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Tritium Plasma Experiment Upgrade and Improvement of Surface Diagnostic Capabilities at STAR Facility for Enhancing Tritium and Nuclear PMI Sciences

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ABSTRACT

Recently, Tritium Plasma Experiment (TPE), a unique high-flux linear plasma device that can handle beryllium, tritium and neutron-irradiated plasma facing materials, has undergone major upgrades in its electrical and control systems. The upgrade has improved worker occupational safety, and enhanced TPE plasma performance to better simulate extreme plasma-material-interaction (PMI) conditions expected in ITER, Fusion Nuclear Science Facility (FNSF) and demonstration fusion power plant (DEMO). The PMI determines a boundary condition for diffusing tritium into bulk plasma-facing components (PFCs) and plays critical role in in-vessel and ex-vessel safety assessments. Enhancing surface capabilities for tritium-contaminated and radioactive samples is crucial for the PMI sciences in burning plasma long pulse operation. The TPE Upgrade and improvement of surface diagnostic capabilities for tritium-contaminated and radioactive samples at STAR facility help enhance tritium and nuclear PMI sciences for the development of reliable PFCs and tritium fuel cycle in ITER, FNSF and DEMO.

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1. Introduction

ITER is designed to produce 500 MW thermal power output with 50 MW heating power input to achieve Q=10 condition for demonstrating the scientific and technical feasibility of magnetic fusion energy. ITER also needs to prove safety characteristics of magnetic fusion device as a basic nuclear facility with unprecedented amount of tritium inventory (< 4000 g-T in site). Safe operation of ITER dictates that in-vessel tritium concentrations retained within plasma-facing components (PFCs) must remain at acceptable levels. Regulatory limits are set to 1000 g-T in vessel and 0.0001 g T/m^3 in the reactor cooling water in ITER [1]. A critical challenge for long-term operation of ITER, Fusion Nuclear Science Facility (FNSF), Demonstration reactor (DEMO) and future fusion reactor will be the development of PFCs that demonstrate erosion resistance to steady-state/transient heat fluxes and intense neutral/ion particle fluxes under extreme fusion nuclear environment.

The behavior of tritium in fusion reactor materials plays a major role in material selections for blanket and divertor components because tritium retention and permeation determine in-vessel inventory levels and ex-vessel release quantities. For example, the decision by the ITER organization to exclude carbon is based on predictions of unacceptable levels of tritium retained in co-deposited carbon layers. Tungsten, a primary candidate material for the future reactor's divertor and first wall, is expected to receive a neutron dose of tens of displacement per atom (dpa) of radiation damage in FNSF and DEMO. Hydrogen and deuterium are widely used in fusion community to simulate tritium behavior as a surrogate due to economical and safety reasons. Diffusion and migration of tritium can be approximated with the use of hydrogen and deuterium since the diffusivity of hydrogen isotopes is equivalent to the inverse ratio of the square root of the masses of the isotopes [2]. One key phenomenon that hydrogen and deuterium are not capable of simulating is tritium decay into helium-3 and its beta emission. The half-life of tritium is 12.3 year, and approximately 5 percent of tritium decays into helium-3 in a year. Tritium decay helim-3 is known to form helium bubble in metal and to have significant effects on fracture toughness and microstructure in metal [3]. Under divertor-relevant high ion flux condition, tritium is migrated in bulk and trapped in radiation-induced trap site (up to 1 at. % T/W) in neutron-irradiated tungsten. In burning plasma long pulse operation, the tritium decay helium-3 from trapped tritium will play a major role in microstructural evolution (e.g. helium embrittlement) in tungsten due to relatively low helium-4 production (e.g. He/dpa ratio of 0.4-0.7 appm [4]) in tungsten. The microstructural evolution from tritium decay helium-3 and irradiation effects (e.g. displacement damage, transmutation, gamma radiation) can also alter near surface material evolution, erosion, dust production, and tritium behavior in burning plasma operation. Plasma material interaction (PMI) determines a boundary condition for diffusing tritium into bulk PFCs. Tritium-beta is reported to induce static charge that levitates dust, and the fundamental properties (e.g. resuspension, transport and combustion) of tritiated and activated dust can be significantly different than that obtained with non-tritiated and non-activated dust [5]. Neutron-irradiation and gamma radiation environment are also known to have profound effects on tritium migration, trapping, and permeation in materials [6,7]. Fully addressing the challenges described above will undoubtedly require a range of modeling and experiments executed at many non-nuclear and nuclear facilities. However, *direct* work with tritium, neutron-irradiated materials, and gamma radiation environment is indispensable as a means of elucidating key physical processes governing tritium permeation and tritium decay helium-3 embrittlement and understanding microstructure and chemical states of tritiated and activated PFCs and dusts.

Synergistic effects of neutron with hydrogen isotope behavior and microstructural evolution were reported recently [8,9], and more experimental database of multiple separate effects and synergistic effects in tritium and nuclear PMI is required for accurately predicting multiple separate effects and synergistic effects on PMI and material response in burning plasma long pulse operation. One of the critical challenges for tritium and nuclear PMI is limited availabilities of surface diagnostics for tritium-exposed and radioactive samples to advance scientific understanding of multiple separate effects and synergistic effects for successful development of reliable PFCs and tritium fuel cycle in burning plasma long pulse operation. This paper discusses the upgrade in one of the only tritium compatible high-flux linear plasma devices and the recent improvement of surface diagnostic capabilities for tritium-exposed and radioactive samples to enhance tritium and nuclear PMI sciences and material sciences in burning plasma long pulse operation.

2. Tritium plasma experiment upgrade

Idaho National Laboratory (INL) operates a linear plasma device, known as the Tritium Plasma Experiment (TPE). The TPE is unique linear plasma device that possesses four specialized elements: (a) the use of tritium, (b) a divertor-relevant high-flux plasma, (c) the ability to handle radioactive materials, as well as (d) beryllium [10]. The TPE is located within the Safety and Tritium Applied Research (STAR) facility, which is a DOE Less Than Hazard Category 3 (LTHC-3) nuclear facility [11], and is licensed to handle tritium inventory up to 16,000 Ci (5.9×10^{14} Bq) and moderately activated/neutron-irradiated materials. Despite its age, the TPE still stands as the only existing high-flux linear plasma device that can handle both tritium and neutron-irradiated material to study tritium and nuclear PMI in fusion nuclear environment [12]. Recently, the TPE has undergone major upgrades in its electrical and control systems, and in November 2015 the TPE has successfully achieved first deuterium plasma via remote operation from outside the contamination area after a significant three-year upgrade. Figure 1 shows the photo of the new TPE power supplies and control center outside the contamination area after the three-year upgrade. Details of the electrical upgrade, enhanced operational safety, and improved plasma performance were summarized in previous publication [13].

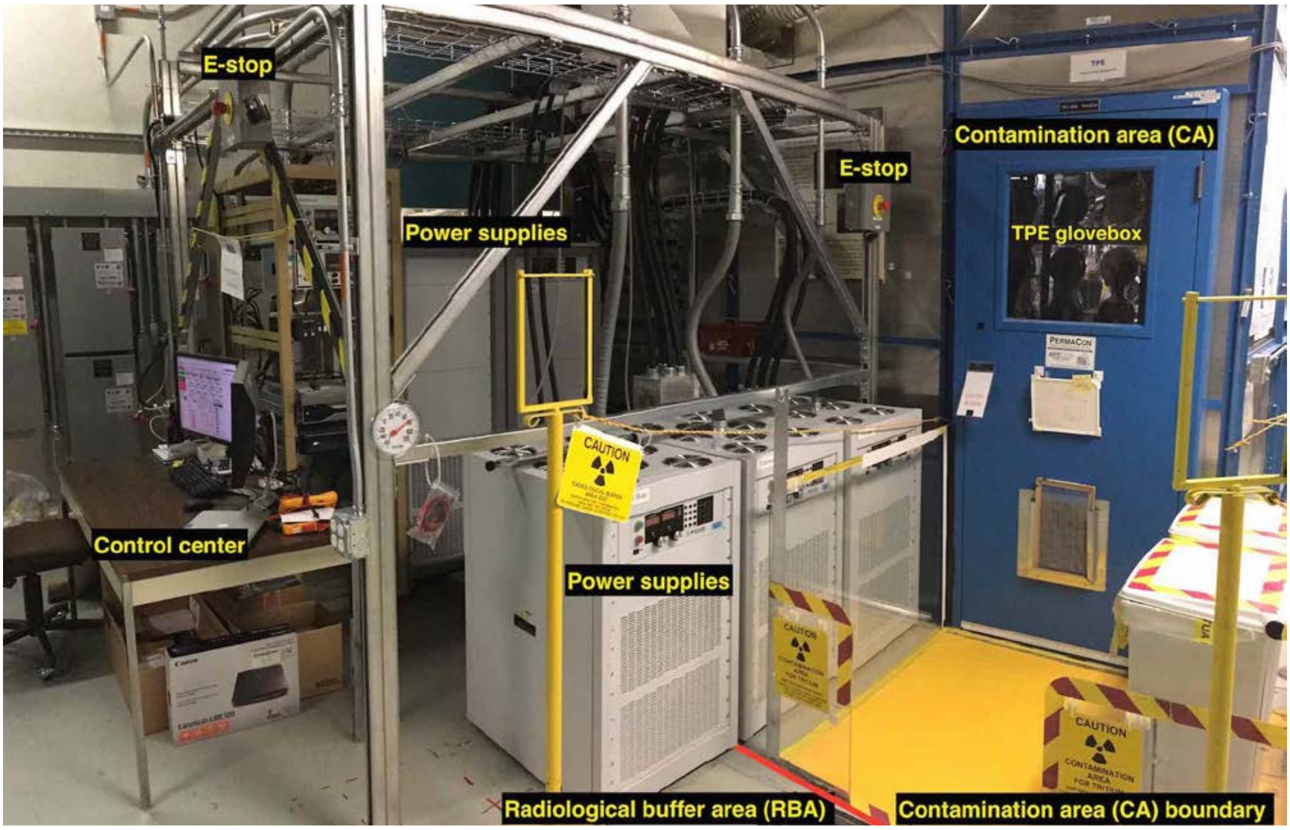

Figure 1: Photo of the new TPE power supplies and control center outside the contamination area after the three-year upgrade

3. Improvements in diagnostic capabilities at STAR

3.1 Improvements in plasma and tritium diagnostic capabilities in the TPE

The STAR facility at INL is a DOE LTHC-3 nuclear facility that is capable of handling both tritium and radioactive materials. The efforts to improve plasma and tritium diagnostics capabilities in the TPE were carried out in parallel to enhance operational safety during the three-year TPE upgrade. Plasma and tritium diagnostic capabilities in the TPE are summarized in Table 1. A single Langmuir probe consisting of a 1 mm diameter (2mm length) tungsten wire surrounded by an alumina insulator is used to obtain the electron density, electron temperature, plasma space potential, and floating potential in the TPE. Details of the single Langmuir probe and the plasma characterization in the TPE are given elsewhere [8]. A fixed range high-resolution spectrometer (Ocean Optics HR-4000, 580-680 nm in wavelength range) is to distinguish and monitor Balmer-alpha emission lines of hydrogen isotopes, H_{α} (656.280 nm), D_{α} (656.104 nm) and T_{α} (656.045 nm), in the plasma. A Czerny-Turner spectrometer (Andor Technology, Shamrock SR-750-A) equipped with a back illuminated 2D (1024×255pixel) CCD camera (Andor Technology, iDUS 420) is recently installed to measure helium ion density in mixed (D, T and He) plasma from *HeI* (447.1 nm) or *HeII* (468.6 nm) emission lines for mixed plasma operation, and to monitor Balmer-alpha and higher n-Balmer lines of hydrogen isotopes and impurity emission lines in plasma. Two ion chambers (Tyne engineering, 10 and 1000 cc tritium ion chambers with tritium dual ion chamber controller model 7000) are also installed in the TPE source gas inlet and vacuum exhaust line to monitor tritium concentration of feed gas and vacuum exhaust. A plasma-driven tritium permeation system is being developed for integration in the TPE under collaboration with Sandia National Laboratories-Livermore [14]. The design allows for control of the membrane cooling by heat sink positioning and varying of the fluid flow; heating is solely from the incident TPE ion flux. It also equipped with a 1000 cc tritium ion chamber (Tyne Engineering) to measure plasma-driven tritium permeation rate in helium purge gas stream with the detection capability of 1 parts per trillion (ppt) tritium gas in helium.

Table 1: Plasma and tritium diagnostic capabilities in the TPE

Diagnostics in the TPE	Tritium detection
Single probe Langmuir probe (ϕ=1 mm, L=2mm)	No[a]
Fixed range high-resolution spectrometer (Ocean Optics HR-4000, 580nm $<\lambda<$ 680nm)	Yes
Czerny-Turner spectrometer (Andor Tech. Shamrock 750, 750mm, f/9.7) with 1024x255 pixels CCD (Andor Tech. DU420A-BV)	Yes
Tritium ion chambers (Tyne Engineering 10 cc and 1000 cc ion chamber)	Yes
Plasma-driven permeation system with 1000 cc ion chamber (Tyne Engineering 1000 cc ion chamber)	Yes

[a] Langmuir probe is compatible with tritium use, but is not capable to distinguish tritium ion density in mixed plasma condition. The TPE do not operate with 100 % pure tritium plasma for safety and inventory reasons.

3.2 Improvements in surface diagnostic capabilities at the STAR

In addition the efforts to improve surface diagnostics capabilities for tritium-exposed and radioactive samples are being carried out at the STAR facility. Surface diagnostics capabilities for tritium-exposed and radioactive samples at the STAR are summarized in Table 2. Thermal desorption spectroscopy (TDS) system is equipped with standard (1-100 amu) and high resolution (1-6 amu) quadrupole mass spectrometers (MKS Instrument, eVision$^+$ and IP Microvision) to distinguish small mass difference between He (4.0026 amu) & D_2 (4.0282 amu), and measure total retention of He and D_2 with linear temperature ramp rate (10-60 °C/min) up to 1100°C. This diagnostic has been providing important database in deuterium retention in neutron-irradiated tungsten studies, and is now being modified to perform tritium thermal desorption spectroscopy with flowing inert gas to enable measuring low tritium concentration (ppt) with ion chamber. A Doppler broadening positron annihilation spectroscopy (DB-PAS) system is recently developed with a single high-purity germanium (HPGe) detector (Ortec, GMX series) [15,16]. The DB-PAS is used to investigate characterizations of radiation defects (e.g. vacancy, vacancy-cluster, voids) and also occupancy of hydrogen isotopes and helium in neutron-irradiated material. Optical microscope (Nikon Optiphot-100) with 2D CCD camera (PAXcam) provides 2D optical microscope image of surface morphologies (e.g. blister formation, cracks, melting etc.) at material surface. A refurbished liquid scintillation counter (Beckman LS6500) was recently installed in the STAR facility, and it will provide us critical information of tritium concentration in tritiated liquid with superb detection sensitivity of one parts per quadrillion (ppq) of tritiated water. This will enable us to perform extremely low permeation measurement at the tritium gas absorption permeation system [17] and plasma-driven tritium permeation system [14] when combined with catalyst beds and bubblers. Imaging plate reader (Fujifilm FLA-7000) enables us to measure 2D image of relative tritium concentration on surface by tritium imaging plate technique and also provide depth profiling of tritium by cutting tritium-implanted sample with a diamond wire saw.

Scanning Auger microscope (Perkin Elmer PHI 660) and X-ray photoelectron spectroscopy (Perkin Elmer PHI 5400) are being transferred from other INL facility and being reassembled at the STAR facility to add critical surface diagnostic capabilities for tritium-exposed and radioactive materials. The scanning Auger microscope is designed to measure low Z elemental composition on the surface and near surface with ion beam sputtering capability, and will provide high resolution, spatially resolved chemical images of elemental composition on surface and also measure depth profile of elemental composition in tritium-exposed and radioactive materials. The x-ray photoelectron spectroscopy will provide elemental composition, chemical and electronic state of each element on surface and also measure depth profile of elemental composition in tritium-exposed and radioactive materials. Glow discharge optical emission spectroscopy (Horiba GO-

PROFILER 2) is being installed in the STAR facility. The glow discharge optical emission spectroscopy is one of the rare surface techniques capable of measuring hydrogen isotope depth profiling in tritium-exposed and radioactive materials with nano-meter resolution [18]. These improvements of plasma and surface diagnostic capabilities at STAR facility will help enhance tritium and nuclear PMI sciences and nuclear material sciences in burning plasma long pulse operation.

Table 2: Surface diagnostic capabilities for tritium exposed and radioactive samples at the STAR

Diagnostic at the STAR facility	Handling tritium exposed sample	Handling radioactive sample[f]
Thermal desorption spectroscopy with 1-100 amu and 1-6 amu quadrupole mass spectrometers (MKS eVision+ and IP Microvision)	Yes	Yes
Positron annihilation spectroscopy with a single high-purity germanium (HPGe) detector (Ortec GMX series)	Yes[a]	Yes
Optical microscope (Nikon Optiphot-100) with 2D CCD camera (PAXcam)	Yes	Yes
Liquid scintillation counter (Beckman LS6500)	Yes	No[b]
Imaging plate reader (Fujifilm FLA-7000)	Yes[c]	Yes[d]
Scanning Auger microscope (Perkin Elmer PHI 660)	Yes[a]	Yes
X-ray photoelectron spectroscopy (Perkin Elmer PHI 5400)	Yes[a]	Yes
Glow discharge optical emission spectroscopy (Horiba GO-PROFILER 2)	Yes[a]	Yes

[a] Surface tritium contamination of sample must be reduced to less than 10,000 dpm/100 cm^2.

[b] Contamination with radioactive material other than tritium must be avoided in tritiated liquid.

[c] Typically a single layer of protective film is inserted between highly tritium contaminated sample (> 10,000 dpm/100 cm^2) and imaging plate to minimize tritium contamination of imaging plate.

[d] Beta and gamma background from radioactive sample must be comparable or smaller than the beta signal from implanted tritium for tritium detection use.

[f] Radioactive material must be contamination free, and its dose rate must be less than 100 mR/hr at 30 cm.

4. Conclusions

The TPE has restarted plasma operation with new electrical and control system in Nov. 2015. The efforts to improve plasma and tritium diagnostic capabilities in the TPE and surface diagnostic capabilities for tritium-exposed and radioactive samples at the STAR facility were being carried out in parallel to enhance operational safety during the three-year TPE upgrade. New acquisition of scanning Auger microscope, X-ray photoelectron spectroscopy and glow discharge optical emission spectroscopy will strengthen the unique capability at the STAR facility to handle tritium-contaminated and radioactive samples. The TPE-upgrade and the improvement of surface diagnostics capabilities at the STAR facility will provide valuable tritium and nuclear PMI database to advance scientific understanding of multiple separate effects and synergistic effects in fusion nuclear environment.

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References

[1] ITER Preliminary Safety Report (RPrS), 3ZR2NC (2011).

[2] R.A. Causey, R.A. Karnesky, and C. San Marchi, “Chapter 4.16: Tritium barriers and tritium diffusion in fusion reactors” in “Comprehensive Nuclear Materials” R.J.M. Konings (Elsevier, 2012).

[3] M.J. Morgan, S. West, and M.H. Tosten, *Fus. Sci. Technol.* **54** (2008) 501.

[4] M. Sawan, *Fus. Sci. Technol.* **66** (2014) 272.

[5] C.H. Skinner, C.A. Gentile, L. Ciebiera, and S. Langish, *Fus. Sci. Technol.* **45** (2004) 11.

[6] M. Ikeya, T. Miki, and M. Touge, *Nature*, **292** (1981) 613.

[7] G.R. Longhurst and R.A. Causey, *Nucl. Technol. Fus.* **4** (1983) 681.

[8] M. Shimada, G. Cao, T. Otsuka, M. Hara, M. Kobayashi, Y. Oya, and Y. Hatano, *Nucl. Fusion* **55** (2015) 013008.

[9] A. Hasegawa, M. Fukuda, K. Yabuuchi, and S. Nogami, *J. Nucl. Mater.* **471** (2016) 175.

[10] M. Shimada, R.D. Kolasinski, J.P. Sharpe, and R.A. Causey, *Rev. Sci. Instru.* **82** (2011) 083503.

[11] “Hazard Categorization and Accident Analysis Techniques for Compliance with DOE Order 5480.23, Nuclear Safety Analysis Report”, US Department of Energy (DOE) Standard DOE-STD-1027-92 (1992).

[12] M. Shimada, G. Cao, T. Otsuka, M. Hara, M. Kobayashi, Y. Oya, and Y. Hatano, *Nucl. Fusion* **55** (2015) 013008.

[13] M. Shimada, C.N. Taylor, R.J. Pawelko, R.D. Kolasinski, D.A. Buchenauer, L.C. Cadwalladar, and B.J. Merrill, *Fus. Eng. Des.* **109-111** (2016) 1077

[14] D.A. Buchenauer, R.D. Kolasinski, M. Shimada, D.C. Donovan, D.L. Youchison, and B.J. Merrill, *Fus. Eng. Des.* **89** (2014) 1014.

[15] C.N. Taylor, M. Shimada, B.J. Merrill, M.W. Drigert, D.W. Akers, and Y. Hatano, *Phys. Scr.* **T159** (2014) 014055.

[16] C.N. Taylor, M. Shimada, B.J. Merrill, D.W. Akers, and Y. Hatano, *J. Nucl. Mater.* **463** (2015) 1009.

[17] R.J. Pawelko, M. Shimada, K. Katayama, S. Fukada, P.W. Humrickhouse, and T. Terai, *Fus. Eng. Des.* **102** (2016) 8.

[18] Y. Hatano, J. Shi, N. Yoshida, N. Futagami, Y. Oya, and H. Nakamura, *Fus. Eng. Des.* **87** (2012) 1091.