Proposal of a testing procedure to qualify ITER window assemblies exposed to high microwave stray radiation

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Abstract

In any Tokamak, the window assemblies are essential to preserve the conditions for successful experiments and to guarantee adequate access for inspection and measurement. In ITER, the materials traditionally used for the windows will be exposed to an exceptionally harsh environment. Moreover, the systematic use of tritium as fuel would make any failure of the primary vacuum containment a particularly dangerous accident. It is therefore essential to understand the potential threats to the integrity of the windows assemblies and define a series of tests to ensure their properties and quality before installation. One specific hazard to the windows is the microwave radiation due to either heating schemes or specific diagnostics. In this paper the main causes of degradation, which could lead to failures under microwave loads, have been identified. A series of laboratory tests have been defined, to assess the quality of the materials and the assemblies, including the coatings for the absorption of the microwave radiation in the ducts leading to the windows. Complete testing procedures and an overview of the main facilities, where the assemblies and materials could be qualified, are also provided.

1. Introduction and background

In any magnetic confinement device for thermonuclear fusion the primary vacuum windows are essential to preserve good conditions for their operation and to guarantee accessibility for measurements. In ITER, they have also important safety implications, given the nature of the fuel to be systematically used: a mixture of deuterium and tritium. Their long-term protection is crucial essential for both the operational safety of the machine and its scientific exploitation.

Although window assemblies have been used for a long time in fusion facilities, there are not many qualification records available. In ITER, the window assemblies will be subjected to much more severe loads than in present devices. Moreover, the diagnostic window assemblies differ in size, material, material grade, assembly type, etc.

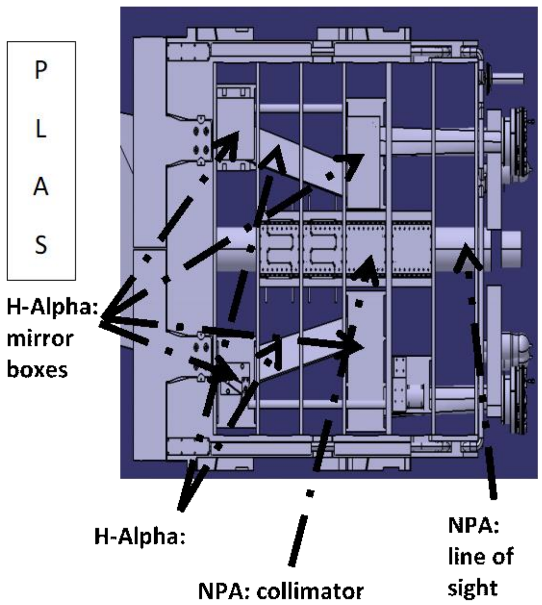

Figure 1: A view of DMS1 port #11.

In ITER, stray radiation at 170GHz and 60GHz is an engineering threat to the integrity of various window assemblies [1, 2]. This work covers the assessment of Electron Cyclotron Resonant Heating (ECRH) and Collective Thomson Scattering (CTS) stray loads, impacting the windows of two diagnostic port plugs, #11 and #12, considered some of the most affected. The diagnostics connected to these port plugs, which may need additional protection, are: a Neutral Particle Analyser (NPA), the Low Field Side Reflectometer (LFSR), Vacuum Ultraviolet (VUV) and H-alpha spectrometers. Indeed, exposure of the windows to ECRH/CTS stray radiation for the duration of a discharge can result in an increase in window temperature and consequently in excessive stresses. Therefore, particular attention must be devoted to the design of the diagnostic conduits, including suitable absorbing coatings, since they can act as transmission lines for the stray radiation [3].

Heat absorption in the window assemblies, due to Electron Cyclotron Resonant Heating and Collective Thomson Scattering radiation exposure, is correlated to two physical material properties: dielectric permittivity and loss tangent.

With regard to the structure of the paper, next section presents the estimates of the loads on the various diagnostics due the EC stray radiation. Section 3 summarise the property of the windows material, fused silica and the main types of tests required to qualify it. The measurements of the windows dielectric properties are covered in Section 4 and an overview of the irradiation tests to be foreseen is provided in Section 5. The summary of the tests to be planned for the window assemblies is the subject of Section 6. The issues presented by the absorbing coatings, to be implemented in some ducts, are described in Section 7. The global tests to qualify the coatings are covered in Section 8, while the measurements of their surface properties are overviewed in Section 9. Section 10 contains the conclusions about the tests procedures of the coatings. Appendix A provides an overview of the facilities, which could be considered for the irradiation tests.

2. Estimates of stray radiation

In ITER, Electron Cyclotron (EC) radiation will be injected from four upper ports and one equatorial port. Its main functions will be heating, stabilization of NTM (Neoclassical Tearing Modes), assisted break-down and current drive. At 170 GHz it is planned to couple to the plasma a maximum of 20 MW of microwave power. In normal operation without unforeseen events, most of the injected power will be absorbed by the plasma, but it is unavoidable that a small fraction will reach the diagnostic windows. Since they are made of dielectric materials, the window discs can absorb a non-negligible fraction of the stray electron cyclotron radiation.

The expected power density of ECH stray radiation striking the First Wall (FW) apertures was evaluated in [1] for different situations:

- Direct beam during start-up: 3 MW/m² for up to 5.5 s around Eq. Port #11 and #17.
- Background power during start-up: 0.13 MW/m² for up to 5.5 s on all FW apertures
- Cross-polarised beam: 1.25 MW/m² localised on the Low Field Side (LFS) FW, around the diagnostic Equatorial Ports #11, #12 and #17.

More details about the expected stray radiation loads at the wall are reported in Table I. The largest power densities at the FW are particularised for mode and port. As shown in the table, the highest stray power densities at 170 GHz are due to the cross polarized and unabsorbed co-polarized ECRH radiation on the inner Wall. The former amounts to 1.25 MW/m^2 for ports #11 and 12; the latter can reach 3 MW/m^2 [1] for ports #11 and #17 but only for 5.5 s during the plasma breakdown. During start-up the ECRH background power density is estimated to be about 0.13 MW/m^2 for up to 5.5 s; this load can be neglected, because it is significantly lower than the other contributions. The same consideration applies to the background density of 20kW/m^2 during normal operation.

At 60GHz, the stray power density is due to the X-polarized collective Thomson scattering. Since the CTS beam is will be on-off modulated with 50% duty cycle, the inner wall peak value of 1.5 MW/m² [2] can be halved for determining the effects on thermal timescales. Again, the stray radiation from multiple reflections can be estimated in the range of 7kW/m^2 [2] and consequently it can also be neglected.

This work covers only the diagnostics installed in port #11 and in port #12., The stray radiation that could reach these diagnostic windows, through their corresponding conduits, has been estimated, taking into account the reduction due to the absorbing coatings. Figure 1 reports a view of the DSM1 (Diagnostic Shielding Module 1) of the Equatorial Port Plug (EPP) #11.

From the estimated stray power densities at the aperture of the lines, reported in Table 1 [4, 5], it is possible to derive the power loads on the by evaluating: 1) the mode-dependent coupling between the incident stray radiation and the first wall apertures and 2) the attenuation of waveguide modes, or their combinations, along the conduit.

Table 1: The estimated stray power densities at the aperture of the conduits for the two possible sources of stray radiation: ECRH and CTS.

Source	*f* [GHz]	Stay power density [MW/m²]	Ports	Duration
ECRH (O-mode)	170	3	#11 #17	5.5s
ECRH (X-mode)	170	1.25	#11 #12	CW
CTS (X-mode)	60	1.5	#11 #12	Modulated 50% duty cycle

To remain on safe side, only worst case scenarios have been investigated in detail [5]. Therefore the highest input power density has been assumed at both 170 GHz and at 60 GHz. Moreover, regarding the propagation along the transmission lines, only the least attenuated modes have been considered. The best-coupled modes radiation pattern has been also calculated for the angle of maximum gain. Sometimes, particularly to save computational resources, an even more conservative approach has been adopted; uniformly illuminated apertures with unit efficiency have been assumed, i.e. the calculation have been carried out in the hypothesis that all the incident power density is coupled to the aperture, independently from the angle of incidence. This is unphysical, because a uniformly illuminated aperture is the most directive antenna, however it is also worse than any physically possible configuration.

In this section we report the results of calculations based on the hypothesis that subsystems outside the torus vacuum are perfectly matched to the incoming stray radiation. This is also not realistic, but representative of the mean power absorbed by the windows. The software tools to perform the actual computations are commercial computer programs; CST Microwave Studio, to determine the attenuation in the ducts, and GRASP[1], to estimate the coupling between stray radiation at the first wall and modes propagating in the diagnostic conduits.

Table 2 summarises the power absorbed by the windows of the investigated diagnostics. The highest value per mode and diagnostic is reported. These estimates have been obtained assuming an absorbed power fraction of ~1.14% at 60GHz and ~3.23% at 170GHz in the fused silica windows.

In the H-alpha lines, very low attenuation is predicted on the basis of the simulations via CST, because of their geometry. The best-coupled modes are TE_{10} and TE_{01}. Their radiation pattern at the angle of maximum gain has been retained, to estimate the maximum coupled stray power. At 170 GHz, the highest power obtained comes from the bottom line of sight, while at 60 GHz the top LoS contributes more.

Regarding the NPA diagnostic, CST simulations indicate that $TE_{12,1}$ is the least damped waveguide mode along the conduit. The probability of incident stray radiation to direct excite this mode is low and appreciable only for far off-axis incidence. Incident stray radiation is coupled more efficiently to the mode TE_{11}, which, on the other hand, is strongly damped by the collimator in the diagnostic conduit.

The core VUV has been modelled as a narrow rectangular waveguide coated with Boron Carbide (B_4C). Unfortunately, at the two frequencies relevant to the present study, important data, such as loss tangent and permittivity, is not available for this material. Boron Carbide is a well-known microwave absorber, though. Consequently, CST simulations have been performed for titanium alumina. Moreover, no computations have been performed at 60 GHz because of the well-known $f^{1/2}$ scaling of attenuation with frequency. Regarding geometrical aspects, the conduit of the divertor VUV diagnostic includes ten irises, causing such a large scattering of the modes that it is impossible to realistically account for them. Simulations with CST do not help either, because of the enormous import of the electromagnetic problem. Again a worst case scenario situation has been considered, neglecting both mode conversion and attenuation and assuming optimum coupling between the incident radiation and the first wall aperture. Even in these highly unrealistic conditions, the continuous power absorbed by the window does not exceed 5W CW and reaches 30 W only during plasma breakdown.

On the contrary, the antennas of the Low Field Side Reflectometry (LFSR) can collect a significant amount of EC stray radiation, reaching potentially dangerous levels for the windows. Designers, aware of the issue, are planning to fit a grating mirror on the first mitre bend, which would provide about 20dB insertion losses at 170GHz.

The collective Thomson scattering diagnostic has been analysed as well. The only relevant stray power contribution, which can reach the windows, is coupled in an extremely narrow angular range. To protect against this unlikely worst case scenario, it is recommended to implement the same countermeasure planned for the LFSR.

To summarise, in general the power reaching the windows is expected to be very low and no special precautions are required. On the other hand, the windows of the LFSR and CTS can be subjected to dangerous levels of stray radiation [5]. For these diagnostics it is therefore recommended to envisage suitable grating mirrors and interlocks. Absorbing coatings are not applicable to LFSR waveguides, while the CTS conduit is already made of B_4C.

In the light of the results just summarised, the effects of gamma and neutron flux on the properties of the window materials (Sections from 3 to 6) constitute the remaining issues, requiring a careful assessment. With

[1] https://www.ticra.com/software/grasp/

regard to the practical measures envisaged to alleviate the most severe cases, absorbing coatings can play a significant role. Consequently, also their properties will have to be assessed, to make sure that they will provide the adequate protection they are supposed to (Sections from 7 to 10).

Table 2 - Absorbed powers in the worst-case scenarios.

Diagnostic	Stray Power Polarisation and frequency	Stray Power Density duration	Worst case absorbed power by the window (absorption fraction equal to 1.14% at 60 GHz and 3.23% at 170 GHz)
Hα port 11	O ; 170 GHz	5.5s	≤ 17 W
	X; 170 GHz	CW	≤ 4 W
	X ; 60 GHz	Modulated	≤ 9 W
Hα port 12	X ; 170 GHz	CW	≤ 3 W
	X; 60 GHz	Modulated	≤ 9 W
LFS port 11	O ; 170 GHz	5.5s	≤ 214 W
	X ; 170 GHz	CW	≤ 1 W
	X ; 60 GHz	Modulated	≤ 36 W
NPA port 11	O ; 170 GHz	5.5s	≤ 2 W
	X ; 170 GHz	CW	≤ 1 W
	X ; 60 GHz	Modulated	≤ 2 W
"Divertor" VUV port 11	O ; 170 GHz	5.5s	≤ 1 W
	X ; 170 GHz	CW	≤ 1 W
	X ; 60 GHz	Modulated	≤ 1 W
"Core" VUV port 11	O ; 170 GHz	5.5s	≤ 1 W
	X ; 170 GHz	CW	≤ 1 W
	X ; 60 GHz	Modulated	≤ 2 W
CTS, port 12	X ; 170 GHz	CW	≤ 296 W

3. Window assemblies of fused silica and the required tests

Based on previous studies, the reference choice for ITER windows is fused silica and therefore in this work only this material is considered. However, given the potentially detrimental effects of neutrons and gamma, specific radiation tests have to be foreseen for this material.

Fused Silica and Fused Quartz are types of Quartz Glass, which can be manufactured with several different processes but whose main constituent is silica in amorphous form. The feedstock of Fused Silica glass is high purity silica sand, which is normally melted in electric furnaces. The resulting material can be translucent or opaque. The opacity is due to very small air bubbles which remain trapped within the material during the manufacturing process.

Synthetic Fused Silica is made from a silicon-rich chemical precursor. Two main grades of the material are normally used for optical manufacturing:

1. UV grade of synthetic FS (KU-1);

2. UV-IR grade of synthetic FS (KS-4V).

Both synthetic fused silica grades are ultra-pure, single component glasses. They present a unique combination of thermal, optical and mechanical properties, rendering them the preferred choice in a variety of processes and applications. Their high purity, typically over 99.9%, guarantees minimum contamination in process applications. Indeed, synthetic fused silica can routinely withstand temperatures above 950°C. Moreover, thanks to a very low coefficient of thermal expansion, both grades can be rapidly heated and cooled without risk of failure due to thermal shock. These qualities, combined with excellent optical transmission compared with most other glasses, render them useful materials for production of superior quality optical windows, beam-splitters, lenses, cold/hot mirrors, beam- combiners, prisms, etc. These materials are also chemically inert to most substances, including virtually all acids, allowing their use also in quite hostile environments.

The dielectric properties, very high electrical resistivity over a wide range of temperatures, combined with low thermal conductivity, render synthetic fused silica the material of choice in many applications.

In any case, given the extreme operating conditions in ITER, a sequence of quite complicated tests is indispensable to qualify the windows for this hostile environment.

The expected loads and stresses can be the root cause of three main types of failure modes:

a) Failure mode A: fast fracture

b) Failure mode B: progressive deformation due to thermal effects

c) Failure mode C: debonding

d) Degradation of the diagnostics-related performances (e.g. optical transmissivity)

Given these potential failures, to guarantee the required properties of the windows in all regimes of operations, the test of the assemblies must be sufficiently detailed and able to detect any problem, from the first stage of manufacturing, testing already the "as-built" conditions. Consequently, a good testing program for the window assemblies would consist of five different steps:

1. Geometrical characterization;

2. Optical characterization;

3. Mechanical and thermal tests;

4. Measurements of dielectric properties;

5. Irradiation tests.

The <u>geometrical properties</u> of the manufactured disks have to be verified first. The quantities to be tested include planarity, thickness, diameter and roughness.

The main goal of the <u>optical tests</u> consists of determining whether the disks present any large cracks. This can be achieved by optical scanning.

With regard to mechanical and thermal tests, the loads on the window assemblies can be divided into manufacturing and operational loads. The former have to be applied during the manufacturing and assembly processes. The operational loads can be grouped into the 5 following categories:

1. Electromagnetic loads
2. Thermal loads
3. Pressure loads
4. Inertial loads
5. Nuclear loads

4. Measurements of dielectric properties

From the perspective of quality control, the microwave parameters of disks, particularly their dielectric properties, can be measured with different Fabry-Perot-resonators [6]. The loss qualification is normally obtained with open resonator setups, consisting either of a spherical mirror and plane mirror or of two spherical mirrors.

The complex permittivity dissipative can be quantified by measuring the resonant frequency and quality factor, defined as the ratio of the mean stored energy and dissipated energy per period of oscillation, first for the empty unloaded and then for the loaded resonator. The loss tangent, the fundamental quantity for the qualification of the dielectric properties of these materials, can then be calculated as the ratio of the dissipative imaginary and real part of the permittivity.

To take into account the potentially crucial aspect of the dielectric properties degradation caused by neutrons and gamma rays, all the tests described in this section should be performed before and after irradiation of the disks.

To summarise, at 170 GHz the following measurements are necessary for an adequate dielectric characterization of the disks:

- Measurement of the loss tangent $\tan\delta$ and the permittivity ε_r, averaged over the entire window.
- Mapping of the whole disk.

5. Radiation effects on dielectric properties: review of the literature

During ITER lifetime, its windows will be exposed to different types of radiation:

a) gammas and neutrons from the plasma;

b) gamma and neutrons from cooling water systems.

The fusion neutrons cause cascades of atomic displacement and transmutation nuclear reactions within the materials. The atomic displacement cascades produce vacancies and interstitials defects (point structure defects). Transmutation reactions induce the formation of impurities (e.g. H, He atoms). High energetic gammas have also the capability of producing displacement in material but to a much lower extent than the neutrons. However, gamma rays are able to ionise the materials, altering some of their properties.

Table 3 summarises the phenomena induced by radiation, which can affect ITER window assemblies.

Table 3 - List of failure modes

Effect	Acronym	Main Dependence	Explanation	Components affected	Failure mode
Radiation-induced absorption	RIA	Total dose Temperature	Optical absorption increases due to the production of defect related absorption bands, leading to light transmission loss	Window transparent material	It affects diagnostic performance
Radio luminescence or radiation induced emission	RL or RIE	Ionizing dose rate Temperature	Light emission due to excitation of defects and impurities	Window transparent material	It affects diagnostic performance
Thermal conductivity decrease	-	Total dose Temperature	Thermal conductivity decreases leading to temperature increases	Window material and frame	Thermal conductivity directly affects the thermal gradients in the ferrule/window leading to possible over stresses and ***cracks***
Enhancement of the dielectric permittivity and loss tangent	-	Total dose Dose rate Ionizing dose Temperature	Dielectric loss increases leading to an enhanced absorption of microwave power	Window transparent material	Increase of ECH absorption increasing the thermal load
Volume changes and transmutation	-	Total dose Temperature	Transmutation products Materials swell, or in some cases shrink; both cause distortion and embrittlement due to internal stresses.	Window material and frame	Bonding might be affected by this effect jeopardizing the sealing of the window as first confinement barrier. Defects on the bonding and/or cracks in the window material may be created.
Radiation enhanced diffusion	-	Ionizing dose Temperature	Radiation directly affects boundary processes such as dissociation or adsorption, introduces defects and impurities into the solid by radiation damage there by modifying tritium transport	Window material and frame	Tritium permeation will increase. No safety implications are expected
Radiation induced segregation	-	Total dose Dose rate Temperature	Radiation produces quantities of point defects. At intermediate temperatures, these defects are mobile and travel to low energy sites boundaries, dislocations, and other defect sinks.	Alloys, window frame and bonding	Bonding will be affected by this effect jeopardizing the sealing of the window as first confinement barrier (micro-cracks that may develop cracks in the bonding area)

Mechanical Properties	-	Total dose Dose rate Temperature	Hardening (H) Loss of ductility (LD) Loss of fracture toughness Loss of creep strength	Window material and frame	Frame and bonding will be affected by this effect jeopardizing the sealing of the window as first confinement barrier

Since the irradiation tests are particularly demanding, the literature [7-10] has been reviewed to see whether there is any information available relevant to the present investigation.

Even if various experimental results on the exposure of fused silica to gamma rays are reported in the literature, the effects depend on many parameters such as impurities and manufacturing procedures. Therefore, the degradation of fused silica exposed to gamma irradiation remains difficult to quantify. Moreover, even if several irradiation tests with neutrons have been performed, the effects on fused silica properties have been documented mainly for the visible and UV region of the spectrum.

One important aspect to consider is that, depending on the fabrication process, fused silica can contain a percent of OH, which can range over more than three orders of magnitude (KU-1 (<2000 ppm) and KS-4V (<0.1 ppm)). However water-free fused silica should be preferentially used, if compatible with operation of the specific diagnostics [11, 12].

According to the literature, the dielectric properties of fused silica windows are affected by long term electron, gamma and neutron irradiation as summarised in the following [13].

1. Prompt effects have been only observed at very low frequencies, consistent with the low Radiation Induced Conductivity (RIC) results, as derived from dielectric measurements in the range from 1 kHz to 20 GHz under electron irradiation in silica. Consequently, the gamma induced extra dielectric loss is expected to be negligible for frequencies above 10 GHz. Measurements of loss and permittivity were performed in the range between 100 Hz and 1 MHz under electron irradiation at 60 and 250 °C. At both temperatures and for dose rates up to 3.5 kGy/s, a very fast increase in the loss (tan δ) was observed at the onset of irradiation, with an almost perfect 1/f dependence below about 100 kHz. It is a well-established fact that radiation enhanced dielectric loss are the sum of two effects: the enhanced DC conductivity (RIC) and the enhanced polarization loss. The 1/f behaviour, combined with the fact that the permittivity does not change significantly, indicates that at these low frequencies the loss is mainly due to RIC. Even if the losses increase with the dose rate linearly, so for an expected dose rate of about 375 Gy/h (0.1 Gy/s), the effect will be negligible at high frequencies, in good agreement with the available RIC measurements.

2. When exposed to a gamma dose of 330 MGy (5×10^{-5} dpa), much higher than expected for a typical ITER diagnostic windows, neither the post-irradiation loss nor the loss measured during irradiation showed any appreciable permanent degradation The deviation from 1/f behaviour above 100 kHz is conjectured to be related to enhanced polarization loss. Under all conditions, the DC permittivity showed no detectable modification.

3. A neutron dose of 10^{21} n/m^2 (~10^{-4} dpa) is maximum achieved as reported in [10]. Below 60 MHz post-irradiation measurements showed a permanent increase in loss by up to a factor of 2. However no effect was observed in the GHz range. Unfortunately, the estimated total lifetime dose for ITER window is higher, around 1.2 10^{22} n/m^2.

A dose comparable to the one expected in ITER, including a 10x safety margin was reached in another study [14]. The loss tangent again did not show any significant increase at intermediate frequencies frequencies. At higher frequencies (90 GHz), some effects were observed; the dielectric loss values oscillated between 0.3 and 1.0 10^{-3} after neutron irradiation.

6. Overview of the tests for the window assemblies

The tests, reported in the literature, cover irradiation levels of the same order of magnitude as expected in ITER. The degradation of silica dielectric properties was never found to reach problematic levels. However impact of the neutron irradiation would deserve need some further tests, to prove that a specific silica grade/brand can effectively meet the design requirements. Presently, a study for DEMO in EUROfusion WP-MAT-FM framework is under way, in which the dielectric loss, including in silica window materials, is under test at doses 3 times higher, namely from around 10^{24} n/m^2 to 10^{25} n/m^2. The first results have already provided very useful information at high neutron doses. Up to a neutron dose of 10^{24} n/m^2, the measured loss tangent at 15 GHz remains still low, with a value around 1.7 x 10^{-4}. Moreover, values of loss tangent show no further degradation from 10^{22} n/m^2 up to 10^{25} n/m^2 in the MHz range, indicating a good safety margin for silica windows; in the GHz range the data are not available yet. Optical absorption bands (radiation induced defects indicator) have also shown a saturation of damage above fluences of 10^{23} n/m^2.

If needed, the required level of irradiation can be performed at SCK-CEN reactor (Belgium) or MTA reactor (Budapest-Hungary). The EUROfusion project irradiation tests were performed in this last reactor.

The post-irradiation dielectric measurements can be made at CIEMAT (frequency up to 20 GHz) and KIT (f>20 GHz), both with a large experience in this technique. At these doses, the dielectric measurements do not require special equipment or laboratories.

Regarding the issue of ageing under microwave loads, so far there is reliable information available in the literature. However, if the microwave radiation is quite low, and the window absorption is maintained at low initial levels, no significant detrimental changes are foreseen and the only expected consequences are minor temperature increases).

Finally, if lower dielectric losses proved to be necessary near 100 GHz, silica could be replaced by a covalent material such CVD diamond.

7. Coatings: overview of the literature

The extreme operating conditions of fusion reactor devices have led to an increasing interest in the field of high performance materials. With regard to coatings of relevance for the present study, absorbing layer ceramics very resistant to thermal shocks, and also with good absorption capability in the range 70-200 GHz, are the most extensively tested [15-17]. In particular, after applying relevant thermal loads, coatings of alumina-titanium were studied with various techniques - micro-analysis with X-ray EDS, ESEM (Environmental Scanning Electron Microscopy), X-ray diffraction analysis (XRD), Differential Scanning Calorimeter (DSC).

Other coatings showed good promise to motivate detailed investigations: Chromium Oxide (Cr_2O_3) and Boron Carbide (B_4C).

Chromium oxide showed the advantages of resistance to high temperatures and higher thermal conductivity with respect to alumina-titanium based coatings. Short-pulse and long pulse loads have been applied to this coating by IFP-CNR [now ISTP CNR] (with the bonding layer, to minimise stresses caused by the different thermal expansion of the coating and the copper). It has shown high resistance to detachment.

Boron Carbide (B_4C) showed better resistance than other coatings to high temperatures and also better maximum microwave absorption. The utilization of B_4C as absorbing coating layer in the inner wall of a bolometric device was patented by IFP-CNR (ISTP-CNR) (Italian Patent No. MI2005A000290 - International Patent Application No. PCT/EP2006/050605 filed on 1st February 2006), granted in US (US7586070B2) and licensed to the Italian company L.T.Calcoli).

The information available in the literature, about the candidate materials for ITER (Al_2O_3-TiO_2, B_4C and Cr_2O_3), has been reviewed. From this analysis it has emerged that, at low power densities, the tests required to fully characterize the coatings are well established and relativity simple to perform [15]. The Millimetre wave absorption of metallic samples with absorbing coatings was measured at frequencies of 170 GHz and 60 GHz.

In any case, only partial information can be found in the literature. Moreover, the properties of these materials in the conditions relevant to ITER have not been exhaustively tested. In particular, the behaviour under irradiation levels typical of ITER remains poorly understood. In the following, we define the main lines of test procedures to fill in the gaps in the characterization of the candidate coating materials for ITER. These procedures will have to be finalised in collaboration with the facilities selected to perform the tests, because various aspects can be defined in detail only on the basis of the equipment and infrastructures available at each site.

These tests are essential to accurately determine the permittivity and loss tangent of the various materials, which are crucial ingredients for the choice of the optimal thickness of the coatings.

8. Coatings under exposure to ITER relevant conditions

In order to qualify the actual suitability of the candidate absorbing coatings, samples of the corresponding overall coating-interlayer-substrate systems must be subjected to particular test procedures able to reproduce the ITER working conditions.

8.1. As built

The "as-built" condition is representative of the reference situation of the considered coatings and relative interlayers and substrates, so this starting point qualification is a crucial prerequisite for the following steps. Moreover it should be noted that the analysis of the as-built condition is crucial to evaluate the result of the chosen production process technology (e.g. plasma spray technology), before starting with the same analysis of the samples subjected to the other tests.

8.2. Tests under high power density microwave loads

The expected effect of high-power density microwave load on the coating under vacuum is essentially a temperature increase in the coating layer, from exposed surface to the opposite side, with the heat flowing mainly to the metallic substrate, where it is then removed by a cooling system, or, lacking an active cooling, dissipated in the bulk, causing a general temperature increase. The heat flow towards the substrate is maintained by a temperature gradient, which keeps the coating temperature higher than the one of the substrate to which it is attached. The amount of this temperature difference depends on the power density deposited in the coating and on the coating thermal conductivity and thickness. Typical conductivities of sprayed coatings are around 2 W/m°K, which give a ΔT of 200° per incident MW/m^2 for 100μm thickness.

The maximum temperature of the coating depends then on the substrate temperature. Supposing an ideal thermal contact, the substrate must remove the same power density at the rear side of the coating with a moderate temperature increase, which in a complex environment can become a difficult task.

Indeed the coating temperature gradient can be not uniform, due to the non-uniform absorption in the coating; this effect is hardly reproducible except with high-power microwave absorption at the maximum power density expected.

Relatively homogeneous and surface-extended power densities of the order of 1 MW/m^2 cannot be obtained in high-power gyrotron installations, where the central power densities usually employed in the beams are much higher than needed, decreasing rapidly to the beam edge, while leaving no possibility for a sample to be placed in a controlled uniform field. Power densities of the order of 1 MW/m^2 over extended surfaces are reached in high power calorimetric loads ("dummy load") used for gyrotron qualification and conditioning. Completely controlled irradiance however is not easily obtained also in this case, due to the interference pattern inside the load. Moreover, insertion of a sample tile properly diagnosed would require ad-hoc design and manufacturing or modification of an existing dummy load to add this functionality. Industrial medium-power gyrotrons have usually lower frequencies, while newly developed high-frequency gyrotrons for NMR have a too-high working frequency. It is therefore hard to find a standard setup for testing the effects of high power applied with microwaves. The heat exposure can somewhat be simulated by applying a high heat flux by a plasma torch flame close to the surface, as done in reference [18] but the simulation is not completely representative of the real microwave heating, occurring in the coating bulk.

According to the literature, the main source of stress in the coatings is due to the fact that the thermal expansion coefficient of the coating (low thermal expansion coefficient) and the thermal expansion coefficient of metallic substrate (high thermal expansion coefficient) are quite different, even if this problem is somehow mitigated by the bonding layer usually applied for the purpose of reducing the stresses.

One possibility to test the coating under a thermal stress, representative to some extent of the cycling microwave exposure, is to perform thermal cycling tests up to the temperature reached at the maximum microwave exposure expected. The reason for this similarity is that, under microwave exposure, the coating temperature is always higher than that of the substrate; this means that, when both are heated to their maximum temperature as in the maximum exposure case, the thermal expansion of the coating with microwave heating is higher than when both coating and substrate are heated at the same (equal) maximum temperature. The stress due to differential expansion in this last (equal temperature) case is not lower than in the microwave exposure case (possibly higher, because the substrate thermal expansion is higher than that of the coating). This gives a way to (slightly) overstress the coating with an oven thermal cycling where the temperature is uniform, provided it is made at the maximum temperature expected. These test conditions however overlook the effects related to short timescale transients that one can expect during tokamak operations.

Ideally, these tests should be repeated after the various exposure campaigns, in particular before and after nuclear irradiation. Tests after nuclear irradiation can be omitted if other test techniques investigating the structural properties of the samples are easier and if the dielectric properties are not modified significantly by nuclear irradiation, according to the information available in the literature.

8.3. Thermal ageing

The thermal ageing test is a fundamental test to verify the possible detrimental effects of long-term exposure to coating elevated temperatures and of the overall coating-interlayer-substrate system. It must be planned in ITER working conditions, considering the temperature levels foreseen for the component, for which the coatings are considered.

This experimental test is normally performed storing the sample (coating-interlayer-substrate system) in a laboratory controlled-climate oven, at the specified test temperature and time. In order to reproduce the working conditions and to avoid external environmental contamination, it can be considered (not mandatory) to perform the test in a suitable oven having vacuum condition or an inert gas filling system. At the conclusion of the test, the sample must be slowly cooled to room temperature, in order to avoid harmful cracks especially on the interface zones.

As example of an experimental procedure, if the temperature of 300°C is considered the maximum application temperature for the interested component in ITER, the test can be performed at four different temperatures, starting from room temperature to 300°C, proceeding in steps of 100 °C. For each temperature step, stationary intervals of up to a maximum of 2000 hours can be applied. However, tests at higher temperatures (e.g. 450 °C) are also recommended to study safety conditions. With different exposure times, it is possible to plot levels of possible damage versus exposure time, in order to extrapolate the actual results for the overall foreseen lifetime and to simulate the in service condition.

A proper use of the Arrhenius equation permits to consider "accelerated thermal ageing test" [19], especially to obtain results for the high temperatures and long times. Nonetheless, in the present context the need to obtain accurate activation energy values requires to perform tests at multiple temperatures, so the use of the more traditional procedure is considered more prudent.

8.4. Thermal cycling

Thermal cycling is a specific form of test meant at evaluating the capability of materials to resist variations, typically periodic, in temperature over a maximum predetermined range. So a generic thermal cycle consists of repeated cycles of heating up to the maximum testing temperature, a steady state and cooling down to the base temperature.

The aim is to simulate and/or reproduce the variable temperature conditions in ITER, in order to verify the formation of micro and macro-defects, caused by stresses consequence of the different thermal expansion coefficients of the coating-interlayer-substrate system.

The definition of the appropriate test procedure is essential and must take into account the final effective working conditions. In the EUROfusion research context, the experimental procedures about thermal cycling test regarded mostly the use, for instance, of plasma technology or electron beam facilities at high temperature and high density, because of the necessity to study and develop plasma facing materials (e.g. tungsten): in this latter case typical maximum testing temperature and base temperature are 1000°C and 250°C, respectively, with pulse duration of 400-500s [20-21]. Nonetheless, in the present work, the samples of the coatings have to be baked under ITER relevant baking conditions (e.g. temperature 240°C, baking duration 500h) [22], so that more conventional experimental procedures, with lower temperatures and heat densities, can be adopted. In this context, heating systems (e.g. oven) in vacuum or in an inert gas (Ar) atmosphere, able to monitor temperature during the heating, steady state and cooling steps of the single cycle, and for all cycles, can be considered. The temperature monitoring should be implemented with thermocouples directly in contact with the sample.

It is recommended that the maximum testing temperature of the cycle be equal to or higher than the one representative of the working conditions, and that the number of cycles is sufficiently large to validate the coating lifetime. As example of an experimental procedure, if the temperature of 250°C is considered the operation temperature for the interested component in ITER, a (safety) thermal cycling test between 100°C and 400°C seems appropriate: after a first acceptance test (3-5 cycles), the actual quality control test should foresee at least 100 cycles on two samples, with equal heating and cooling rates as equal as possible (symmetric cycle). In general, these rates depend on the difference between the two temperatures and on instrumental limits, but usually they should not be lower than 50°C/h.

8.5. Water exposure

An overall qualification of the coatings should include the study of their behaviour under partial or complete water immersion and under exposure in an atmosphere maintained at 100 % relative humidity. Indeed water can cause the degradation of coatings, so actual information on coating resistance to water is helpful in predicting its service life.

The basic principles and operating procedures for testing water resistance of coatings, in terms of water immersion and exposure in an atmosphere at full humidity for the coatings and coating-interlayer-substrate system, are reported in the ASTM D870/02 and ASTM D2247/02 standards

The water exposure tests must be included into the initial test group, to verify the actual integrity and functionality of the samples: for this reason these tests are recommended only for the as-received samples, to detect the presence of possible water-related failures after a fixed period of test time.

8.6. Neutron irradiation exposure

During ITER operation, the radiation that will reach the coatings and the windows has a different nature: a) the gamma and neutron sources from the plasma fusion reactors; and b) gamma and neutrons from cooling water system (N16, N17).

Obviously, since this irradiation is to be performed in a fission reactor, also gammas will be present, thus recreating the realistic environment of ITER.

Because of the difficulty of doing on-line tests in a reactor, only off-line tests are foreseen for the neutron irradiation. The characteristics of the tests should be as follows:

1. Type of test: Off-line irradiation
2. Neutron flux spectrum: note that the spectrum in the fission reactor should be as similar as possible to the one of a fusion reactor. Cadmium shields may be needed in order to remove the excessive contribution from thermal neutrons.
3. Total Fluence: 1.2 E18 n/cm^2 +/- 10%
4. Temperature of the test: 150 °C +/- 10% (constant monitoring)

Characteristics of the tests in terms of size and of irradiation rig must be defined with the facility where the irradiation is going to take place. Once the activation exposure is measured, samples will be transported to a hot cell when necessary.

The main properties that need to be measured after irradiation are:

a) Structural integrity (Optical microscopy, SEM, Energy Dispersive Spectroscopy, X-Ray Diffraction)
b) Dielectric properties (loss tangent, permittivity)

The tests to evaluate the structural integrity of the absorbing coating are explained in the next section. A list of candidate facilities where the neutron irradiation and the PIE could be carried out is reported in Appendix A.

8.7. Gamma irradiation exposure

From the available literature, it is not possible to assess the relevance of the gamma ray exposure and its effects. At this point it is therefore difficult to specify which test will be required.

Moreover, the gamma radiation expected in ITER should be characterized by a gamma flux of 330 Gy/h with an energy of about 6-8 MeV. At present, facilities with a gamma source able to generate this power do not exist.

9. Test methods for integrity, micro-structural and surface properties

9.1. Scope and procedures

In order to qualify the absorbing coating before and after the different test conditions, and to verify the differences between them, important investigations are to be considered, based on ITER requirements up to the end of the work lifetime. Such investigations and the corresponding results are to be developed also taking into account the effects of the vacuum condition and the expositions to the ITER environment.

The aim is to observe structural and micro-structural features, including their evolution during and after the test conditions, which can be harmful for the correct operation of components, in which the coatings are essential. In this context, suitable observations and tests are to be developed by using proper samples and/or mock-ups, on which the coating-interlayer-substrate system and the coatings surface are to be characterized. In particular, the following issues will have to be checked and analysed:

a) coating inhomogeneities in thickness and roughness: it is important to evaluate the quality of the process technology (e. g. plasma spray process) and the corresponding parameters chosen for the coatings production;

b) presence and formation of micro and macro-defects (i.e. pores, cracks, inclusions, detrimental phases): these could be nucleation points for progressive crack propagation or contaminations during the working conditions, possibly leading to unexpected catastrophic rupture. Furthermore, micro-cracks and especially porosity can absorb air and volatile elements, leading to excessive outgassing;

c) presence of excessive residual stresses at the interfaces of the coating-interlayer-substrate system: these residual stresses can cause harmful crack formations. Such a coating-interlayer-substrate system is characterized by the three different thermal expansion coefficients, where the interlayer is produced to provide a soft intermediate interface between bulk and coating. Nonetheless, the overall system can be subjected to thermal (constant or cycling) loads involving important modifications of the chemical and microstructural status, giving rise to residual stresses into the interface zones;

d) loss of adhesion and toughness properties: it is important to obtain indications and data about the mechanical resistance of the coatings, so that the same coatings maintain an actual functional adherence during ITER lifetime.

These issues involve the use of metallographic investigation techniques and specific surface tests, each of which is to be considered mandatory, pertinent or helpful depending on the test conditions to which the coating sample has been subjected. However, it should be noted that all presented investigations are recommended mandatory for the as-built samples, not only to evaluate and to optimize the best process parameters of the considered production technology, but also to obtain first data (reference condition) that will be compared to that developed after every foreseen test condition.

The conventional approach to manufacturing coatings is based on plate shape samples for the substrate, on which the interlayer and the coating are deposited. However, it is crucial that shapes and especially dimensions (thickness of the overall coating-interlayer-substrate sample included) of the as-built and tested samples are preliminarily agreed with all the considered facilities, in order to be consistent as much as possible with all the testing procedures and to minimize possible sample modifications that could involve changes of the material properties.

It is important to underline that the analysis on the samples subjected to neutron exposure must mandatorily take into account specific and accredited facilities, considering that the value of 2μS/h (source: STCF ISIS) is the maximum radioactive emission rate above which the samples cannot be managed and analysed by conventional procedures. This can imply the exclusion of some presented investigation techniques or their consideration after complete decay.

9.2. Optical microscopy

Optical metallography can provide useful information needed to characterize the microstructure of coatings. In general, under the specific resolution of an optical microscope, coating structure such as morphology of layers and surfaces, inclusions, pore size and distribution, coating-substrate interfacial macro-contamination can be verified. Moreover a direct visual observation of possible macro-defects and cracks on the interlayer/bulk surface (e.g. due to a possible not optimized plasma spray process) is possible, in order to verify the actual integrity and homogeneity of the coatings and interlayers.

For a complete characterization, it is recommended to perform such observations both on the coating surfaces and on the prepared metallographic cross-section sample about the coating-interlayer-substrate system. On this latter sample, it is important to develop thickness analysis of the coating with statistical basis: at least 15 equidistant points must be chosen where to measure the coating thickness. The details of the tests must be agreed with the considered facilities: standard practices as ASTM E3-11, ASTM B487-85 and the referenced ones are desirable.

Optical metallography is to be considered mandatory to analyse the first general characterization of the coatings, as built and after the specific test conditions.

9.3. Scanning electron microscopy (SEM)

SEM observations at high magnification are necessary to verify the microstructural aspects as the presence of micro-cracks, micro-porosity, unexpected inclusions and chemical phases, which can be harmful for the resistance performances and the compliance with the vacuum requirements.

The importance of SEM use regards the possibility to spot and focus on micro-defects in more detail and at high resolution by the detection of secondary electrons (SE), whereas the limits of optical microscopy do not permit to observe such details. Furthermore it is possible to observe different chemical composition areas (called compositional contrast) by detection of back-scattered electrons (BSE), in order to individuate possible presence and shape of expected/unexpected phases.

Performing SEM observations, both on the coating surfaces and on the prepared metallographic cross-section samples of the coating-interlayer-substrate system, is recommended, since valuable for surface analysis.

In general defects and micro-defects are due to inhomogeneous or unexpected conditions of local stresses or chemical interactions, that can result from the incorrect plasma spray process and/or harmful working conditions. For this reason, SEM observations are to be considered mandatory for the coatings in study, as-built and after every foreseen test conditions. Also the water exposure test can cause the degradation, oxidation or corrosion of coatings, so SEM observation after this test condition is recommended.

It should be noted that SEM observations must be performed with a good statistical basis, to reach reliable conclusions: conventional procedures involve the acquisition of 15 images, at least, for every microstructural aspect under study.

However methods and details of the tests must be agreed with the considered facilities: standards practices as ASTM E3-11, ASTM 1920-03, ASTM B748-90, ASTM E1245-03, ASTM E1829-97 and the referenced ones [23] would be appropriate.

9.4. Energy dispersive spectroscopy (EDS)

In order to analyse the nature of the observed microstructural features by identification of chemical elements (Al, Ni, Br, O, Cr...) and their relative proportions (atomic % or weight %), EDS measurements are usually performed with the SEM observations. The determination of the actual distribution of the chemical elements on the analysed specific zones (coatings, interlayer, bulk and relative interfaces), visualized by SEM observations is recommended. To evaluate the benefit of focusing EDS compositional analysis on local points/well defined zones or of developing element maps, can prove useful, depending on the specific observed features to be analysed. Moreover, concentration profiles of the chemical elements along perpendicular lines to the interface planes are important, to verify and to check possible diffusion phenomena of chemical elements and their evolutions between coating, interlayer and substrate, before and after specific test conditions. ASTM E1508-98 standard is a good reference.

9.5. X-ray diffraction (XRD)

The presence of expected/un-expected phases and compounds, and excessive residual stresses on the interfaces coating-interlayer-substrate, can lead to crack formation and/or possible fracture under the actual ITER working conditions.

In this context, X-ray diffraction can detect the residual stresses, determining the differences before and after the foreseen tests. Consequently, estimating the presence of corresponding micro-strains can be very useful: by the detection and the analysis of the resulting diffraction spectra, it is possible to identify the crystallographic details of the observed micro-structural details and to calculate the lattice inter-planar spacing d of the unit cell.

Thus, when a material is uniformly strained, elongation and contraction are produced within the crystal lattice, changing the inter-planar spacing of the lattice planes and involving a shift in the diffraction pattern: the change in d can be evaluated and the strain within the material deduced. Furthermore, when the material is not uniformly strained, for instance because of generic micro-structural defects, a broadening of the characteristic peaks is observed. Therefore, the effects of the plasma spray process and of the foreseen test conditions can be evaluated analysing and comparing the micro-strain conditions obtained by the corresponding detected X-ray diffraction spectra. In order to measure the local evolution of strain due to the different test conditions, it is suggested to focus the X-ray beam simultaneously on substrate, interlayer and coating, considering the prepared metallographic cross-section sample. The XRD analysis should be developed at room temperature (as built and after test conditions) by X-ray diffractometer, verifying the reproducibility of the obtained results to have a statistical basis. For this reason, at least three repeated acquisitions of XRD spectra at the same condition (for precise measurements, acquisition step of $\Delta\theta=0.02°$ and 10 sec can be considered) are recommended. In order to guarantee consistent testing procedures and results, in general the procedures issued by the International Centre for Diffraction Data (ICDD), regarding useful guidelines on sample preparation, testing procedures, and analysis methods, are recommended. These guidelines are especially useful for the most common diffraction applications such as phase identification, texture analysis, and quantitative analysis.

9.6. Roughness measurements

The primary aim of a surface coating is not only to protect from high-temperature but in general to withstand the operative conditions of the external environment. In particular, coating surface roughness is an important parameter considering that, in general terms of engineering surfaces, excessively rough surfaces are. Excessive surface roughness can imply the presence of deep cavities and/or pits: these can act as nucleation points for future cracks. The peaks of the rough surface can shear off, overly reducing the coating thickness or even exposing the substrate (interlayer or bulk) material. Furthermore, they could catch contaminants and air/gas, degrading compliance with vacuum requirements. For these reasons, roughness measurements are recommended on the surface of the as-built coating, also to evaluate the proper plasma spray process parameters. Comparing shape and dimension of the used powders (e.g. by SEM observations) to the roughness properties can be helpful.

Characteristic qualitative results, obtainable by the chosen experimental tests, are especially Ra (average length between the peaks and valleys of the profile and the deviation from the mean line) and Rz (the vertical distance from the highest peak to the lowest valley within five sampling lengths). In general the terms, definitions and parameters for determination of surface roughness are provided in EN ISO 4287 standard.

The most common test method involves drawing roughness profiles with a stylus across a surface, by contact-type instrument (contact profilometer), in which the same stylus is connected to a detector for the electrical signal processing. Instead of that, optical profilometer (no-contact profilometer), can be considered for more reliability.

With that, the instrumental system can measure small variations of the vertical displacement as a function of position, tracing 2D profiles and, to reduce number of measurements on the same statistical base, 3D images of the in study surface profile. For a more precise and in details surface profile determination, atomic force microscopy (AFM) can be considered.

9.7. Hardness and micro-hardness test

Hardness is defined as the resistance to a localized permanent indentation: it is investigated using a specific and standard indenter at a fixed load, and the hardness value is determined by measuring the characteristic shape and dimension of the resulting imprint. It is performed by specific standard instrumentation, depending on the standard test method that is chosen. The most used methods are Rockwell hardness test and Vickers test that change on the basis of indenter type, characteristic features to observe and to calculate to assess the hardness value, the scale of the foreseen hardness. In the case of Vickers test, micro-hardness test (with a micro-indentation to evaluate) can be performed. In order to perform the test procedures in terms of reproducibility and reliability, ASTM E384-17 standard can be considered.

Useful indications, regarding the possible changes of the resistance properties (e.g. toughness, wear resistance) of the coating, can be obtained also considering that the softer coatings are in general more susceptible to erosion-corrosion mode of degradation.

Hardness tests can be performed on the surface coating, in order to determine the hardness value (before and after the foreseen test conditions). In addition it is possible to observe possible decohesion or crack propagation starting from the remaining indentation imprint. Developing micro-hardness (Vickers hardness) profiles, along perpendicular lines to the interface planes on the prepared metallographic cross-section sample about the coating-interlayer-substrate system, is recommended before and after the foreseen test conditions.

At least 15 repeated measurements must be performed to obtain reliable results from statistical point of view.

9.8. Scratch test

To investigate the critical loads for the coatings adhesion properties, scratch tests are a widely used methods. Indeed they are typically performed to determine the adhesion of coatings to substrates, simulating abrasion mechanisms with suitable instrumentation. About ITER requirements on lifetime performance, scratch tests would be useful to obtain information on the actual adhesion of the coatings, considering that the same coatings will be inevitably subjected to a minimal level of abrasion and wear by the work environment.

A standard scratch test device implies moving a stylus over a sample surface by two possible procedures. In one case, a progressive linearly increasing load is applied, until failure occurs, at which point the a critical load is determined. Another alternative consists of a constant load scratch test, in which series of scratch tests are performed, each one at constant normal loads, again until the critical load, at which the coating fails, is reached. In general the samples are examined with optical microscopy, during and after the test. The most important parameter of the test for the scratch adhesion value is the lowest critical load at which the studied coating fails (L_C), since it can be used for comparative coatings evaluations.

For the specific test ASTM C1624-05 standard can be considered, and it should be considered for general characterization of the coatings, as built and after the specific test conditions.

9.9. Outgassing test

Outgassing tests are able to evaluate materials and products for potential and existing outgassing risks. In particular, under working conditions, gas released from the structural material can poison ITER plasmas, so it is important to evaluate the outgassing properties before and after the foreseen test conditions.

Outgassing test methods, and the corresponding apparatus and procedures, usually depend on the particular nature of the analysed gases and sample typology, by static or dynamic measurements of the same gas release. The most common method can be considered the "throughput method" [24] and in general dynamic flow methods are the most used in EUROfusion research context [25-28]. The characteristic output, for which the outgassing properties should be monitored and analysed, is the *outgassing rate*, that is the amount of gas emitted per unit time and exposed surface at a specified time after the evacuation starts, and it is measured in Pa m^3 s^{-1} m^{-2} (Pa m s^{-1}): the outgassing rate of the coating samples (in particular the samples of the coating-interlayer-substrate system) related to the materials used or foreseen in the ITER vacuum systems, must be consistent with the limits given in the ITER vacuum handbook [29].

Table 4 – Summary of the qualification tests. The facilities with the capability of performing the tests the tests reported in the table are discussed in Appendix A.

Test	Measured quantity	Information derived	Relevance on as-built sample	Relevance after exposures
Dielectric properties at low power	permittivity and loss tangent	Choice of the optimal thickness	mandatory	mandatory after thermal ageing/cycling
Optical microscopy	Microstructural aspects	Verify the actual homogeneity of the coatings	mandatory	mandatory after thermal ageing, microwave exp. and neutron exp.
SEM/EDS	Microstructural aspects	Presence of micro-cracks, porosity, unexpected inclusions	mandatory	mandatory after thermal aging, microwave exp. and neutron exp.
XRD	Crystallographic structures and chemical composition	Individuate phases and the presence of this residual stresses in the system bulk-interlayer-coating	mandatory	Mandatory for thermal ageing/cycling Useful after neutron exposure

Roughness measurement	Surface roughness	Nucleation points for future cracks. compliance of the vacuum requirements	mandatory	Helpful after thermal aging
Hardness/ Micro-hardness	Resistance to a localized permanent indentation	Possible changes of the resistance peculiarities. possible wear	useful	Useful after thermal aging, thermal cycling
Scratch test	Adhesion properties	Obtain the critical loads that are related to adhesion properties between coating and substrate	mandatory	mandatory after thermal aging/cycling
Outgassing	*Outgassing rate*	Potential and existing outgassing risks	mandatory	mandatory after thermal aging/cycling

As an example, typical operating conditions (baking cycle and measurement conditions) for an outgassing test are:

- Ramp up from room temperature to 150 °C : 15 hours,
- Steady temperature at 150 °C for 72 hours,
- Ramp back to 100 °C : 4 hours,
- Steady temperature at 100 °C for 24 hours.

Moreover the test procedures, especially if referred to specific standards (e.g. ASTM 595/15), can involve the measurement of data as, for instance:

- *total mass loss* (TML): loss of material outgassed from a specimen that is maintained at a specific constant temperature and operating pressure for a specified time. It is expressed as a percentage of the initial specimen mass.

- *collected volatile condensable material* (CVCM): material quantity of outgassed matter from a test specimen that condenses on a collector maintained at a specific temperature for a specific time. It is expressed as a percentage of the initial specimen mass.

The use of the latter standard parameters, and of the associated ones, implies the consideration of acceptance limits that must be mandatorily determined during the definition of ITER test conditions.

Indicative information and interesting research approach about outgassing properties can be given by performing thermal cycling test and observing the effects by the introduced investigation techniques, nonetheless the outgassing efficiency of the material under study on the context of an actual ITER application must consider the aforesaid official procedures.

10 Summary of the proposed tests for the coatings

In Table 4, a summary of the proposed tests for the absorbing coating qualification with their main characteristics is reported.

In Figure 2, the scheme of the overall tests sequence for the coating qualification is shown.

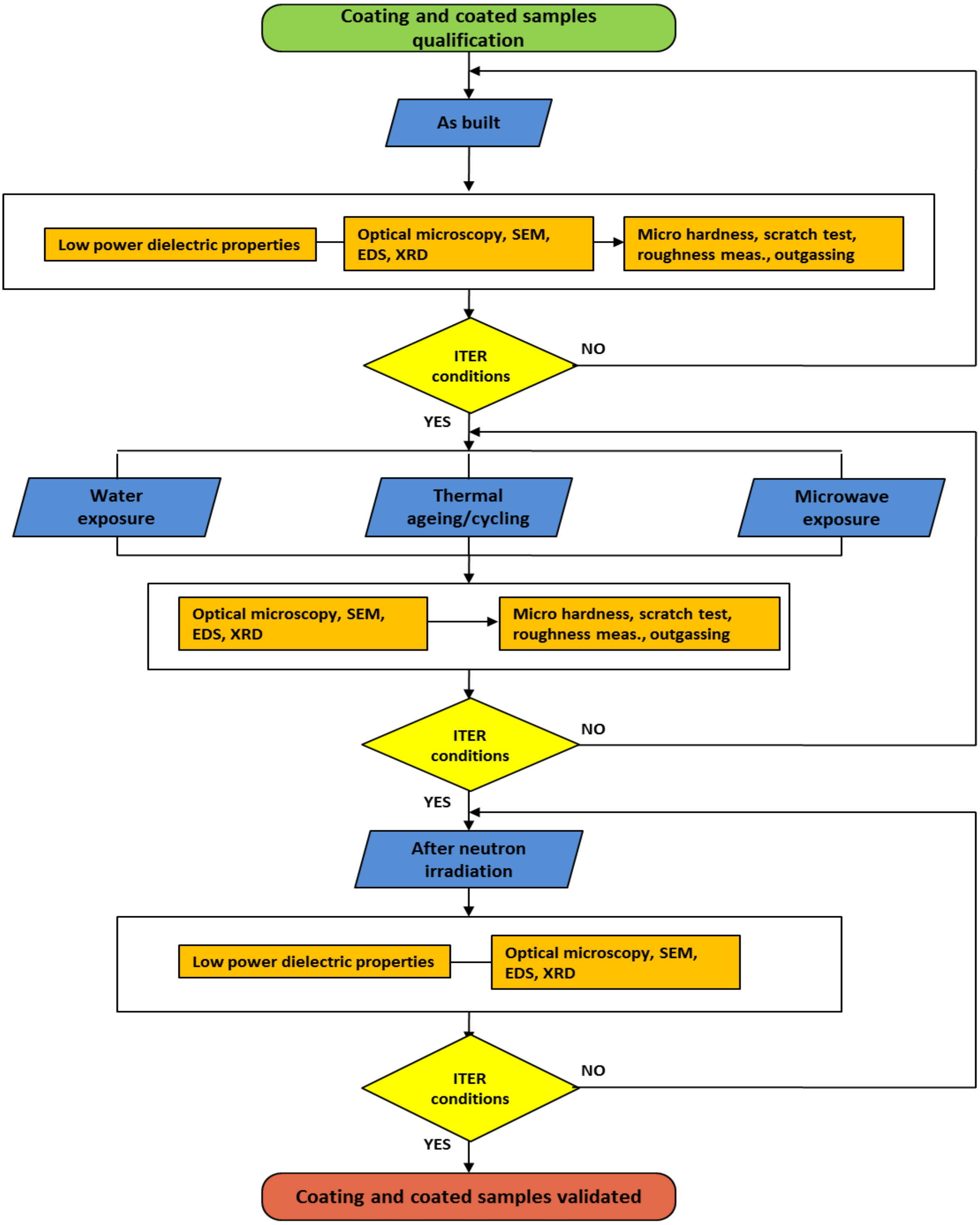

Figure 2 – Overview of the qualification scheme

A concise summary of the proposed tests is provided in the form of a matrix (see Table 5).

Colours indicate the importance of the tests:

- mandatory (red): the essential, unavoidable tests that have to be performed in order to provide an actual and reliable qualification of the coatings in ITER working conditions
- useful (green): the complementary tests, not considered strictly indispensable, able to improve the mandatory information for the assessment of the coatings;
- helpful (yellow): the additional tests, developed to study in depth the aspects obtained by the mandatory and useful tests, in order to obtain an overall comparison among all the considered tests.

Table 5 - Proposed test matrix for the qualification of the coatings

Test condition	**As built**	**Thermal ageing/ cycling**	**Water exposure**	**Microwave exposure**	**Neutron exposure**
Low power dielectric properties	(red)	(red)			(red)
Optical microscopy	(red)	(red)	(red)	(red)	(red)
SEM/EDS	(red)	(red)	(red)	(red)	(red)
XRD	(red)	(red)	(yellow)	(yellow)	(green)
Roughness measur.	(red)	(yellow)	(yellow)	(yellow)	
Hardness/Micro-hardness	(green)	(green)	(yellow)	(yellow)	
Scratch test	(red)	(red)	(yellow)	(yellow)	
Outgassing	(red)	(red)	(green)	(yellow)	

Mandatory (red)	Useful (green)	Helpful (yellow)

As a final summary, it can be stated that the situation of the test requirements for the coatings is quite clear, including the priorities, except for the gamma ray exposure, for which not enough experience has been gathered to make exact pronouncements about its relevance. In any case, compared to the neutrons, the gamma rays are expected to have a significantly lower impact. The only other aspect, which will probably need a specific investigation, consists of the requirements in terms of leak tests, to meet both ITER vacuum standards and safety criteria in case of overpressure.

Disclaimer

The views and opinions expressed in this paper do not necessarily reflect those of the ITER Organization.

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APPENDIX A. Candidate facilities for nuclear irradiation tests

In the following, the main candidate facilities to perform irradiation test on the coatings are reviewed. The information has been derived from the web sites of the facilities and from email interactions with their contact persons. Table A1 provides a more concise synthesis of the main facilities and of their main characteristics.

A1. Culham Centre for Fusion Energy

The Material Research facility (MRF) at Culham provides academic and industry users with a unique resource for micro-characterisation of materials. It meant to provide convenient lab access, to bridge the gap between the university or small industrial labs and large facilities at nuclear licensed sites. The facility supports research in lifetime extension for today's power stations, nuclear new build and Generation IV, and fusion. A new MRF building is operational, allowing to handle radioactive materials, with hot cells and micro-characterisation of neutron-irradiated samples. It provides the capacity of cutting, polishing and encapsulating individual Charpy-style samples up to the TeraBecquerel level, for both on-site studies and back at the user's facility.

The initial on-site analysis equipment, already available for non-active research, are a dual beam Focused Ion Beam, a Nanoindenter, Thermal Desorption Spectroscopy and a Scanning Electron Microscope with EBSD and EDS.

EQUIPMENT

a. Material processing

Higher throughput of lower active materials

b. Sample Preparation;

Sample Cutting, mounting and polishing
Sample FIBing

c. Material characterisation;

On-site PIE of active materials – nanoindenter, outgassing (TDS) Handling & inspection of beryllium & tritiated material, SEM (EDX, EBSD, TKD),

d. Material transfer;

Transfer of lower active materials for off-site PIE

To perform the tests on activated sample the activity limits are:

1. 3.75 TBq 60Co source for hot cells

2. 3.75 GBq 60Co source for instrument cells

3. <10 μSv/h at external wall of shielding

A2. Irradiation Center Karlsruhe (KIT)

The Irradiation Centre in Karlsruhe was inaugurated in 2001 and has provided irradiation studies of silicon sensors for the current CMS Tracker at the LHC at CERN.

Now, the experiments for the LHC are completed, but the next challenge will be the HL-LHC

The collider will be upgraded to ten times higher peak luminosity, posing a much more significant challenge to the detectors. This requires further R&D to investigate the radiation limits of silicon sensor technology and related electronics.

With regard to radiation hardness studies, the HEP community can provide irradiations with 25 MeV protons, generated with a Compact Cyclotron operated by ZAG Zyklotron AG, a private company located on the Campus North of KIT.

To test the effects of pure ionising radiation, an X-ray setup with a dose rate of up to 40 kGy/h is also avaialable.

Note that, they irradiate with 23MeV protons and not neutrons and they have no means for post-irradiation characterizations. They suggested contacting the KNMF (http://www.knmf.kit.edu) for the PIE tests but it not clear if the KNMF can handle activated samples.

A3. BR-2

To estimate the evolution of materials under irradiation, SCK•CEN can organise tests at the BR2 and the LHMA laboratory (Laboratory for High and Medium Activity with specially protected cells or hot cells).

Further information can be found at the web site: https://www.sckcen.be/en/Research/Infrastructure/BR2

A4. HFR (Petten)

Owned by the European Commission and operated by the Dutch Nuclear Research and consultancy Group (NRG), the High Flux Reactor (HFR) in Petten is one of the most powerful multi-purpose materials testing reactors in the world. Operated at 45 MW, the HFR is a tank in pool type light water-cooled and moderated reactor. It offers a variety of irradiation facilities in the reactor core, in the reflector region and in the poolside facility. It is used for different commercial applications ranging from production of medical radio-isotopes to doping of silicon and nuclear R&D.

Examples for research carried out in the HFR are:

1. Materials testing to ensure safe long term operation of existing nuclear power plants
2. Irradiation tests and qualifications of innovative fuels and functional/structural materials for use in next generation fission reactors
3. R&D for Fusion Reactor technologies, e.g. first wall components

Material Characterization and Qualification

NRG has extensive experience in the development, improvement, characterisation and qualification of existing and new nuclear materials. All aspects can be covered by NRG:

1. manufacture and processing of (radioactive) samples of the quality required;
2. design and build of irradiation facilities on site;
3. irradiation under controlled neutron flux, neutron spectrum and temperature during irradiation: measurement of temperature, flux and dimensions;
4. after irradiation: testing, examination and characterisation in a flexible, shielded hot cell environment treatment and storage of the nuclear waste generated.

A5. LVR – 15

In this facility, Material Irradiation Research and Services provide the following tests: 1) Studies on the compatibility and corrosion behaviour of structural materials in various environments from helium to liquid metals and molten salts 2) Assessment of corrosion damage mechanism under model operational conditions, depending on the level of mechanical stress and composition of the aquatic environment 3) Experiments in the experimental facilities of the research reactor LVR-15, including continuous monitoring of experimental conditions 4) Irradiation experiments focused on material behaviour research and influence of radiation and chemical parameters 5) Study of structural materials' behaviour in the aquatic environment under chemical parameters and radiation conditions 6) Evaluation of corrosion behaviour of structural materials in steam circuits and liquid metals 7) Assessment of local and surface damage mechanism and the nature of corrosion attack in operational conditions 8) determination of corrosion behaviour of fuel cladding materials, including eddy current measurements.

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A6. TRIGA II – Pitesti – SS Core

The largest family TRIGA research reactor, TRIGA 14 MW, is owned by the Institute for Nuclear Research in Pitesti, Romania and commissioned 37 years ago. Originally designed to be operated with HEU nuclear fuel, now TRIGA 14 MW reactor core has been fully converted to LEU nuclear fuel. The core conversion, indispensable to ensure the continuous operation of the reactor, took place gradually, using fuel manufactured in different batches by two qualified suppliers. After completion of the conversion, a modernization program for the reactor systems was undertaken with two main objectives: safe operation and competitive reactor utilization to satisfy the current and future demands and requirements. Radioisotopes production (131I, 125I, 192Ir etc.) for medical applications is also carried out at the TRIGA 14 MW reactor. A method for 99Mo99Tc production from fission is under development. The facility is expected to operate for another 15-20 years, supporting new tests for future generations of reactors, producing radioisotopes production thanks to more efficient new technologies, sustaining research on safety.

A7. BOR 60

Nowadays, JSC "SSC RIAR" operates the majority of Russia's high-flux research reactors: MIR, SM, BOR-60, VK-50, RBT-6 and RBT-10/2.

RIAR's reactors provide a full range of capabilities to test fuel and materials of all types of existing power reactors as well as advanced and innovative ones: water-cooled thermal reactors, including those with boiling and pressurized water, gas-cooled, fast and other types of reactors.

All the above reactors have the state, characteristics and operating parameters comparable with the best world's ones. Nowadays, they are operated at the design parameters and with a capacity factor achieving 0.7.

The experimental base of JSC "SSC RIAR" allows implementing a wide range of tests and examinations of any fuel, structural and other materials, including pre-irradiation examinations of their physical and mechanical properties, irradiation and post-irradiation examinations of irradiated samples in the hot cells.

In addition, at the reactors the personnel have experience in irradiation of components and materials for ITER project.

ITER microwave components can be irradiated in the test reactor RBT-6 equipped with a KORPUS facility with a neutron flux density of $\sim 9 \cdot 10^{10}$ n/cm2·s1.

Besides, RIAR has all equipment to perform the following examinations:

- Optic microscopy;

- SEM (they also have scanning microscopes equipped with energy-dispersion spectrometers, e.g. Supra 55 WDS VP);
- XRD;
- Mechanical tests and hardness measurements;
- Measurements of temperature conductivity and thermal properties.

RIAR has a wide experience in conducting mechanical tests and examinations of various materials.

As for measuring the dielectric properties, they claim to have such experience and, in case of interest, they could upgrade their test and measurement equipment and use the up-to-date techniques to conduct these measurements.

A8. Joyo

The Japan Atomic Energy Agency (JAEA) owes the experimental fast reactor Joyo, which is the first sodium-cooled fast reactor (SFR) in Japan. Joyo reached its initial criticality as a breeder core (MK-I core) in 1977. In 1983, the reactor increased its thermal output up to 100MWt, which allowed starting the irradiation tests of fuels and materials to be used mainly in other SFRs. By 2000, using the MK-II core, thirty-five duty cycle operations and many irradiation tests were successfully completed.

In 2003, the core was then modified to the MK-III version. To obtain higher fast neutron flux, the core was modified from one region core to two-region core with different Pu fissile contents. Consequently, the reactor power increased up to 140 MWt.

After Fukushima NPP accidents, new nuclear safety regulations were issued. As of 2018, Joyo is under review by NRA following the new safety regulations. To meet the new disaster prevention guidelines, the thermal power of Joyo will be reduced to 100MWt.

Experimental Facilities in Joyo

The fuel region is divided into two radial enrichment zones to flatten the neutron flux distribution. The fissile Pu content (239Pu + 241Pu) / (U + Pu)) is about 16 wt% in the inner core fuel and about 21 wt% in the outer core fuel. The fuel region is surrounded by a 25 cm thick radial stainless steel reflector region.

Materials Monitoring Facility (MMF)

PIEs for fuel claddings, wrapper tubes, structural materials, control materials etc. are performed in the MMF. The MMF has several hot-cells such as concrete cells of α-γ sealed type, β-γ type, and lead cells. In the α-γ sealed type, fuels are removed from the segmented fuel pin, and the mechanical tests are conducted such as tensile tests, creep tests and transient burst tests and so on. In the β-γ type cells, a mechanical test, a metallurgy test, dimensional and density measurements can be conducted. The field-emission transmission electron microscopy (FE-TEM) and TEM are equipped to observe the microstructure of irradiated material.

Preliminary irradiation test proposal

Following an exchange of emails, the contact person of the facility as provided the following preliminary proposal for the irradiation tests.

Fused silica windows and small samples can be irradiated in UPR or EXIR at a specific, controlled temperature with neutron flux of about 10^{10}-10^{11} n/cm^2 s. Irradiation condition can be selected lithium, sodium, or inert gas.

After irradiation, these specimens will be transferred to PIE facilities neighbouring Joyo.

Possible examinations with current equipment are: a) Scanning Electron Spectroscopy (SEM) b) Optical microscopy c) X-Ray Diffraction (XRD) d) Outgassing (QMS) e) Electron Probe Micro Analyzer (EPMA) f) Hardness and Micro-hardness

The Joyo Facilities does not have the equipment to perform measurements of dielectric properties. On the other hand, the contact person has stated that they would be prepared to investigate whether other institutes within the JAEA could perform the tests.

Table A1 - Summary of the Testing Facilities

Name		**Type**	**Location**	**Microwave Tests**	**Comments**
Material Research facility (MRF)		--	Culham (Oxfordshire)	Could be performed in collaboration with Strathclyde University	Activity limits: 3.75 TBq 60Co source for hot cells 3.75 GBq 60Co source for instrument cells <10 µSv/h at external wall of shielding
Irradiation Center Karlsruhe (KIT)		---	Karlsruhe	additional information to be provided	Irradiation only with 23MeV protons and not neutrons
BR-2 (with LHMA laboratory)		Thermal Neutron Material Test Reactor (MTR)	Belgium		Tests to reliably estimate the evolution of material properties under irradiation
HFR (Petten)		Thermal Neutron Material Test Reactor (MTR)	Netherlands		After irradiation: testing, examination and characterisation in a flexible, shielded hot cell environment treatment and storage of the nuclear waste generated.
LVR – 15		Thermal Neutron	Czech Republic	additional information to be provided	Materials and fuels tests.

		Material Test Reactor (MTR)			Isotope production. Neutron scattering
TRIGA II – Pitesti – SS Core		Thermal Neutron Material Test Reactor (MTR)	Romania		Materials and fuels tests. Isotope production. Neutron scattering
BOR 60 JSC “SSC RIAR”		Sodium Fast Reactor	Russia	ITER microwave components can be irradiated in the test reactor RBT-6 equipped with a KORPUS facility with a neutron flux density of ~ $9 \cdot 10^{10}$ n/cm2·s1	JSC “SSC RIAR” allows implementing a wide range of tests and examinations of any fuel, structural and other materials (pre and post irradiation).
Joyo		Sodium Fast Test Reactor	Japan	The contact person has stated that they would be prepared to investigate whether other institutes within the JAEA could perform these tests.	PIEs for fuel claddings, wrapper tubes, structural materials, control materials etc. are performed in the MMF. The MMF has several hot-cells such as concrete cells of α-γ sealed type, β-γ type, and lead cells.