Is There a Need for International Standardization in Evaluating Coatings for Tritium Permeation Barriers?

A. Iaboni[1], E. Gallo[1], C. Alberghi[3], G. Centomani[1], F. Di Fonzo[2], G. Fierli[3], M.E. Gennaro[1], D. Martelli[3] , A. Stinchelli[3]

(1) Eni SpA, piazzale Enrico Mattei 1, 00144, Roma, Italy

(2) ENEA, Località Brasimone, 40032 Camugnano, Bologna, Italy

(3) x-nano S.r.l. Via Rubattino 81, 20134 Milano, Italy

IAEA
International Atomic Energy Agency

Technical Meeting on Tritium Breeding Blankets and Associated Neutronics
02-05 set 2025
IAEA Headquarters, Vienna, Austria

ENEA Agenzia nazionale per le nuove tecnologie, l'energia e lo sviluppo economico sostenibile

eni

x_nano invisible matters

Introduction

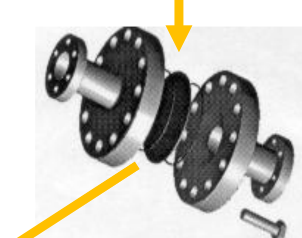
Fusion Power Plants must keep **Tritium losses** to the environment **below 1g/year** [1]. Current estimates for DEMO tritium release might achieve some grams in a few days [2]. To meet safety requirements **Tritium Permeation Barriers (TPB) are necessary,** with **Permeation Reduction Factors (PRF) of at least 10^3,** [3]. In this framework ceramic coatings are being investigated due to their promising properties, including strong adhesion to the substrate and high corrosion resistance. The presence of such coatings typically shifts the tritium permeation mechanism toward a surface-limited regime.

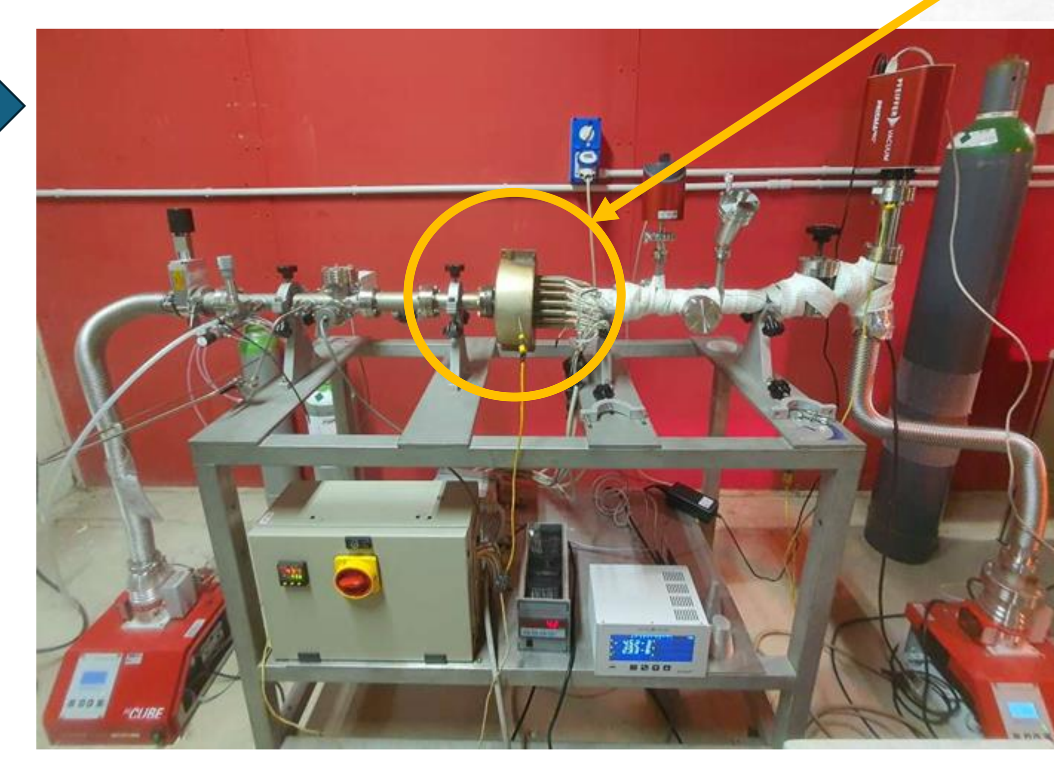
$$J_{Diffusion\ Limited} \propto \frac{\sqrt{p}}{t}, \quad J_{Surface\ Limited} \propto p \quad \Rightarrow \quad PRF \propto \frac{1}{t\sqrt{p}}$$

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_s mm	d_f µm	P_s x10^{-11} molH_2/s/m/$Pa^{0.5}$	P_f x10^{-18} molH_2/s/m/$Pa^{0.5}$	Ref
Al_2O_3	1000	0.5	1	1.30	25.9	29
Cr_2O_3	1000	1.6	10*	0.017	0.72*	31
Cr_2O_3/Al_2O_3	3500	0.5	1	1.30	7.41	34
Er_2O_3	1000	0.5	1	1.30	25.9	35
Er_2O_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
CrWN	100	0.5	4.4	1.30	1140	48
CrN	117	0.5	2.6	1.30	576	48
Cr_2N	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

[4]





Experimental Set-up

Hydrogen permeation tests were performed using the ENEA PERI-II experimental facility [5]. Gas driven measurements have been performed by means of **Continuous Flow Method** (CFM). Conditions relevant for a fusion power plant environment have been experimented involving:

- Sample temperature **up to 500°C**,
- **Hydrogen/Deuterium** partial pressure between **0.1 Pa and 500 Pa.**

Sampling Realization

Al_2O_3 has been deposited on sample disks of **40-50 mm** diameter and thicknesses between **0.5-3 mm** by means of Atomic Layer Deposition (ALD) and by Pulsed Laser Deposition (PLD). Three types of steels - AISI 316L, Nitronic 50 and Incoloy 800H - have been employed as substrates. Prior coating deposition, substrates were subjected to electropolishing to improve the surface finish (SEM has been performed after the treatment to check the surface quality).

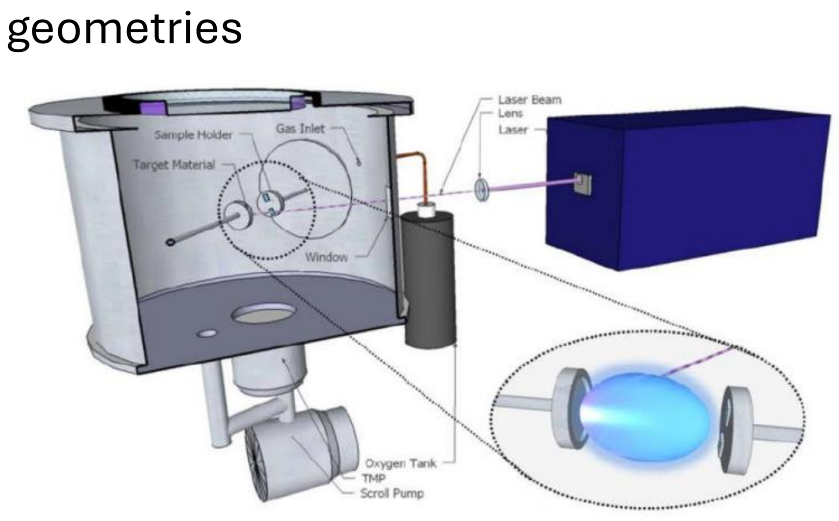
PLD

High-power laser vaporizes atoms from a target material housed in a vacuum chamber. The ablated material condenses on the substrate resulting in a film growth [6].

✅ **Versatile** → compatibility with a variety materials with high reproducibility and deposition rate

✅ Deposition of **dense and crack-free amorphous** thin **films**

❌ **Line-of-sight** technique→ suitable for planar or simple geometries



Schematic representation of the PLD equipment

ALD

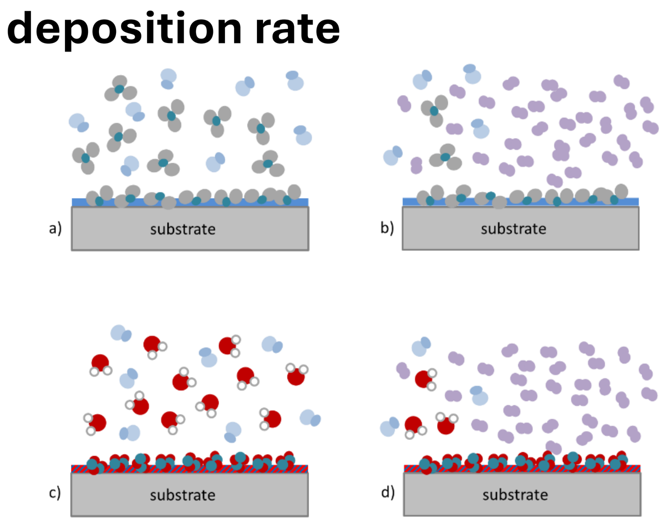
Alternated insertion of:
1- Gaseous precursor that is chemically absorbed on the substrate;
2- Coating material reacting with the precursor.
These steps are repeated in succession until the target thickness is reached [7].

✅ Accurate **control of the thickness**

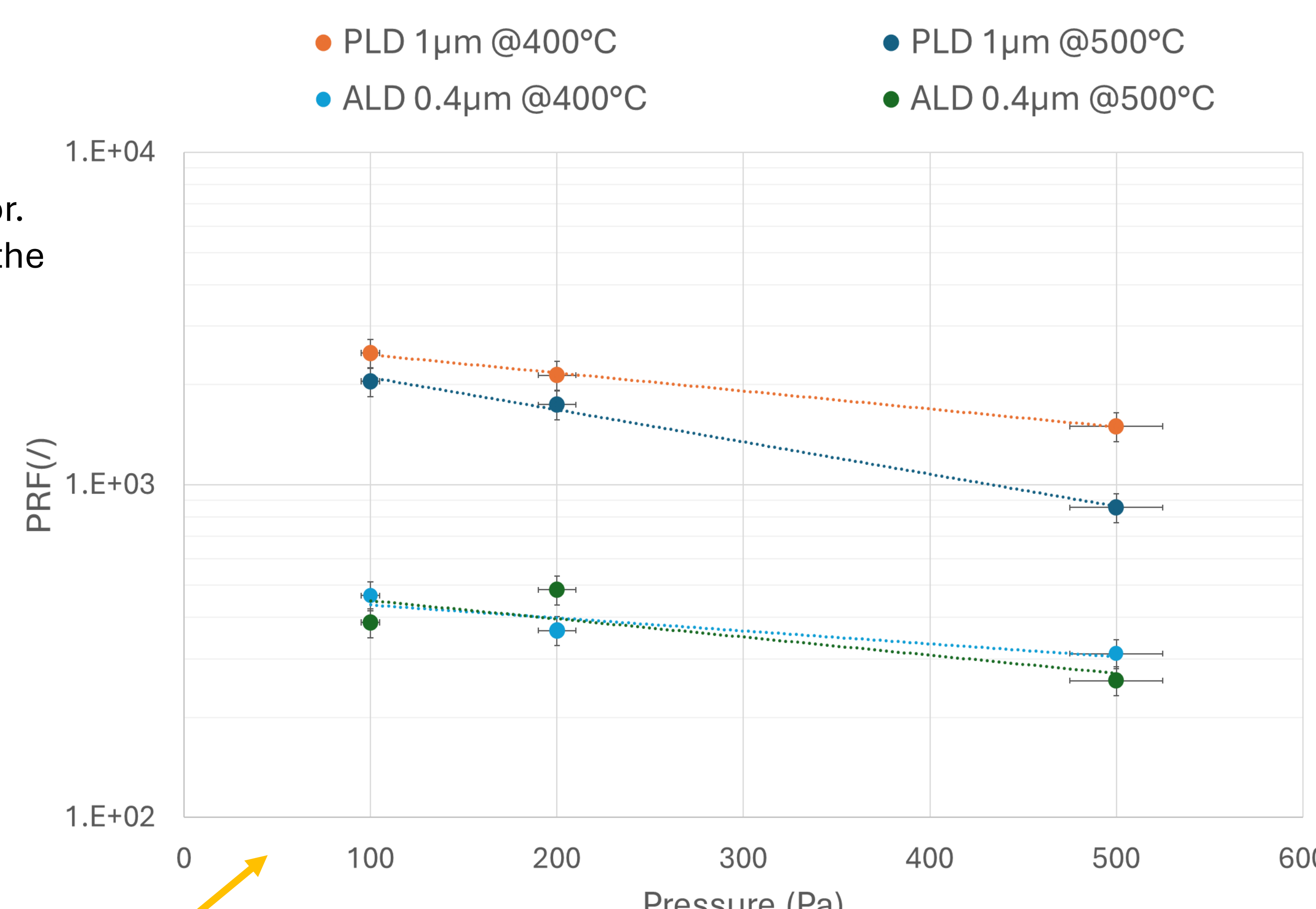
✅ **Large batch processing capability**

✅ **Uniformity on complex surfaces with minimal access**

❌ **Low deposition rate**



Description of an ALD cycle



Experimental Results

Good comparison has been evidenced with literature data on pristine samples [8], [9], [10]. **PRFs** have been measured in the order of **10^3 for PLD** and **10^2 for ALD.** A **suited substrate surface preparation protocol**, capable of removing the carbonaceous contaminations present on steel surfaces (responsible for the uncoated spots), **could unleash a higher potential for the ALD technique.**

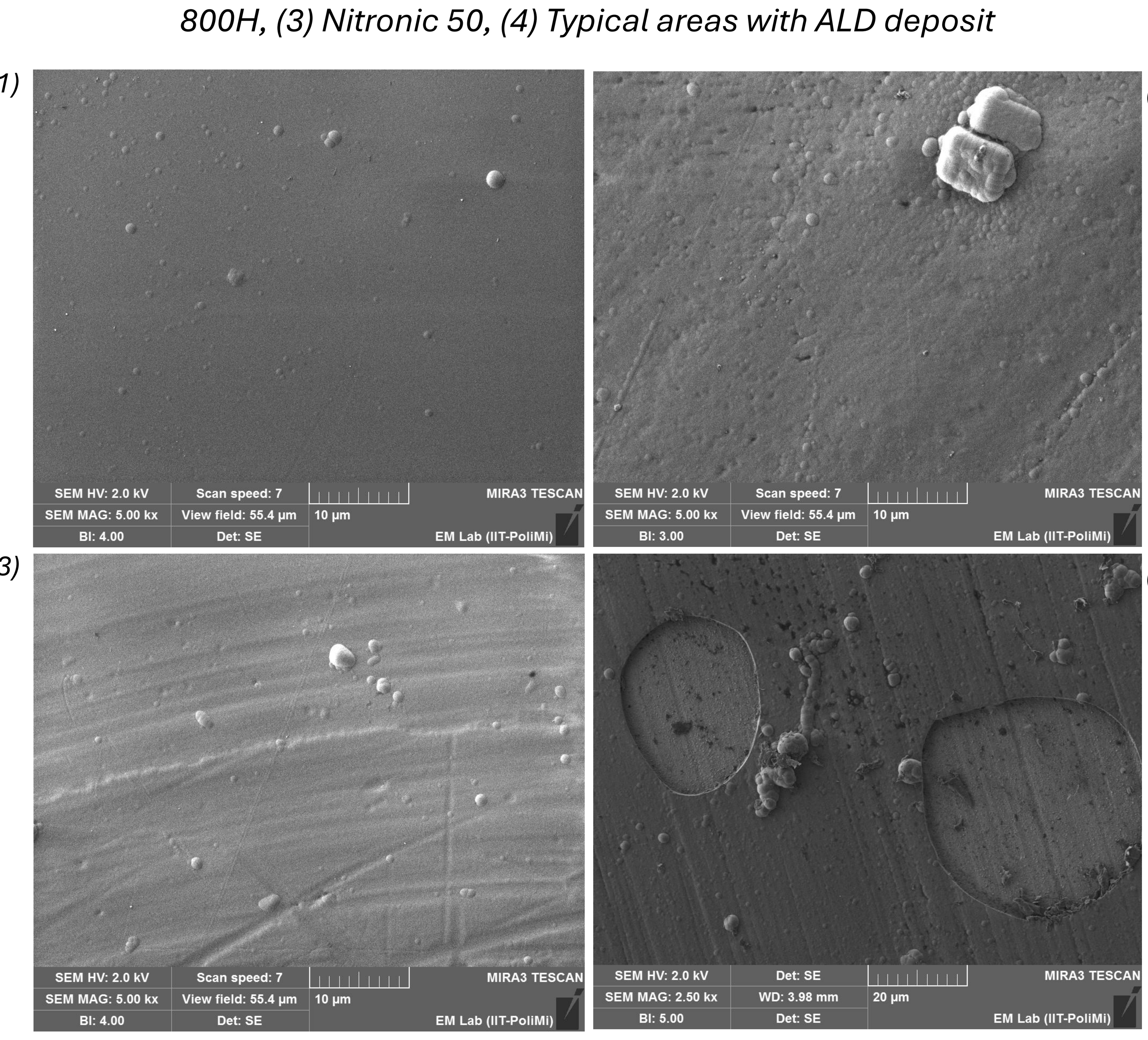
Post measurement characterization of the coatings have been performed in ENI laboratories by:

- **SEM** microscopy to check surface morphology and homogeneity of the deposition;
- **XRD** analysis to detect any crystalline phases;
- **Ellipsometry** to measure sample thickness and indirectly coating thickness;
- **Profilometry** to evaluated the surface roughness of the coated-side.

A portion of the samples has been subjected to thermal cycling or thermal shock processes in x-nano laboratories to verify the stability of the deposition at high temperatures.

Following measurements of ion-induced damage will give insights regarding the amount of Hydrogen trapped in the samples coating.

ALD as-depostied coatings by SEM on: (1) AISI 316L, (2) Incoloy 800H, (3) Nitronic 50, (4) Typical areas with ALD deposit



Need for international standardization for testing protocols

Discrepancies are often observed among experimental results reported in literature, which may arise from:

- Different environmental testing conditions;
- Variations in sampling techniques and pre-/post-measurement characterization;
- Extremely low permeation rates, approaching the instrument's limit of detection (LOD).

A collaborative infrastructure with **shared methodologies** for hydrogen permeation experiments is encouraged to **accelerate the validation of TPB** performance under reactor-relevant conditions and provide valuable support for the design of future fusion power plants.

References

[1] IAEA, NF-T-1.8, 2015
[2] 10.1016/j.fusengdes.2020.111759
[3] 10.1016/j.fusengdes.2021.112573
[4] 10.1016/j.nme.2019.04.001
[5] 10.1016/j.fusengdes.2008.12.091
[6] 10.1007/s11082-022-03786-6
[7] 10.1021/cr900056b
[8] 10.1016/0022-3115(88)90038-4
[9] 10.1016/0022-3115(88)90319-4
[10] 10.1016/S0022-3115(00)00188-4