**2359-24**

**Joint ICTP-IAEA Workshop on Physics of Radiation Effect and its Simulation for Non-Metallic Condensed Matter**

***13 - 24 August 2012***

# R&D and qualification of non-metallic materials for DEMO reactor (long term operation conditions)

S. M. González de Vicente

*European Fusion Development Agreement (EFDA)*
*Garching*
*Germany*

# R&D and qualification of non-metallic materials for DEMO reactor (long term operation conditions)

**Joint ICTP-IAEA Workshop on Physics of Radiation Effect and its Simulation for Non-Metallic Condensed Matter**

**August 21, 2012**

**S. M. González de Vicente**

***Materials Responsible Officer, EFDA***

The Abdus Salam International Centre for Theoretical Physics

IAEA

**Contributions from E. R. Hodgson and R. Vila (CIEMAT)**

# OUTLINE

## INTRODUCTION

1. Optical materials.
2. Dielectric Materials.
3. Breeder Materials: Li-ceramics and eutectic LiPb.
4. Coating materials for Blankets.
5. Future plans

# INTRODUCTION

**ITER , DEMO and any future Fusion Power Plant is really an electromagnetic machine. Therefore once its *structural integrity* can be assured, there is still a great effort to guarantee its *functional integrity.***

**All the magnetic coils, heating systems and safety diagnostics rely strongly on AC/DC electrical and/or optical properties of materials and components.**

**Finally tritium generation also needs dedicated systems and materials.**

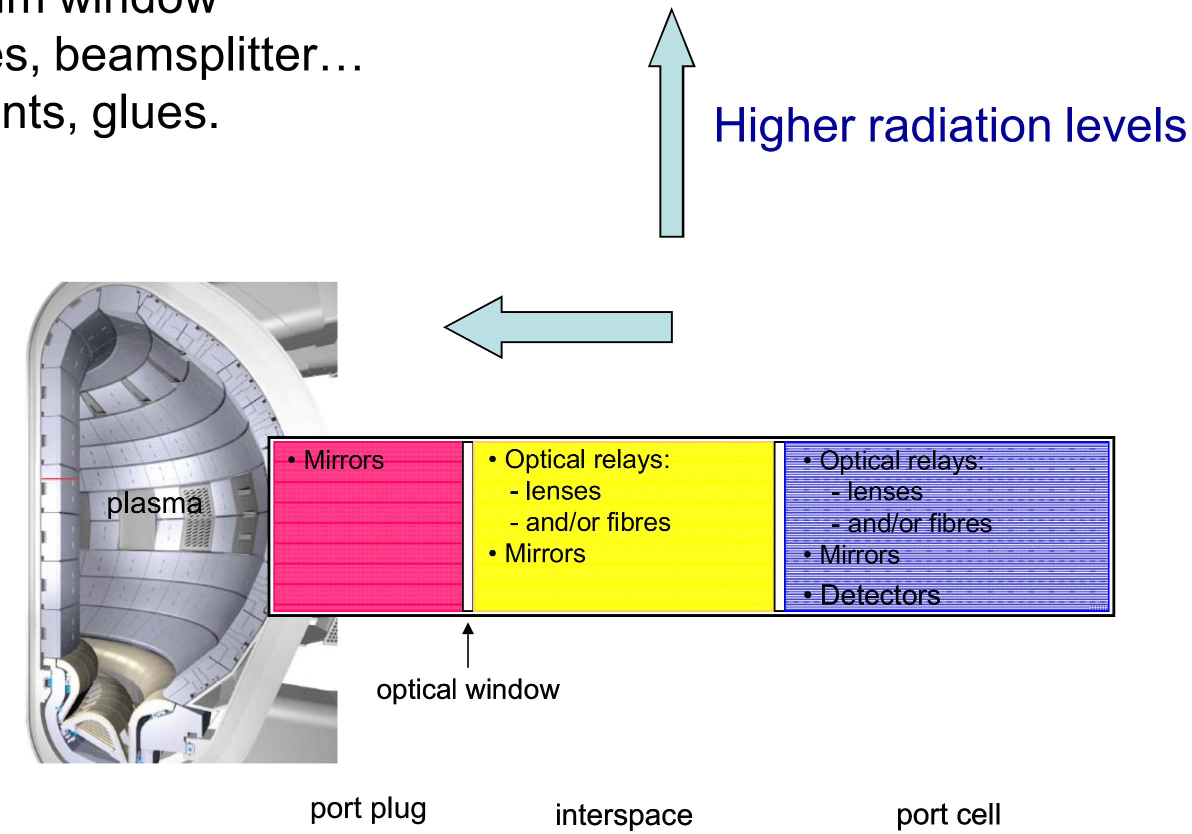
**The integrity and preservation of their specific physical properties under severe environment ( neutron and gamma irradiation, temperature,..) should therefore be a relevant activity inside EU included in the “DEMO Design and R&D ”.**

1. **Optical materials.**
2. **Dielectric Materials.**
3. **Breeder Materials: Li-ceramics and eutectic LiPb.**
4. **Coating materials for Blankets.**
5. **Recommendations: Summary**

# 1. Optical Materials.

## ❍ Application of materials:

* Mirrors ( first and secondary)
* Vacuum window
* Lenses, beamsplitter…
* Cements, glues.




| | | | |
|---|---|---|---|
| Neutron/γ loads (W.m$^{-3}$) | HIGH ($10^3$ to $10^7$) | « LOW » < $10^3$ | « VERY LOW » |

# 1. Optical Materials.

- **Main candidates**:

Refractory metals, Mo, Rh, Ta… (1st mirrors)

Dielectric mirrors (possible secondary mirrors)

Fused silica : **KU1, KS-4V** (broadband window) (now commercially available again)

Diamond (broadband window)

Sapphire (broadband window, birefringent)

Rad. resistant Schott Glasses (VIS and for lower n doses)

Conventional IR transmitting materials (Ge, Si, ZnSe, ZnS, BaF2, CaF2, YAG..)

(also for lower n doses, but better IR properties)

→ (Radiation, T, Atmosphere and Functional/performance requirements) → (But (DEMO is still too unclear)

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# 1. 1 Mirrors ( first and secondary)

All the optical diagnostics and even ECR heating system have mirrors to reach the first wall/plasma → large impact on tokamak operation.

- First Mirrors will suffer, in addition to high n doses, from erosion /co-deposition effects from the plasma.

- Therefore two types of experiments are needed:

1. Experiments as close as possible to DEMO relevant conditions are required for plasma interaction:

- Assessment of the performance of candidate mirrors, such as **sc Mo and rhodium-coated,** under **erosion-** or **deposition-** dominated conditions in existing devices (tokamaks and plasma devices) → material choice.

- **Mitigation of deposition** of Be and C compounds (will exist in Demo?)

→ Develop techniques to decrease and if possible, to avoid C and Be deposition. Options : shutters, baffles, optimization of duct geometry and gas puffing to limit particle flux to mirrors. The issue of Be deposition is still an open question that must be addressed.

- Cleaning of deposited layers - mirror surface recovery.

→ Develop efficient, simple and robust technique to clean mirror surfaces from deposits. Techniques include both plasma- and laser-based cleaning techniques. Otherwise, the development of an engineering solution for quick mirror replacement will be necessary.( We did not found a working system for DEMO like)

# 1. 1 Mirrors ( first and secondary)

- Erosion and deposition modelling is required for the geometry and different first-mirror positions in DEMO structure under plasma operation conditions, including impurity transport towards the mirrors.

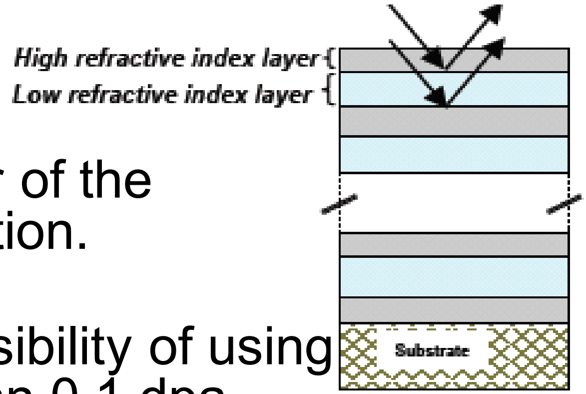
2- Experiments based on neutron irradiation effects. (in-reactor testing)

- Predictive modelling of irradiation effects and their impact on the optical characteristics of mirrors.
  → Simulate and assess impacts caused by alpha, gamma and neutron irradiation on the optical and surface properties of the main candidate mirror materials.

To summarize, the determination of life-time of plasma facing mirrors used in optical systems in DEMO operation conditions must be studied.

*Nevertheless is a field quite active compared to other functional materials.*

# 1.1 Dielectric mirrors




- Further work to better understand the behaviour of the microstructures of these materials under irradiation.
  (example: developing interlayer stress)
- Some irradiation experiments suggests the possibility of using dielectric mirrors to much higher dose levels than 0.1 dpa.
  Hence, **higher dose neutron and gamma irradiation** experiments would be needed.
- The size of typical diagnostic mirrors (Ø ~100 mm) will be much larger than that of the standard test samples used for irradiation experiments. Therefore, similar tests should be performed with the **prototypes of diagnostic mirrors**.
- Requires knowledge of **heat transfer** to cooling device, as different crystallization threshold of the layers can play a role at temperatures over 230ºC.
- The use of a mirror substrate whose irradiation-induced changes are closely matched to the behaviour of the mirror leads to a more radiation tolerant system. This behaviour should be assessed by new neutron irradiation campaigns.

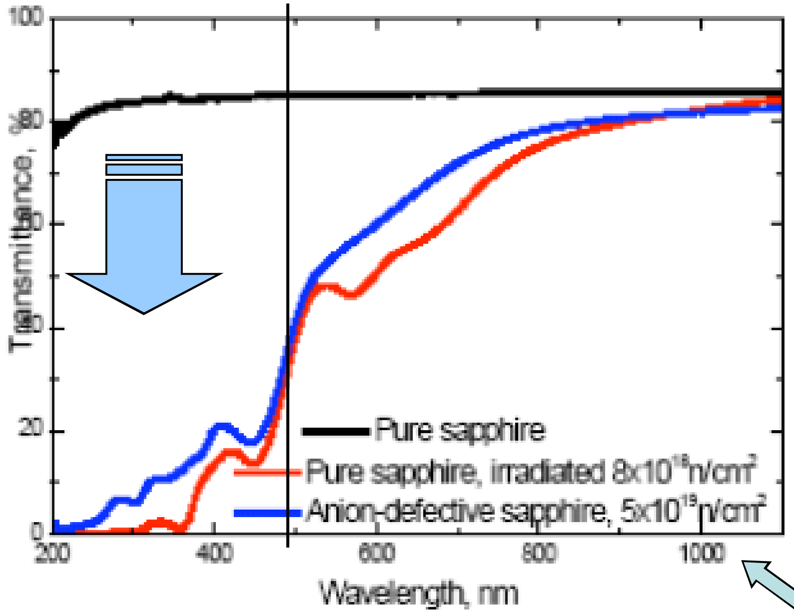
# 1.2 Vacuum Windows

Needed for a dual purpose: Optical **broadband** (normally VIS_IR) + **vacuum** tight window (severe conditions)

- Combination of wide spectral transparency and mechanical strength

High dose neutron irradiation of Sapphire

~$10^{23}$ n/m²



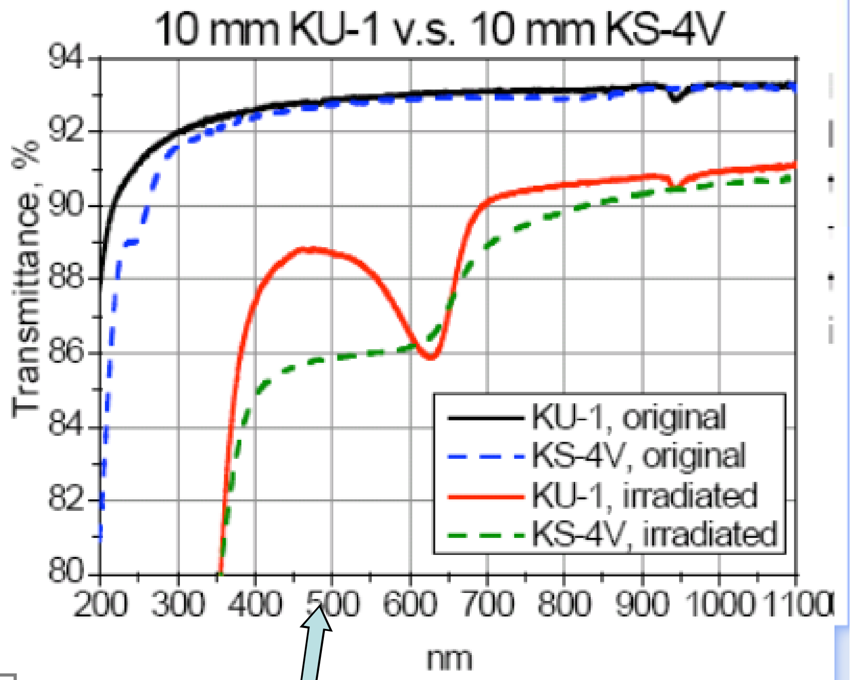

From Orlovskiy. IEA meeting 2011

Total loss in %T in UV
severe loss in VIS

High dose neutron irradiation of SiO2

~6.$10^{20}$ n/m²

10 mm KU-1 v.s. 10 mm KS-4V




Total loss in %T in UV
Low loss in VIS
( BUT lower dose)

# 1.3 Optical components.

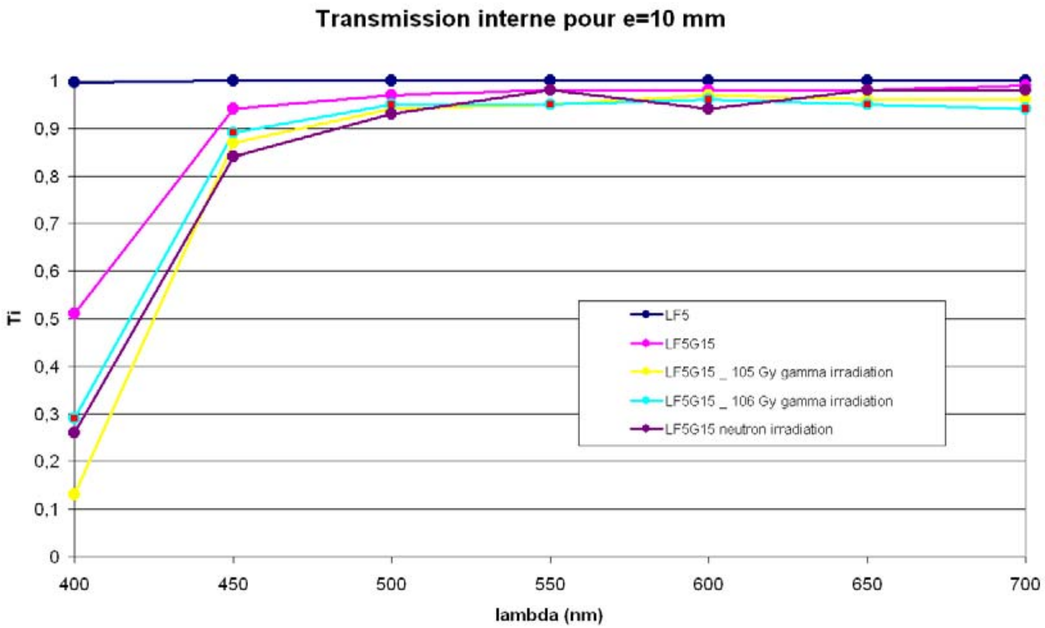
In this case Optical properties are dominant. Most will work **in only one range :** UV**,** VIS or IR.

- Optimal spectral transparency
- Wider Refraction index range (crown /flint )
- But lower radiation hardness.

Typical commercial solution

Example
Schott LF5 G15**.**
**n & gamma test**

Test neutrons : fluence < **1,5.E20** n/m²
Test gammas : 1E4 Gy, 1E5 Gy, 1E6 Gy




No high dose irrad.

Schott data

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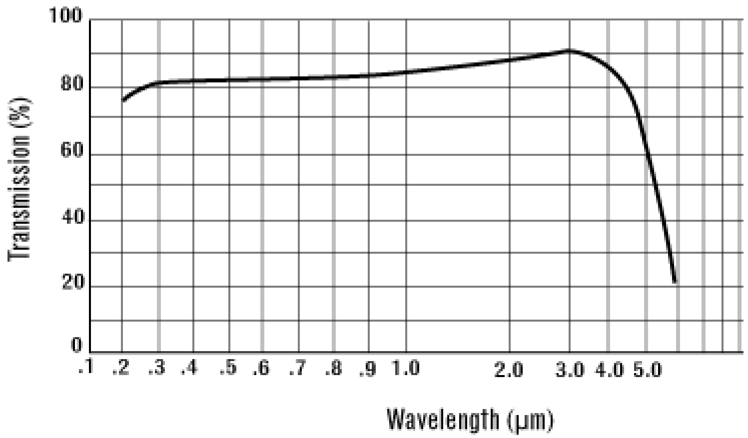
**A special comment must be made in the case of IR >3µm.:**

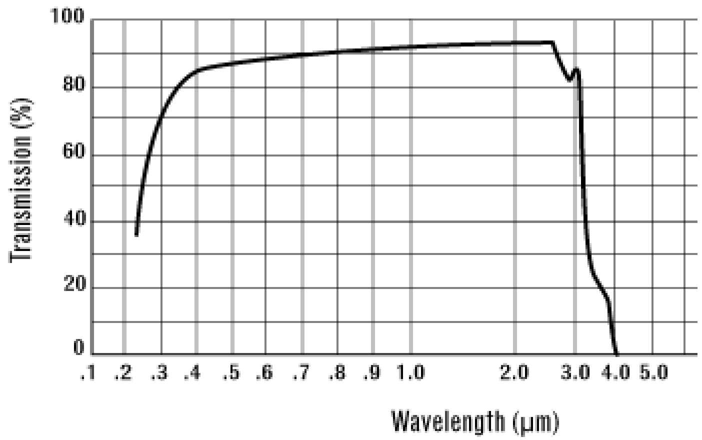
For conventional IR transmiting materials (Ge, Si, ZnSe, ZnS, BaF2, CaF2, YAG) **There are almost no published data about transmission of irradiated IR materials**

Typical Fusion Window materials are not very performant in 3-5 µm

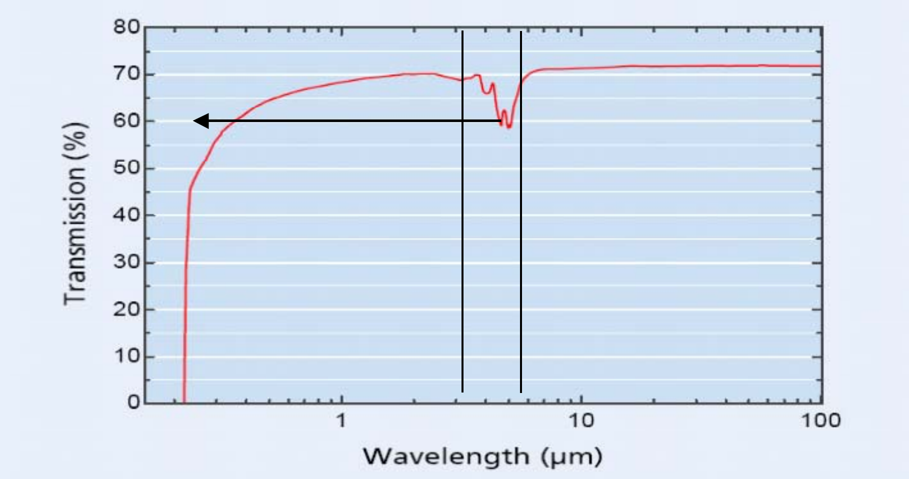
**Sapphire (Al2O3 )**



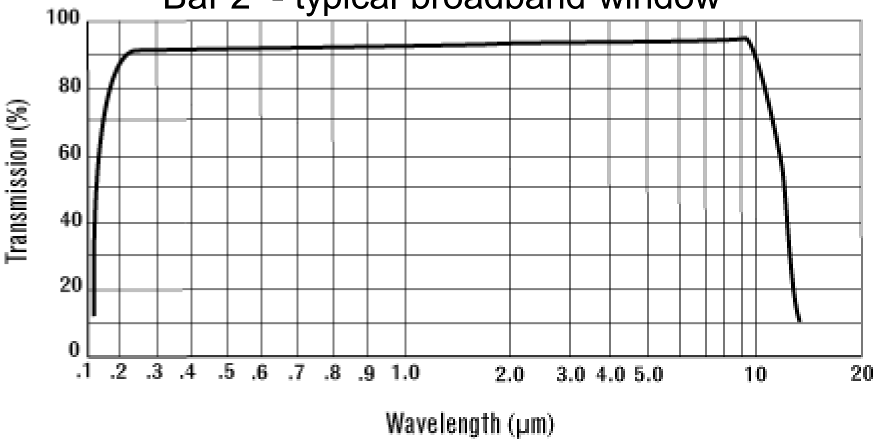

**IR-grade Fused Silica ($SiO_2$)**



**CVD Diamond** is a better choice for IR transmission although it has some intrinsic absorption just in the 3-5 μm region




BaF2 - typical broadband window




There are better traditional materials for IR, BUT radiation effects are not well known in this range

# 1.4 Coatings and glues

Coatings and glues needed for lenses will be located in medium or low irradiation areas in the optical diagnostics chain:

- Only space qualification experiments have been found, but the dose for these experiments are lower than that expected in fusion devices.

  → Some irradiation experiments are required to the levels of gamma and neutron fluences expected in the structure of DEMO or similar fusion reactors.

  → For optical and/or for mechanical properties (and with larger samples)

1. Optical materials.
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# 2.Dielectric Materials.

**These are the core of the DC electric and AC/RF dielectric applications**
(**H&CD** and **diagnostic** systems)!

- very broad range of frequencies: → DC to 170 GHz !
- Located in many different configurations.

⇓

- Relevant conditions; dose rates and doses, temperature, applied V ...
- Radiation effects/damage of concern.

- Present situation; experimental data (EU, JA, RF, US) exists for: **$Al_2O_3$, MgO, BeO, $MgAl_2O_4$, AlN, $Si_3N_4$, $SiO_2$, diamond**

But most available data limited to ≤ 0.1 dpa
However sufficient to indicate potential problem areas

- Testing programme needed

# What we need to measure?

- For DC: Conductivity ( and its degradation =RIED)
- For AC/RF: Insulator subjected to an electric field E of frequency ω (and with permittivity ε)

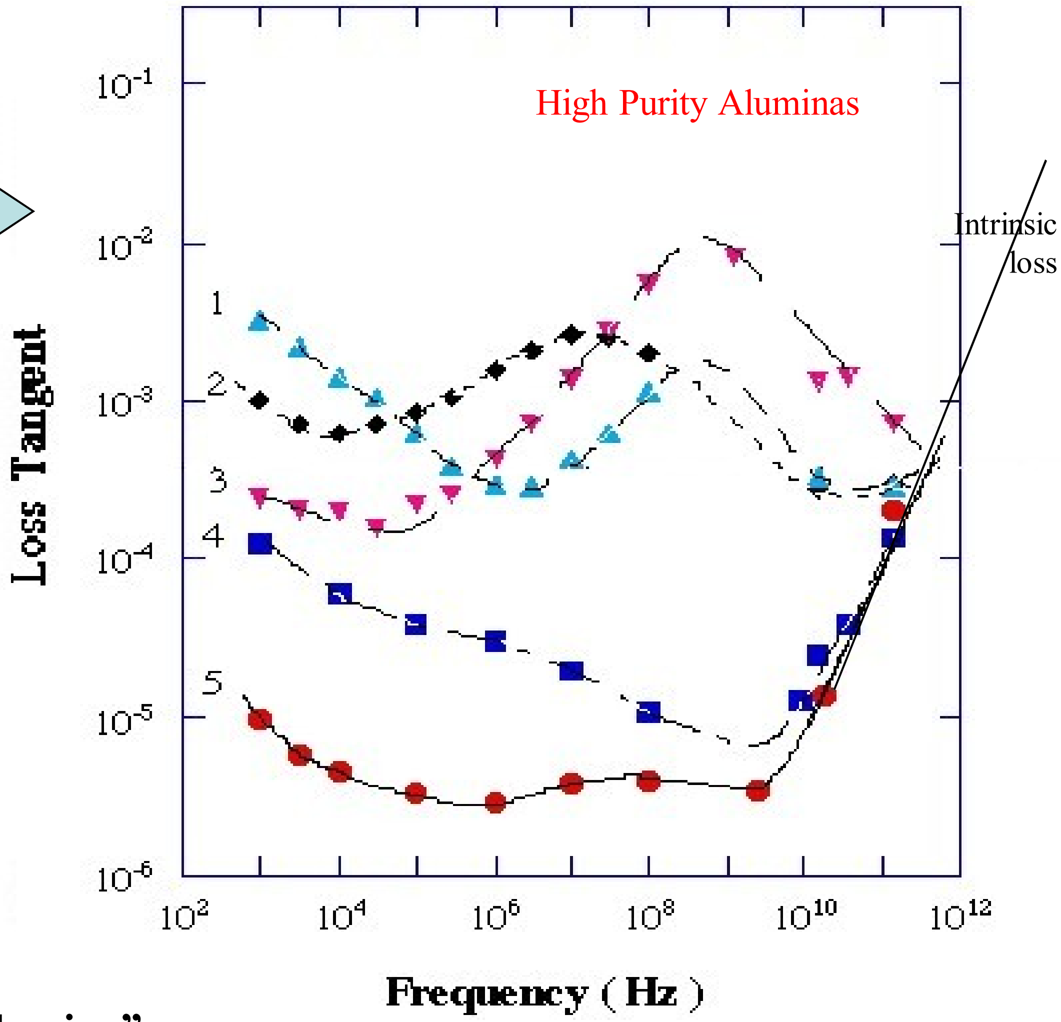
**Absorbed Power** in dielectrics = $E^2 * \varepsilon * \omega * \tan\delta /2$

**Loss Tangent** = Key Factor

**Loss Tangent change orders of magnitude with material , T, frequency…**

# Wide variation in dielectric loss with material purity

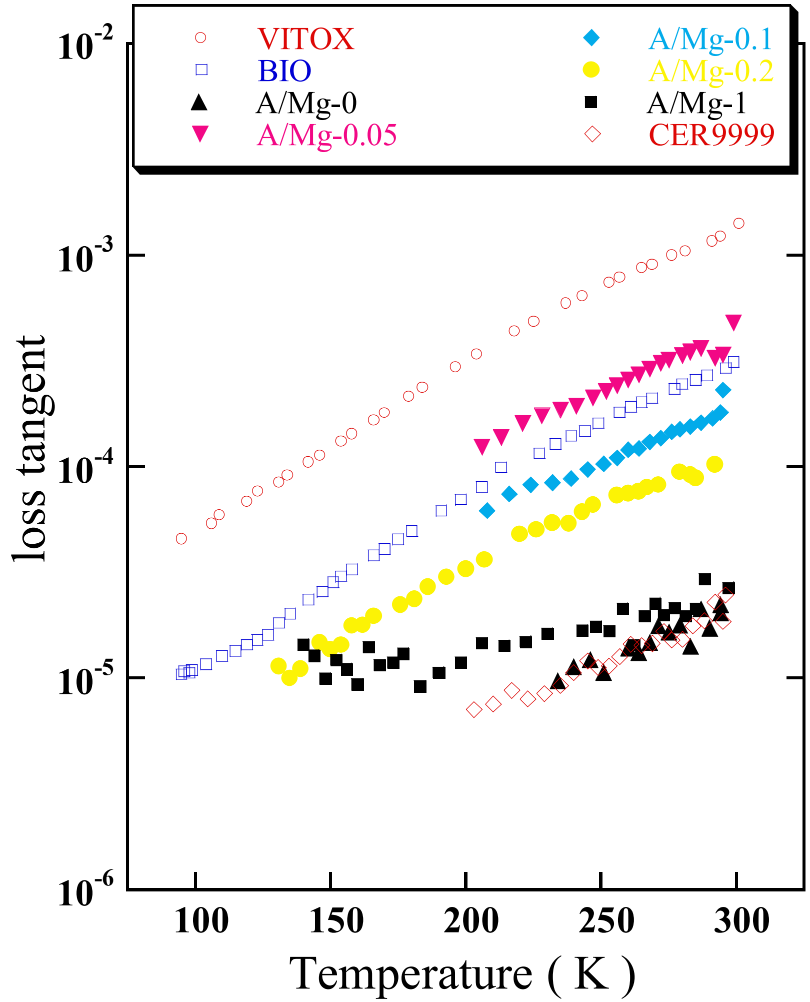
- HP Aluminas show a wide range in loss!
- High purity materials : best – S.C. sapphire
- Need to measure over a wide frequency range.
- Impurity related (Mg, Fe,...) at ppm levels

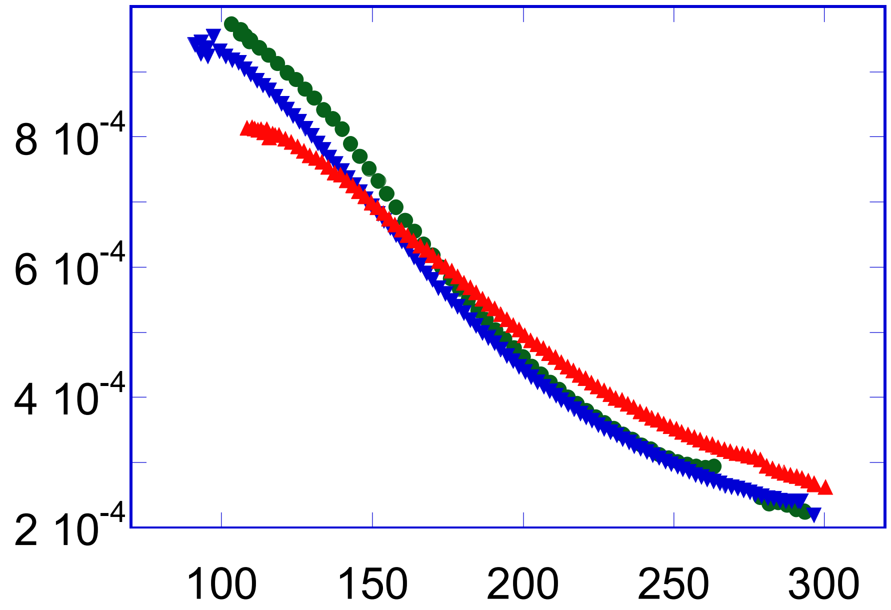



Concept: we cannot speak of Tanδ of "alumina"

# T effects

Alumina ——— ←-→ Silica




Important implications for Fusion applications !

# Radiation levels for insulators

- **Beyond ITER** (1st wall: DEMO ≈ 80 dpa, PP ≥ 150 dpa)
  Dose rates probably very similar (1st wall loading limit)
  Ionizing doses up to 100s GGy
  Displacement doses: some dpa's

We do not have results even close to these values!

| For Insulators | Dose dpa | Dose GGy |
|---|---|---|
| ITER | < 0.3 | < 10 |
| DEMO | ≈ 8 | ≈ 250 |
| PP | > 15 | > 470 |

# Temperatures

Nuclear heating ≥ hours ⇒ Components at > 200 C (?)

**Also - most in vacuum (influence observed)**

# 2.Dielectric Materials.

## Possible / expected effects ITER to DEMO/PP

- **Dose rate**

  Several studies: Initially behaviour very similar to ITER, but as damage accumulates should expect changes in the dose rate effects on physical properties

- **Dose**

  For the considerably higher doses (Gy and dpa) can expect important influence of extended defects and structural damage.

  Changes in the physical and thermo-mechanical properties

- **Transmutation**

  Nuclear reactions will introduce "impurities", for DEMO/PP will become important

**Maximum appm/year transmutation products for alumina (1 MW/m$^2$ 1st wall)**

| | H | He | C | N | Na | Mg | Si |
|---|---|---|---|---|---|---|---|
| ITER | 29 | 50 | 62 | 3.4 | 7 | 44 | 1 |
| DEMO | 774 | 1340 | 1660 | 91 | 187 | 1170 | 27 |
| PP | 1470 | 2550 | 3150 | 173 | 355 | 2220 | 51 |

(Based on L.H. Rovner and G.R. Hopkins, 1976)

## 2. Dielectric Materials.

# Work has been focused on ITER and few on long term

Radiation effects in insulators for **H&CD, diagnostics, and RH**

Mainly physical properties:

- Electrical conductivity - **RIC**, **RIED**, surface effects
- Dielectric loss and permittivity - Hz to GHz (only 1 in-reactor)

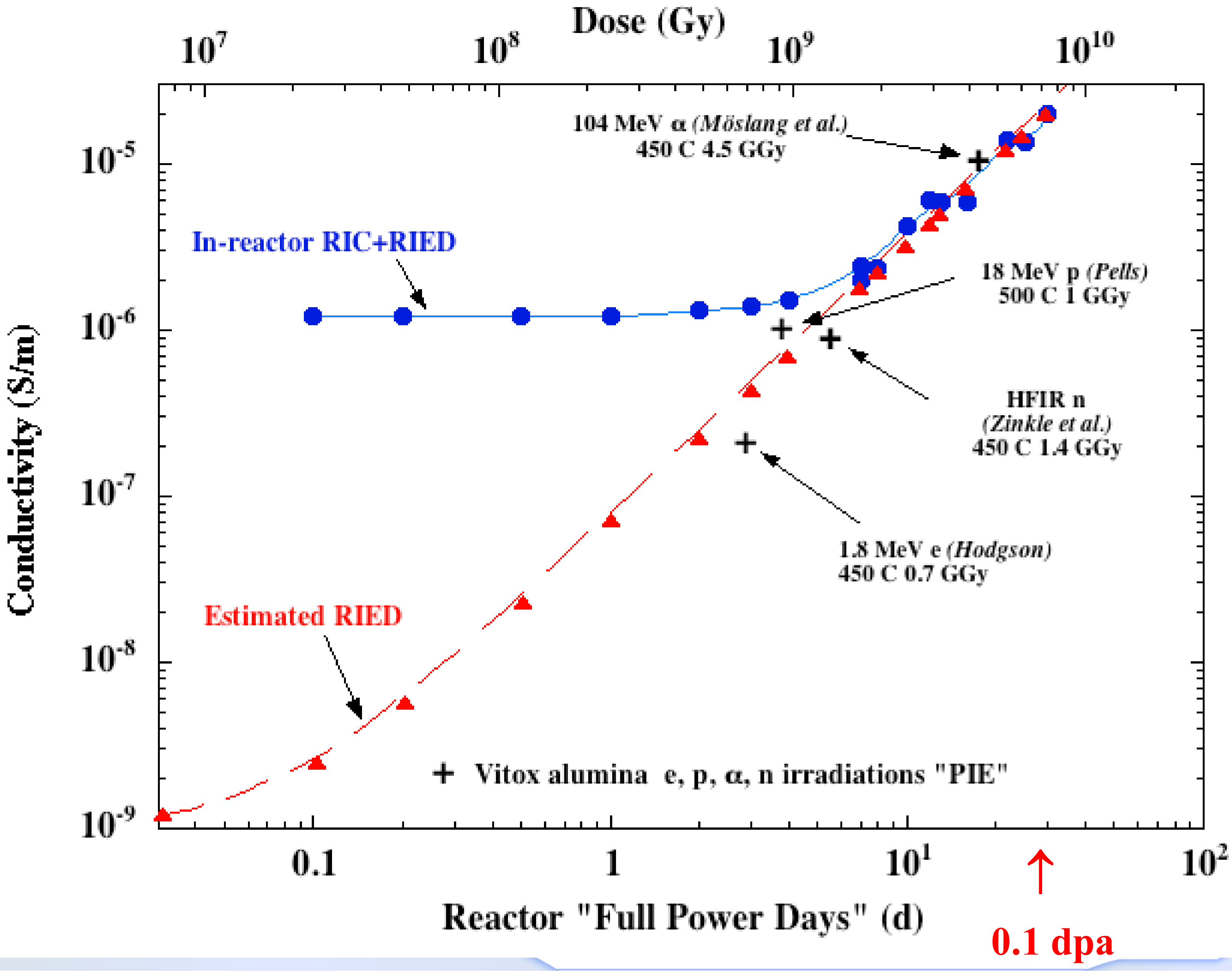
Limited recent work on thermo-physical and mechanical properties

- Thermal conductivity - diamond, silicas
- Mechanical strength – vacuum test, polymers/elastomers

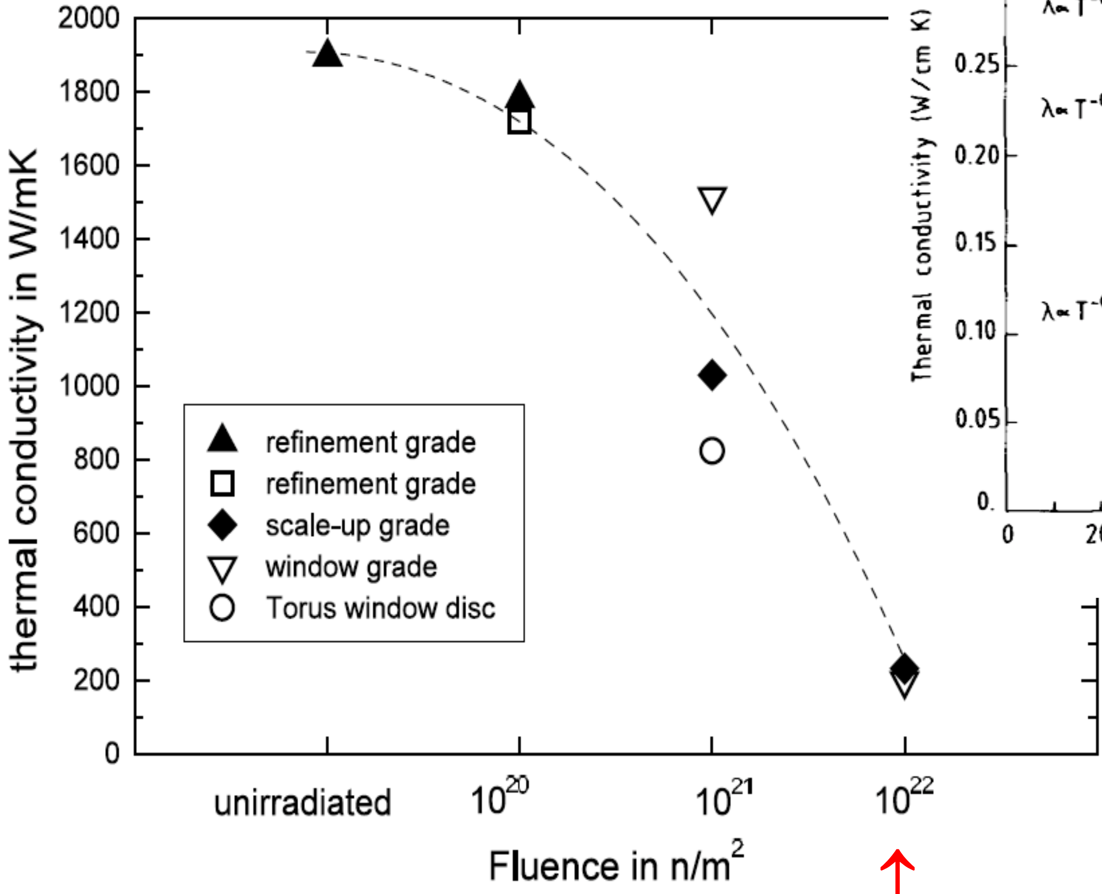
Other Components:

- Bolometers, pressure gauges, MI cables, Hall sensors,...
- Also many components for **RH !**

# JMTR in vac RIED *Kyocera A479SS* 510 C 500 kV/m *(Shikama et al.)*

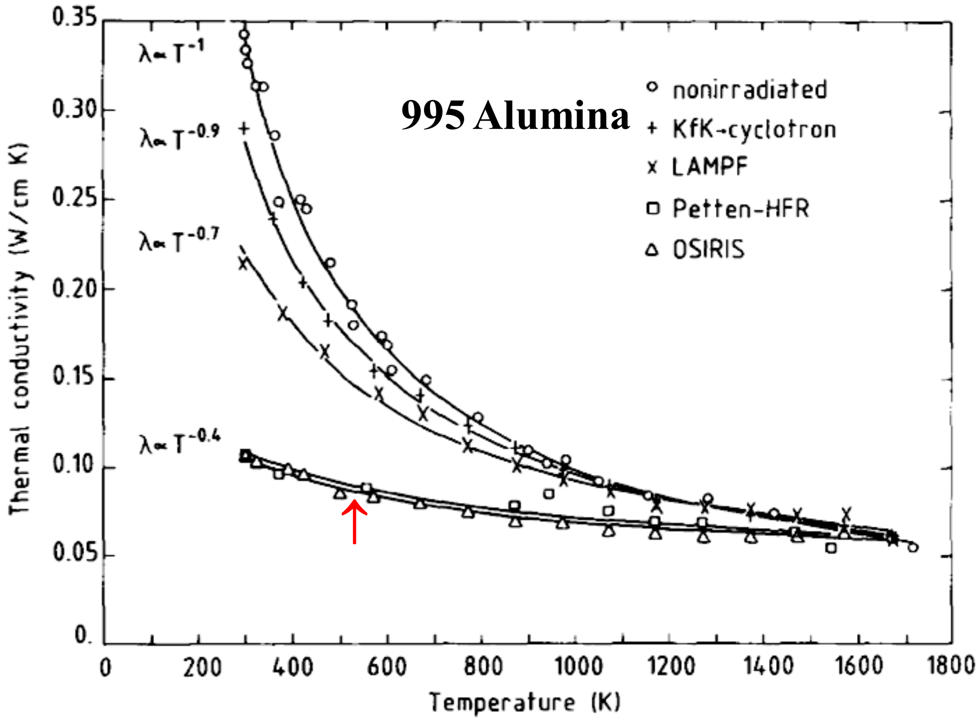


# Thermal conductivity reduction
# Neutron irradiated window grade CVD diamond and Alumina




(Heidinger et al. FED 2001)

**0.001 dpa**




Irradiation conditions

| | Fluence (part./cm$^2$) | Max. energy (MeV) | Irradi-ation temp. (K) | Damage dose (dpa) |
|---|---|---|---|---|
| KfK-Cyclotron | $1 \times 10^{17}$ | 100 | ~ 700 | ≤ 0.001 |
| LAMPF | $5 \times 10^{20}$ | 100 | ~ 600 | 0.5 |
| Petten-HFR | $2.8 \times 10^{20}$ | > 0.1 | 473 | 0.4 ← |
| OSIRIS | $2.1 \times 10^{21}$ | > 1 | 823 | 5 ← |

(Rohde & Schulz JNM 1990)

2. Dielectric Materials.

# Present knowledge / data base

## DC applications

- Best available **general insulating** material is alumina (high purity, small grain size)
- RIC < 10-6 S/m @ 1 kGy/s
- Limitations in use set by RIED:
- T ≤ 200/250 C
- E < 1 MV/m
- At these temperatures thermal conductivity reduced to lower saturation limit by < 0.4 dpa
- Surface degradation is important

## AC/RF applications

- For frequencies ≤ IC alumina is again the best choice
- Alumina (and sapphire) keep loss < $10^{-4}$ up to 0.1 dpa (PIE data)
- Limitations in use set by RIED:
  T ≤ 200/250 C
  E < 1 MV/m
  BUT very little *in situ* data
- At these temperatures thermal conductivity reduced to lower saturation limit by < 0.4 dpa
- Again, very few *in situ* measurements
- For ECRH, diamond is at present only option. BUT thermal conductivity degradation is a serious problem
  → It will depend on design values and conditions

# DEMO/PP future testing programme

- **<u>Where</u> - Present irradiation testing facilities:**
  **Fission reactors with accelerators for backup role**

- **<u>What</u> – Actual candidate materials to extend and complete database.**

- **<u>How</u> - Over the years experience gained, and solutions found for in-situ testing of insulating materials**

  ***BUT so far solutions not 100 % satisfactory hence more work needed.***

  **first development / improvement of systems required**

  ***In reactor + temperature control + in vacuum + electrical connections***

- **MUST examine RIC & RIED to high dose.**
- **The relevance of surface degradation needs to be examined**
- **Effort to make *in situ* loss tangent measurements (again !)**

# Conclusions

1. We will need ceramics/insulators in DEMO and beyond:
Severe limitation to operation, control and safety functions.

2. Assuming a Tokamak type device, extrapolate expected conditions:
Dose, dose rate, temperature, environment etc..

3. Most available data limited to $\leq$ 0.1 dpa, but sufficient to indicate problems.

4. To move ahead quickly need presently available irradiation testing facilities:
i.e. fission reactors with accelerators for backup role.

5. *In situ* testing is essential.

6. Modelling was started years ago with encouraging results ($Al_2O_3$, $SiO_2$) but this work vanished years ago.

7. Make best use of experience and available expertise for in-situ testing:
Problems to be faced; temperature control, vacuum, applied electric field ...

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# 3.1 Lithium Ceramics as Solid Breeder

**Main candidates: Li-Silicates and Li-Titanates**

**Proposal of other lithium-rich ceramics**

In order to increase the lithium inventory, other compounds enriched with lithium can be interesting such as:

— $Li_6SiO_5$
— $Li_8SiO_6$
— $Li_4TiO_4$
— $Li_4Ti_5O_{12}$

Mixed compounds corresponding to the system LiO-SiO2-TiO2, improving the structural integrity of the pebbles.

→ Different compositions suggested: $Li_4SiO_4/Li_2TiO_3$ and $Li_4SiO_4/Li_4TiO_4$.

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# 4. Coatings for structural steels

**Review of coating activities towards a double purpose in DEMO blankets: corrosion and T-permeation barriers**

**- Candidate technique: Aluminium rich layer and subsequent oxidation heat process→ $Al_2O_3$**

*Techniques:*

HDA (hot dip aluminizing) and CVD aluminizing processes.

CVD coatings interaction with the Pb-Li eutectic ⇒ layer degradation

Experiments very limited ⇒ laboratory conditions.

Only small metal pieces have been coated.

**- Fe-Al-scales which act simultaneously as T-permeation and corrosion barriers.**

*Techniques:*

HDA: Great influence of the diffusion temperature in the formation of the coating.

Advantages: the layer act as T-permeation and corrosion barriers.

Disadvantage: Method no reproducible at industrial scale.

# 4. Coatings for structural steels

Nowadays. The **electrochemical deposition** technique has been developed to obtain a reproducible method and suitable to industrial processes on large and complicated structures.

- A very limited research has been performed on self-healing techniques and clearly further experiments are needed.
- Scarce studies on coatings for structural steels. Further effort is necessary.
- The next step would be the design and the realisation of irradiation experiments.
- Once the proper coating has been obtained it is necessary to submit the coated samples to neutron irradiation and even to study the behaviour of these samples under operation conditions (Tritium permeation and/or Li Breeder corrosion)

# 4. Coatings for structural steels

- More work is needed to optimize heat treatment in RAFM steels to achieve industrial relevance and be able to coat 3D structures like boxes used as breeding units
- Compatibility studies of the structural materials in contact with Li ceramics breeders should be systematically evaluated : corrosion attack and the need of coatings on the HCPB concept.
- Development of corrosion barriers is essential for elimination of loop blockage risk due to precipitations.
- Corrosion testing of newly developed / manufactured Al barriers.
- Further work in 2012 is including review on **insulating coatings** from MHD point of view.

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# General conclusions / Future works to be done

- “In-situ “ measurements are mandatory for insulators , RF components, and also at higher doses.
- Some data are *strongly* absent, as radiation effects in IR bands (3-5 and 10-12 μm). Very few on *in-situ* loss-tangent.
- The general view is the extension from ITER to DEMO is far from completed. We should start before IFMIF for functional materials: normally n spectrum is softer and we can use reactors and accelerators.
- Future definition of H&CD systems and diagnostics included in DEMO will help to reduce the range of functional materials, but until then we need to research on all of them, in case DEMO design change or we find some *show-stopper*.

# Thank you for your attention!

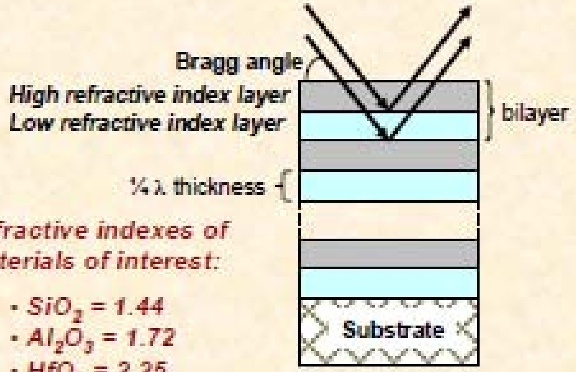

*Courtesy of C. Alejaldre*

# Dielectric Mirrors (background theory)

Coatings which allow the reflection of a specific wavelength, while blocking others. Composed of alternating multilayer films of high and low refractive index materials of quarter wavelength thickness.

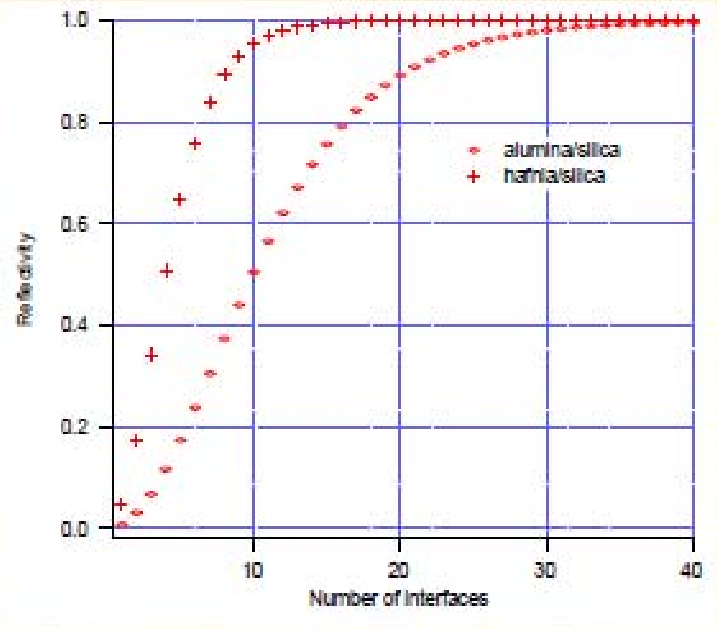
*The reflectivity (R) of a lossless multilayer stack of N successive quarter wave layers of alternating high ($n_{Hi}$) and low ($n_{Li}$) refractive index.*

$$R = \left(\frac{1-b}{1+b}\right)^2 \quad \text{where} \quad b = \Pi_{i=0}^{N}\left[\frac{n_{Li}}{n_{Hi}}\right]$$




*Refractive indexes of materials of interest:*

- $SiO_2 = 1.44$
- $Al_2O_3 = 1.72$
- $HfO_2 = 2.25$




*Calculated reflectivity of two UV coatings vs number of interfaces (number of interfaces is equal to twice the number of layer pairs)*