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Counter-current isotope swamping in a membrane reactor: The PERMCAT process and its applications in fusion technology

D. Demange*, M. Glugla[1], K. Günther, T.L. Le, K.H. Simon, R. Wagner, S. Welte

Karlsruhe Institute for Technology, Institute for Technical Physics, Tritium Laboratory Karlsruhe, Postfach 3640, D-76021 Karlsruhe, Germany

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ABSTRACT

A PERMCAT reactor is a catalytic membrane reactor that combines a Pd/Ag membrane and a catalyst bed. It has been developed to ensure very high tritium recovery from the unspent fuel of fusion machines using deuterium tritium mixtures. The PERMCAT process takes advantage of simultaneously unlocking chemically bound tritium via heterogeneously catalysed isotope exchange reactions and removing tritium via its selective permeation through the membrane. The PERMCAT reactor operated in the counter-current isotope swamping mode allows a very low tritium activity at the outlet of the component to be maintained.

Two main issues have been solved to achieve efficient and reliable PERMCAT operation. Firstly, the mechanical design has to cope with the elongation and deformation of the membrane resulting from thermal expansion and lattice parameter increase under operation with hydrogen. Secondly, the catalyst material has to be chosen in order to promote isotope exchange reactions while minimising the numerous side reactions that occur especially when the mixture contains carbon oxides. This paper presents a general overview of the R&D performed at the Tritium Laboratory Karlsruhe for PERMCAT technology. Technical solutions to solve both issues together with relevant experimental results including processing tests with tritium are discussed.

1. Introduction

Energy production using nuclear fusion reactions has been identified as a future alternative to present nuclear fission power plants. The nuclear fusion reaction between deuterium and tritium is the most suitable in term of efficiency. Tritium being radioactive, special requirements for fuel handling and processing have to be observed. In particular, the unspent tritium has to be recovered so that it can be reused while minimising its release in the environment. For this purpose, a closed loop for the inner-fuel cycle [1] is required as shown in Fig. 1. One of the key systems is the tokamak exhaust processing (TEP) system that separate and recover practically all hydrogen isotopologues (noted Q_2, with Q = H, D, T) coming from the torus, while the rest of the stream is routed for a latter processing step before final release to the stack.

The gas to be processed in TEP at a total flow rate of typically 200 Pa m^3 s^{-1} mainly consists in un-burnt Q_2 and He as product of the fusion reactions. In addition, the extreme conditions in the torus during operation and plasma–wall interactions produce impurities with amounts and compositions depending on the first wall materials. Traces of carbon- and oxygen-containing species are expected due to some residual oxygen from venting procedures, and the use of carbon-based materials for plasma facing components. The tritium recovery requirement for TEP is very high [2]: expressed in term of decontamination factor (DF), a DF of 10^8 means that 99.999999% of the tritium is recovered by TEP and transferred to the isotope separation systems. Such a high efficiency can only be achieved with a multi-stage process.

At the Tritium Laboratory Karlsruhe (TLK), the technology for the D–T fuel cycle is studied at the technical scale and with relevant tritium concentrations. In particular, the high requirements for TEP have been fully demonstrated using the so-called CAPER process [3] shown in Fig. 2. This multi-stage process for tritium recovery relies on permeation and heterogeneously catalysed reactions. Permeation through Pd/Ag membranes is a straightforward process to recover the molecular tritium. The recovery of tritium chemically bound in molecules, e.g. in methane CQ_4 or in water Q_2O, asks for additional efforts, and heterogeneously catalysed reactions are promoted in combination with permeation. The combination of two reactors (catalytic reactor + permeator) operated in a closed loop is used in the impurity processing stage (2nd stage). While the CAPER process relies on hydrocarbon cracking and water-gas-shift reactions [4], the HITEX process based on isotope swamping [5] has also been studied. The possibility to combine both the catalytic

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* Corresponding author. Tel.: +49 7247 82 8439; fax: +49 7247 82 2835.
E-mail address: david.demange@kit.edu (D. Demange).

[1] Present affiliation: ITER Organisation, Department for Central Engineering and Plant Support, Cadarache, 13108 St Paul-Lez-Durance, France.

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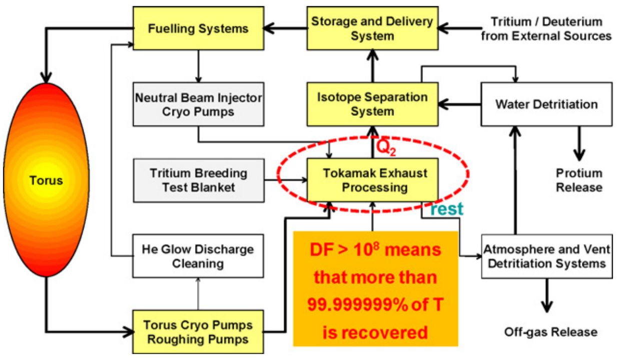

Fig. 1. The closed inner-fuel cycle of the ITER fusion machine with yellow blocks showing the systems that close the fuel cycle (DF means decontamination factor, Q_2 reflects the 6 hydrogen isotopologues with Q = H, D or T). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of the article.)

reactor and the permeator in a single component has also been demonstrated using two totally different approaches. The palladium membrane reactor (PMR) process [6,7] using water-gas-shift reaction has been applied to water detritiation. Alternatively, the so-called permeator catalyst reactor (PERMCAT) process [8–10] as final clean-up stage of CAPER relies on isotope exchange reactions.

The principle of the PERMCAT process based on counter-current isotope swamping is shown in Fig. 3. The gas to be detritiated is fed into the impurity side of the reactor that contains the catalyst bed, while fresh (tritium-free) hydrogen is supplied in purge side in a counter-current mode. The process can be decomposed in three main steps: first the fresh hydrogen permeates through the membrane into the impurity side, then isotope exchange reactions occurring at the catalyst surface liberate tritium in the molecular form, finally the molecular tritium permeates back to the purge side where it is recovered and removed. The hydrogen counter-current swamping allows keeping the impurity outlet at a very low tritium level, thus ensuring very high DF and effective tritium recovery. A logarithmic tritium concentration profile along the length of the reactor is anticipated from model and simulations. A DF to the square is expected by doubling the length; thus, the length of the reactor is crucial for detritiation performances.

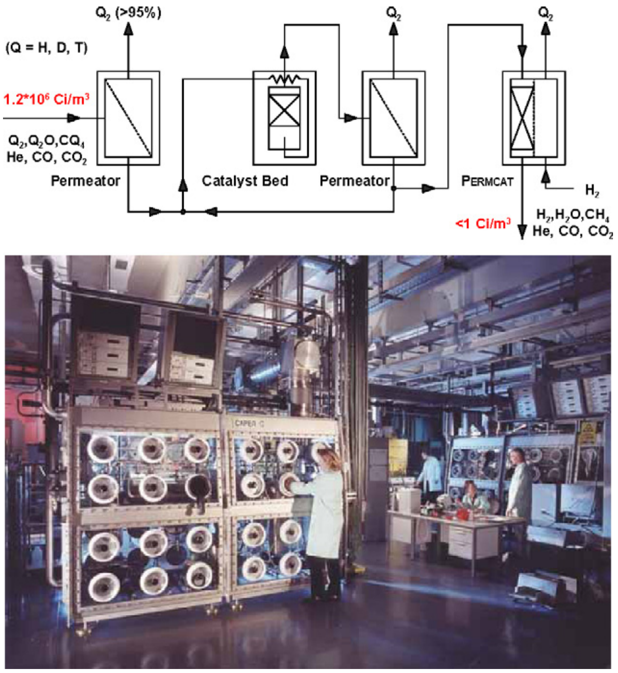

Fig. 2. The CAPER process (top) and facility (bottom) at the Tritium Laboratory Karlsruhe for the experimental demonstration of TEP systems, comprising as final clean-up a catalytic membrane reactor (Q = H, D or T).

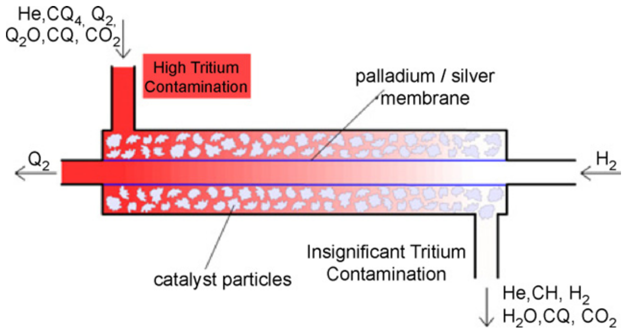

Fig. 3. Principle of the PERMCAT process based on the counter-current isotope swamping technique (Q = H, D or T) (from Ref. [9]).

Several issues have to be solved to ensure reliable and effective PERMCAT operation. On the one hand, both the thermal expansion of the membrane at the nominal operating temperature (400 °C) and the lattice parameter increase of Pd/Ag resulting from hydrogen absorption yield to mechanical stresses of the membrane. For this reason, the membrane cannot be straightforwardly fixed at both ends as shown in Fig. 3. On the other hand, the catalyst material promoting isotope exchanges reactions has overcome the complex side chemistry that might occur in particular if the gas to be processed contains carbon oxides. This paper presents the technical solutions used to solve the mechanical issue, describes the PERMCAT chemistry and catalyst material optimisation, and reports on relevant experimental results including processing tests with tritium using in the CAPER facility operated at TLK.

2. PERMCAT mechanical issue

If the membrane reactor is highly integrated in a complex architecture of many systems, which is the case for the PERMCAT reactor within the fuel cycle of a fusion machine (Fig. 1), it has to withstand some non-nominal operation modes due many off-normal events. A loss of performance cannot be tolerated so that the mechanical design has to propose technical solutions for robust component enabling reliable operation.

2.1. Pd/Ag membrane under hydrogen atmosphere

A dedicated study has been carried out to observe the mechanical behaviour of a Pd/Ag membrane operated under hydrogen ambiances [11]. An apparatus has been built that enables visual inspection of a Pd/Ag membrane at different temperatures (between room temperature and 400 °C) and under different hydrogen atmospheres up to 1 bar at both sides of the membrane. The outer tube made of quartz allows *in situ* observations during operation. In particular, the length of the membrane is continuously measured at the millimetre scale.

A finger-type membrane (400 mm long, 100 μm thick, 2.3 mm outer diameter) together with its dedicated inner capillary tube has been used for these tests. A rather limited elongation of the membrane is observed during nominal operation at 400 °C and 1 bar H_2. Here, the thermal expansion is the main source (80%) of the elongation measured at 0.6% relative to the length of the membrane. However, the pictures in Fig. 4 highlight the most drastic effects that could be encountered. Compared to the picture on the top showing the initial membrane at room temperature under inert atmosphere, both pictures at the bottom reflect the dramatic

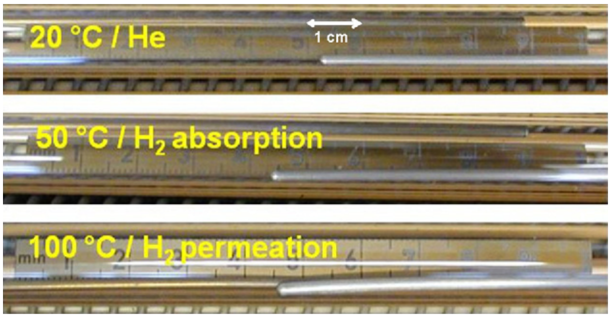

Fig. 4. Elongation and deformation of a “finger-type” Pd/Ag membrane (400 mm) during off-normal operation (hydrogen atmosphere and temperature below 100 °C).

effects during operation at a temperature of 100 °C (or lower) and operation at 1 bar hydrogen at both sides (absorption) or at one side (permeation). In case of a loss of heating capacity resulting for example from an involuntary shut-down operation the membrane can elongate up to more than 2% and can become deformed. To some extent, deformations and change in length are irreversible and remain after the experiment. Under such off-normal operation in a catalyst membrane reactor, the membrane integrity and specificity could be affected due to the presence of the catalyst bed.

2.2. *Different PERMCAT mechanical designs*

Fig. 5 shows the different PERMCAT mechanical designs that have been tested. On the top, Fig. 5(a) the first generation of PERMCAT reactor is based on a three coaxial tubes pattern using a finger-type Pd/Ag membrane, together with an inner capillary tube. The catalyst bed is enclosed in the shell of an outer stainless steel tube. Such arrangement tends to make more complicated the design and production of multi-tube PERMCAT reactors for technical scale applications.

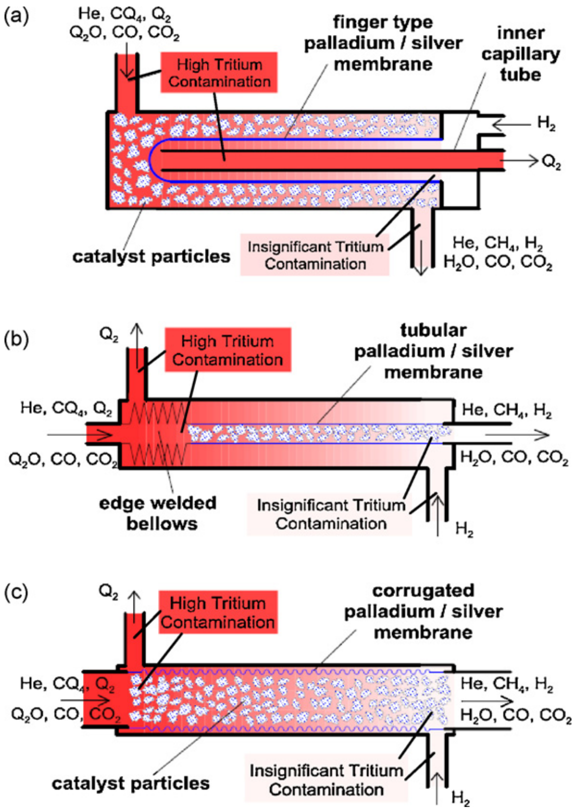

Fig. 5. Different mechanical designs for single-tube PERMCAT reactor: (a) finger-type membrane and additional inner capillary tube, (b) straight membrane coupled with a stainless steel edge welded bellow, and (c) corrugated membrane (from Ref. [10]).

Fig. 6. View during manufacturing of a tritium compatible PERMCAT reactor mounted on a CF250 flange with 2 single-tube units in line (on the left) and the PERMCAT unit comprising a corrugated membrane (on the right).

Upgraded PERMCAT mechanical designs [11] shown in Fig. 5(b) and (c) simplify the pattern to a 2 coaxial arrangement. The first option comprises a tubular membrane joined with a stainless steel metal bellows. The bellows has to be extended during the manufacturing process so that it can compensate any membrane elongation during operation. Using a special corrugated membrane the PERMCAT manufacture can be greatly simplified. Therefore significant efforts have been made to produce defect-free Pd/Ag corrugated membranes [12]. Starting from a commercial tubular Pd/Ag membrane and using standard tools and methods for corrugation process (hydraulic forming), the production of corrugated membranes has been optimised to yield reproducible corrugations without any cracks. This can be achieved if the initial ductility of the membrane is increased prior to the corrugation process. An annealing temperature of about 1000 °C has been found to be adapted. A tritium compatible PERMCAT reactor shown in Fig. 6 has been built using two single-tube units in line comprising such corrugated membranes. Selected experimental results obtained with this reactor are presented in Section 4.

3. PERMCAT chemistry

The catalyst material has to promote isotope exchanges reactions, but other chemical reactions can also take place. Depending on the chemical composition of the gas to be processed, and in particular if it contains carbon oxides, a complex chemistry involving several competitive and interdependent reactions cannot be excluded. This may decrease the nominal detritiation performances of the PERMCAT process. A dedicated study to characterise the catalytic properties of different materials and supports has been performed.

3.1. PERMCAT chemistry and side reactions

The catalyst bed of the PERMCAT reactor is assumed to support isotope exchange reactions between protium being supplied for swamping through the Pd/Ag membrane tube and tritium bound in molecules. For example the isotopic exchange between protium and tritiated methane (1) or tritiated water (2) are promoted at the surface of the PERMCAT catalyst material (Q = H, D, T):

$$2H_2 + CQ_4 \leftrightarrow CH_4 + 2Q_2 \quad (1)$$

$$H_2 + Q_2O \leftrightarrow H_2O + Q_2 \quad (2)$$

Beside the pure isotope exchange mechanism, a complex chemistry can also proceed in parallel. In particular, if the gas mixture contains carbon oxides, many competitive and interdependent reactions are anticipated. Hydrocarbon cracking occurs at the surface of the catalyst material [13] leading to carbon deposition according to the methane decomposition reaction (3). The syngas reaction (4) can be an additional source of carbon deposition. This carbon can be consumed by carbon dioxide according to the Boudouard reaction (5) thus producing carbon monoxide:

$$CQ_4 \leftrightarrow C + 2Q_2 \quad (3)$$

$$CO + Q_2 \leftrightarrow C + Q_2O \quad (4)$$

$$2CO \leftrightarrow C + CO_2 \quad (5)$$

Carbon oxides can react with molecular hydrogen introduced in excess for purge according to different methanation reactions such as dry reforming of methane (6), steam reforming of methane (7) and (8). The inverse water-gas-shift (WGS) reaction (9) is also anticipated:

$$2CO + 2Q_2 \leftrightarrow CQ_4 + CO_2 \quad (6)$$

$$CO + 3Q_2 \leftrightarrow CQ_4 + Q_2O \quad (7)$$

$$CO_2 + 4Q_2 \leftrightarrow CQ_4 + 2Q_2O \quad (8)$$

$$CO_2 + Q_2 \leftrightarrow CO + Q_2O \quad (9)$$

All these side reactions ((3)–(9)) handicap the process in a whole since these reactions tend to immobilise hydrogen (and tritium) in molecules. Thus it does not participate to the isotope exchanges so that the efficiency for tritium removal through the perm-selective membrane is reduced. The detritiation performances could be drastically affected (reduction of decontamination factor at a given throughput, or reduction of the throughput at a given decontamination factor). In addition, water production and its potential condensation at the impurity outlet of the PERMCAT reactor could become a significant problem. Therefore a dedicated study has been performed to identify and chose the most suitable catalytic material.

A numerical simulation of PERMCAT has been performed with a simplified geometry (1 dimension) and a simplified chemistry including isotope exchanges reactions (1) and (2) plus dry reforming reaction (7) and water-gas-shift reaction (9). A PERMCAT experiment containing relatively high amounts of carbon oxides (5 and 8% for CO and CO_2, respectively) has been used to benchmark the simulation [14]. Analysing the concentration profile of CO, CO_2, Q_2, CQ_4 and Q_2O along the reactor, it seems that methanation reaction is very effective at the entrance of the reactor as long as CO is present in the mixture then the inverse water-gas-shift reaction predominates.

3.2. Catalyst material optimisation

Many transition metals are known to have a high catalytic activity with respect to the methane–deuterium isotope exchange reaction [15]. The unwanted methanation reaction is known to proceed via a carbon–oxygen bond rupture consecutive to a carbon oxide dissociative adsorption [16] and is a structure sensitive reaction [17,18]. The methane–deuterium isotope exchange reaction proceeds via a carbon–hydrogen bond rupture and is a rather structure insensitive reaction [16].

A dedicated study to characterise the properties of different catalyst materials on different supports has been carried out [19]. Numerous catalysts have been prepared, characterised and compared along with commercially available materials. Aluminium oxide and silica were used as catalyst support materials. Experiments have focused on isotope exchange reactions between methane and deuterium as well as methanation of carbon monoxide. With regards to the isotope exchange reaction between methane and deuterium, it has been demonstrated that Pt-based and Pd-based catalysts give the best performances. However, it has been observed that methanation reaction is strongly promoted by the Pt-based catalyst. This is not the case for Pd-based catalyst, however, a drastic reduction of the methane–deuterium isotope exchanges has been observed in the presence of carbon monoxide. The adsorption of CO on the catalyst is assumed to block the reaction sites for isotope exchange reactions. This proves that for the particular use of the PERMCAT process as final clean-up stage of the TEP systems (i.e. process complex gaseous mixtures potentially containing carbon oxides), the catalyst material optimisation cannot only focus on pure isotope exchange reactions; side reactions and side effects have also to be carefully considered. As the best compromise, the commercial nickel catalyst supported on kieselguhr (NIKKI 111) has been chosen for PERMCAT reactors. This choice for catalyst material will be eventually revisited for other specific applications other than the nominal one, for example the process of tritiated water.

4. Processing performances

The PERMCAT reactors are developed to be finally used with tritium. In order to limit radioactive tritium exposure for workers, all the processes and components are operated inside a glove box. This tends to make more difficult late changes after integration into the systems. Therefore, prior to its use with tritium, it is mandatory to check all functionalities of the PERMCAT reactor. In addition, a first evaluation of the processing performances is also very useful in order to operate with tritium only PERMCAT reactors that show satisfying processing capabilities. The methodology for the inactive (without tritium) commissioning of PERMCAT reactors enabling the comparison between different reactors is presented, and experimental results with tritium focussing on side chemistry are discussed.

4.1. H_2O/D_2 isotopic exchanges and inactive commissioning

Several PERMCAT single-tube units based on different mechanical designs have been tested using isotope exchange reactions between water vapour and deuterium. Details of the experimental setup and procedure have been already reported [11]. In short, distilled water (H_2O) is fed as vapour from a vaporiser and mixed with helium as carrier gas (typically 1 to 1 mixture) while pure deuterium (D_2) is fed in counter-current. At the outlet of the PERMCAT impurity side, the water is collected in a cold trap operated at the liquid nitrogen temperature. After more than 6 h of continuous operation, the collected water is recovered. The amount of deuterium in the water (D at% in HDO) directly reflecting the process efficiency is measured using infra-red spectroscopy at 2510 cm^{-1} (O–D bond stretching). Both upgraded design presented in Section 3.2 have been tested according to this procedure.

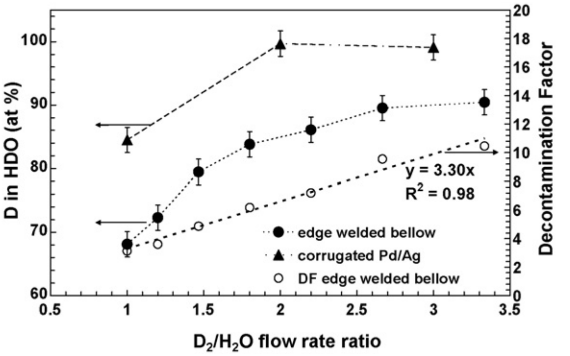

Fig. 7. Processing performances obtained using H_2O/D_2 isotope exchange reactions for both upgraded PERMCAT mechanical designs, and influence of the swamping effect.

Part of the study has focused on the decontamination factor obtained for different swamping ratio (i.e. the ratio between the deuterium flow rate and the water flow rate) since it reflects well the overall (economical) efficiency of the PERMCAT process. Fig. 7 shows superimposed the results obtained with both upgraded designs, using the same water vapour inlet of 15 mL/min and swamping ratio ranging between 1 and 3.3. As expected from the large differences between these two mechanical designs [11] (about the same length but the ratio between the amount of catalyst and the permeation area differs significantly) contrasting processing capabilities have been measured. Using the PERMCAT reactor comprising edge welded bellows (Fig. 5b), it is verified that an increase in the swamping ratio gives higher amounts of D in HDO. Expressed in HDO in term of decontamination factor (right *y*-axis), it is shown that the decontamination linearly increases with the swamping ratio. However, the processing capabilities of this reactor are far lower compared to the ones measured with the PERMCAT reactor comprising a corrugated membrane. With the later one, more than 99% of D (DF higher than 100) is measured in the collected water at a swamping ratio of 2. Unfortunately, the precision of about 2% relative for measurement of D in HDO does not allow a precise determination of the DF at these high deuterium concentrations, therefore it is not displayed on the figure.

Such large differences in processing capability are attributed to the significant difference between both reactors. The ratio between the amount and the permeation area is 0.16 and 0.06 g/cm^2 for the corrugated-type and bellows-type reactor, respectively. The best results are obtained with the PERMCAT reactor that contains relatively to the permeation area the largest amount of catalyst material, giving for the water vapour a larger residence time in the reactor. For this reason, this corrugated membrane PERMCAT reactor (Fig. 6) has been selected and installed in the CAPER facility for further characterisation with tritium.

4.2. Tests with tritium in CAPER and parametric study on side reactions

The current version of PERMCAT process in the CAPER facility has been described in details [12]. The detritiation performances at the mid-pathway of the process are monitored in real time with a ionisation chamber placed between the 2 corrugated membrane single-tubes PERMCAT in line. This is of great benefits when the PERMCAT is operated at outlet tritium levels close to the residual background level.

Within the CAPER facility, a mock-up section allows the production of a large variety of tritiated gaseous mixtures with gas composition and tritium activity relevant for the TEP systems. In total, 18 runs (some lasting up to 3 days of continuous operation, 825 h in total) have been performed with the corrugated membrane reactor. More than 1600 L of different gaseous mixtures (with up to 9% CQ_4, 4% Q_2, 4% CO_2) have been successfully processed without any problem. In total 3.8 g tritium (1.4×10^{15} Bq) have been recovered. In general, very good detritiation performances have been obtained and DF higher than 10^5 have been regularly measured. Along this parametric study, some runs have been performed to experimentally study the side reactions using mixtures containing Q_2, CO_2 and CQ_4.

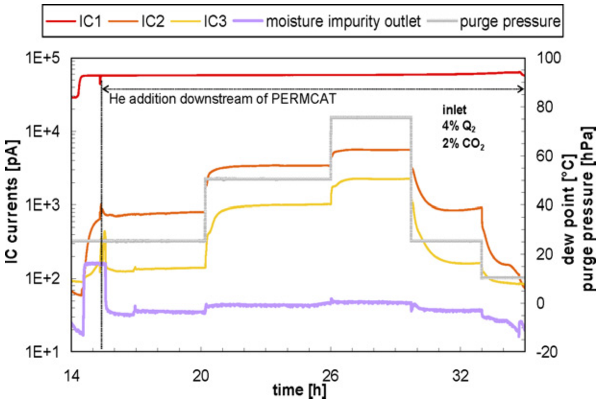

Fig. 8. Processing performances measured with tritium and influence of the hydrogen swamping pressure on the side reaction (IC1, IC2, IC3 are the tritium monitors at the inlet, mid-pathway and outlet of the PERMCAT impurity side, respectively). (For interpretation of the references to colour in text, the reader is referred to the web version of the article.)

Fig. 8 shows a part of the process of a gas mixture containing in a helium matrix the following species: 4% of Q_2 (Q = H, D or T) with a specific activity of 2.0×10^{15} Bq/m^3 plus 2% of CO_2 (vol%). This gas mixture is fed into the PERMCAT unit from a 120-L vessel at a constant flow rate of 10 mL/min, and the H_2 purge gas flow is set at 10 mL/min. The pressure of the impurity flow is maintained constant at 1200 hPa and the effect of varying the H_2 purge pressure between 10 and 75 hPa is shown in Fig. 8. The signals of the ionisation chambers (IC2 and IC3 give the activity of the impurity side at the mid-pathway and at the outlet, respectively) show that the detritiation performances are strongly influenced by the purge gas pressure. An increase in purge pressure tends to reduce the detritiation efficiency as shown by the IC2 and IC3 signal increases. The best detritiation performance for this experiment is measured for 10 hPa H_2 swamping pressure, which is the lowest value used during this test. It confirms that in presence of carbon oxides, the PERMCAT process promotes side reactions. A decrease in the purge pressure can limit the consumption of CO and CO_2 by H_2 and reduce the formation of water and methane via the inverse water-gas-shift and methanation reactions. Besides tritium signals, the humidity level measured at the PERMCAT impurity outlet confirms the occurrence of side reactions. Immediately after starting the process, the humidity sensor shows saturation (violet curve in Fig. 8). This reflects a significant water production along the reactor since no water is present in the feed stream. In order to avoid water condensation and to see an evolution of this signal for different operating conditions, fresh helium is added downstream of the unit to decrease the dew point down to a reasonable value. Then, the humidity level follows the evolution of the ionisation chamber signals, with the lowest humidity level obtained with the lowest purge pressure. Thus, the H_2 partial pressure for isotope swamping is a key parameter that has to be optimised according to the gas mixture to be processed.

5. Conclusions

This paper gives an overview of the R&D performed at TLK for the PERMCAT technology. The process and reactors are studied for more than one decade, including technical scale units and experiments. Accordingly, PERMCAT can now be considered as a mature technology.

Different technical solutions have been tested to improve the robustness of the membrane reactor so as to ensure integrity of the reactor and reliable operation for its use in nuclear environment such as processing the fuel of future fusion machines. The upgraded mechanical design using special corrugated Pd/Ag membranes greatly simplifies the manufacturing process.

The catalyst material has been selected as the best compromise between the promotion of isotope exchange reactions and limitation of side reactions. Nickel-based catalyst allows optimum process of a large variety of tritiated gaseous mixtures that will be produced in the fusion machine. However, for a specific tritiated stream exempt from carbon oxide (side reactions insignificant) other catalyst materials could be more suitable.

Several experimental campaigns have been carried out, including tests with tritium using the CAPER facility. Along these tests, the flexibility and versatility of the PERMCAT process have been demonstrated. In particular, it has been proven that PERMCAT is compatible with high tritium levels. Inactive tests without tritium have also demonstrated good compatibility with high water partial pressures. Therefore other applications than the original one (final clean-up of the exhaust gas from the torus) are presently under evaluation. For example the tritium recovery from very highly tritiated water, where conventional techniques for water detritiation may not be well adapted, could be performed with PERMCAT.

In any case, technical scale application will require the use of multi-tube membrane reactors to deal with higher throughputs. The production of such technical PERMCAT reactors is well advanced [20]. Additional efforts for PERMCAT numerical simulation are required to include radial effects and kinetics for chemistry. This will be greatly valuable for the scale-up and optimisation of PERMCAT designs.

Acknowledgments

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