essential



- Mandatory limit of 0,002 g/d (less than 1 g/y) of tritium release
- T release without any mitigation strategies: HCPB \approx 1 g/d & WCLL \approx 37 g/d (respectively 0,35 % and 11 % of the T produced in the blanket)
- **No fusion power plant can be built without suppressing the permeation of tritium**

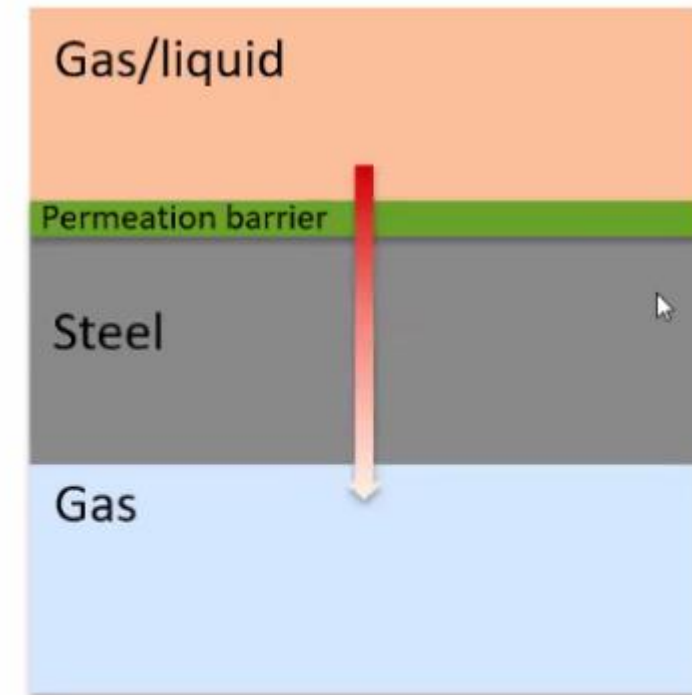
Fig. 1. Generic view of the tritium migration path in DEMO coolant.

- The basic processes to be studied in a circuit consists of three domains: Liquid Metal, Solid, Gas.



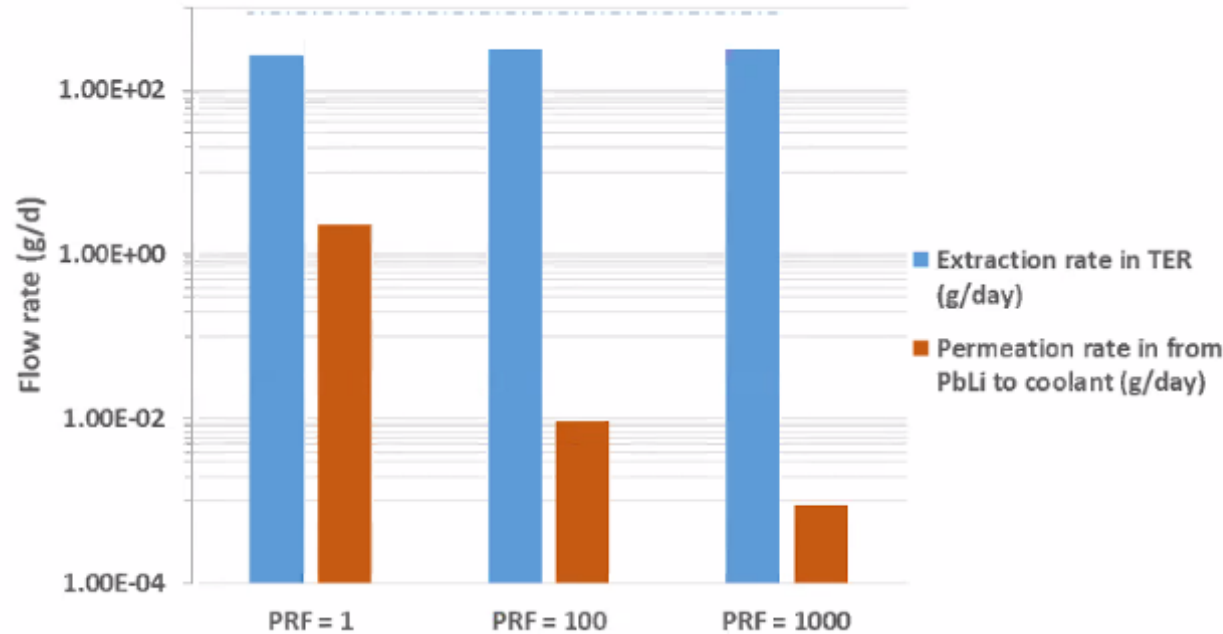
Apply a barrier on all the surfaces where high tritium permeation fluxes are expected

- The main parameter to take in account is the Permeation Reduction Factor (PRF), which is the ability of the coating to suppress permeation
- The PRF depends on the operative temperature, on the material, and on the number of defects present in the coating
- A PRF value above 1000 (at 450 °C) is necessary to satisfy the limit in tritium release
- Protective barriers also useful for corrosion protection of the structural steels or for suppression of MHD effects



$$PRF = \frac{J_{uncoated}}{J_{coated}}$$

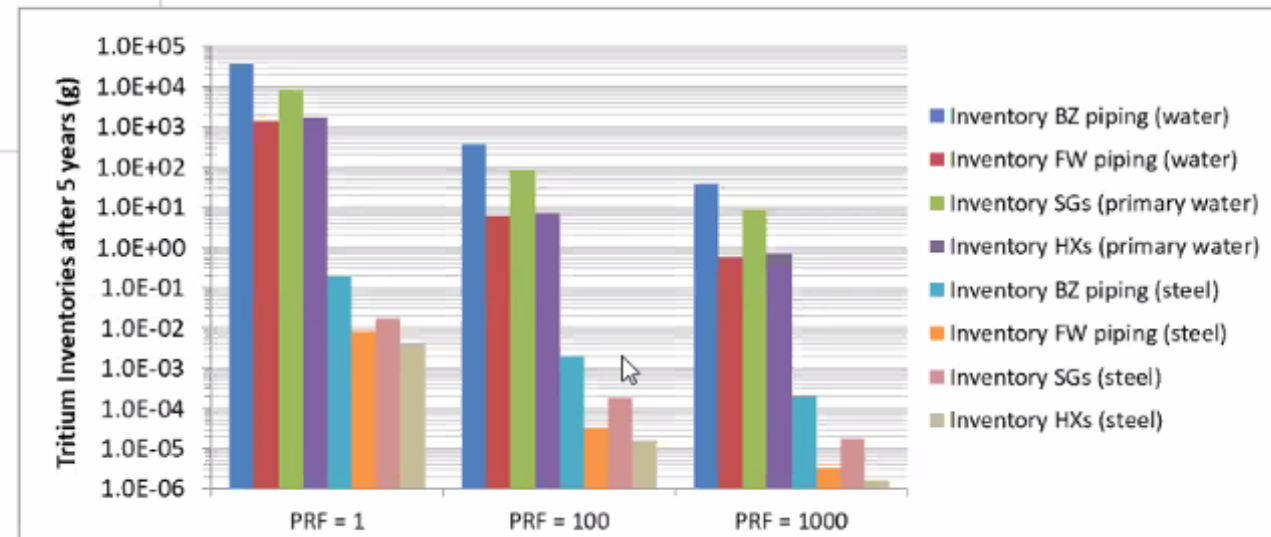
PB effect in WCLL (C. Moreno, 2nd Tritium School)



It can be noticed how the reduction in the permeation rates increases slightly the concentration in PbLi but reduces very significantly the concentration in the water circuits.

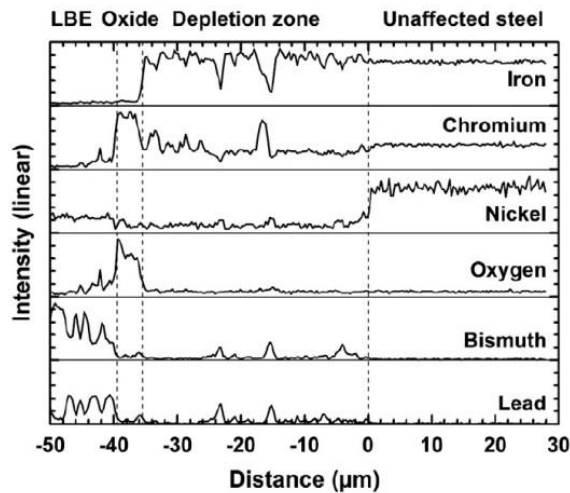
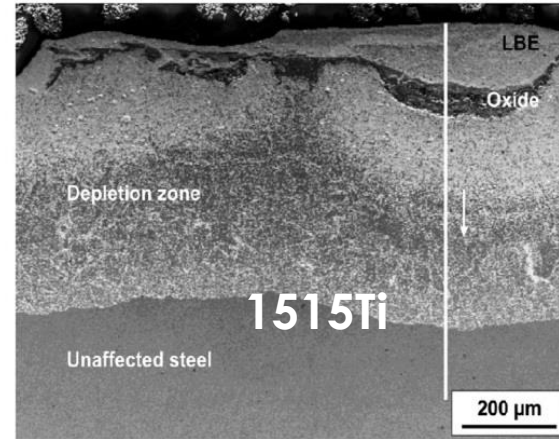
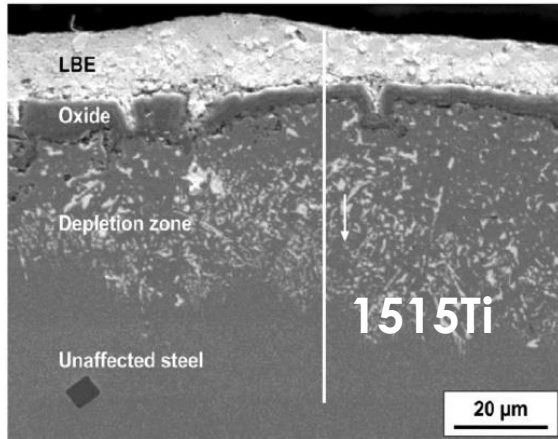
Values after 5 years.

Inventories in coolant decrease as PRF is increased.

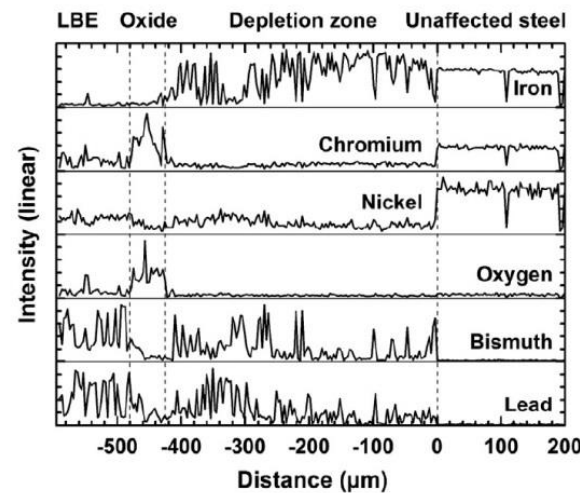


HLM Corrosion Mitigation Strategies

Ni leaching in austenitics (23000 h @ 550°C, 10⁻⁶ wt.% O)

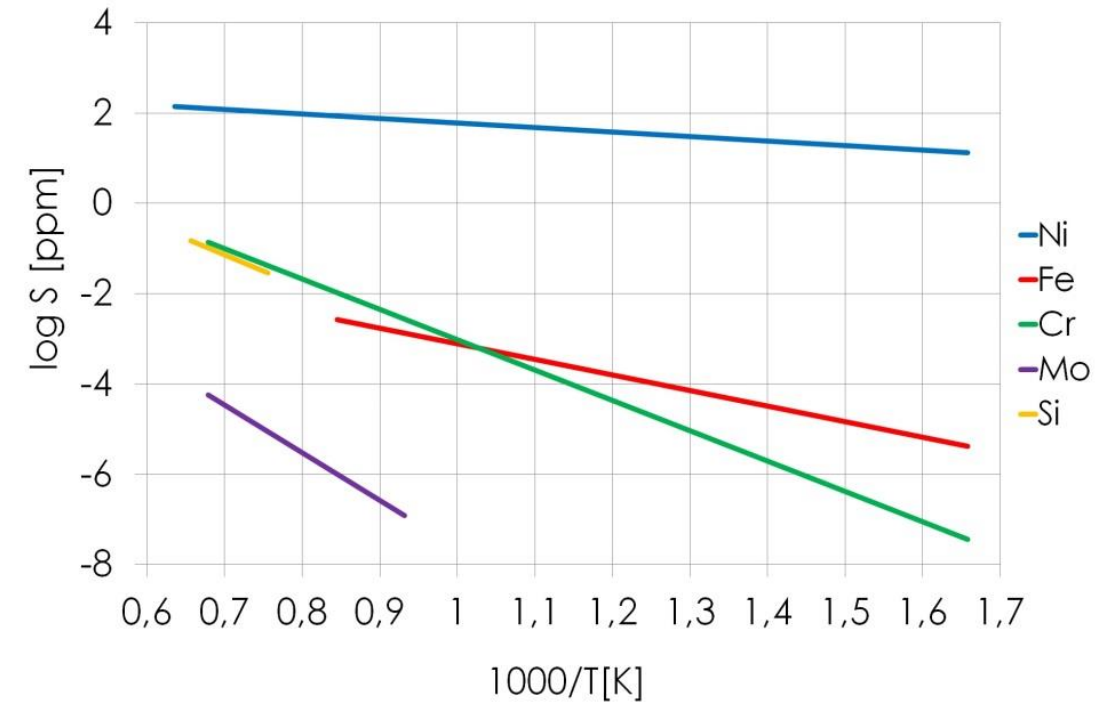


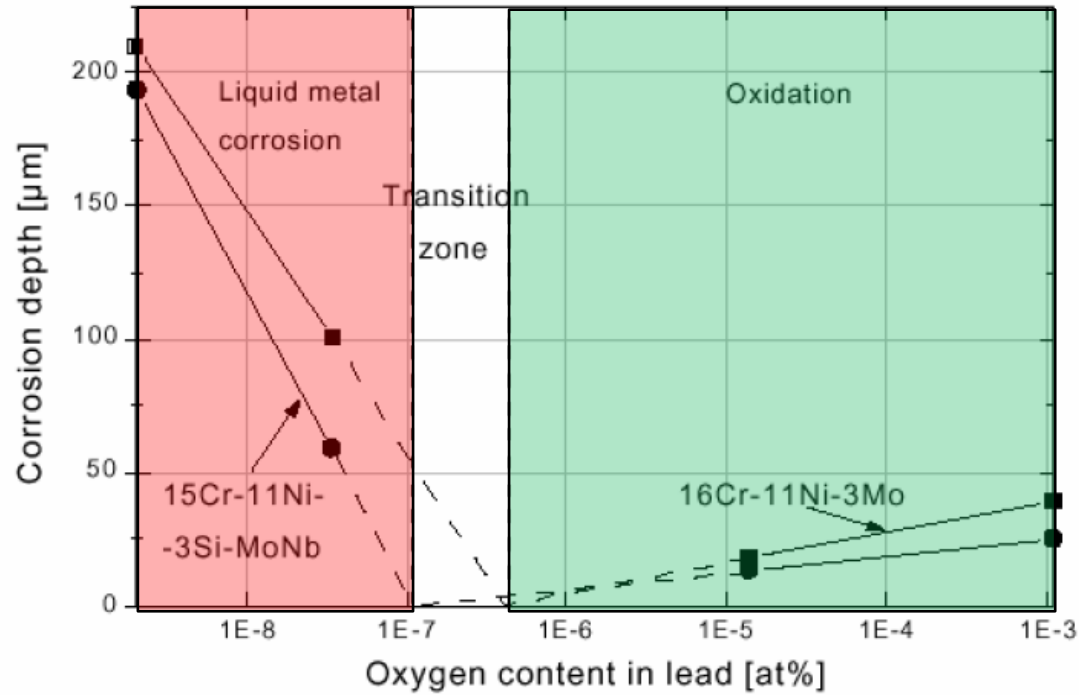
(a)



(b)

Solubility of Ni in lead is very high



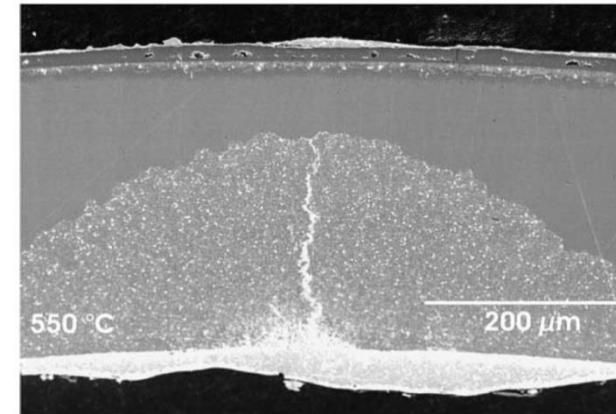


Austenitic steels exposed to HLM for 3000 hours at 550°C

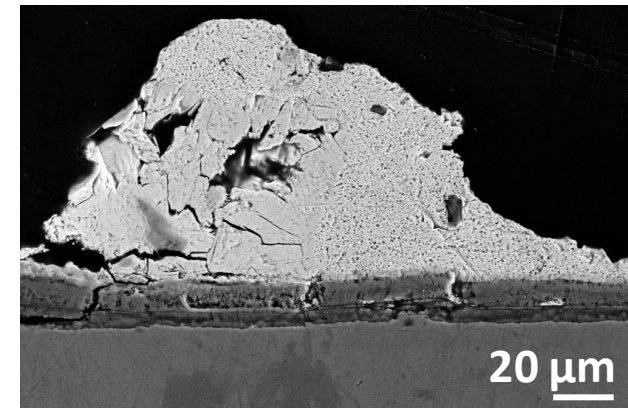
V. Gorynin et al. – Metal Science and Heat Treatment - 1999

In-situ passivation is not viable for $T > 500^\circ\text{C}$ (will be exceeded by fuel cladding)

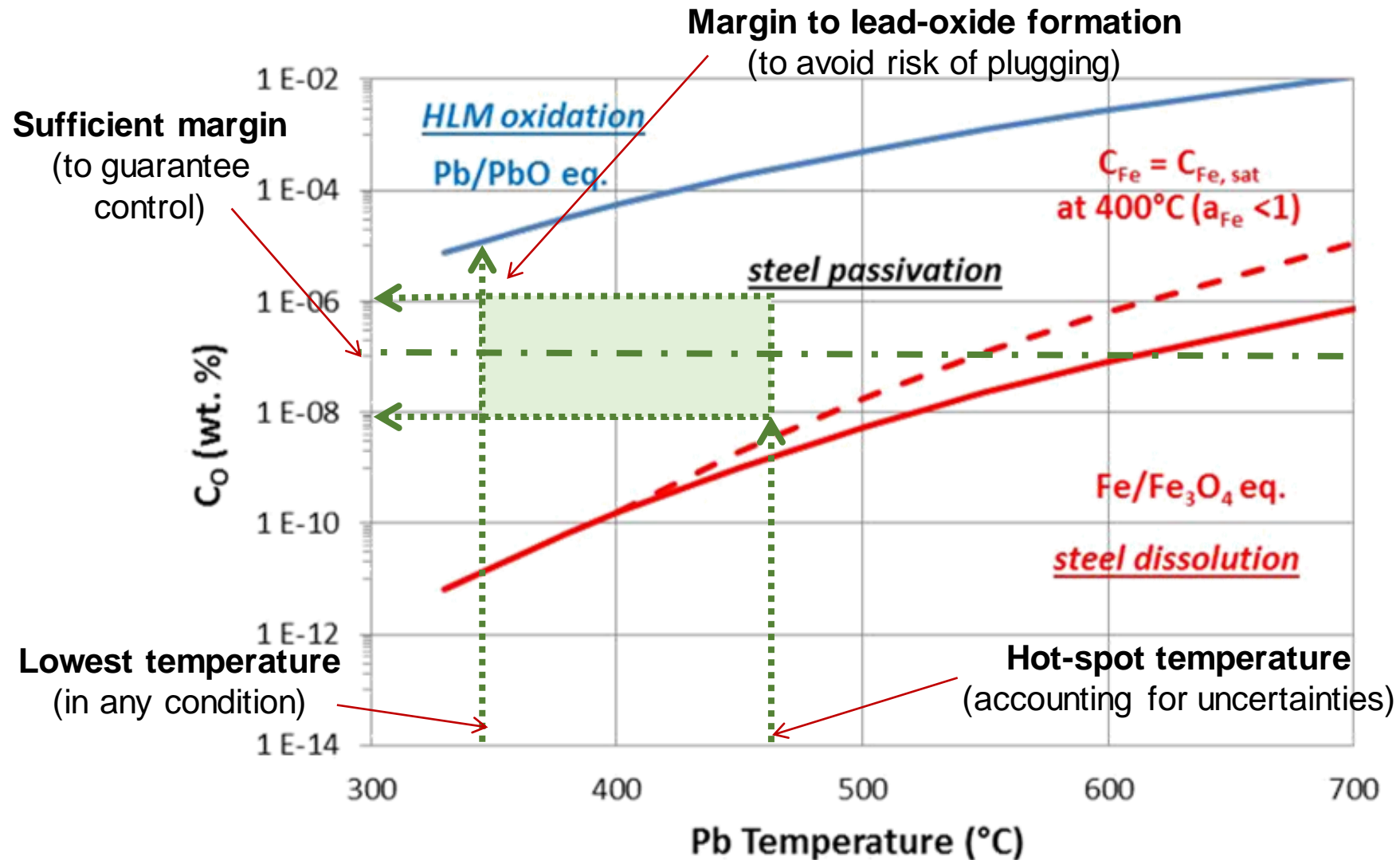
G. Mueller et al. – J Nucl Mater – 2004

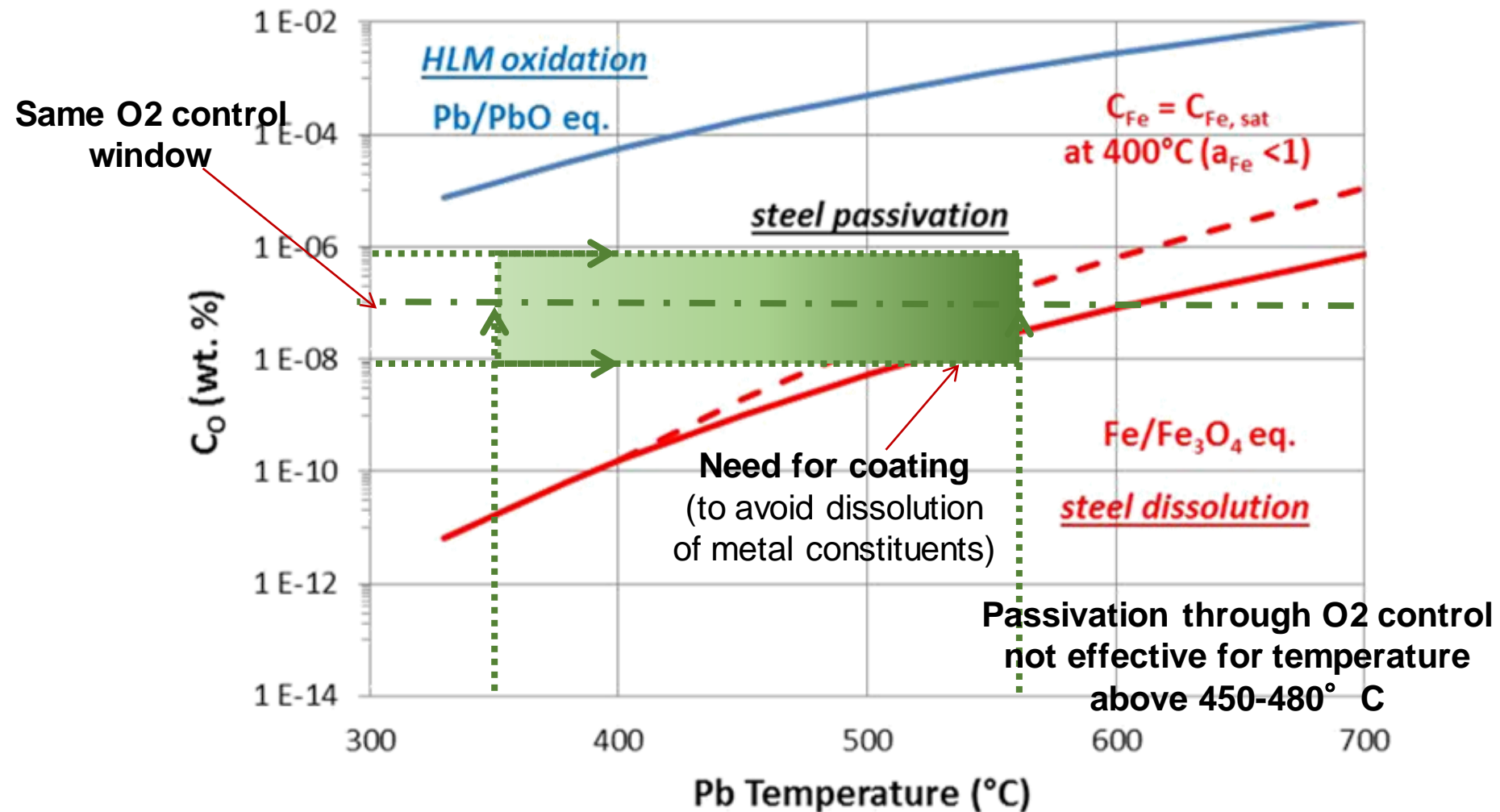


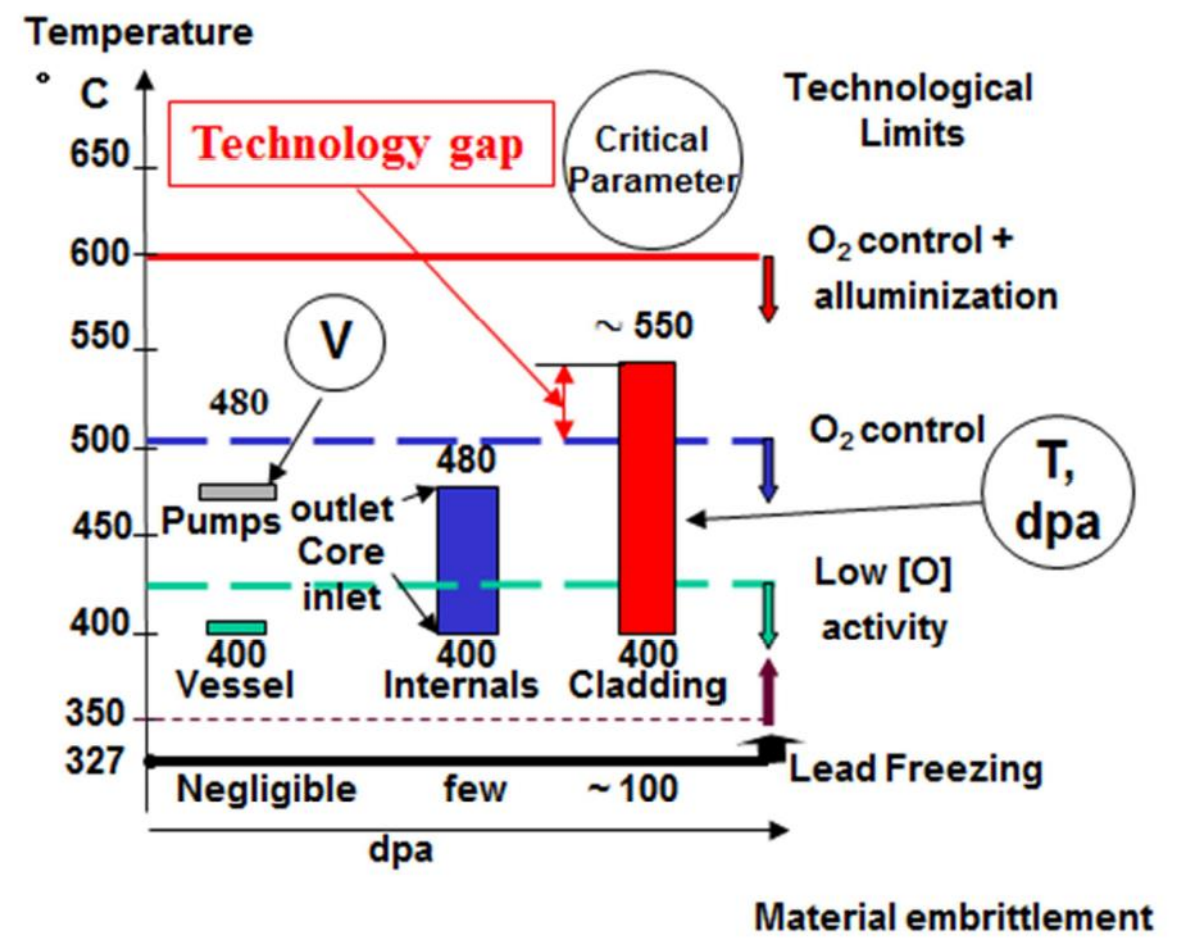
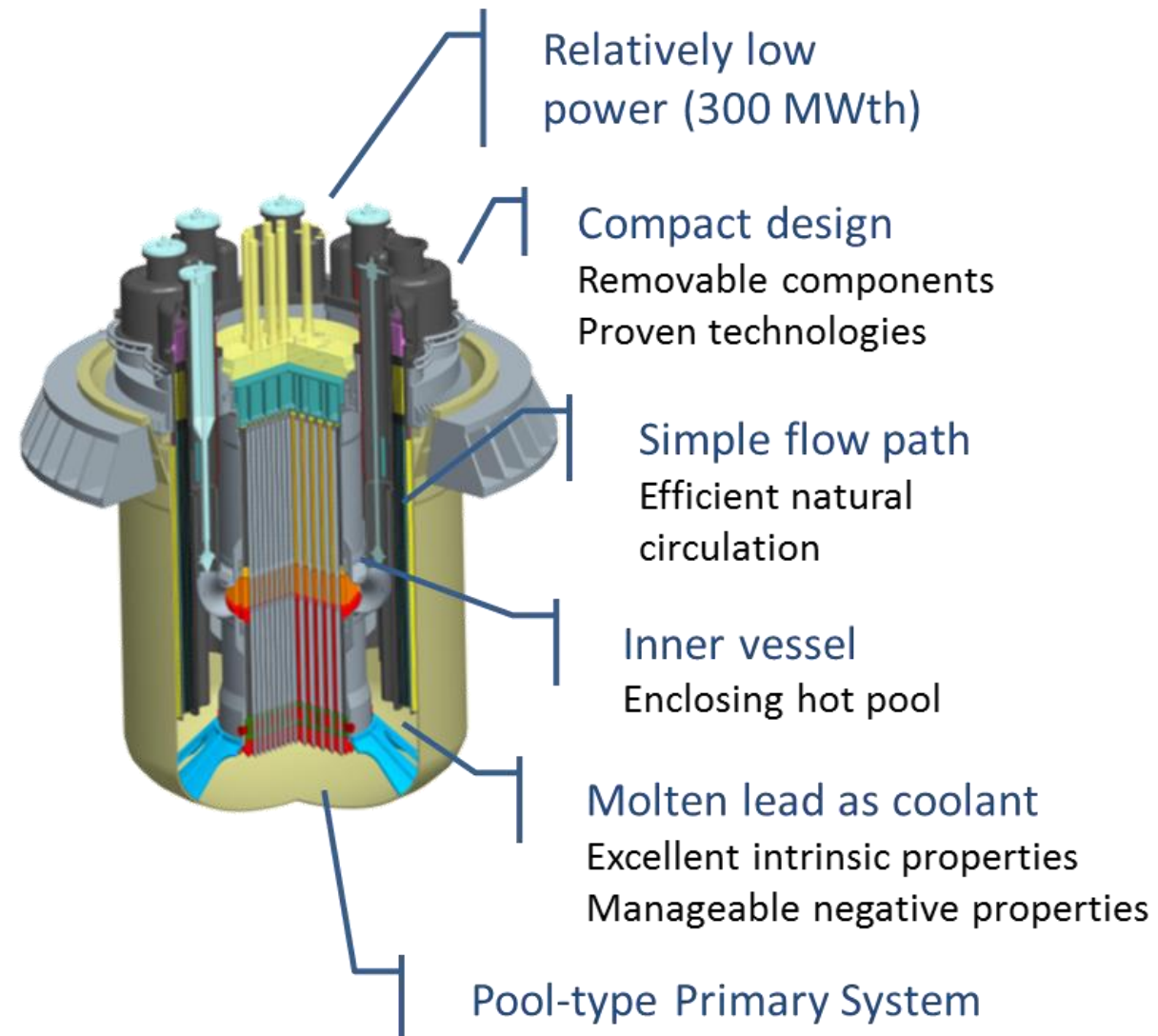
Dissolution



Oxidation

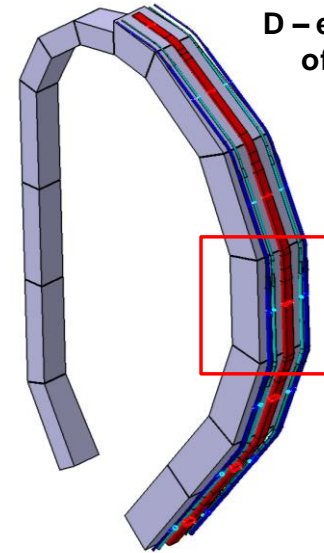
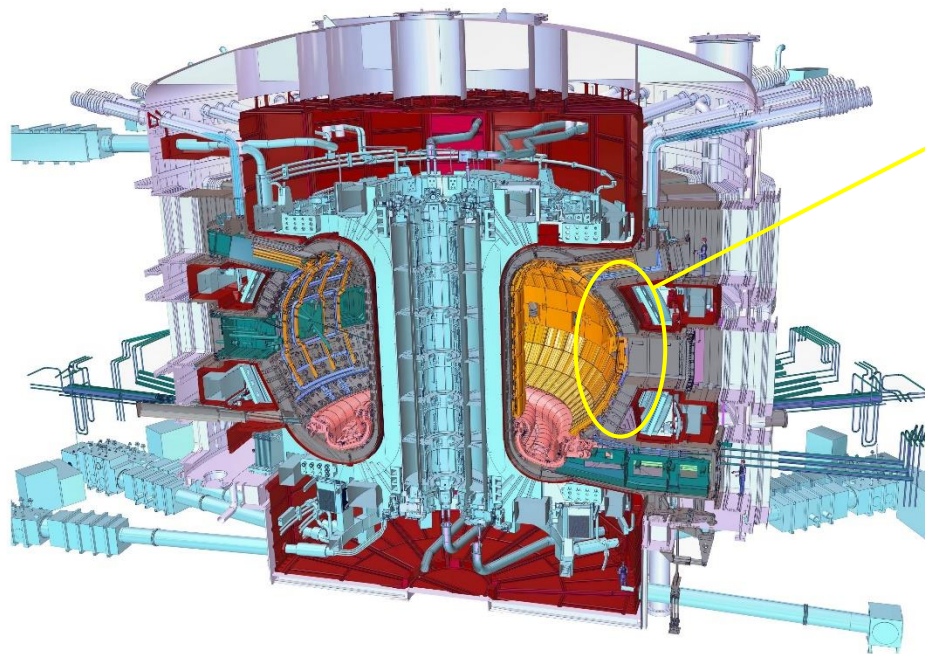




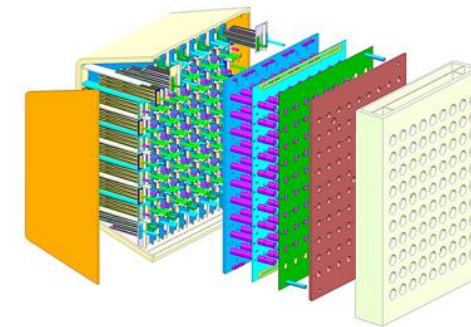


➤ **Pb-Li based Breeding Blanket (BB)**

- first wall heat removal
- neutron moderator/multiplier (thanks to Pb)
- tritium breeder through nuclear transmutations



D – element of the first wall
of DEMO reactor core



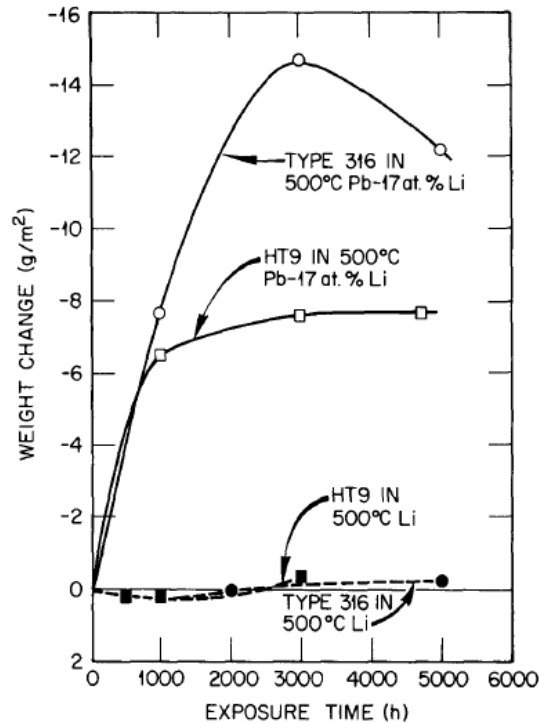
Section of the Breeding
Blanket component

➤ **Working conditions for fusion:**

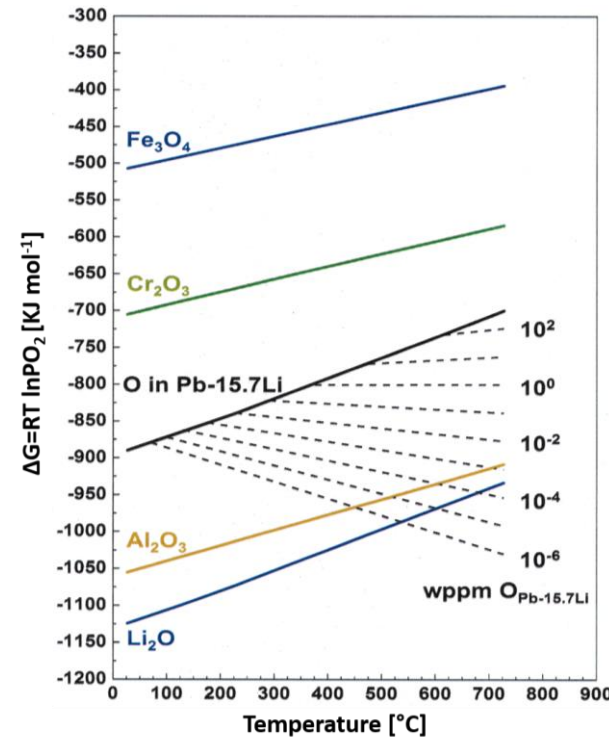
- up to 200 dpa
- 450 – 550 °C
- **NO SUITABLE MATERIALS**

➤ **HLM corrosion operated by lead-lithium eutectic (LLE): the combined action of Pb and Li**

Corrosion rates in pure Li VS Pb-Li



P.F. Tortorelli et al. – J
 MATER ENERG SYST – 1982



Thermo-dynamical potential of Pb-Li VS different oxides

Credits to S. BASSINI
 from ENEA

⇒ **Lithium presents high affinity with oxygen: it tends to reduce (any) metal oxides, depending on the conditions**

Requirements for environmental barrier coatings for structural materials in fission (fuel cladding, core components) and fusion (BB)

Mechanical performance

H₂/D₂/T₂ permeation

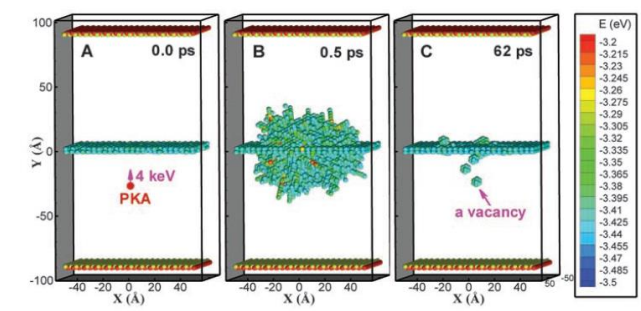
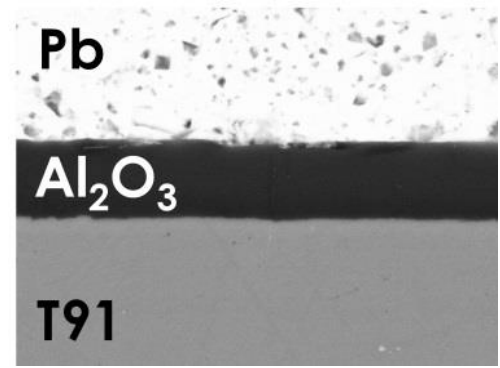
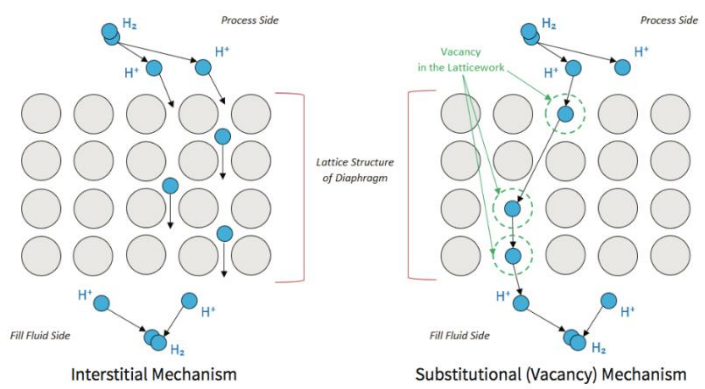
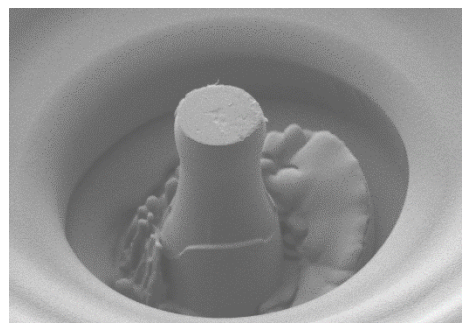
Corrosion resistance

Radiation tolerance

Ductility, hardness, strength

Amorphous structure

Interstitial emission from GBs



X.M. Bai et al. – Science - 2010

Thin films in fusion

Table 2
State of the art efficiency of some selected dielectric materials, recently recognised as HPBs, expressed as P_f at 400 °C.

	PRF	d_a mm	d_f μm	P_a $\times 10^{-11}$ molH ₂ / s/m/P ₂ ^{0.5}	P_f $\times 10^{-18}$ molH ₂ / s/m/P ₂ ^{0.5}	Ref
Al ₂ O ₃	1000	0.5	1	1.30	25.9	29
Cr ₂ O ₃	1000	1.6	10*	0.017	0.72*	31
Cr ₂ O ₃ /Al ₂ O ₃	3500	0.5	1	1.30	7.41	34
Er ₂ O ₃	1000	0.5	1	1.30	25.9	35
Er ₂ O ₃	1000	0.5	1.3	1.30	33.7	36
SiO ₂	1	0.15	0.2	0.13	1711	40
BN	100	0.1	1.5	0.13	193	42
TiN	100	0.1	1.5	0.13	193	42
TiN	1100	0.35	1.7	0.13	5.7	43
TiN	1000	0.1	1.7	0.13	21.8	43
TiAlN	6800	0.35	1.7	0.13	0.92	44
TiAlN	20,000	0.5	5	1.30	6.5	45
SiN	2000	0.5	0.5	1.30	6.5	25
WN	38	0.5	2.3	1.30	1570	48
Cr-WN	100	0.5	4.4	1.30	1140	48
Cr-N	117	0.5	2.6	1.30	576	48
Cr ₂ N	236	0.5	2.2	1.30	241	48
AlCrN	350	0.5	4.5	1.30	333	48
ZrN	4600	0.5	1.4	1.30	7.9	48
TiC	10	0.1	1	0.27	2750	52
TiN+TiC	100	0.5	1 + 0.25	1.30	324	53

Influence of annealing atmosphere on the deuterium permeation of Y₂O₃ coatings

Wu Yunyi^a, Shuai Li, Di He, Xiaopeng Liu, Shumao Wang, Lijun Jiang

Yttria deposited by MOCVD + annealing 700 °C.

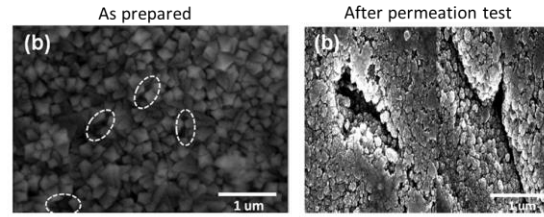
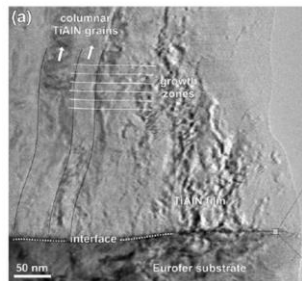


Table 1 – The PRF values of the coating S2 in the first and second permeation measurement cycles.

Temperature (°C)	500	550	600	650	700
First cycle	412	332	242	181	102
Second cycle	276	197	131	87	43

Hydrogen permeation through TiAlN-coated Eurofer '97 steel

Paul J. McGuinness^{a,*}, Miha Čekada^b, Vincenc Nemanic^c, Bojan Zajec^c, Aleksander Rečnik^a



Deposition using four unbalanced DC magnetron sources. Targets: **bulk titanium with aluminium plugs**. Total thickness of 5 μm (2 μm/h). The deposition temperature was about 450 °C.

MAX PRF = 20000 @400 °C. After 10 days the PRF dropped to 5800

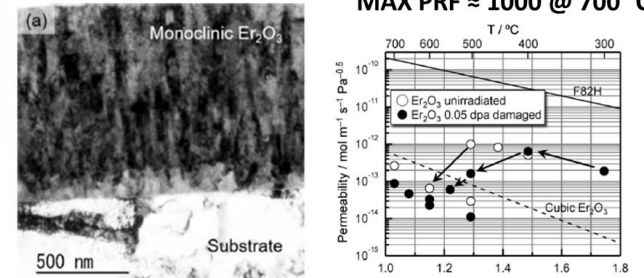
Deuterium permeation through monoclinic erbium oxide coating

Takumi Chikada^{a,*}, Hikari Fujita^a, Masayuki Tokitani^c, Yoshimitsu Hishinuma^c, Takayuki Terai^b, Yasuhisa Oya^b

Erbia deposited by vacuum arc vapor deposition (1.3–1.6 μm).

The substrate temperature was @ 600 °C

As deposited
MAX PRF ≈ 1000 @ 700 °C



Al–Cr–O thin films as an efficient hydrogen barrier

Denis Levchuk^a, Harald Bolt^a, Max Döbeli^b, Simon Eggenberger^c, Beno Widrig^c, Jürgen Ramm^{c,*}

Coatings by **cathodic arc evaporation** @ T = 550 °C, thickness 1 μm.

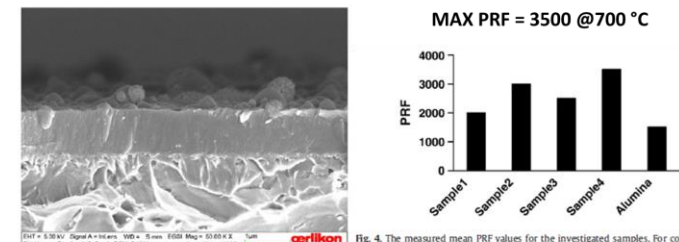


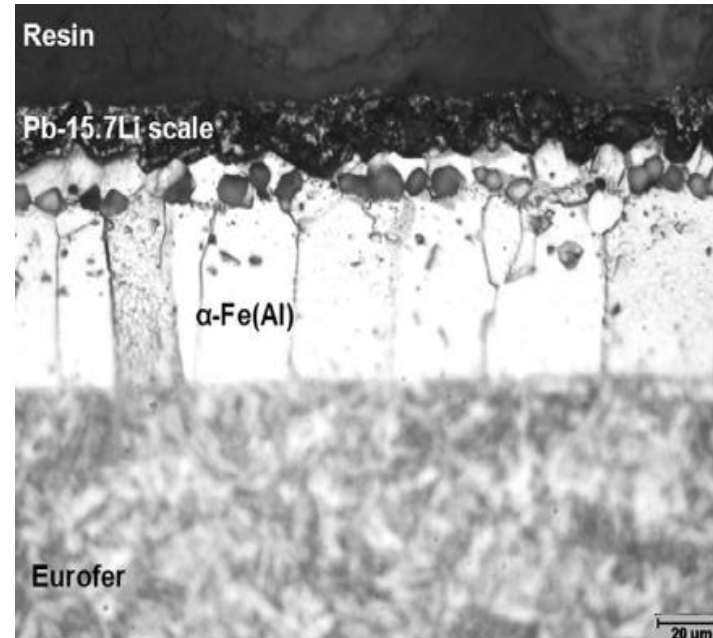
Fig. 4. The measured mean PRF values for the investigated samples. For comparison, data of a 1 μm thick alumina film deposited at 720 °C from [4] is given.

Electrochemical processes:

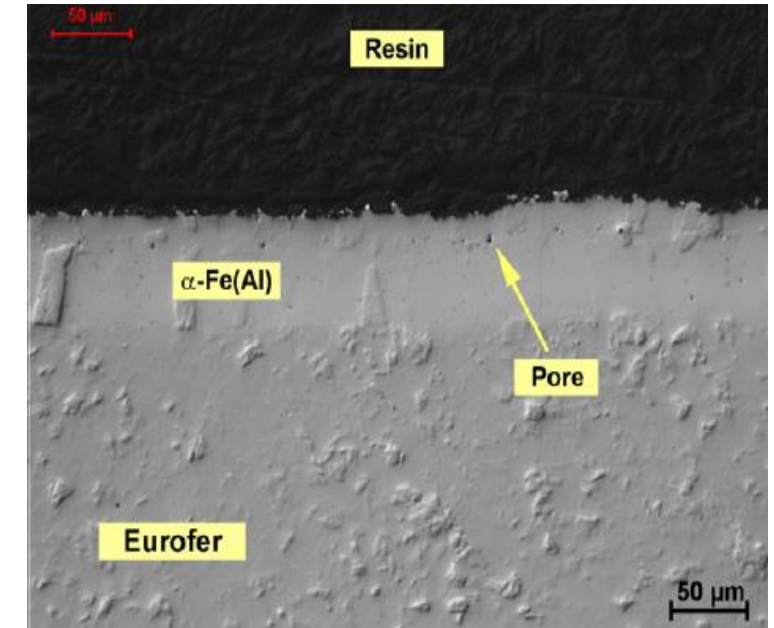
- **ECA** (organic electrolyte)
- **ECX** (Ionic liquids)

The formation of the functional surface requires specific heat treatments:

- 913K/4h in Air/Ar
- 1253K/0.5h + 1033K/1.5h in air
- Natural cooling down



Exposed ECA



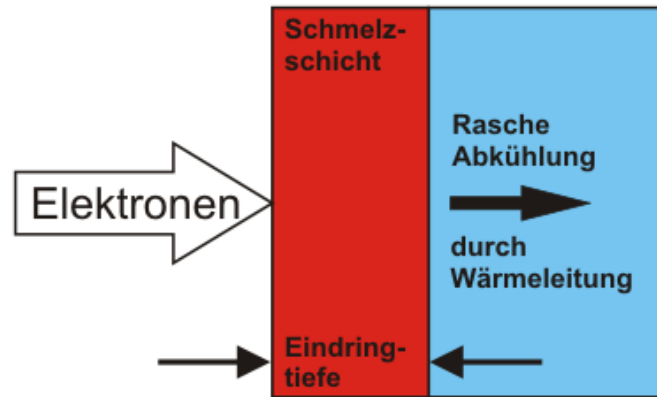
Unexposed ECX

J. Konys et al., *Fusion Engineering and Design* 87 (2012) 1483-1486.

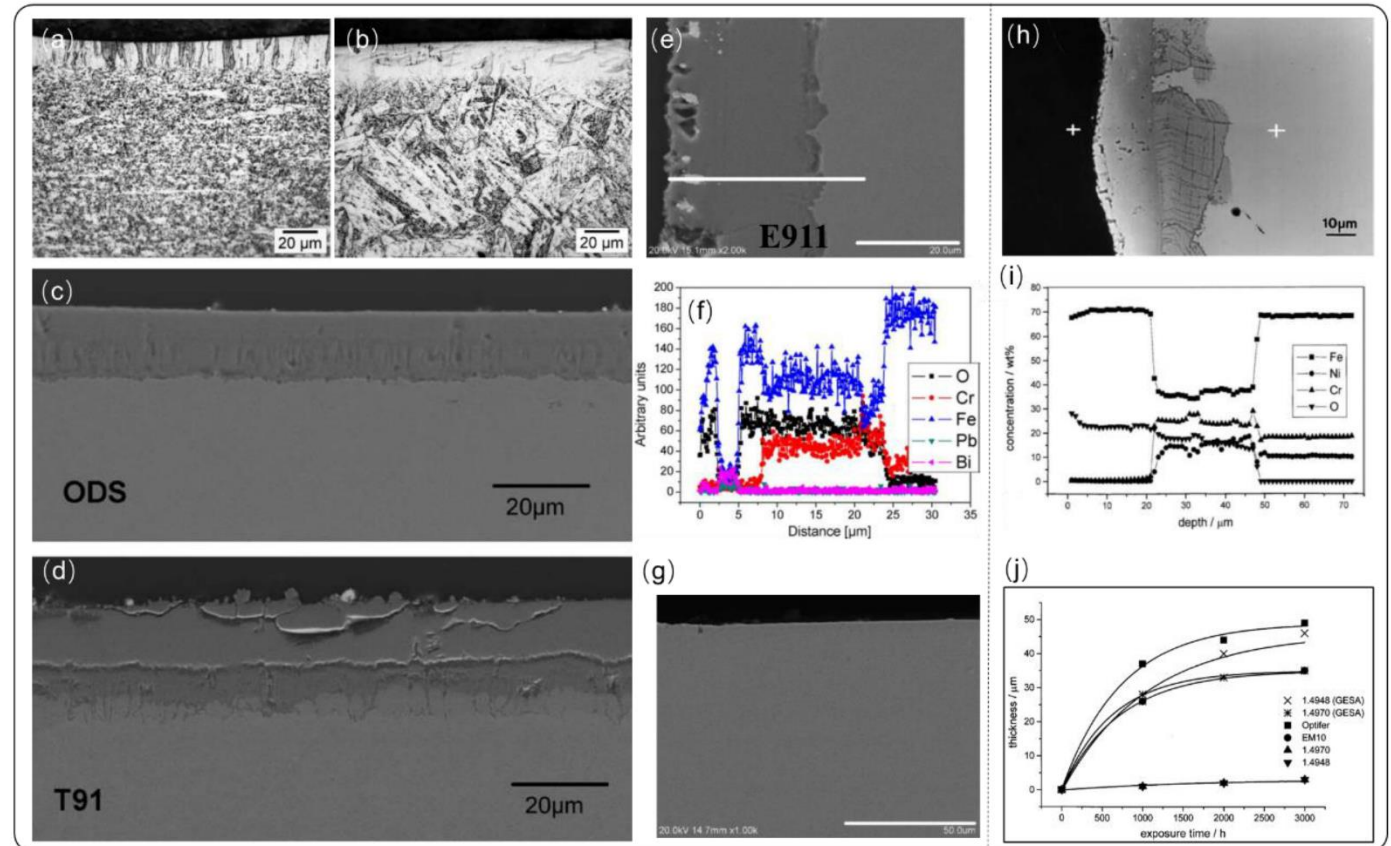
W. Krauss et al., *Journal of Nuclear Materials* 417 (2011) 1233-1236.

Both techniques shown a good barrier behaviour against corrosion (5000h in flowing Pb-17Li at 823K and $0.22\text{m}^*\text{sec}^{-1}$).

ECA **PRF** at beginning of life ~ 1000



- Eindringtiefe der Elektronen: 10 - 100 μm
- 3 -20 J/cm^2 werden benötigt zum Schmelzen
- Rasche Abkühlung durch Wärmeleitung ins Grundmaterial (10^7 - 10^9 K/s)
- Effizienter Energieeintrag



Gepulste Elektronen Strahl Anlage (GESA) treated coatings and corrosion performance. (a,b) Cross-sectional images of Al-coating on oxide dispersion-strengthened (ODS) and E911. (c–e) Cross-section images of the Al-coated ODS, T91 and E911 after corrosion test. (f) Element distribution of E911 after GESA treatment. (g) Cross section of ODS with Al alloying. (h) Cross section of 1.4948 steel after GESA treatment. (i) Element distribution from EDX energy spectrum of EP911. (j) Summary of oxide layer thickness at 550 °C in flowing lead [65,66]. **H. Wang Coatings 2021, 11(3), 364**

Non equilibrium synthesis of ductile amorphous ceramic coatings for nuclear applications by Pulsed Laser Deposition

Acta Materialia 61 (7), 2662-2670, 2013
Corrosion Science 77, 375-378, 2013
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Ceramics International 47 (24), 2021
Materials Characterization 178, 2021

“Ductile” amorphous ceramics

enhanced

enhanced

enhanced

enhanced

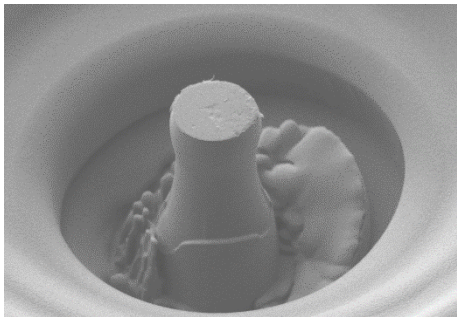
Mechanical performance

H₂/D₂/T₂ permeation

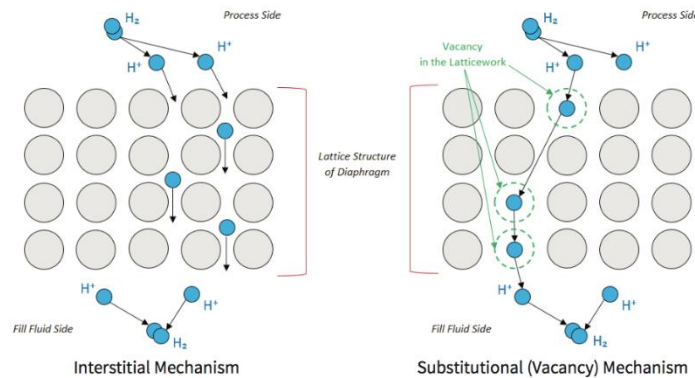
Corrosion resistance

Radiation tolerance

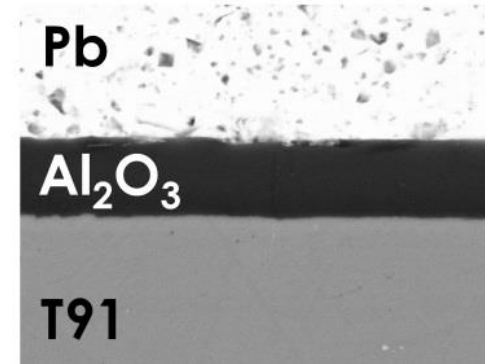
Ductility, hardness, strength



Amorphous structure

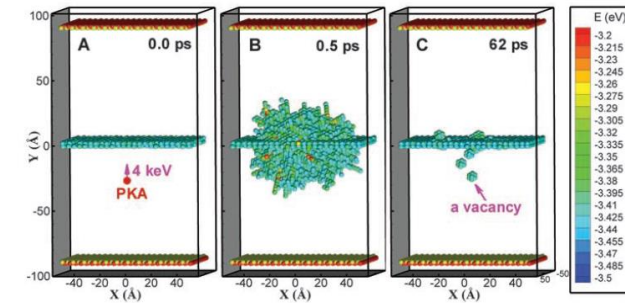


F. Garcia Ferré et al., SCI REP (2016)
E. Frankberg et al., Science (2019)



F. Garcia Ferré et al. – CORROS SCI – 2013

Interstitial emission from GBs



X.M. Bai et al. – Science - 2010

“Ductile” amorphous ceramics

enhanced

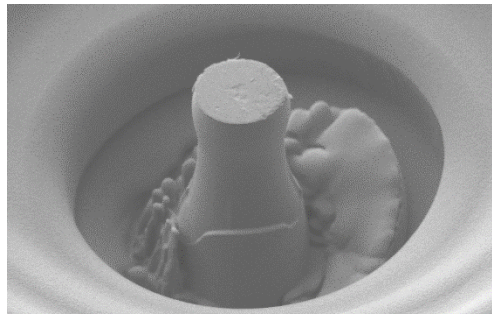
enhanced

enhanced

enhanced

Mechanical performance

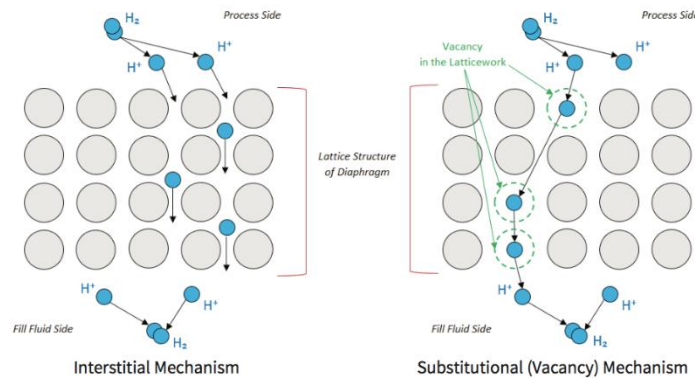
Ductility, nanohardness



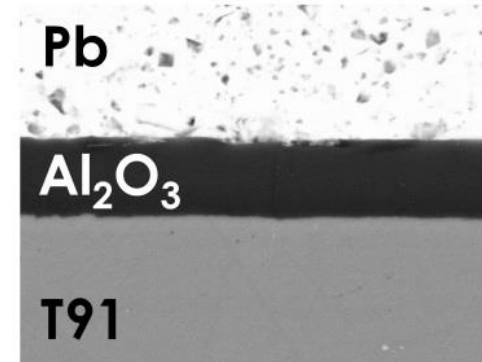
F. Garcia Ferré et al., SCI REP (2016)
 E. Frankberg et al., Science (2019)

H₂/D₂/T₂ permeation

Amorphous structure



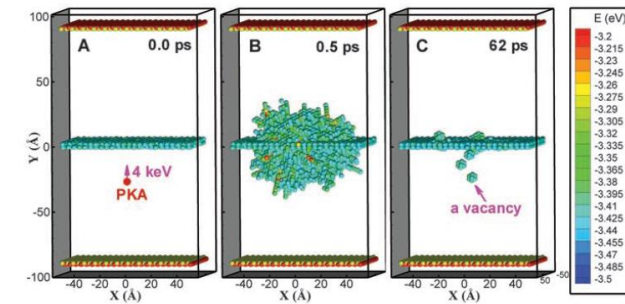
Corrosion resistance



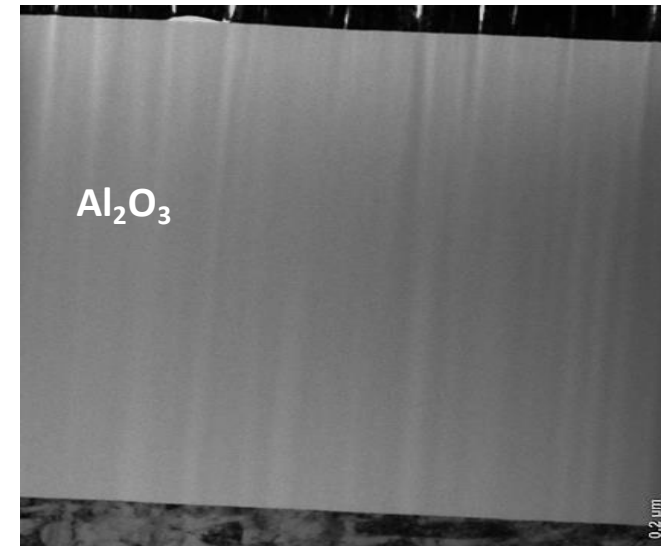
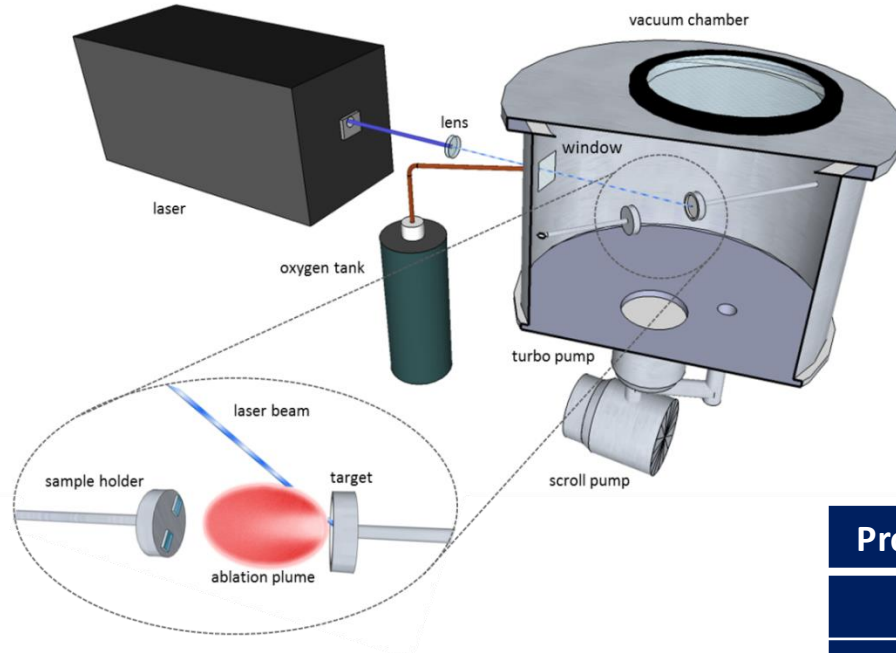
F. Garcia Ferré et al. – CORROS SCI – 2013

Radiation tolerance

Interstitial emission from GBs



X.M. Bai et al. – Science - 2010

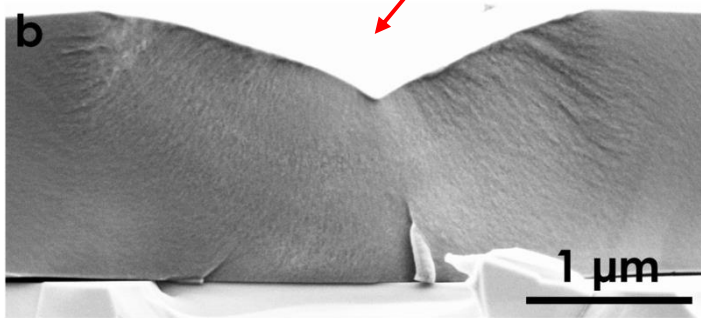
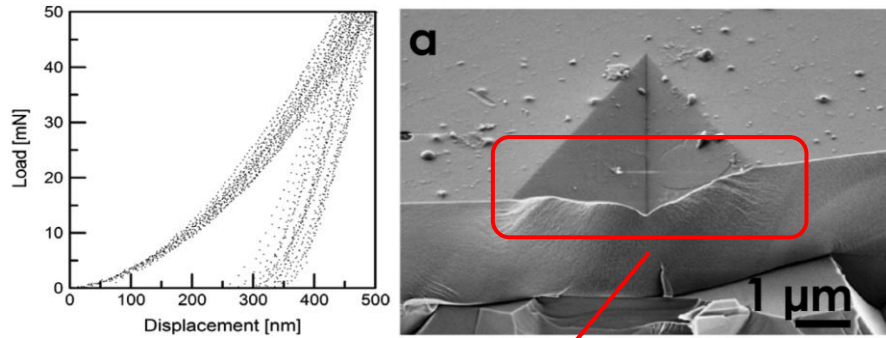


- ✓ amorphous Al_2O_3 is mechanically matched to steels
- ✓ out-of-equilibrium process, large freedom on chemistry!
- ✓ density of the amorphous material \approx crystalline (3.5 g/cm³ for Al_2O_3)
- ✓ impervious to gases
- ✓ room T process

Property @RT	Sapphire	PLD Al_2O_3	AISI 316L
ν	0,24	$0,295 \pm 0,025$	0,3
E [GPa]	345	$193,8 \pm 9,9$	200
G [GPa]	175	$75,5 \pm 3,8$	80
B [GPa]	240	$159,2 \pm 11,8$	140
H [GPa]	27,8	$10,3 \pm 1$	4
H/E	0,059	$0,049 \pm 0,007$	0,025

H/E parameter index of wear resistance and fracture toughness

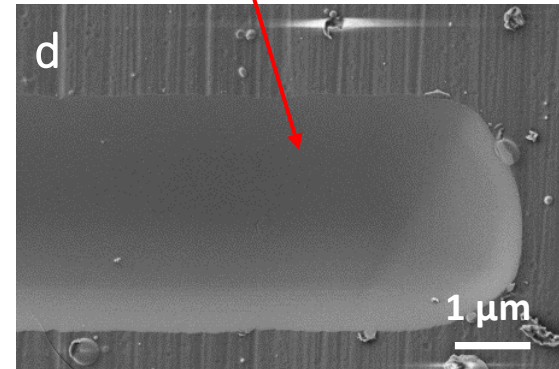
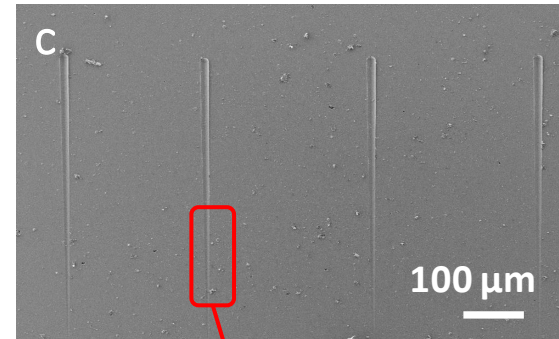
- Nanoindentation Tests**



metal-like behavior under plastic strain

Plastic work 62%

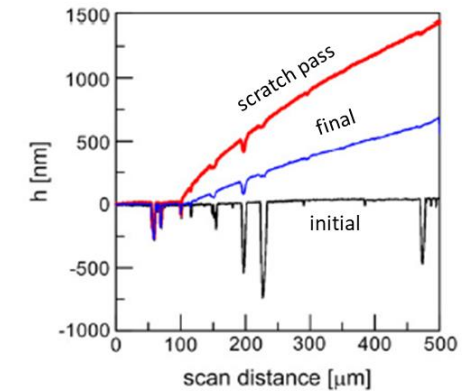
- Nanoscratch Tests**

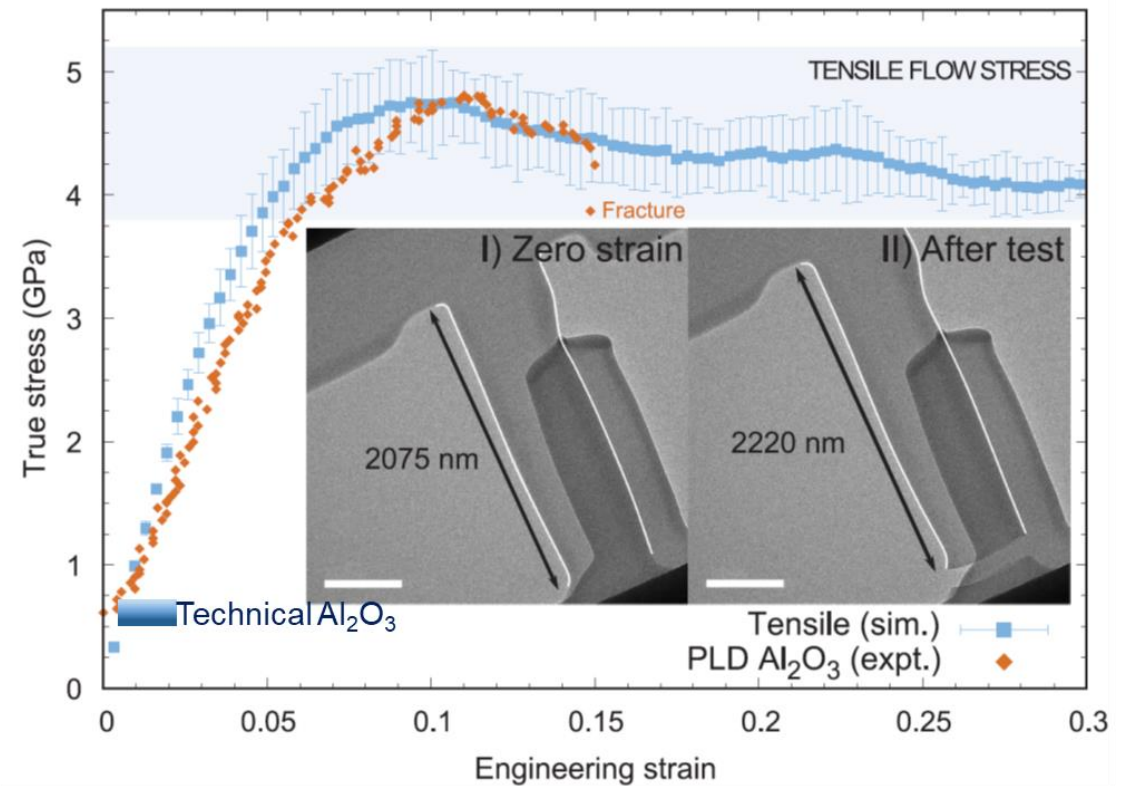
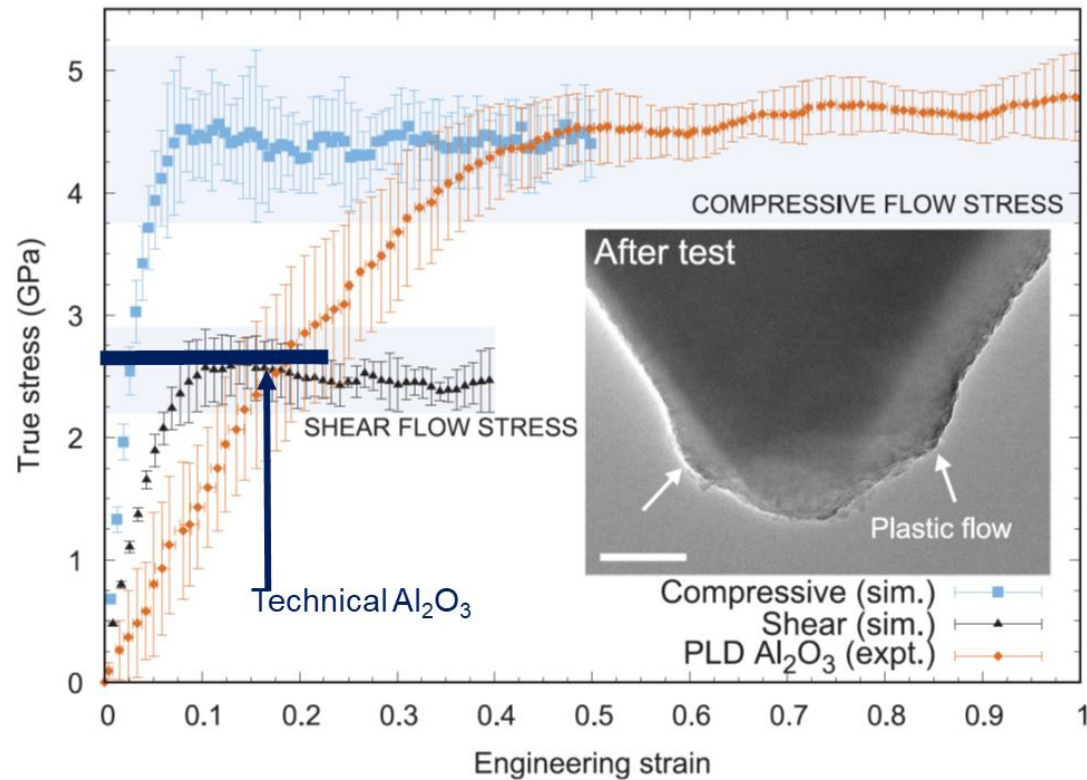


strong interfacial bonding

**No delamination
 No critical load**

**Strong interfacial
 bonding**





In situ TEM results:

-compression: 100% deformation

-tensile: 15 % total elongation, 7% plastic deformation to failure

GLASSES
Science Highly ductile amorphous oxide at room temperature and high strain rate
 AAAS

E. Frankenberg et al. Science 864-869, 2019

“Ductile” amorphous ceramics

enhanced

enhanced

enhanced

enhanced

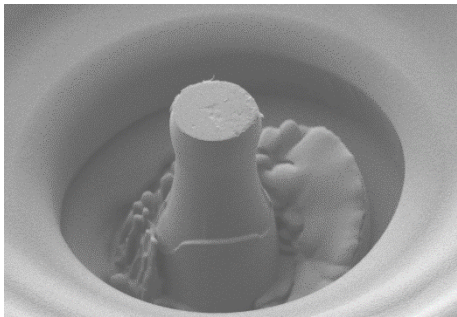
Mechanical performance

H₂/D₂/T₂ permeation

Corrosion resistance

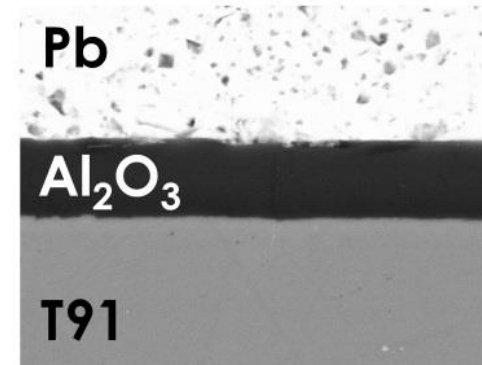
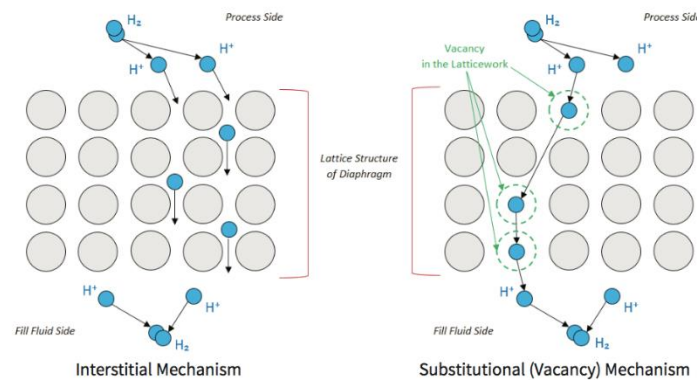
Radiation tolerance

Ductility, hardness, strength



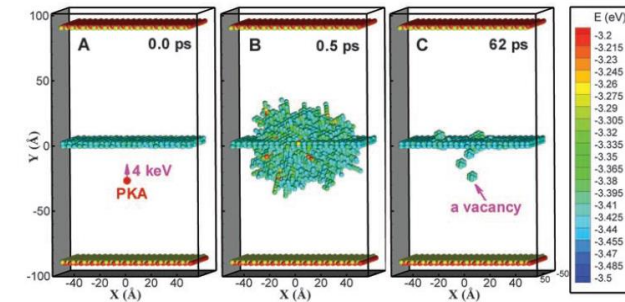
F. Garcia Ferré et al., SCI REP (2016)
 E. Frankberg et al., Science (2019)

Amorphous structure

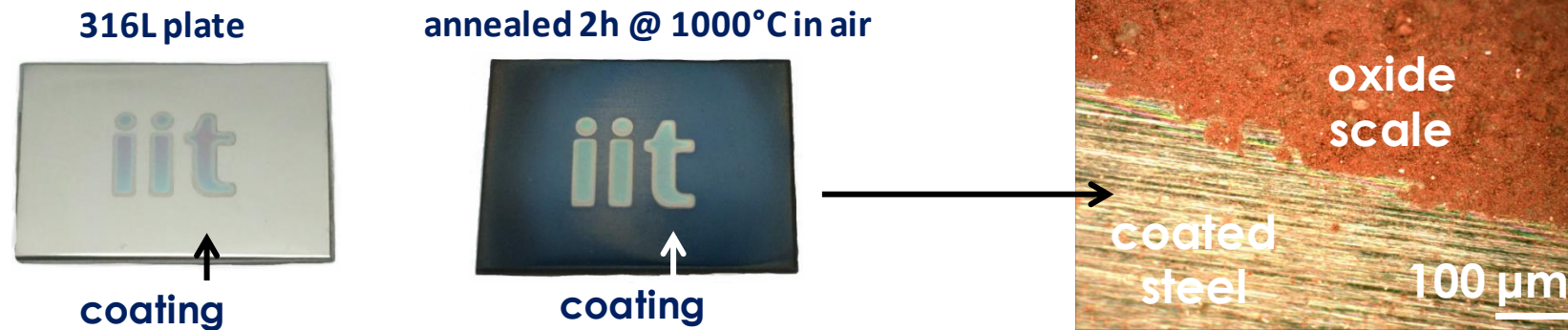


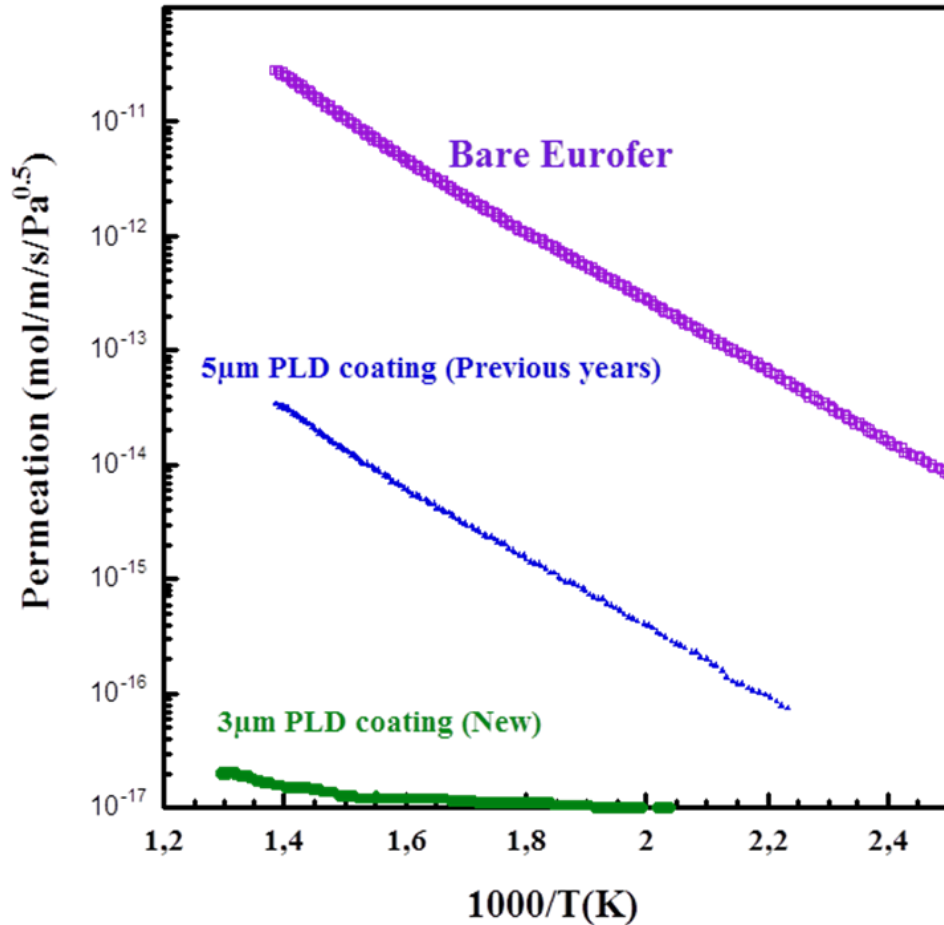
F. Garcia Ferré et al. – CORROS SCI – 2013

Interstitial emission from GBs

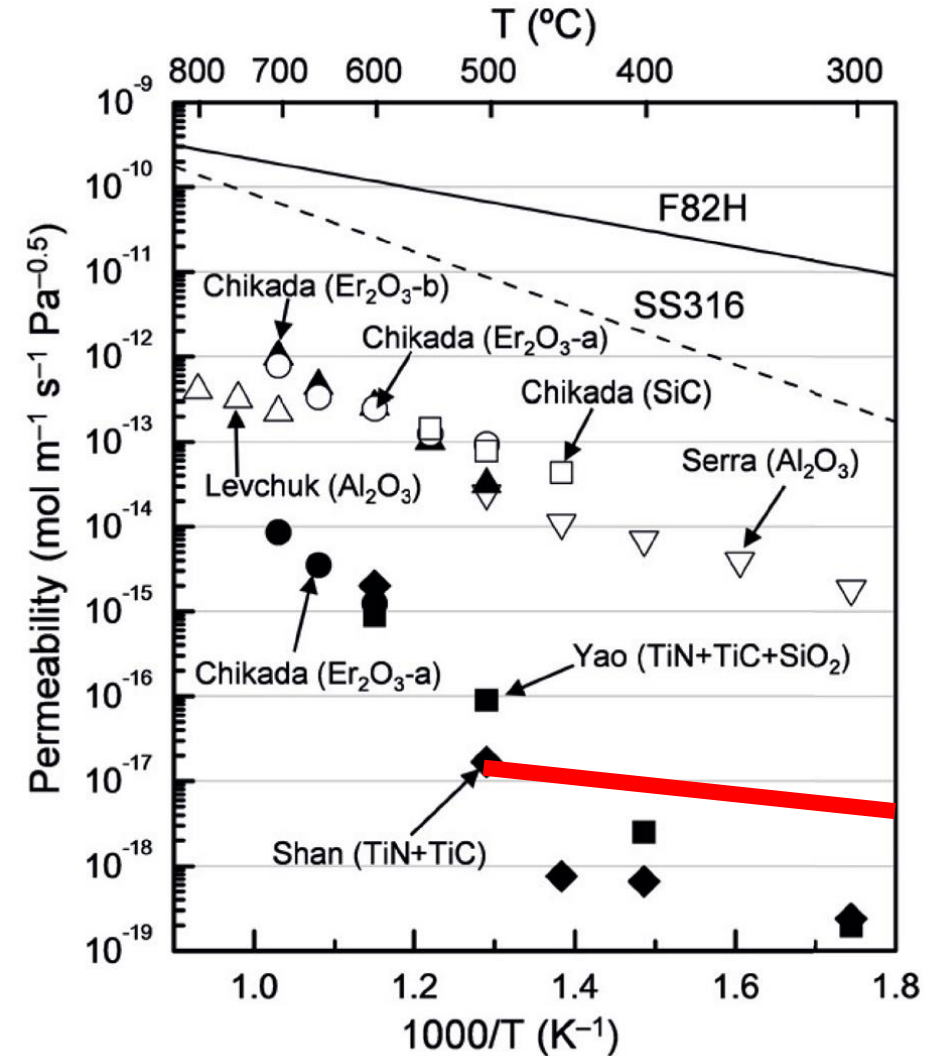


X.M. Bai et al. – Science - 2010





$$PRF = \frac{J_{Bare}^D}{J_{Coated}^D} > 10^5 @ 450^\circ\text{C}$$



“Ductile” amorphous ceramics

enhanced

enhanced

enhanced

enhanced

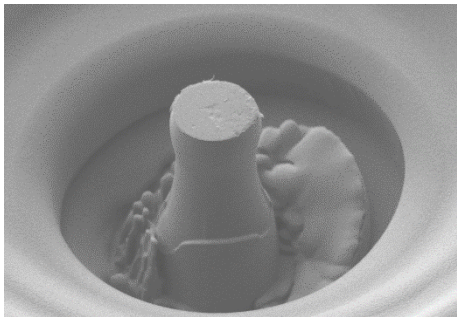
Mechanical performance

H₂/D₂/T₂ permeation

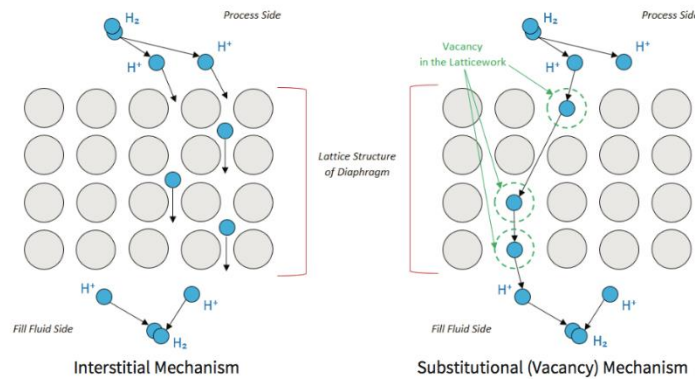
Corrosion resistance

Radiation tolerance

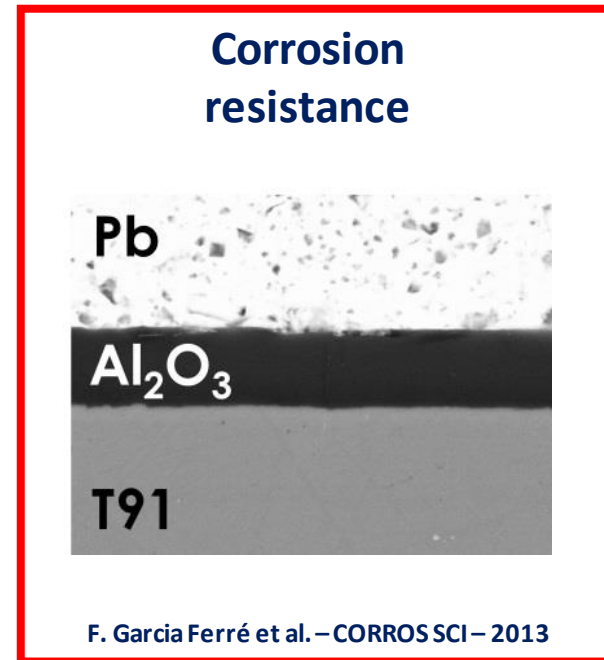
Ductility, hardness, strength



Amorphous structure

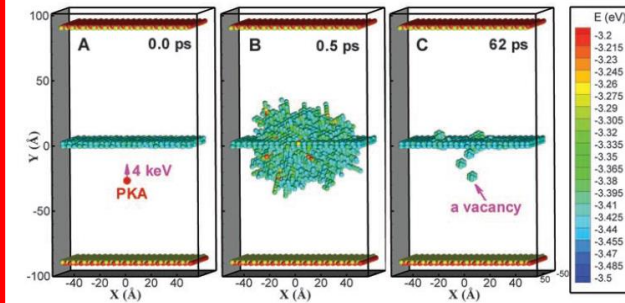


F. Garcia Ferré et al., SCI REP (2016)
 E. Frankberg et al., Science (2019)



F. Garcia Ferré et al. – CORROS SCI – 2013

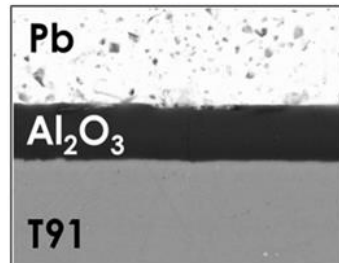
Interstitial emission from GBs



X.M. Bai et al. – Science – 2010

Stagnant lead (LFR)

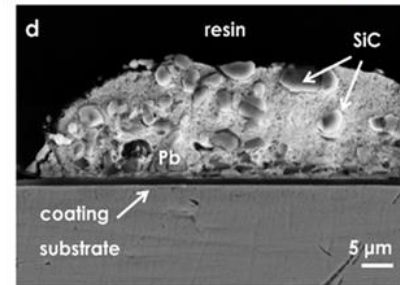
- 500 h @ 550 °C
- No oxygen control



Garcia Ferrè F. et al.,
 Corr. Science, 2013

Stagnant lead (LFR)

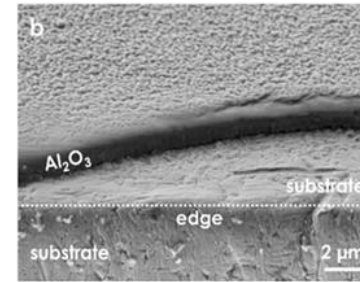
- 10'000 h @ 550 °C
- 10^{-3} - 10^{-8} wt.% O₂



Garcia Ferrè F. et al.,
 Corr. Science, 2017

Stagnant lead (LFR)

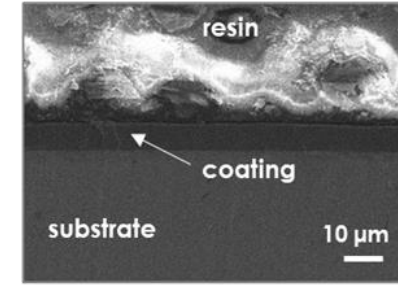
- 1'000 h @ 550 °C
- pre-irradiated coating



Garcia Ferrè F. et al.,
 Corr. Science, 2017

Flowing lead (LFR)

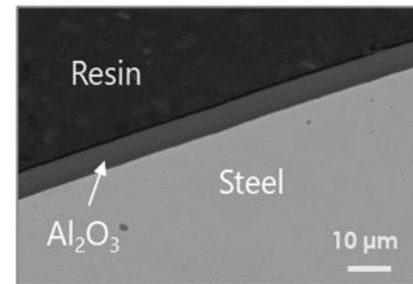
- 200 h @ 525 °C
- 10^{-4} wt.% O₂



Vanazzi M. et al.,
 IAEA technical report, 2021

Stagnant Pb-55Bi (LFR)

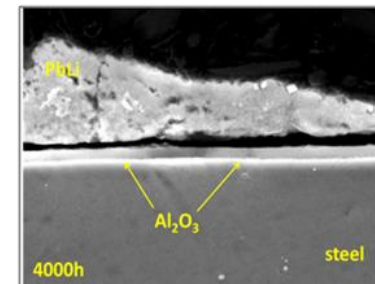
- 1'000 h @ 500 °C
- 10^{-10} wt.% O₂



E. Charalampopoulou, et al.,
 Mat. Char., 2021

Stagnant Pb-16Li (BB)

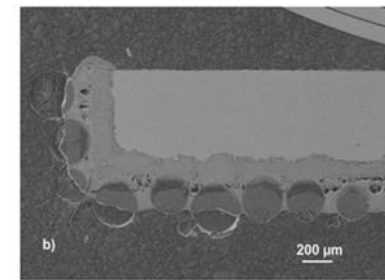
- 8'000 h @ 550 °C
- 10^{-8} wt.% O₂



Iadicicco D. et al.,
 Fus. Des. and Eng., 2019

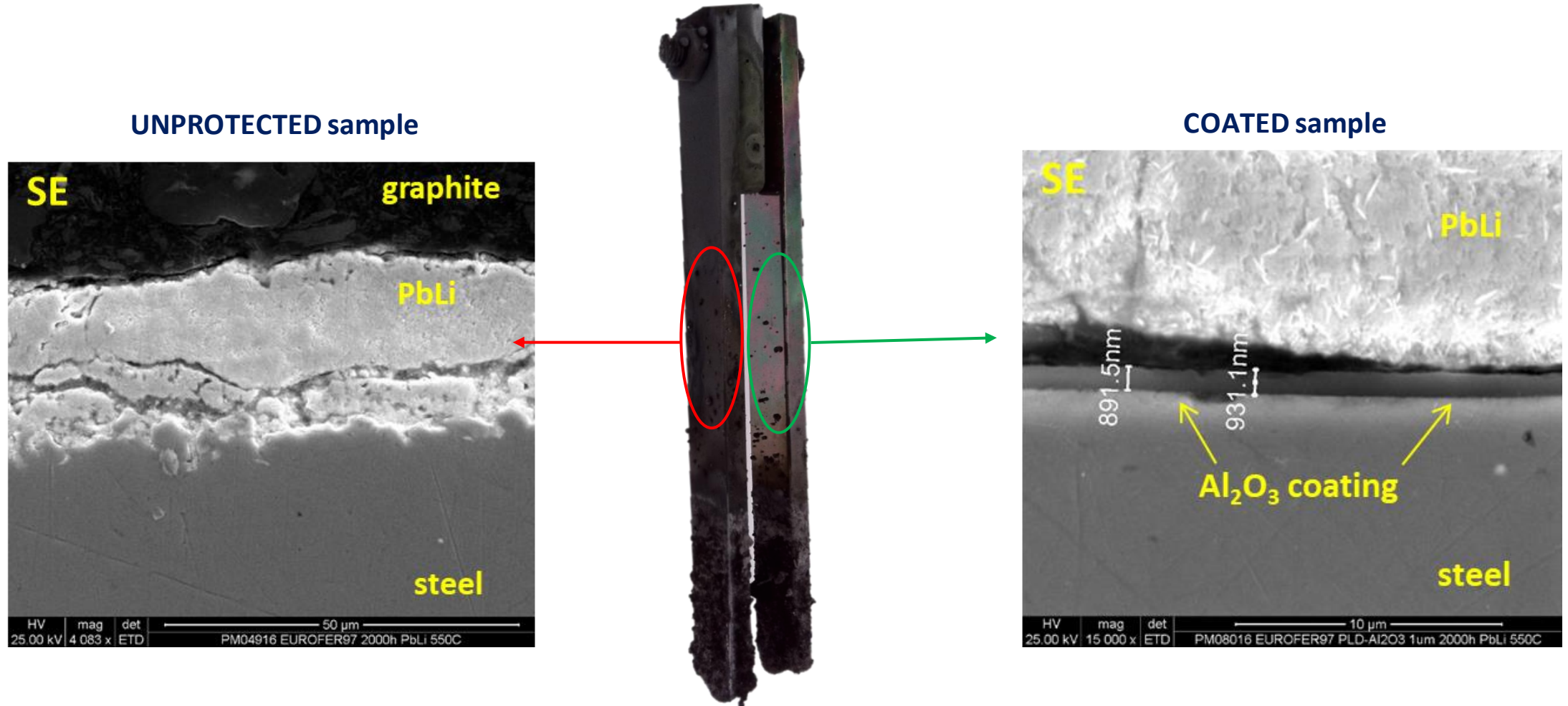
Li₄OSi₄ pebbles (BB)

- 730 h @ 800 °C
- Pebbles bed system

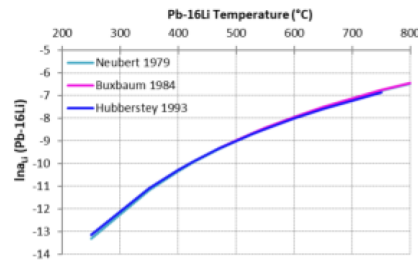


Hernandez T. et al.,
 J. of Nuc. Mat., 2019

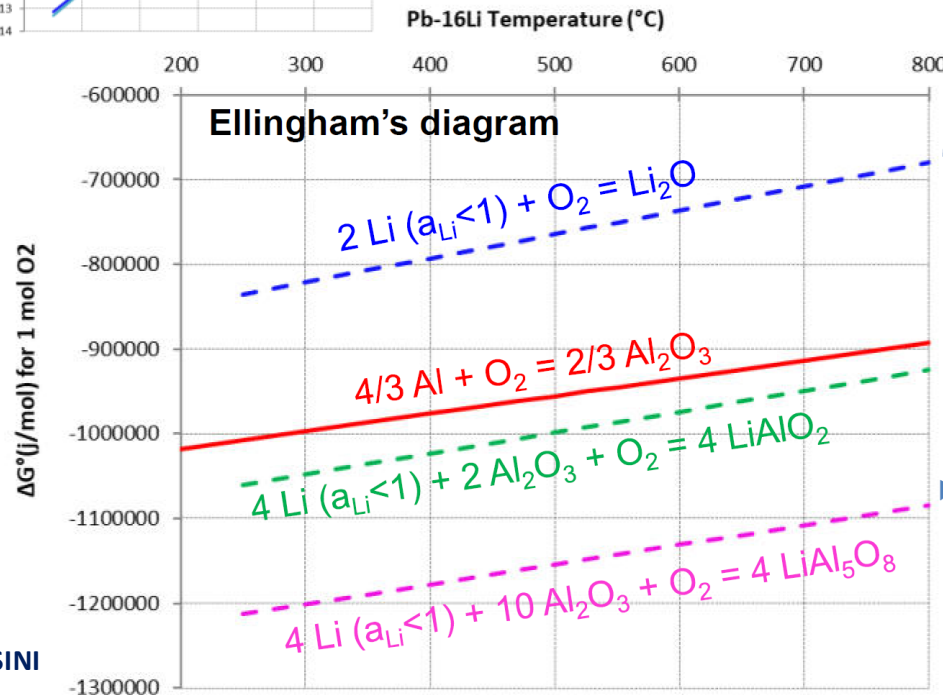
- **Stagnant LLE Corrosion Test on alumina-coated EU-97 steel (collaboration with ENEA)**
- **Test temperature 550 °C - up to 8'000 hours - O₂ % monitored (dissolutive @ 10⁻⁸ wt.% regime)**



Thermodynamics of Al_2O_3 in PbLi



a_{Li} activity from Hubberstey 1993
 ΔG°_f Li_2O , Al_2O_3 , LiAlO_2 from Barin
 ΔG°_f LiAl_5O_8 from NBS table



- Al_2O_3 is stable in liquid PbLi eutectic thanks to the low Li activity (Hubberstey 1997).
- Experimentally, LiAlO_2 formation occurred in PbLi eutectic on crystalline Al_2O_3 layer at 800°C after 1000h (Pint 2008).
- LiAlO_2 formation was observed on crystalline alumina powder in PbLi exposed to air at 550°C after 48h. No formation under vacuum, slow kinetics under flowing inert gas (Jain 2014).
- LiAl_5O_8 and LiAlO_2 formation from Al_2O_3 is thermodynamically favored in PbLi. LiAl_5O_8 is more stable, in agreement with our experiments which shows about LiAl_5O_8 formation.
- Effect of kinetics should be investigated: likely LiAlO_2 formation is promoted at high temperature and with source of oxygen (air or inert gas flowing) whereas LiAl_5O_8 is formed at lower temperature with minor oxygen availability (e.g. inert gas overpressure in our experiments).

“Ductile” amorphous ceramics

enhanced

enhanced

enhanced

enhanced

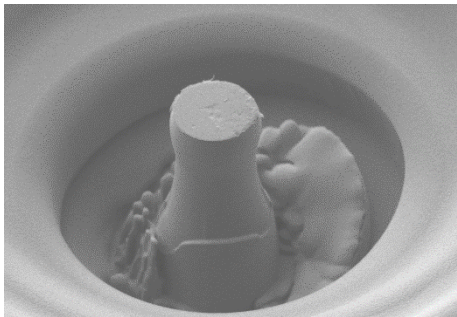
Mechanical performance

H₂/D₂/T₂ permeation

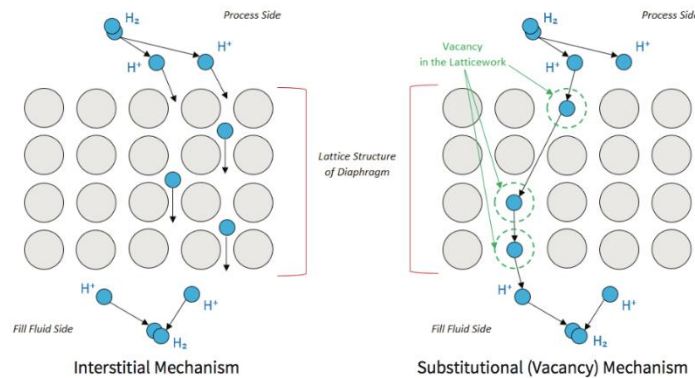
Corrosion resistance

Radiation tolerance

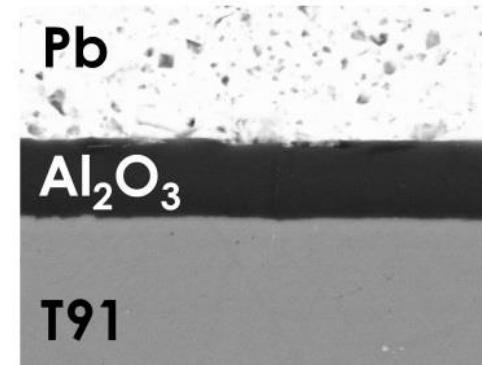
Ductility, hardness, strength



Amorphous structure

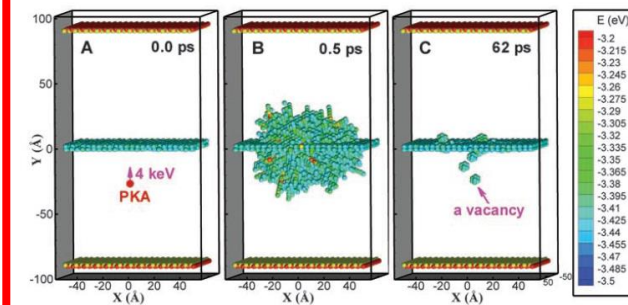


F. Garcia Ferré et al., *SCI REP* (2016)
E. Frankberg et al., *Science* (2019)



F. Garcia Ferré et al. – *CORROS SCI* – 2013

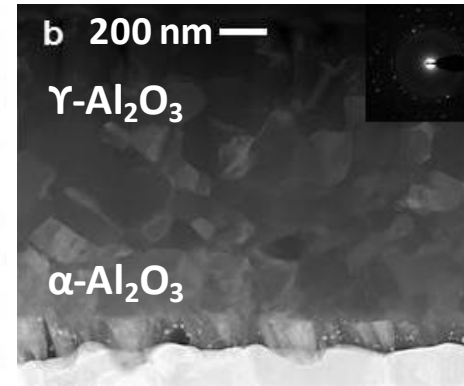
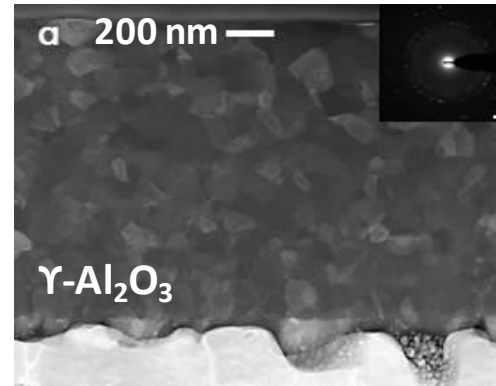
Interstitial emission from GBs



X.M. Bai et al. – *Science* – 2010

moderate dpa

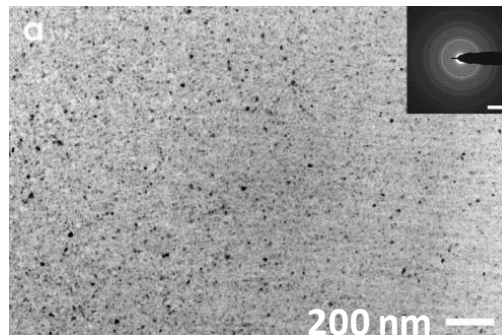
ultra-fine nanoceramic
GB-driven deformation
highest fracture toughness



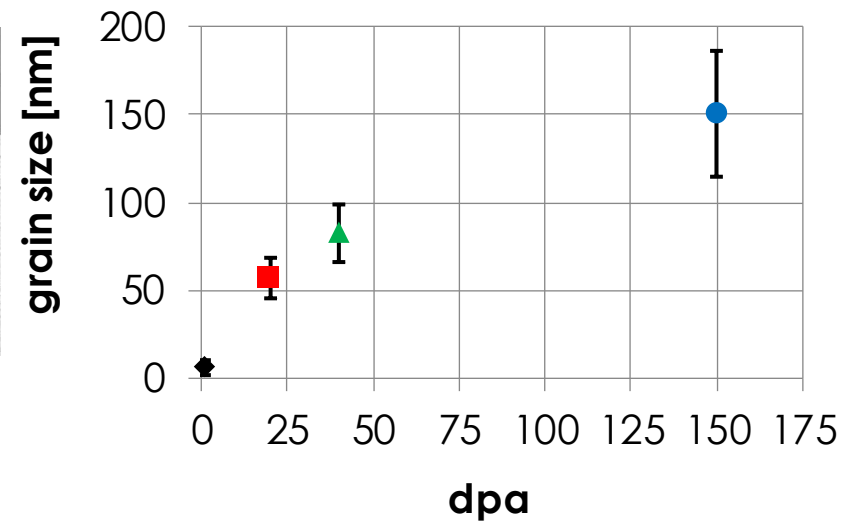
high dpa

fine nanoceramic
GB-driven deformation
sub-linear grain growth

pristine

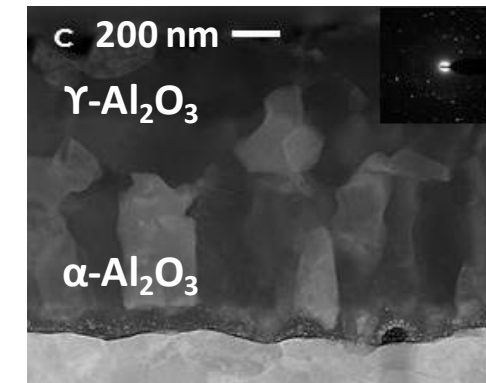


bi-phase nanocomposite
shear banding
highest fracture strength



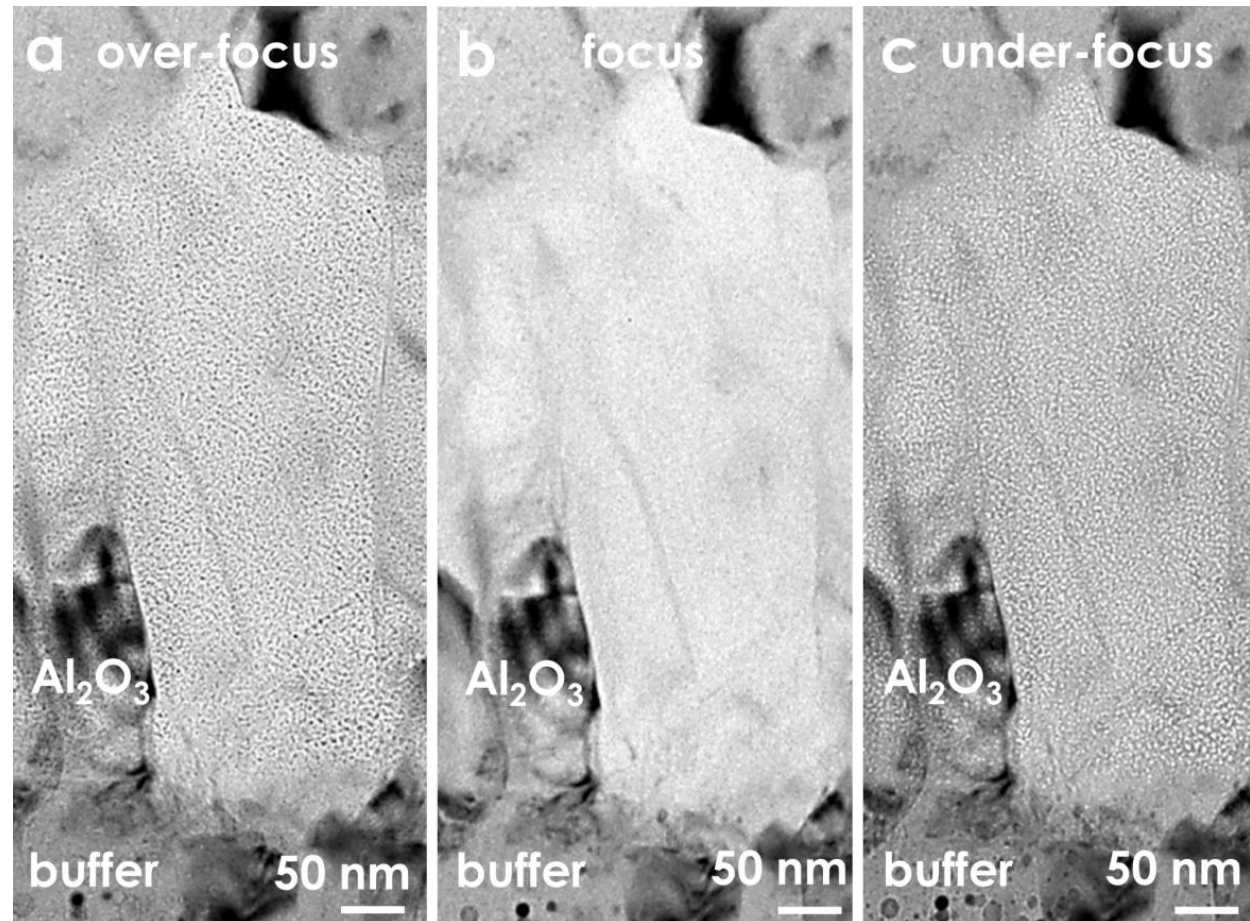
Sublinear grain growth

end-of-life dpa



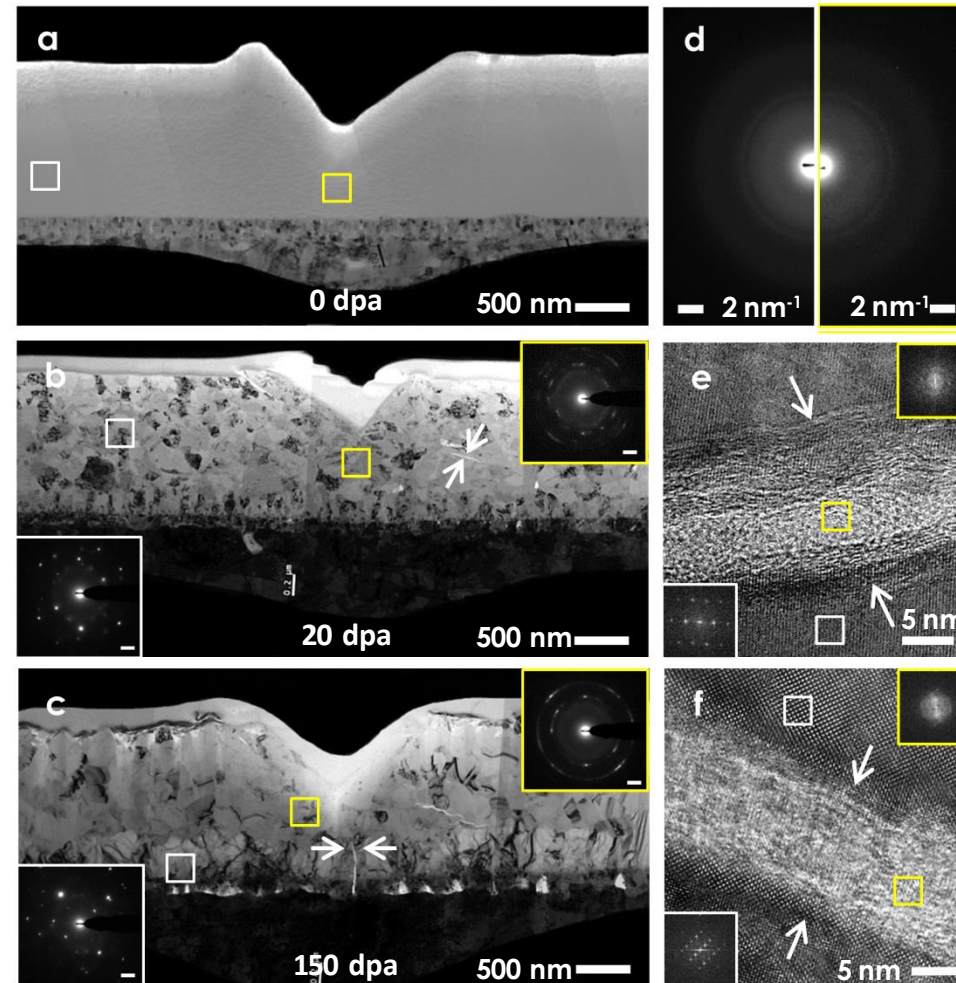
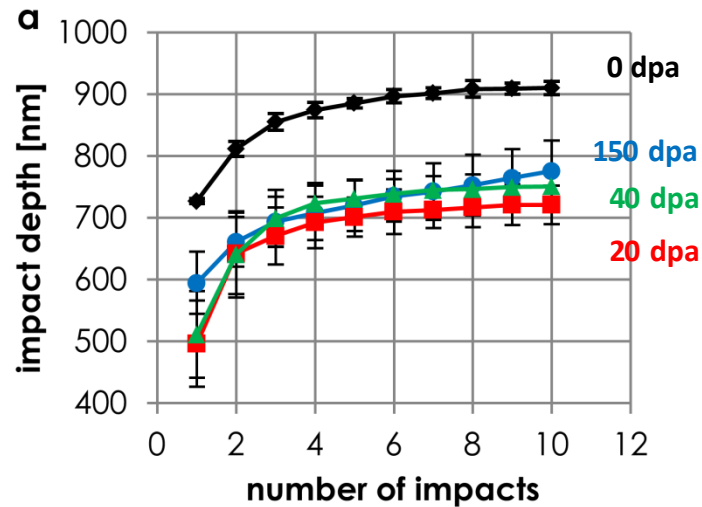
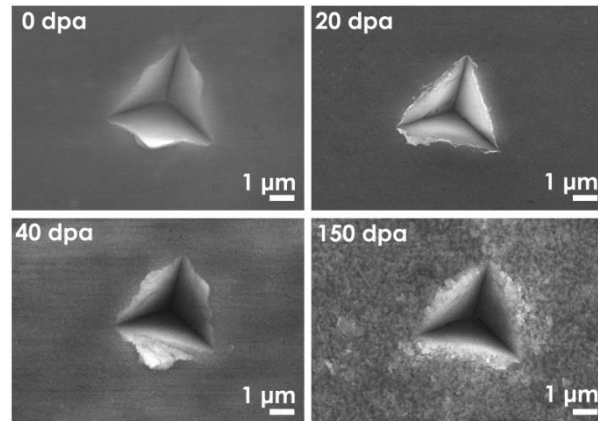
nanoceramic
GB-driven deformation
highest stiffness

Heavy ion irradiation (Au + W): swelling at 150 dpa



- Large number of small voids, only in a monolayer of grains, at the interface with the buffer layer
- These grains are alpha-alumina. All the other grains are gamma-alumina

Heavy ion irradiation (Au + W): nanoimpact



Impact energy is dissipated more efficiently in irradiated samples

Conclusions

➤ **Qualification tests for Lead Fast Reactor and Breeding Blanket applications: a follow up**

- Corrosion tests in **static/fluent Pb, static LBE and LLE** up to **10'000 hours**
- Combined tests of **permeation** with **D₂ under electron irradiation**
- **Engineering of mechanical response** in nano-ceramic coatings

➤ **Evolution of barrier coatings under irradiation: design and understanding**

- **Role of the irradiation parameters** on the material changes (ion type, T, flux, etc.)
- Formulation of a **structural/mechanical model** to **predict the evolution of alumina**
- **Control of the crystallization** by **chemical doping: improved performance**

➤ **Future development for the technological licensing**

- Continuation of **relevant tests** (corrosion, permeation, etc.) with an **integrated approach**
- Collection of **ions VS neutrons experiment results** to support the present data
- **Towards high entropy stabilization of ductile amorphous oxides under irradiation**

Collaborators



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National Centre for Nuclear Research
ŚWIERK

Instytut kategorii A+, JRC collaboration partner

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Łukasz Kurpaska



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Heavy ion irradiations
CABET Celine
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Norwegian University of
Science and Technology
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Teresa Hernandez

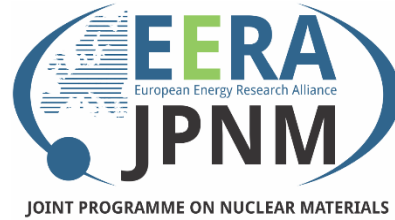


Dr. Jinh Hu
Dr. Wei-Ying Chen
Dr. Meimei Li

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MINISTERO DELLO
SVILUPPO ECONOMICO



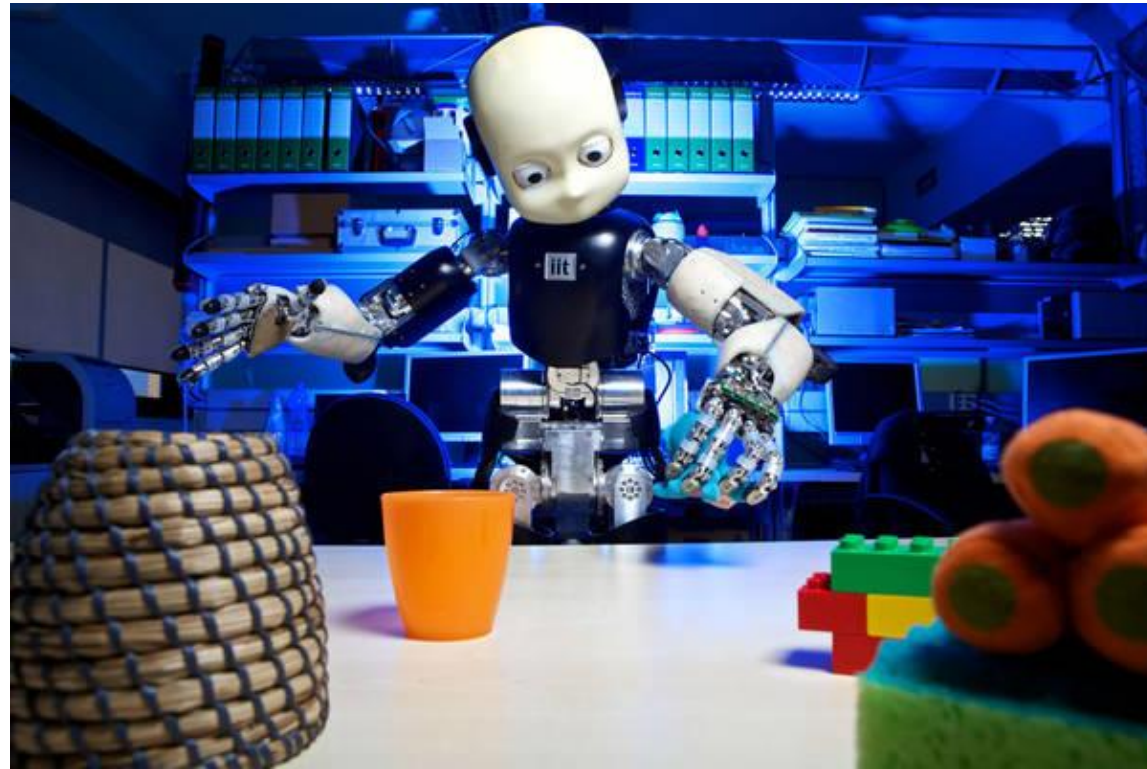
EUROfusion



GEMMA



First Prize at SOFT 2020 Innovation Prize



Thank You
for your attention!