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ITER R&D: Auxiliary Systems: Tritium Systems

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1. Introduction

The challenging requirements for the ITER fuel cycle tritium systems are summarised below.

1. Dynamic control for the pulse plasma operation
   - fuel supply shall meet all DT plasma operation scenarios with a wide range of throughputs and isotopic compositions,
   - tritium processing at high throughputs of tokamak exhaust shall allow rapid recycling for plasma operation and
   - ZrCo metal hydride bed for tritium storage and rapid delivery shall meet fuelling requirements,
   - rapid and accurate tritium measurement for tritium accountancy shall be possible,
2. Enhancement of tritium safety
   - fuel cycle tritium systems shall be designed to minimize tritium inventory,
   - minimization of environmental tritium release at all ITER operation states (including maintenance) and for accident scenarios, and
   - minimization of tritium contaminated waste production.

These requirements called for an extended range of detail design studies, and R&D program for new chemical processes, key equipment and instruments.

This chapter describes results of the R&D implemented throughout the ITER EDA.

2. Development of a Tokamak Exhaust Process

2.1. Objectives

The processes of this system are: front-end permeators process for separation of hydrogen isotopes and impurities, impurity processing for the recovery of tritium from tritiated chemical species, and final detritation for the rejection of residual gases. Objectives of the specific R&D items of this system are: (a) verification of long term tritium durability of the Pd/Ag alloy membrane, (b) development of special catalysts for conversion of tritiated impurities, (c) development of a high efficiency final detritation process, and (d) achievement of an overall detritation factor (DF) greater than 10^8.

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Table 1
Summary of long term permeator tritium durability tests at 350°C

T_2 gas exposure duration (h)	He-3 content in alloy (appm)	He-3 bubble conc. (10^{16} cm^{-3})	He-3 bubble dia. (nm)	Lattice parameter change ($\Delta a/a$, %)	Tensile strength (σ_u, MPa)	0.2% Yield stress ($\sigma_{0.2}$, MPa)	Elongation (δ, %)
0	–	–	–	–	730	570	8.2
385	73	–	–	0.07	690	530	19.7
938	205	2.7	2.4	0.05	710	480	22.5
1458	330	2.4	2.8	0.05	710	490	22.5
2003	466	1.8	3.8	0.02	725	510	20.5
3625	840	3.0	3.9	0.00	730	525	17.0

2.2. Verification of long term tritium durability

In the reference design of the ITER tritium plant, Pd/Ag alloy membranes (nominal thickness ~80–100 μm, grain size in the micro structure ~10 μm) are utilised as separators of elemental hydrogen isotopes and impurities in various tritium process systems such as the front-end permeator system, the impurity process system, the final detritation process system, the water detritation tritium recovery stream, and the neutral beam regeneration gas stream.

Long term T_2 gas exposure tests (T_2 pressure 0.2–0.45 MPa, exposure temperature 300–350 °C), including post exposure material tests such as ^{3}He concentration measurement, microstructure analysis, X-ray diffraction analysis, stress–strain tests and positron annihilation analysis were implemented at the Tritium Laboratory of VNIINM. The results (see Table 1) revealed that, although very little swelling and progressive formation of ^{3}He bubbles and clusters were observed in the microstructure, no marked deterioration in the mechanical strength of the membrane specimens has occurred in the present maximum test duration up to 3625 h [1]. This fact indicates that the Pd/Ag membrane permeator in the front–end permeator system can be operated for 4–5 years of ITER operation with 10% machine availability per year. Use of ITER-scale permeators at various institutes (JAERI/TPL [2], LANL/TSTA [2] and FzK/TLK [3]) has demonstrated that these components can successfully operate over several years of integrated lifetime in representative gas compositions.

2.3. Development of special catalysts

Tokamak exhaust during plasma operation and wall conditioning is expected to contain various impurities such as tritiated water vapor, tritiated hydrocarbons, tritiated ammonia, carbon monoxide, carbon dioxide, He, etc. Development [4,5] of special catalysts for the conversion of tritiated impurity mixtures into elemental tritium gas in the front-end permeator and the impurity processing system, and for enhancement of hydrogen isotope exchange reactions in the final detritation process, was the key issue to achieve overall tritium decontamination $DF > 10^8$ (see Table 3). Tritium laboratory teams (JAERI, LANL, ARSRIIM, FzK, Chalk River, ISPRA, Fontenay aux Roses) in all the Home Teams have worked on the tritium exhaust processing (TEP) R&D including various options of process configuration aiming at this target value of the DF. Table 2 shows the catalysts selected for the ITER design of these processes [5]. The catalytic reactor using those catalysts can be utilized for tritium recovery (O_2 gas baking method) from co-deposited layer

Table 2
Tritium process catalysts selected for ITER Tokamak exhaust process system design

Process	Catalyst	Main reactions	Research Institutes
Front-end process	Japanese NIKKI-111 catalyst (50% nickel on kieselguhr)	methane cracking at 450 °C $CQ_4 \rightarrow C + 2Q_2$	TLK at FzK
Impurity process	Japanese NIKKI-111 catalyst (50% nickel on kieselguhr)	watergas shift reaction at 200 °C $H_2O + CO \leftrightarrow H_2 + CO_2$	TPL at JAERI[a] TLK at FzK[b]
Final detritation	Japanese NIKKI-111 catalyst (50% nickel on kieselguhr)[c]	Hydrogen isotope exchange reaction in counter current membrane reactor	TLK at FzK[d]

[a] First implementation was accomplished at TPL, JAERI in early 1980.

[b] Tritium durability more than 6 years has been verified throughout the operation of an engineering scale test facility (CAPER/CAPRICE) since early 1990.

[c] (AI_2O_3 or SiO_2) catalyst was favourable for hydrogen isotope exchange reaction efficiency, however it was finally rejected because of its property of being poisoned by CO.

[d] Tritium demonstration tests using an engineering scale of new test facility (CAPER) are on going.

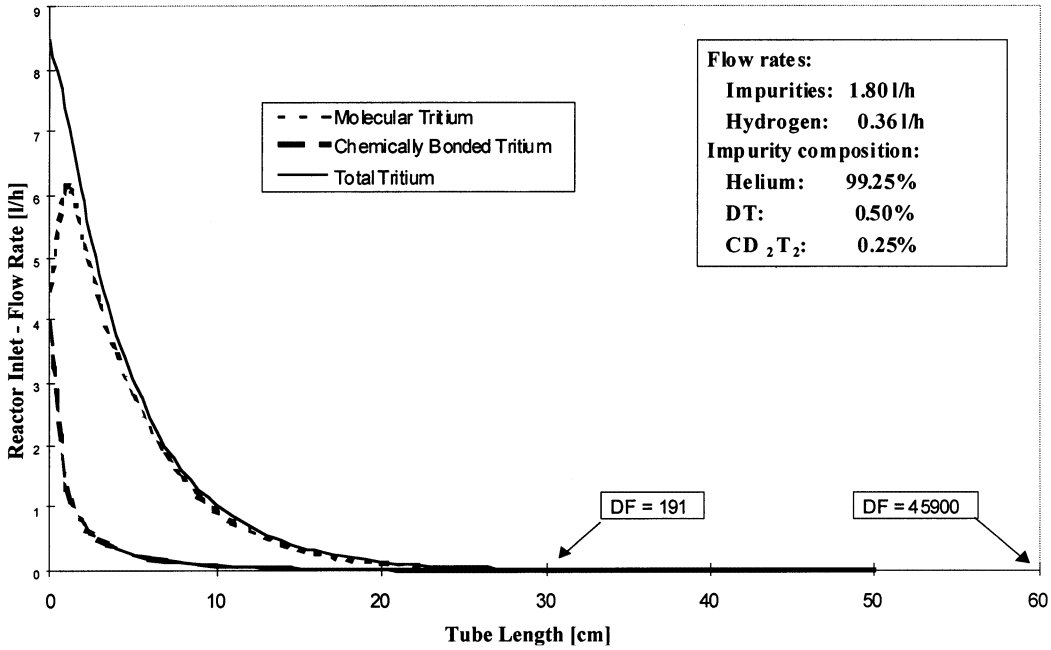

Fig. 1. A correlation between the decontamination factor and the length of a counter current membrane reactor (PERMCAT).

Table 3
Tritium decontamination factor for the Tokamak exhaust processing (TEP) system

	Front-end permeator process	Impurity process	Final detritation process
Experimentally determined DF	10–100	~1000	~100 000
Design DF	30	350	10 000

in the torus vessel. Confirmatory R&D is in progress.

2.4. Development of a high efficiency final detritation process

Experiments on several options have been carried out including oxidation/electrolysis combined with a Pd/Ag membrane permeator (JFCU; JAERI) [2], hydrogen isotope exchange reactor combined with a Pd/Ag membrane permeactor (HITEX; Chalk River) [6], Pd/Ag membrane reactor packed with a Pt/Alumina-catalyst (PMR; TSTA/LANL) [7], Counter current Pd/Ag membrane reactor packed with a catalyst (CAPER model; FzK) [8]. The latter three options employ essentially the same processing principle, i.e. its equilibrium chemical reactions of tritiated impurity conversion such as $Q_2O + CO \leftrightarrow Q_2 + CO_2$ (where Q means hydrogen isotope atoms). That can be shifted to the right by removing Q_2 with a Pd/Ag membrane permeator. Based on the experimental results of the options, the counter current Pd/Ag membrane reactor was selected for ITER because of its achievable decontamination factor (DF) greater than 10^4 (Fig. 1). An engineering scale of test stand (CAPER) was newly constructed at FzK for long term tritium processing demonstration tests under the ITER R&D program. Table 3 shows the experimentally determined DF and the design value for each process of the ITER TEP system [8]. From these system-

atic experimental campaigns, it was confirmed that an overall DF of 10^8 for the present ITER design can be achieved, with a reasonable margin.

3. Development of ZrCo metal hydride beds for ITER fuel cycle

3.1. Objectives

Two candidates for metal tritide bed technology, i.e. the uranium powder hydride (UQ_3) bed and the zirconium cobalt alloy powder hydride bed ($ZrCoQ_3$), were proposed for the ITER tritium storage and delivery system. The ZrCo hydride bed technology was selected because of its low chemical reactivity with air (in case of an air ingress accident) compared to the potential high pyrophoricity of a uranium powder bed, and to avoid the potential complication of employing a nuclear material. Objectives are to clarify the characterisation of the materials and to develop tritium inventory measurements for the fuel cycle.

3.2. Material characterisation

Extensive additional key R&D such as the disproportionation-reproportionation of this material, kinetics of rapid desorption (endothermic reaction) and absorption (exothermic reaction), and isotope effect during rapid fuel delivery were carried out in order to meet the ITER operational requirements. X-ray diffraction analysis (see Fig. 2) shows complete recovery of the disproportionated material which is easily attained by vacuum heating [9] ZrCo is dissociated into ZrH_2 and $ZrCo_2$ when it is heated to a high temperature >450°C at high Q_2 pressure $> \sim 1$ bar. It has been fully demonstrated, through the use of ZrCo beds for a decade, that no disproportionation occurs as long as the hydrogen delivery pressure is kept at <1 bar during delivery of Q_2 at 350 °C [9]. Fig. 3 indicates that the ITER requirement of rapid delivery (200 Pa m^3 s^{-1}) could be achieved by a full-scale ITER storage system. However, it was found that an improvement of the ZrCo powder packing design will be needed to increase the heat transfer rate to overcome a rapid temperature drop due to the endothermic reaction of the Q_2 desorption [10]. Fig. 4 shows a typical result indicating that no appreciable composition change occurs in all ranges of stoichometric change during rapid delivery of a 50%H–50%D mixture from $ZrCoQ_{2.19}$ under vacuum pumping [10].

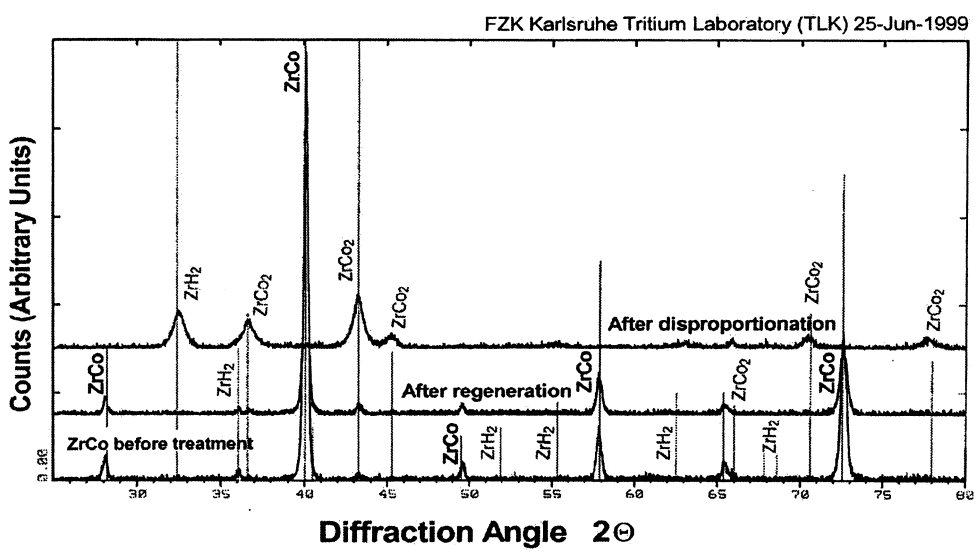

Fig. 2. X-ray diffraction analysis of disproportionation and reproportionation of ZrCo alloy.

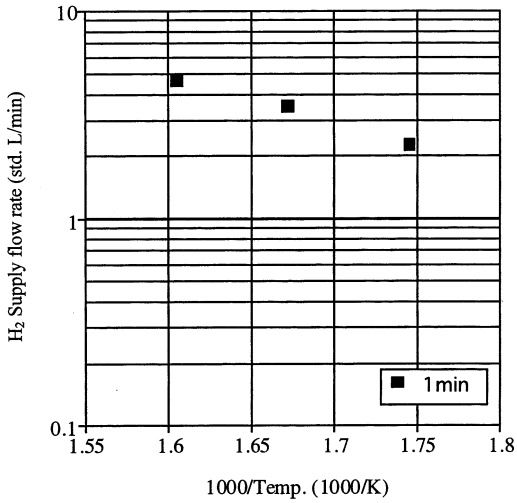

Fig. 3. A typical result of rapid delivery in the first 1 minute (Using a 1/10 scaled model of the ITER ZrCo bed).

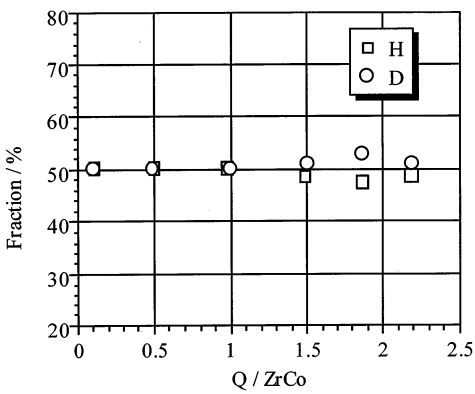

Fig. 4. Fluctuation of hydrogen isotope gas composition during rapid supply from the ZrCo bed, H:D = 50:50 mixture stored to ZrCoQ2.17 initially.

3.3. Development of ZrCo beds for rapid accountancy

Tritium inventory measurements of the ITER fuel cycle will be required daily. This is only possible by the accurate measurement of the tritium inventory in the fuel cycle tritium storage and delivery system. The site overall tritium accountancy can be achieved by using a special calorimeter developed for tritium shipping containers (ZrCo tritide bed of 100 g T capacity). This accountancy measurement will be implemented in the ITER long term tritium storage system vault, where tritium shipping containers are received, and tritium is delivered to the ITER fuel cycle storage beds. Such a self-assaying hydride bed was first proposed and tested at LANL/TSTA [11]. A full-scale design of the ITER fuel cycle ZrCo bed with in-bed calorimetry has been developed, and a scaled model (1/5 of the ITER bed) has been routinely used (JAERI/TPL). Fig. 5 shows that the accuracy of tritium inventory measurement is in the range of 97–98.5% in 8 h, and 99.5–100% after 24 h following rapid delivery at 350 °C and rapid recovery of T_2 gas [10].

3.4. Development of tritium calorimeters

Tritium calorimetry measures the thermal power produced by the β decay of tritium; 1 g of tritium generates 0.324 W. The main advantage of measuring tritium by calorimeter is that it is a non-destructive method and can be used to determine tritium in gases, liquids and solids. Several calorimeters based on different principles have been developed for routine tritium accountancy in the ITER [11–13]. With the progressive improve-

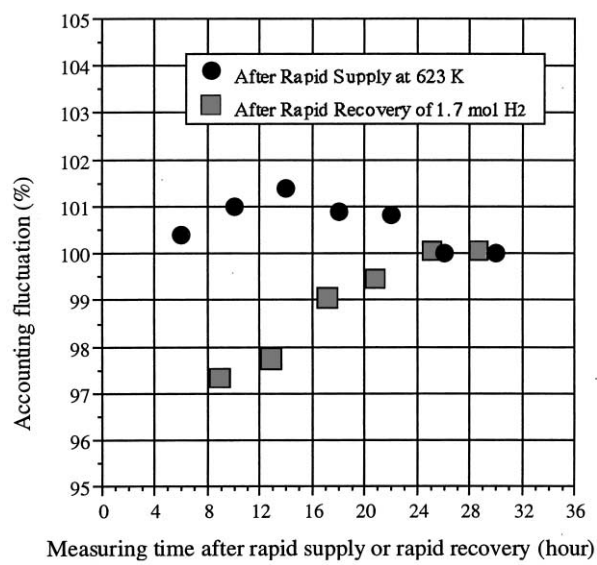

Fig. 5. Uncertainty of tritium inventory after rapid supply and recovery as a function of measuring period.

Table 4
Progress with tritium calorimeters at TLK

Year	Dynamic range (μW)	Detection limit (μW)	Detection limit (Bq)	Sample volume	Calorimeter type
1995	1×10^{3}–5×10^{6}	1000	10^{12} (30 Ci)	1.2 l	ANTECH isothermal
1998	7–5×10^{6}	7	7×10^{9} (0.2 Ci)	0.5 l	Inertial guidance IGC-A
1999	1–10×10^{6}	1	10^{9} (0.03 Ci)	0.5 l	Inertial guidance IGC-V

ment of the detection limit achieved with the TLK calorimeters (Table 4), the method with different measuring volumes can now be applied to other tasks such as the determination of residual tritium in plasma-facing components, tritium shipping containers after unloading, etc.

4. Dynamic control of the Hydrogen Isotope Separation

4.1. Objectives

The ITER hydrogen isotope separation system (ISS) receives a DT stream from the torus, a H_2 stream from the water detritation system, and a D_2 or H_2 stream from the neutral beam injectors; and is composed of a four column cascade: one column for the production of T_2 and 50D–50T, two columns for D_2, and one for hydrogen rejection. Despite dynamic changes in flow rates and compositions in these ISS feed streams, constant product purity in each product stream has to be maintained to meet the plasma fuelling requirements and the environmental tritium release guideline. Objectives are to develop the dynamic control of the ISS.

4.2. ISS development

Extensive tests of the dynamic control of the ISS cascade have been carried out with a full cascade configuration in the LANL/TSTA [14], and with a small ISS test loop at JAERI/TPL [15]. A dedicated test column based on the ITER design specification was constructed to demonstrate dynamic control of the two key columns (T_2 production and H_2 rejection columns) in the ITER ISS. A test result of the T_2 production column when using a D_2–H_2 mixture as an alternative to the of T_2–D_2 mixture, is shown in Fig. 6 [16]. In this case, five successive cycles were performed, with cycle times equal to that expected for ITER-FEAT cryopump regeneration. The composition setpoints were 4% for the top product and 96% for the bottom. The column feed flow was the sum of the indicated product flows at each point in time. From this figure it is found that the column bottom product compositions can be maintained at their setpoints while the feed composition was changed stepwise between 50 and 75%D. Similar dynamic tests were carried out successfully for the hydrogen rejection column, and this column control method (proportional mass flow control) was fully applied to the ISS design and to the ITER fuel cycle dynamic simulation code.

5. Detritation of highly contaminated solid materials

5.1. Objectives

To minimize residual tritium in the solid wastes produced from the ITER tokamak and the tritium plant, development of a practical tritium recovery technology is a key issue for ITER waste management. Objectives are to develop detritation methods from the solid wastes such as the co-deposited carbon.

5.2. Development of detritation methods

To minimize residual tritium in the solid wastes produced from the ITER tokamak and the tritium plant, development of a practical tritium recovery technology is a key issue for ITER waste

management. A range of methods such as purging with different moisture concentrations, ozonation, ultra-violet (UV) irradiation, oxygen baking, and oxygen radio frequency (RF)-plasma exposure have been investigated [17,18]. Oxygen RF-plasma exposure removed tritium from two-dimensional carbon fibre composite (CFC)-samples ten times more effectively than oxygen baking at the same O_2 gas pressure. UV irradiation was the most effective of the methods investigated. The purging method has the advantage of enabling detritation in shadowed regions of a material. Table 5 shows

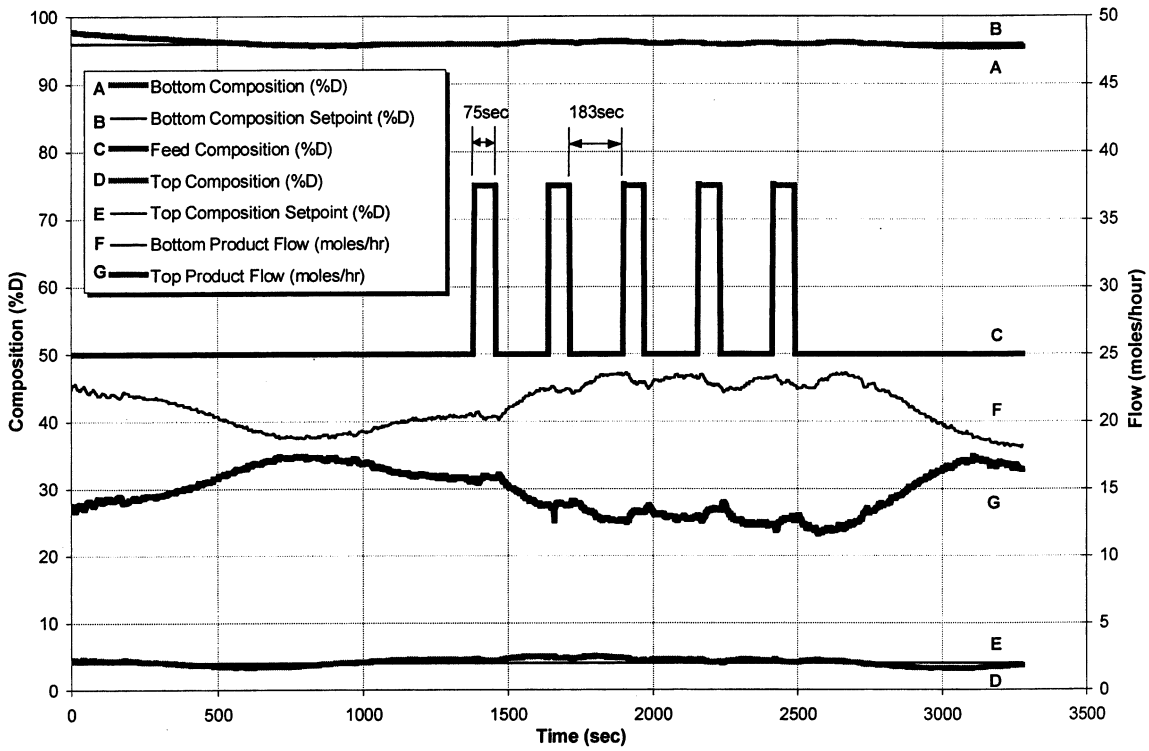

Fig. 6. Demonstration of dynamic control of cryogenic hydrogen isotope separation column. Feed composition was stepwisely changed for five successive cycles.

Table 5
Ex-situ tritium recovery from JET graphite tiles

Temperature (°C)	Duration of treatment (days)	Released activity (%)	Cumulative released activity (%)	Remaining activity (%)
25	0.42	0.3	0.3	99.7
200	4.42	5.7	6	94
200	5.34	0	6	94
300	6.26	16.3	22.3	77.7
400	7.01	64.1	86.4	13.6
400	7.8	0	86.4	13.6
400	10.67	0	86.4	13.6
800	10.84	13.5	99.9	0.1

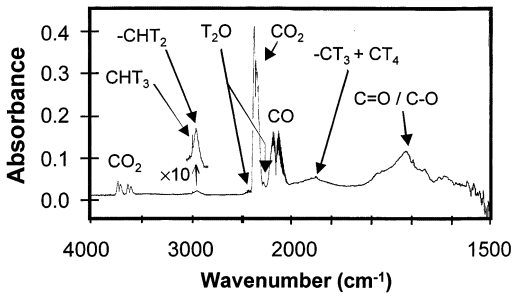

Fig. 7. Formation of organic compounds due to β-ray induced reaction in a T_2–CO gas mixture.

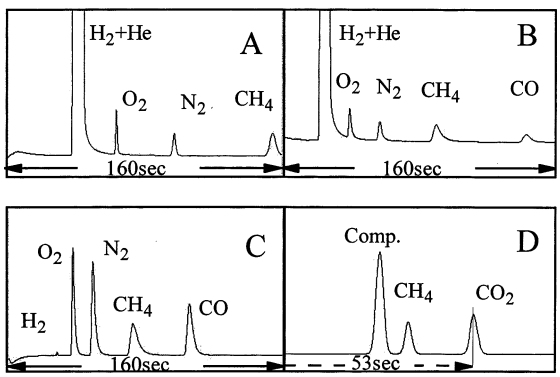

Fig. 8. Rapid gas analysis using four-channel micro-GC.

the tritium release characteristics for graphite tiles installed in JET during the ex-situ tritium recovery process by using high temperature baking [19]. It should be noticed that the tiles used in this measurement have previously been exposed to plasmas in JET and then have been placed in a moist air stream at progressively increasing temperatures. The amount of tritium present on the tiles used in this measurement is considered to be a relatively small percentage of the total amount of tritium trapped by the co-deposited layers formed during plasma operation.

5.3. Investigation of tritium re-deposition

An oxygen erosion method which employs a low pressure Q_2 gas exposure at 240–350°C has been proposed as a promising solution for in-situ tritium recovery from the co-deposited carbon layers. However, it is anticipated that tritium re-deposition along the pipeline from the torus to the tritium recovery plant might occur due to β-ray enhanced side reactions in the mixture of various chemical species (DT, DTO, CO_2, CO, tritiated hydrocarbon, excess O_2 and carrier gas N_2), which are produced by this method. Fig. 7 shows infrared absorption spectra of reaction products of a T_2–CO mixture (55 kPa, 300 K, 10 days after mixing, reaction rate $\sim 5\%$/day). Analysis of the reaction products revealed that solid particles and/or condensate of organic compounds such as aldehydes (RTCO) carboxylic acids (RCOOT), etc were formed. Kinetic experimental work of these reactions is on going using simulated gas mixtures [20].

6. New analytical instruments

Use of micro gas chromatographs (Micro-GC) for characterizing tritium systems was first proposed by LANL/TSTA [21]. Since then, an integrated four channel micro gas chromatograph (micro-GC) has been fully developed through routine use at TPL/JAERI, and is now the reference equipment for the ITER tritium plant central analytical system. Fig. 8 shows the measurement of a gas mixture simulating typical tokamak exhaust as an example, and indicates that all species can be analysed in less than 160 s, which is approximately 1/15 the time for conventional gas chromatography. A micro-GC with a cryogenic column was also developed for hydrogen isotope analysis [22].

Low memory scintillation-type tritium detectors coupled with a photo-multiplier were developed [23]. The measurement range for the tritium concentration is 2×10^4–10^{10} Bq/m^3. A fibrous scintillation detector for in-line direct measurement was also developed [24]. The applicable tritium concentration range is ~ 1 ppm–100%.

Laser Raman spectrometers have been employed for quantitative measurement of gaseous tritium and tritiated species at FzK, JAERI and LANL. While at JAERI the use of optical fibres was fully developed for real time, multi-channel tritium process control (hydrogen isotope separation system), and routinely used over 5 years at

the TPL/JAERI [25], an external resonator was employed at FzK to improve the sensitivity of the method by more than two orders of magnitude [26]. Reflection time-of flight (TOF) type mass spectroscopy, which is characterized by its high sensitivity due to the low background effects by β rays from tritium decay, was developed [27]. These new technologies will be applicable to various tritium processing systems.

7. Conclusions

The ITER fuel cycle is composed of a complex configuration of different tritium processing subsystems. In order to meet the challenging requirements (dynamic features in pulse plasma operation and the tritium safety) of ITER, extensive but systematic R&D has been carried out under the ITER R&D program. The research teams in the four Home Teams have proposed different ideas and/or principles for different chemical processes and components. A scheduled review process has been introduced in this R&D program to assess the achievement of all the R&Ds. Selection of key processes and components has been implemented by taking into account, not only the typical result of specific R&D, but also reliability and simplicity in operation, durability of components and the cost of the tritium subsystem. Choices of key subsystems integrated with different processes were also made by specific demonstration tests, including long term operation using tritium.

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