CONFERENCE PRE-PRINT

ANTICIPATING TRITIUM IMPACT AND TRANSFER IN FISSION AND FUSION POWERPLANTS
Results from the TITANS project

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Abstract

TITANS (Tritium Impact and Transfer in Advanced Nuclear ReactorS) was built to contribute to research and innovation on cross-cutting activities required to improve knowledge on tritium (T) management with a multidisciplinary approach. Operation of fusion devices and future GEN IV reactors will generate higher amounts of tritium than existing facilities, therefore safety and social acceptability require to tackle potential permeation through the confinement materials and T release in the environment. TITANS pursues challenges identified at each step of the tritium life cycle: develop tritium release mitigation strategies, improve waste management and refine knowledge in the field of radiotoxicity, radiobiology and dosimetry. Progresses carried out on corrosion barrier development and evaluation of hydrogen isotopes behaviour are first presented: SiC anti-corrosion coating deposited on Eurofer97 were developed and proved efficient to prevent corrosion during LiPb exposure. Evaluation of deuterium (D) permeation highlighted that the Permeation Reduction Factor of the SiC-coating deteriorates after exposure to PbLi, while T exposure highlighted the increase of surfacic trapping sites linked with corrosion for non-coated samples: additional testing to enhance multilayer coatings acting as both corrosion and permeation barriers will be pursued. Activities on T detection techniques are then tackled: solid-state NMR (Nuclear Magnetic Resonance) investigations provided unprecedented insights into the speciation and isotopic exchange processes of tritium within cementitious matrices and getter systems. Despite the radiological constraints, the results confirm the capacity of NMR to probe hydrogen isotope incorporation at the molecular scale, thereby offering a powerful characterization methodology for sequestration materials. Last but not least, the study of the genotoxic effects of hydrogenated and tritiated particles of steel and cement on human lung macrophages is introduced. Particles did not impact cell viability but induced genotoxic effects; interestingly, tritiated particles induced significant DNA damage, with the lowest concentration inducing the most DNA damage.

1. INTRODUCTION AND CONTEXT

The TITANS project (Tritium Impact and Transfer in Advanced Nuclear ReactorS) [1], supported by Horizon Europe, was established to provide cross-cutting activities between fission and fusion programs to improve knowledge on tritium (T) emission management. Operation of fusion devices and future fission reactors will generate higher amounts of T than existing facilities. Safety and social acceptability require to tackle potential permeation and T release in the environment at each step of the T life cycle: develop T release mitigation strategies, improve waste management and refine knowledge in the fields of ecotoxicology, radiotoxicology and dosimetry. Progresses were obtained on 3 main objectives:

— **Tritium handling**: enhancement of permeation barriers is crucial to limit contamination of materials and dissemination in the environment, therefore upgrading of potential solutions (notably via surface treatment) along with validation in T environment has been studied. Another dissemination source concerns tritiated particles produced during dismantling phases: feedback from practical T decommissioning activities [2] has been carried out in parallel with binder matrix development to immobilize T metallic dust with minimal T release. Last but not least, management of tritiated water is anticipated via the design of a mobile water detritiation facility;
— **Tritium control:** T measurement and modelling are of crucial importance for real-time safety monitoring in future devices. Advances in T measurements are tackled through the development of gas chromatography, autoradiography, and novel measurements techniques in tritiated solid (Nuclear Magnetic Resonance (NMR), dusts and aerosols using Nuclear Reaction Analysis [3]), along with solutions to measure T in liquid metals. In parallel, benchmarking of various fusion and fission T transport codes from system to detail level has been carried out to adequately estimate T inventories in future setups;
— **Tritium protection:** dosimetric studies tackled accidental exposure to T steel or cement dust by discussing the behaviour of T aerosol in the environment, the biological effects in a mussel's population of T particles exposure, the bio kinetics of T particles ingress through skin and the risk assessment of genotoxic effects on human macrophages. These data will be key to establish a radiation dose-response relationship for various biological models.

All results from the project are available on TITANS site [1]; one scientific achievement for each theme is detailed below.

2. TRITIUM PERMEATION THROUGH EUROFER EXPOSED TO LIPB CORROSION AND PROTECTED BY SIC COATING

Increase T levels in future nuclear devices, combined with the foreseen high temperature operation, is expected to increase permeation through the confinement materials. On the other hand, stability on the first wall materials can be jeopardized by the chemical operational conditions: most notably, liquid metals (such as LiPb) are to be used as heat transfer fluids, as they allow much higher efficiency than traditional coolants at high temperature, yet they are highly corrosive. Providing a corrosion protection of structural materials in contact with them is therefore a crucial issue; innovative barriers development has to integrate closely the impact on hydrogen isotope behaviour, in particular the evolution of the obtained Permeation Reduction Factor (PRF). Development and test of a corrosion-barrier solution deposited onto the material of interest, while keeping the structural properties and low cost of the steel (here, Eurofer97), was carried out in CIEMAT, along with the subsequent exposure to LiPb. In parallel, estimation of permeation, inventory and desorption of various H isotopes was led on CEA experimental set ups to estimate the coating impact on T trapping and potential leakage out of the system.

2.1. Corrosion coating barrier and LiPb exposure in CICLO

A dedicated study carried out at CIEMAT and UPM compared the properties of diverse coatings (Al_2O_3, AlN and SiC) deposited by sputtering in planar and non-planar geometries [4]. Al_2O_3 was ruled out as it was observed that in these coatings Li penetrates throughout the entire thickness in a significant amount, which under neutron irradiation could have some important drawbacks as lithium will transmute into He and T. AlNx coating proved to be insufficiently adhesive, as during some tests coatings completely delaminated from the Eurofer substrate. Therefore, SiC coating was chosen as the best candidate for the permeation study after LiPb exposure. Before deposition, all Eurofer97 substrates were mirror polished, using a 0.04 μm particle size colloidal silica suspension in a vibratory polisher for the last polishing step. Then, substrates cleaning was performed with ultrasonic washing

in alkaline detergents, rinsing with de-ionized water, cleaning with isopropanol and air-drying. Substrates to be coated were introduced in the deposition chamber and underwent a plasma etching process in Ar atmosphere to remove surface impurities, before deposition of the 1.1 µm thick SiC coatings performed by RF sputtering. Further details about the deposition process and optimization of coating properties can be found in [4].

Static PbLi corrosion experiments took place on the CICLO device [5]: Eurofer97 and SiC-coated Eurofer97 samples were introduced in magnesia crucibles. Subsequently, PbLi ingots were polished in order to remove the native oxide layer and placed on gratings on top of the crucibles. During melting, this grating retains the remaining PbLi oxides, ensuring high PbLi purity during the corrosion experiments. Once the samples are in the oven, two thermal cycles at 100°C and 150°C are performed in vacuum in order to degas the oven walls. After this, Ar is introduced in the oven and the temperature is increased to 450°C, where it remains for the duration of the experiment (1500 h). While Eurofer97 samples showed the typical signs of degradation by PbLi, SiC-coated samples did not present any degradation: after extraction and cleaning, the samples were sent to CEA for the deuterium (D) permeation and T inventory experiments.

2.2. Permeation Reduction Factor (PRF) determination

The PRF was measured at CEA using the Hypertomate gas-driven permeation (GDP) experiment presented in [6]. In this experiment, while the sample is held at a constant temperature using an oven, gas (H_2, D_2 or a mixture of both) is introduced in the upstream section. As permeation occurs, the resulting increase in downstream pressure is measured via a pressure jauge assisted by a mass spectrometer. At a given temperature, this operation is repeated several times with varying upstream pressure (ranging from 50 to 500 mbar). The experiments were conducted in Hypertomate with gaseous D_2 at increasing temperatures ranging from 300°C to 500°C with 50°C steps. At each temperature, a minimum of 6 permeation cycles were performed. In parallel, a tritium gas exposure and inventory study was carried out in Saclay Tritium Lab: this way, additional trapping sites potentially present at the surface can be evaluated with increased sensitivity thanks to autoradiography. Coupling D permeation at high temperature with T surfacic inventory allows a stronger assessment of hydrogen isotopes behaviour, combining PRF estimation with potential additional trapping sites in the material as a function of surface conditions.

The Permeation Reduction Factor (PRF) is a quantity that is used to evaluate the efficiency of a given coating regarding the decrease of the permeation flux through the sample. It is defined in Eq. 1 as followed:

$$\mathrm{PRF} = \frac{J_{\mathrm{perm,\ uncoated}}}{J_{\mathrm{perm,\ coated}}}$$

EQ. 1. Permeation Reduction Factor definition.

The PRF primarily depends on the coating type and varies with the temperature of operation. The most straightforward way to evaluate the PRF of a given coating technology is to conduct the analysis on two identical samples (in terms of composition, geometry/thickness, and preparation), one of which is coated and the other uncoated. If the analysis is performed this way, the PRF formula can be applied directly for each temperature. In the comparison between the SiC-coated and non-coated Eurofer97 samples, thickness variation is non negligeable: thicknesses of the reference bare Eurofer97 and LiPb samples are similar, while the SiC-coated is 50 % thinner and the SiC-coated exposed to LiPb is half as thick. Yet the direct evaluation of the PRF given in Eq.1 can only be performed if both samples have the same thickness or if they are both in Diffusion-Limited Regime (permeation flux is proportional to $1/e$ where e is the thickness of the sample). Yet it appears from the results that permeation flux is affected by surface constants in some cases (notably SiC-coated Eurofer exposed to LiPb): permeation modes and thickness both varying between the 4 sample conditions, a direct evaluation of the PRF is therefore out of reach. A workaround to this issue was proposed by assuming Diffusion-Limited Regime (DLR) and Surface-Limited Regime (SLR - permeation flux is affected by surface constants only and does not depend on the thickness of the sample) as upper and lower bounds.

Results obtained on Hypertomate highlighted first that permeation-wise, Eurofer exposed to LiPb is the same as bare Eurofer: local oxidation did not seem to jeopardize D permeation. However, the T retention at the surface changes drastically as autoradiography reveals a modification in retention in the first layers of the material: LiPb exposure and corrosion has created additional sites for T trapping that appear strongly inhomogeous at the surface as can been seen in Fig. 1.

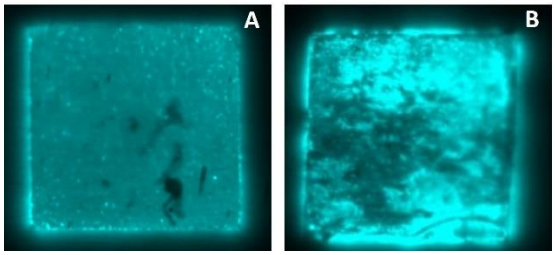

FIG. 1. Autoradiography after T gas loading and desorption at 200°C (blue intensity proportional to T activity) for non-coated Eurofer (A) and non-coated Eurofer after LiPB exposure (B): although permeation was non affected by surface corrosion, inhomogeneities appearing at the surface strongly increase T retention locally. Samples are 9×9 mm^2.

Comparison between lower and upper bounds PRF estimations for SiC-coated Eurofer and SiC-coated Eurofer exposed to LiPb permeation are displayed in Fig. 2 as a function of temperature. SLR evaluation results in a higher PRF, as surface processes are slowing down the overall permeation. SiC-coated Eurofer yields a PRF ranging from 10 to 16, while it ranges between 2 to 10 for SiC-coated Eurofer after LiPb exposure, showing that the exposure to PbLi degrades the PRF. Overall, these PRFs are relatively low, and an enhancing the permeation barrier effect is required for long-term use in future devices.

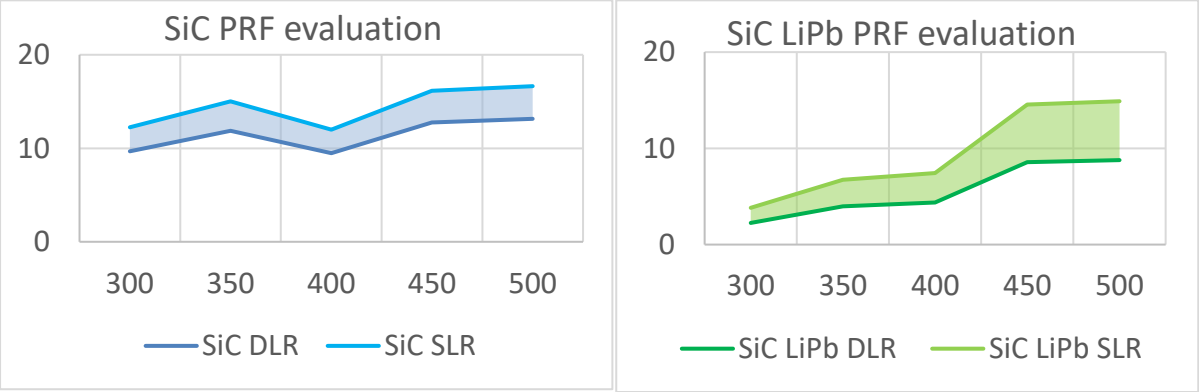

FIG. 2. PRF evaluation for the Eurofer with SiC layer (on the left) and for the Eurofer with SiC layer after LiPB exposure (on the right) considering the lower and upper bounds by estimating the two limiting permeation regimes: Diffusion-Limited Regime (DLR) and Surface-Limited Regime (SLR).

3. TRITIUM MEASUREMENTS: DEVELOPMENT OF THE NUCLEAR MAGNETIC RESONANCE (NMR) TO MEASURE TRITIUM IN SOLIDS

Solid-state NMR has been established as one of the most powerful spectroscopic method for structural and dynamical investigations of a wide range of materials. High Resolution techniques such as Magic-Angle (sample) Spinning (MAS) is now the most widespread approach for acquiring high-resolution spectra aiming at a quantitative identification of all chemical species of the probed nucleus. Potential use to characterize chemicals bonds formed by tritium are very appealing, as T (or ^{3}H) is the most sensitive nuclei in the periodic table; a major challenge is to meet the complex safety requirements concerning preparation and conditioning of tritiated samples for NMR study. At CEA Saclay, an NMR platform for Tritium MAS NMR studies exists, with Helium-3 NMR capabilities [7]. As innovative T characterization tools are needed, TITANS explored potentialities of ^{3}H NMR for studying representative solids for tritium sequestration and contaminated materials, focusing on cements-based systems: MKPc-based materials that are currently investigated as potential candidates for tritium sequestration matrices, and CEM-I cements which are used for production of tritiated dusts (in relation with radiotoxicology studies carried out in the project).

After initial cement preparation, samples in powder form were tritiated in Saclay Tritium Lab: after exposure of the samples to 400 mbar T_2 during 18h, a delayed T-degassing period of about 1 month was observed, before samples could be packed in NMR containers. For each sample about 20 mg of powders were packed in flamed-sealed pyrex tubes for a total activity of less than 100 MBq. T NMR spectra with a reasonable signal-noise ratio

could be collected with 5h to 10h of acquisition time. The spectra could provide qualitative information about the speciation of the trapped tritium in the matrices for MKPc without T-getter (MnO2/Ag2O). The NMR data suggest an isotopic exchange between ^{3}H and ^{1}H on structural (or cristallographic) molecular water molecules in K-struvite (the main phase formed in hydrated MKPc materials). This interpretation is supported by ^{1}H-hydrogenation experiments using exposure to $^{1}H_2$ gas of deuterated MKPc samples.

^{3}H NMR signal could be detected as well in CEM-I particles, but attempts to prepare NMR rotors (sample container for MAS NMR that are spun up to 10000 Hz during experiments) failed because of the continuous degassing of the tritiated-MKPc (incorporating tritium getters). This led to a surface contamination higher than 4Bq/cm^2 that prohibits the rotor to be transferred from the tritium laboratory to the NMR laboratory. This failure led us to investigate the degassing behaviour of the tritium getter after hydrogenation (with T_2 and D_2 gas). NMR results for deuterated-getters suggest that an isotopic $^{2}H/^{1}H$ exchange (catalysed by Ag) occurs with air humidity, as well as in the case of ^{3}H. This can explain the contamination of the MAS NMR rotor prepared with MKPc sample incorporating T-getter particles. In parallel, regular NMR experiments were performed on different CEM-I samples used for the dust production for the radiobiology studies and significant variations in the hydration of the particles are measured whereas no carbonation is observed: future application to T samples will provide a unique insight for the fine characterization of the chemical bonding of T.

The solid-state NMR investigations provided unprecedented insights into the speciation and isotopic exchange processes of tritium within cementitious matrices and getter systems. Despite the radiological constraints that limit extensive experimentation with tritiated samples, the results confirm the capacity of NMR to probe hydrogen isotope incorporation at the molecular scale, thereby offering a powerful characterization methodology for sequestration materials.

4. RISK ASSESSMENT OF GENOTOXIC EFFECTS ON HUMAN MACROPHAGES AFTER TRITIUM PARTICLES EXPOSURE

TITANS aims to improve the knowledge in the field of radiotoxicity of tritiated particles that could be emitted during the decommissioning of a nuclear power plant: after inhalation, the major target is macrophages that uptake particles in order to eliminate it from the lung. As a consequence, studying genotoxic effects on THP-1 macrophages will allow us to assess the most sensitive indicator of effect; dose reconstruction to cell nuclei and their genomic content for in vitro exposures will also allow to make predictions on the expected level of DNA damage, which can serve as a biological indicator of dose ("bio-dosimeter") and be directly correlated to experimental data. Previous work in TRANSAT [8] on the impact of steel and cement particles on human epithelial lung cells [9] has been expanded during TITANS by evaluating genotoxic effects for the lung immune system on 3 points: internalization of particles in the macrophages, quantification of chromosomal damage and quantification of the cellular antioxidant defense and the reactive oxygen species.

The study of the internalization of particles was carried out at the flow cytometer AMUCICYT platform (available for hydrogenated particles only): as the main role of THP-1 monocytes/macrophages is the elimination of cellular debris and particles in the lungs, internalization is of particular importance to assess radiation damage to the cell nucleus by T β-emission. Cellular uptake of the particles by the cells after a 24h exposure to hydrogenated cement and stainless steel (SS316L) particles were performed by flow cytometry by measuring the increase in scattering of the laser light (see Fig. 3). Results highlight that both types of particles are internalized, with significant levels at 50 µg/ml for stainless steel particles and at 100 µg/ml for cement particles.

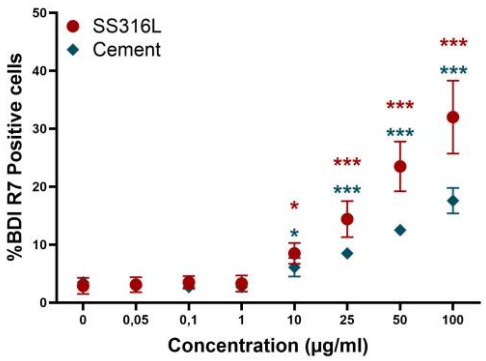

FIG. 3. Uptake of stainless steel (SS316L) and cement particles by macrophages observed by flow cytometry. Bright Detail Intensity (BDI)R7 increases for cells with complex structure, i.e. having internalized a particle.

Quantification of DNA stand breaks and chromosomal damage was carried by the γH2AX assay [10]. Hydrogenated stainless steel particles do not induce DNA damage, whereas tritiated particles induce a significant increase at all concentrations (1-100µg/ml) following 2-hours and 24-hours exposures. However, DNA damage was observed when cells were exposed to hydrogenated cement particles at 50 µg/ml and 100 µg/ml for 2h and only at 100 µg/ml for 24h. Additionally, exposure to tritiated cement particles at all concentrations (1-100µg/ml) for 2-hours and 24-hours triggered DNA damage. For chromosomal damage, both types of particles significantly increased loss of chromosomes and chromosomal breakage for all concentrations, but a dose-dependent effect was observed only with tritiated particles.

Cell viability after exposure to particles was studied to determine cytotoxicity after 2h and 24h after exposure to hydrogenated or tritiated particles up to 200 µg/ml. For hydrogenated cement particles, we observed a slight cytotoxic effect after 24h at 200 µg/ml (66.1% of cellular viability). For tritiated cement particles, we observed no cytotoxicity at 2h at 24h at concentrations up to 200 µg/ml. These results were the basis to define the concentration range for the quantification of the reactive oxygen species (ROS), where production of reactive oxygen species (ROS) was followed over a period of 24h. Hydrogenated stainless-steel particles displayed a significant increase in ROS production that was both concentration-dependent and time-dependent. Using tritiated stainless steel particles, the increase in ROS production was higher than using hydrogenated ones for 30-min, 60-min, 2-hours et 4-hours exposure duration. For hydrogenated and tritiated cement particles, the increase was significant at various tested concentrations ranged between1 and 100 µg/ml, but small. These increases tended to be higher with tritiated than with hydrogenated particles but the control exposure with tritiated water (HTO) of similar T activity did not increase ROS production, hence highlight an impact specific to tritiated particles.

Last but not least, modelling of tritium dose deposition at the cellular level was carried out through simulations performed using the Monte Carlo radiation transport code PHITS for dosimetric assessment and characterization of the radiation field in cell nuclei. Analytical functions of the initial DNA damage yield, relying on a database of results obtained with the biophysical track-structure code PARTRAC, have been then used to estimate tritiated particle biological effectiveness based on DNA damage induction. Due to the short range of the electrons emitted during tritium decay, the exposure of cell population is highly inhomogeneous: in other words, the electron energy is released only to nuclei of cells which have internalized a particle. In this sense, to properly evaluate the dosimetry, at least two dose ranges should be considered: the first one interests cells not directly in contact with a tritiated particle, receiving dose only from tritium released to the medium, and it is not significant. The second range interests cells directly in contact with a tritiated particle. The dose to a nucleus of a cell in contact with a tritiated steel particle is of about 1 Gy (99 cGy), which is significant as well as the estimated initial damage yield associated (57 DSBs).

To summarize, the THP-1 macrophages exposure to cement and SS316L particles for 2 and 24 h did not induce significant cytotoxicity; a significant increase in the production of ROS was only witnessed for SS316L particles. It is of particular matter that significant chromosomal damage occurred after exposure to both hydrogenated and tritiated particles, hence impact of such particles even in non-tritiated environment should be evaluated; however, only tritiated particles induced a dose-dependent response. Tritiated particles induced significant DNA damage with the lowest concentration inducing the most DNA damage, highlighting the need to extend radio toxicity studies to low-activity exposures.

5. CONCLUSIONS AND PERSPECTIVES

Thanks to a large and diverse consortium combining T expertise throughout 21 partners in the EU and the UK, TITANS was able to achieve significant progress towards a safe and sustainable use of tritium in future nuclear devices. The comprehensive coupling of coating development, exposure to LiPb and hydrogen isotope permeation and inventory characterization allowed the rise of SiC as a promising candidate for Eurofer97 corrosion protection, although additional developments will be needed to achieve an outstanding permeation barrier; the methodology used is set and ready to test additional coatings or innovative solutions. During TITANS, the importance of dust studies grew for its impact on several areas from decommissioning to worker exposure dose, up to tritium

transmission in the environment, transcending both fission and fusion communities and strengthening the measurement needs: no diagnostic is currently available to detect in real-time tritium activities in dust potentially generated during operation, maintenance or decommissioning activities. This is all the more important for devices where tritium permeation will be higher, as more materials will be exposed to tritium, therefore creating more potential dust sources during dismantling phases with a larger range of chemical composition. TITANS first solid-state NMR investigations raised a key point on the requirement of a good knowledge of the tritium degassing behaviour of the studied materials with the necessity of real T experiments to evidence potential isotopic exchanges. To get rid of leaks issues on the future studies, pressurized samples will be possible thanks to a new NMR instrument compatible with adequate probe heads. Concerning the radiotoxicity study, results show that the tritiation of particles can enhance their genotoxicity. As macrophages and monocytes are the main targets in case of an accidental exposure, all the more as macrophages can remain for years in the lungs, there is a need to further study the immunotoxicity and the long-term effects these particles could have. A better characterization of the presence of quasi-ultrafine particles, their potential localization in the cells and their specific toxicity is also of crucial interest.

Beyond these results, the most important fruit of TITANS may be the multidisciplinary and transverse community which grew over the scientific exchanges: it materialized in various training and dissemination efforts open to scientists from both fusion and fission fields, in the hope to reinforce knowledge on the challenges ahead and prevent T issues to become Achilles' heel for future nuclear solutions to the energy transition.

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