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SIMPLE CATALYTIC CELL FOR RESTORING He LEAK DETECTOR SENSITIVITY ON VACUUM SYSTEMS WITH HIGH D_2 BACKGROUNDS

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GENERAL ATOMICS

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ABSTRACT

The DIII–D National Fusion Facility at General Atomics focuses on plasma physics and fusion energy science. The DIII–D tokamak is a 35 m^3 toroidal vacuum vessel with over 200 ports for diagnostic instrumentation, cryogenics, microwave heating, and four large neutral beam injectors. Maintaining vacuum in the 10^{-8} Torr range is crucial for producing high performance plasma discharges. He leak checking the DIII–D tokamak and the neutral beamlines has historically been difficult. D_2 is used as the fuel gas in most plasma discharges and neutral beams. After plasma operations, D_2 out-gassing from the torus walls and internal beamline components can exceed 10^{-4} std cc/s. The mass of the D_2 molecule (4.028 u) is indistinguishable from that of the He atom (4.003 u) to a standard mass spectrometer leak detector. High levels of D_2 reduce leak detector sensitivity and effectively mask the He trace gas signal rendering normal leak checking techniques ineffective. A simple apparatus was developed at GA to address these problems. It consists of a palladium based catalyst cell and associated valves and piping placed in series with the leak detector. This reduces the D_2 throughput by a factor greater than 10,000, restoring leak detector sensitivity. This paper will briefly discuss the development of the cell, the physical processes involved, the tests performed to quantify and optimize the processes, and the operational results at DIII–D.

I. INTRODUCTION

A novel application of catalytic chemistry for use in helium (He) leak detection on a deuterium (D_2) contaminated high vacuum system was developed at General Atomics. The DIII–D National Fusion Facility at General Atomics focuses on plasma physics and fusion energy science research. The work is based on developing plasma confinement and plasma shaping schemes in the DIII–D tokamak. D_2 is used to fuel most plasma discharges. The core of the tokamak is a 35 m^3 toroidal vacuum vessel constructed of Inconel 625 stainless steel with over 200 ports for diagnostic instrumentation, cryogenics, microwave heating, and four large neutral beam injectors (Fig. 1). Except for port openings, the inner wall of the vacuum vessel is fully covered with graphite armor tiles [1].

Effective leak checking is an important prerequisite for achieving and maintaining vacuum in the 10^{-8} Torr range that is crucial for producing high performance plasma discharges. He leak detection at DIII–D has historically been difficult. The difficulty lies in the substantial inventory of D_2 that saturates the graphite armor tiles which protect the inner walls of the tokamak from the plasma discharges. Leak checking the neutral beamlines has also been difficult, since D_2 resides in the beamline inner walls and internal components as a consequence of D_2 beam operations. The mass of the D_2 molecule (4.028 u) is indistinguishable from that of the He atom (4.003 u) by a standard mass spectrometer leak detector. High partial pressures of D_2, caused by outgassing from the system components, reduce leak detector sensitivity and effectively mask the He trace gas signal, rendering normal leak checking techniques ineffective for all but large leaks.

After plasma operations, D_2 outgassing from the torus walls and armor tiles can exceed 10^{-4} std cc/s. In the past, before leak checking, the torus was subjected to a prolonged bake at 350°C then allowed to cool to ambient temperature. A minimum of twenty-four hours elapsed before leak checking could begin. The bake reduces the leak detector background level to an equivalent He leak rate of ~10^{-6} std cc/s, allowing limited use for larger leaks. These background levels preclude leak checking in the more sensitive ranges, necessary for finding leaks smaller than 10^{-6} std cc/s, without additional baking or waiting extended periods of time for D_2 levels to decrease due to normal pumping.

Leak checking the neutral beam injectors also presents a formidable challenge. Each 17 m^3 neutral beamline is a vacuum vessel consisting of three 96 in. diameter aluminum spool pieces joined with Buna o-ring sealed flanges. Numerous ports for access, diagnostics, cryogenics and water cooling also rely on o-ring seals. The beamlines cannot be baked because of the o-ring seals and possible thermal expansion damage to the internal 4×10^6 ℓ/s cryo-pumping panels.

Down time between periods of D_2 beam operations is often not sufficient for D_2 outgassing to decrease to levels where effective leak checking can take place. This was the impetus for seeking a method to reduce D_2 backgrounds sensed by the leak detector.

Previous methods for dealing with high D_2 backgrounds have employed cryopumping, titanium gettering and Zr/Al gettering. These methods were not easily portable, required complex systems, were short lived, or required high vacuum for operation and/or extremely high temperatures for activation. The Zr/Al getter has been shown to achieve a best case reduction of D_2 levels by a factor of 250 [2].

A different approach was pursued at DIII–D. Instead of differentially pumping D_2 from the sample stream to the leak detector, the possibility of converting the D_2 to heavy water (D_2O, 20.023 u) by catalytic reaction was explored.

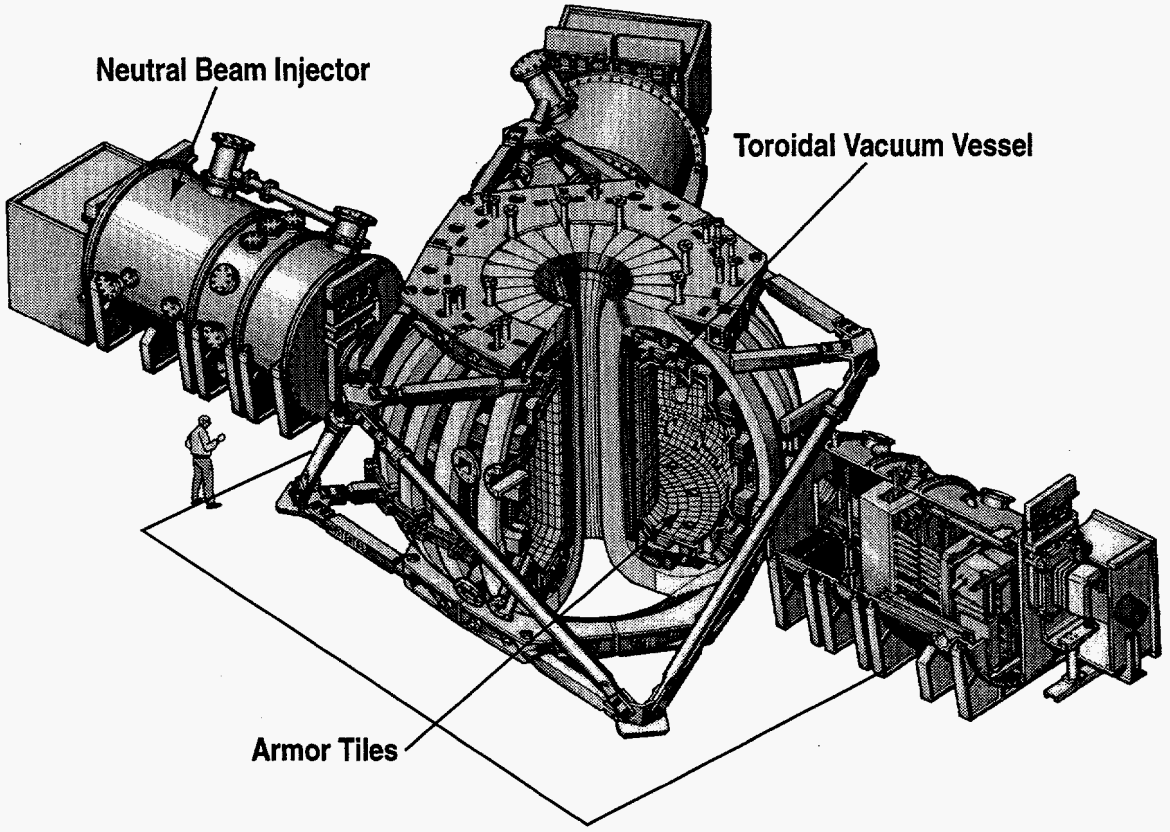

Fig. 1. Cutaway view of DIII–D tokamak and three of four beamlines.

II. CATALYST CELL THEORY AND PRINCIPLES

Catalysis is primarily a surface effect. The catalyst typically is a body centered cubic (BCC) lattice material that provides a binding site for the reactants in a chemical reaction. In this case, a deuterium or an oxygen atom is adsorped onto the interstitial voids ("dimples") of the BCC lattice forming a quasi-face centered cubic (FCC) type structure at the lattice surface. The work functions of the surface and the adsorped atom are both depressed, effectively lowering the energy threshold for reaction. The adatom will latch onto the next available reactant, forming an adsorped D_2O molecule on the catalyst surface. The application of bulk heating to the catalyst provides energy for the reaction, and energy for the adsorped molecule to leave the binding site [3].

At DIII–D, the small leaks that degrade plasma performance are typically air leaks. This fact was exploited by using the residual oxygen present in a chemical reaction with the outgassing D_2 to form heavy water. The sample stream of the leak detector is passed through a catalyst filled reaction chamber that facilitates the formation of D_2O. The resultant heavy water molecules are easily distinguished from helium by the mass spectrometer. Ideally, the catalyst will convert enough of the D_2 background to restore sufficient leak detector sensitivity to He for effective leak checking.

Platinum and palladium are used as catalysts in the pharmaceutical, nuclear, specialty and fine chemical industries. Consultation with industry experts [5] led to the choice of palladium for a proof of principle experiment as it offered the best performance for use with hydrogen and its isotopes.

III. CELL CONSTRUCTION

The prototype cell was assembled from a 1 in. foreline trap [4] with its molecular sieve material removed and replaced with 700 cc's of a commercially available precious metal catalyst. The catalyst consisted of 0.5% palladium supported on 1/8 in. o.d. alumina spheres. The cell was evacuated, and baked with the trap's existing heater at a temperature of 200°C, until a base pressure of 5×10^{-5} Torr was achieved. The first use of the cell was during a successful leak check of the DIII–D tokamak, where a reduction of D_2 background levels by a factor of more than 700 was achieved. This initial success led to the fabrication of several cells of different designs to investigate the effects of path length, catalyst quantity, and temperature on the removal of D_2. Standard vacuum hardware dimensions and the desire to retain the conductance typical of the 1 in. tubing used for our leak checking fittings dictated the minimum size of the cell (Fig. 2).

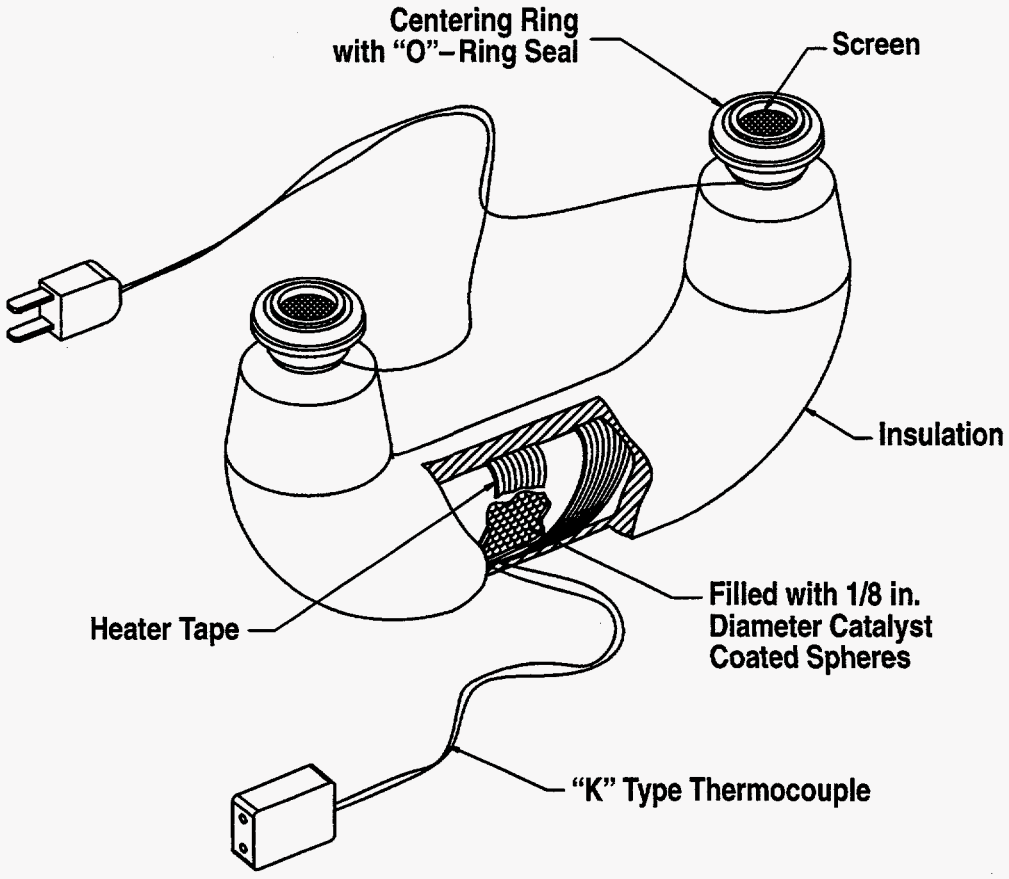

Fig. 2. Final version of catalyst cell with a volume of 333 cc.

IV. TESTING

A series of tests exploring the operating envelope for the catalytic removal of D_2 were performed. An existing vacuum chamber with a residual gas analyzer (RGA) installed for testing the outgassing of materials was used (Fig. 3). A regulated source of D_2 gas fitted with a precision leak valve was connected to a manifold that directed the gas flow either through the catalyst filled cell or directly into the test chamber, bypassing the cell.

The precision leak valve was adjusted to provide a D_2 leak rate that approximated the maximum outgassing rate observed for the DIII–D tokamak. All tests were begun with the catalyst cell evacuated, baked, and brought to stable temperature and pressure conditions. The RGA was configured to periodically record ion current readings for a few selected masses, the most significant being, D_2 (mass=4), D_2O (mass=20), N_2 (mass=28), and O_2 (mass=32). The D_2, O_2, and D_2O readings documented the effect of the catalyst in the test cell. The expected result being a decrease in D_2 and O_2 with an increase in D_2O, indicating the reaction of D_2 and O_2 to form heavy water. The N_2 reading was used as a check for other changes not due to the catalytic process. The cell was placed on-line and a set of baseline RGA ion current readings was taken allowing comparison of initial vacuum conditions for different catalyst cell configurations at the beginning of each series of tests. The leak isolation valve was then opened and test gas flow directed through the catalyst cell. After temperatures and pressures had stabilized, a "cell test" set

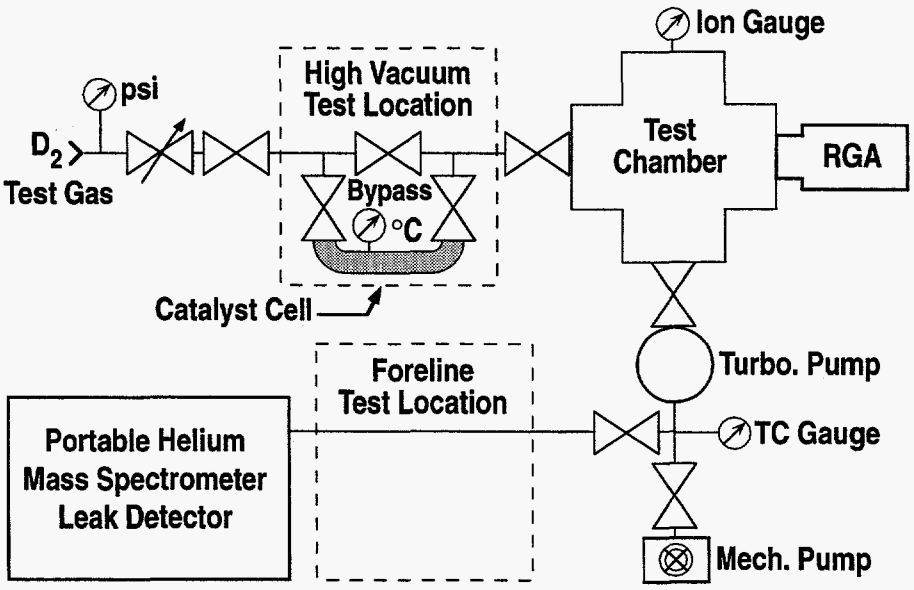

Fig. 3. Diagram of setup utilized for testing each catalyst cell.

of RGA readings was taken. The catalyst cell was then isolated and the bypass valve opened, sending the test gas directly into the test chamber. A "bypass" set of RGA readings was taken. Comparing the cell test and bypass set of readings allowed calculation of the effective reduction ratio of D_2 for each test. Test results are summarized in Table 1

Most tests were run with the catalyst cells in the high vacuum test location (Fig. 3) in order to gather data with the RGA. To duplicate actual leak checking conditions, additional tests were run with the catalyst cell and manifold connected between the inlet of a leak detector [6] and the foreline of the turbopump. For maximum sensitivity during leak checking the mechanical pump is isolated and all flow is directed through the leak detector which provides the backing for the turbo pump. Results from the high vacuum and foreline test locations showed no appreciable differences (Fig. 4).

TABLE 1
Catalyst Cell D_2 Reduction Test Results

Test #	Cell Geometry	Catalyst Volume (cc)	Temperature (°C)	Cell Test D_2 Ion Current (A)	ByPass D_2 Ion Current (A)	**D_2 Reduction Ratio** (bypass/cell test)
1	Mole. Sieve	700	48	7.00×10^{-10}	2.50×10^{-7}	$\mathbf{3.6\times10^{2}}$
2	Mole. Sieve	700	85	6.00×10^{-10}	3.00×10^{-7}	$\mathbf{5.0\times10^{2}}$
3	Mole. Sieve	700	218	3.00×10^{-10}	2.00×10^{-7}	$\mathbf{6.7\times10^{2}}$
4	Mole. Sieve	700	197	2.00×10^{-12}	2.00×10^{-8}	$\mathbf{1.0\times10^{4}}$
5	1.5 in. tube	730	40	1.20×10^{-12}	1.60×10^{-7}	$\mathbf{1.3\times10^{5}}$
6	1.5 in. tube	730	100	1.10×10^{-12}	1.40×10^{-7}	$\mathbf{1.3\times10^{5}}$
7	1.5 in. tube	730	200	2.00×10^{-12}	1.50×10^{-7}	$\mathbf{7.5\times10^{4}}$
8	1.5 in. tube	365	24	2.20×10^{-12}	1.20×10^{-7}	$\mathbf{5.5\times10^{4}}$
9	1.5 in. tube	333	22	1.40×10^{-12}	3.50×10^{-8}	$\mathbf{2.5\times10^{4}}$
10	1.5 in. tube	333	95	2.10×10^{-12}	4.10×10^{-8}	$\mathbf{2.0\times10^{4}}$
11	1 in. tube	700	31	1.20×10^{-12}	4.10×10^{-8}	$\mathbf{3.4\times10^{4}}$

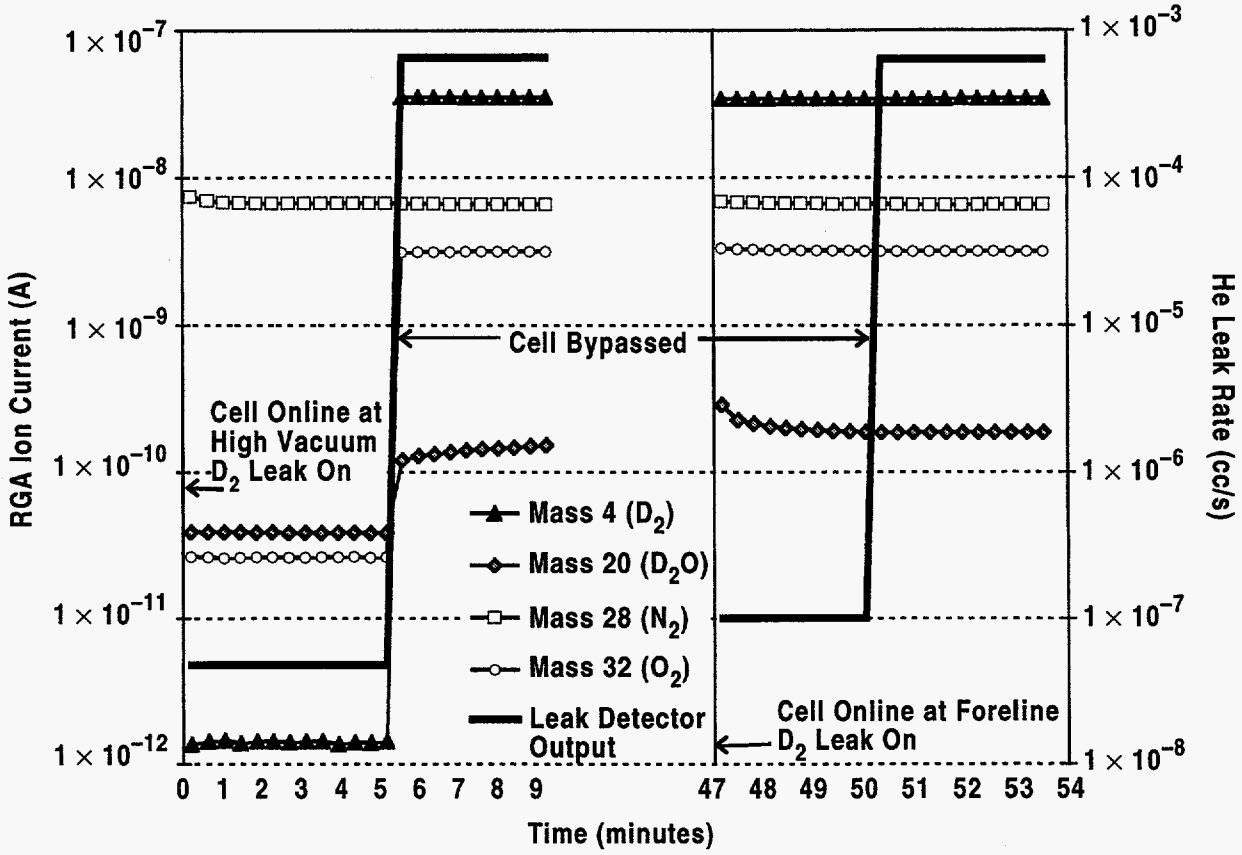

Fig. 4. Comparison of leak detector and RGA test results for 333 cc catalyst cell in high vacuum versus foreline locations.

A. Cell Geometry

Three different cells were fabricated to investigate the effects of path length. Improvements in D_2 reduction were obtained by placing the catalyst inside 1 1/2 in. tubes with containment screens at each end. The increase in path length compared to that provided by the molecular sieve container resulted in more opportunities for interaction with the catalyst and a greater reduction of D_2. Tests with a 1 in. tube, twice as long, containing a similar amount of catalyst were also performed. D_2 reduction was lower and a degradation of leak checker response time due to decreased conductance was also observed (Fig. 5).

B. Catalyst Quantity

For ease of comparison, the preliminary tests of catalyst-filled tubes used about the same quantity of catalyst as had been used in the prototype. Halving the quantity of catalyst resulted in only a minor decrease in the effectiveness of the D_2 reduction but had a direct effect on the length of time for the un-heated catalyst to reach saturation. The cell containing 365 cc of

catalyst reached saturation in ~twenty-four hours with D_2 flowing at (a He equivalent leak rate of) ~5×10^{-4} cc/s. This provides more than sufficient capacity, without heating, for a full day's leak checking activities at DIII–D (Fig. 6).

C. Cell Temperature

According to the catalyst supplier [5], temperatures of 60°C are needed for reliable catalysis, although evidence of D_2O production was detected even at 22°C. Heating the cell above the temperature threshold of 60°C accelerates the catalytic formation of D_2O and facilitates its desorption. This frees binding sites for additional reactant adsorption and increases the partial pressures of D_2O in the test chamber. The reduction ratio of the tubular cells did not change significantly when the bulk temperature of the catalyst was heated above the 60°C threshold. This demonstrates that the dominant early effect is adsorption (Fig. 7). However, heating the cells above 200°C causes the catalyst cell to outgas D_2 at a high rate, resulting in an undesirable higher background.

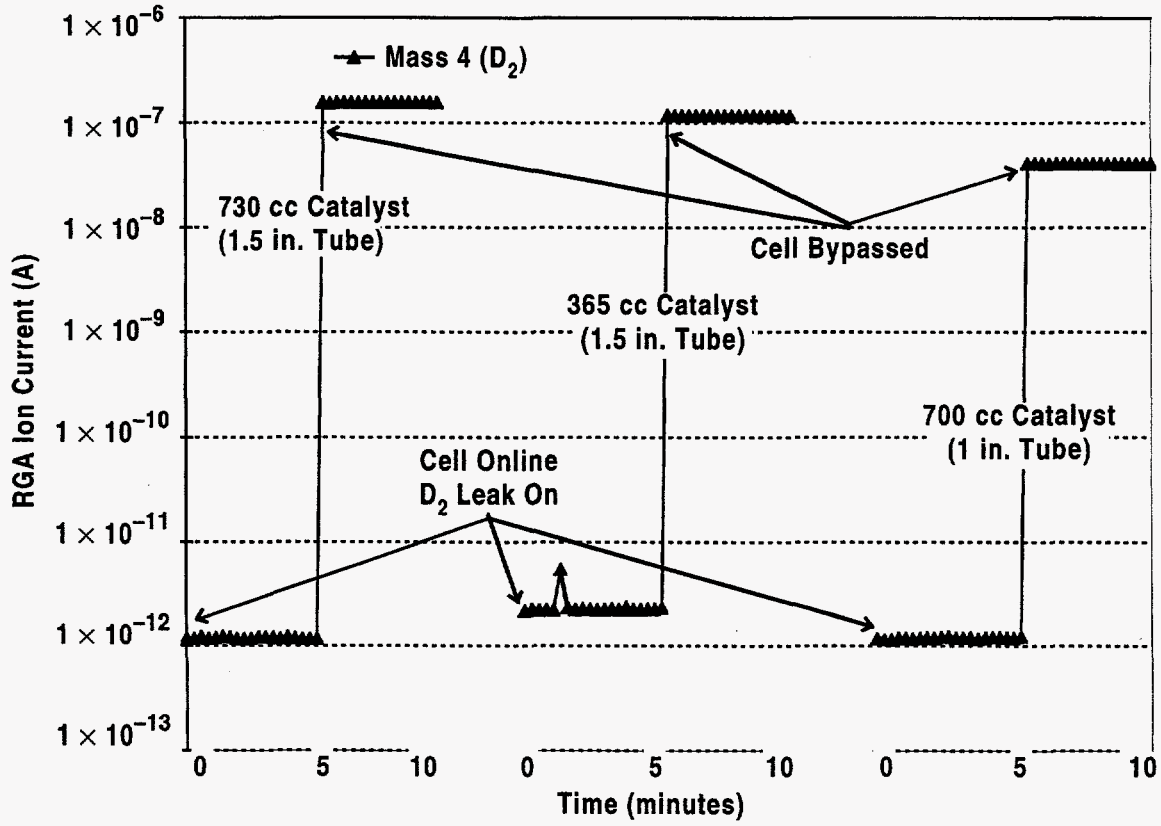

Fig. 5. Comparison of D_2 reduction test for three different catalyst cell configurations.

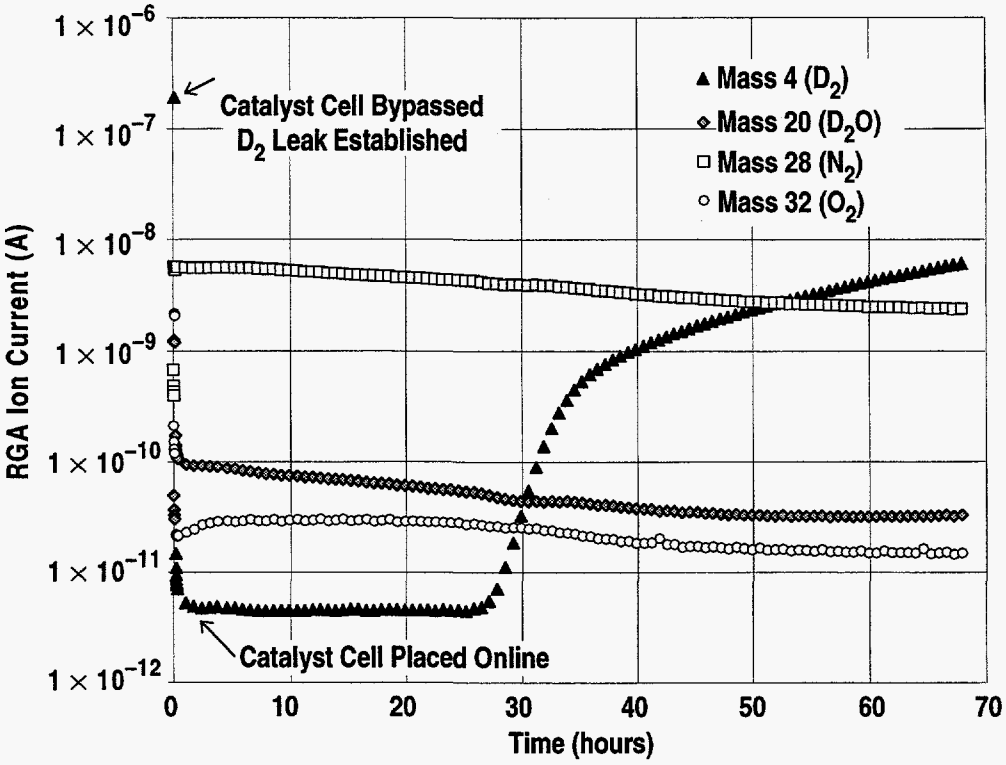

Fig. 6. 365 cc catalyst cell saturation test.

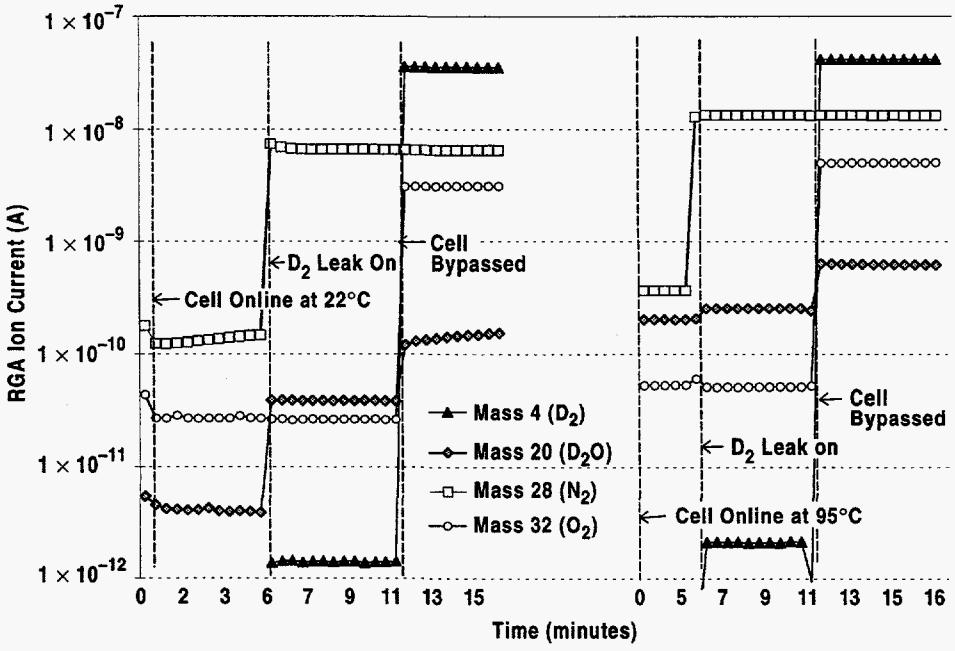

Fig. 7. Comparison of 333 cc catalyst cell test results @ 22°C and 95°C.

V. OPERATIONAL RESULTS

A catalyst cell has been used for every leak check of the DIII–D machine and neutral beamlines since the successful inaugural use of an experimental cell in September 1997. At that time, an intermittent leak had severely impacted DIII–D operations for several weeks and all attempts to locate the leak had failed. Each time the torus was baked to reduce the D_2 background levels sufficiently for effective leak checking, the leak disappeared and then reappeared after resumption of experimental operations. The prototype catalyst cell being prepared for leak checking a neutral beamline was completed and placed on the inlet of a leak detector connected to the DIII–D tokamak (Fig. 8). Leak rate background levels measured by the leak detector before installation of the cell were 7×10^{-6} std cc/s. After the cell was valved in, the leak rate background levels fell below the most sensitive range of the leak detector ($<10^{-8}$ std cc/s), improving sensitivity by a factor of more than 700. The leak was found shortly thereafter and machine operations resumed the next day. During the next maintenance period, the prototype catalyst cell was finally used for leak checking a neutral beamline. The initial background level was 2.6×10^{-5} std cc/s. The cell reduced the background to 1×10^{-7} std cc/s and a small leak that had eluded detection for more than six months was found within two hours.

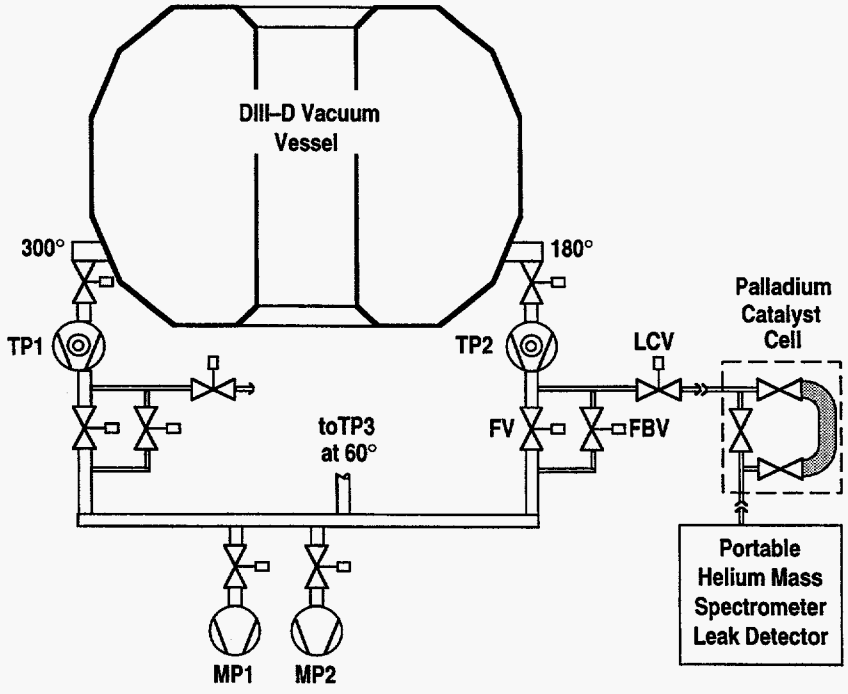

Fig. 8. Diagram of the leak detection set-up on DIII–D. Two of the three 5000 ℓ/s turbo pumps are isolated. The foreline valves on the third are shut and all exhaust flow is pumped through the catalyst cell by the portable leak detector.

VI. CONCLUSIONS

This device provides a significant improvement in He leak checking capabilities, especially immediately following D_2 plasma operations. The time elapsed from end of operations to beginning of leak checking has been reduced from more than a day to less than an hour. The restored sensitivity has increased the efficiency of leak checking procedures and permitted detection of smaller leaks. Maximum leak checking sensitivity is still limited by increased He background levels arising from occasional He plasma experiments and He glow discharge conditioning of the vessel wall.

Bench-top tests indicate a repeatable reduction in D_2 background levels by a factor $>10^4$. The process can be summarized in two steps:

1. Deuterium and oxygen are adsorped onto the catalyst surface, reducing their partial pressures in the test chamber; this is the dominant early effect.
2. Catalytic formation of D_2O begins. Heating the cell above the temperature threshold of 60°C accelerates the catalysis and facilitates the desorption of the D_2O. This frees binding sites for additional reactant adsorption and extended operation.

Leak detector operational background levels are usually reduced by a factor of $\sim 10^3$ with less of a reduction apparent at lower initial D_2 background levels. Since bench-top tests indicate the D_2 contribution to the background is reliably reduced by a factor $>10^4$, the remaining background can be attributed to the presence of helium.

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