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TITLE RESULTS OF EXPERIMENTS ON CERAMIC ELECTROLYSIS CELLS AND PALLADIUM DIFFUSERS FOR APPLICATION TO FUSION REACTOR FUEL CLEANUP SYSTEMS

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RESULTS OF TRITIUM EXPERIMENTS ON CERAMIC ELECTROLYSIS CELLS AND PALLADIUM DIFFUSERS FOR APPLICATION TO FUSION REACTOR FUEL CLEANUP SYSTEMS*

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Abstract: Tritium tests at the Tritium Systems Test Assembly have demonstrated that ceramic electrolysis cells and palladium alloy diffusers developed in Japan are possible components for a fusion reactor fuel cleanup system. Both components have been successfully operated with tritium for over a year. A failure of the first electrolysis cell was most likely the result of an overvoltage on the ceramic. A simple circuit was developed to eliminate this mode of failure. The palladium diffuser tubes exhibited some degradation of mechanical properties as a result of the build up of helium from the tritium decay, after 450 days of operation with tritium, however the effects were not significant enough to affect the performance. New models of the diffuser and electrolysis cell, providing higher flow rates and a more tritium compatible designs are currently being tested with tritium.

Introduction

The experiments described in this paper are performed under the continuing collaborative program between the U. S. Department of Energy and the Japan Atomic Energy Research Institute (JAERI), beginning in 1984. Early results of the experiments are describe in Ref. 1 and 2. This paper presents the results obtained since those papers on the Ceramic Electrolysis Cell (CEC) and Palladium-alloy Diffuser experiments. These two components are being tested as possible elements of a Fuel Cleanup system (FCU) for a tritium-burning fusion reactor. These components were first tested with hydrogen and deuterium at the Japan Atomic Energy Research Institute (JAERI). All tritium tests have been conducted at the Tritium Systems Test Assembly (TSTA).

Electrolysis Cell

The CEC uses a solid oxide ceramic electrolyte which is not affected by tritium[3]. The CEC decomposes tritiated water in the vapor phase. The output of the cell is a stream of hydrogen isotopes free of oxygen, with small amounts of water, and a waste stream of oxygen free of hydrogen isotopes. The cell has the advantages of eliminating both the high tritium inventory necessary in liquid electrolysis cells and the radiation damage in cells with a solid polymer electrolyte.

Initial CEC tests with tritium were performed on two single-tube systems. The first cell failed (the ceramic tube cracked) after approximately five months operation with tritiated water. A second identical single-tube CEC was then installed and tested. It was operated successfully for one year. Then a series of tests to induce failure were performed. When this cell was disassembled the ceramic tube was found to have failed. A second CEC model, containing ten tubes, is now installed at TSTA and being tested with tritium. This cell has vacuum jacket insulation and is designed to handle a higher flow rate of tritiated water.

Palladium Diffuser

The diffuser contains proprietary multi-component palladium-alloy tubes which are operated at 300°C, where hydrogen isotopes diffuse through the palladium membrane while impurities such as helium, methane, and ammonia will not permeate the palladium-alloy tubes. Two diffusers have been tested at TSTA with tritium and impurities. The first model tested contained 12 tubes. This diffuser was designed both to study the long term operating parameters related to continuous operation with tritium and also the effect of impurities in the tritium stream on the permeability of the palladium diffuser. The diffuser was not designed with a secondary containment philosophy.

The second diffuser contains 35 tubes of the same dimension as the first. The design flow capacity of the second diffuser is approximately one-eighth that of the TSTA process loop flow. This model has an improved engineering design (secondary containment and heat shields) that would be typical of a diffuser for application in a fuel cleanup system. The

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design reduces the heating requirements and the tritium diffusion through the outer shell surrounding the palladium-alloy tubes.

The first diffuser was successfully operated in a closed circulation loop for approximately 450 days with both pure tritium and tritium plus impurities. After completion of the tests, the diffuser was cut apart and mechanical tensile tests were performed on the tubes to determine if the properties had changed as a result of the tritium tests. The second diffuser is currently being tested at TSTA. Permeation measurements, wall diffusion and impurity separation efficiency have been measured.

In addition to the loop tests performed on the diffuser, a test program was initiated to determine if the palladium alloy tubes would experience any embrittlement effects (degradation of ductility) caused by the build up of helium-3 in the tube wall from the decay of tritium permeating through the palladium tubes. The alloying of the tubes should mitigate or prevent the effect of beta embrittlement. No formation of the beta phase at ambient temperature and 1000 torr of hydrogen has been seen[2].

ZrCo Getter Tests

JAERI personnel have developed a ZrCo alloy for use for solid storage of tritium[4]. Properties for storage of hydrogen and deuterium are similar to those for hydrogen on uranium. Two of these beds are being used for supplying and storing tritium for the current CEC and diffuser experiments. This paper will present initial results of this work.

Applications to Fusion Reactor Systems

The TSTA fuel cleanup system [5,6] is based on hot metal beds or cryogenic molecular sieve beds for the separation of impurities from the process gas stream. Hot metal beds are also used for recovery of tritium from impurities. Uranium beds operating at 750 K, are used to recover tritium from tritiated water and uranium beds operating at 1150 K are proposed for the recovery of tritium from impurities such as methane and ammonia. The hot metal bed systems have the disadvantages of requiring relatively high temperature for the containment of uranium and the formation of solid waste (uranium carbides, oxides and nitrides) which must be disposed of.

A FCU based on palladium diffusers and ceramic electrolysis cell has been proposed by Yoshida, Konishi and Naruse[7]. The JAERI FCU system uses a palladium diffuser to separate impurities from the process gas, and a CEC to recover tritium from water made in the oxidation of the impurities. JAERI is currently designing a FCU for testing at TSTA. The design of this FCU is based on the results of the tritium tests of the CEC's and the palladium diffusers at TSTA. If approval is obtained, construction of the FCU will begin in 1988 in Japan. The JAERI FCU will then be first tested at JAERI with hydrogen and deuterium. The system will then be shipped to TSTA and installed as part of an upgrade of the TSTA process flow loop. Comparison tests will then be conducted between the operating properties of the TSTA FCU and the JAERI FCU.

CEC Experiments

Description of Ceramic Electrolysis Cell and Tests

Two models of CEC's have been tested at TSTA with tritium. The first was tested to demonstrate operation with tritium and the feasibility of long term operation of a ceramic cell with tritium. The second model has an improved engineering design and can process tritiated water (0.35 mol/hr of tritiated water) at flow and composition conditions similar to those existing in the operation of the TSTA FCU.

Model 1: Tests were performed using two identical cells, consisting of a single yttria-stabilized zirconia tube [1] with platinum electrodes on both the inner and outer surfaces. The tube is closed at one end. Electrical contact is made by mechanical contact with a platinum screen. Voltage for the electrolysis was supplied by a potentiostat which also measured current and voltage. Water vapor in a carrier gas flows into the inside of the ceramic tube through a alumina tube which extends to the bottom of the zirconia tube. The water is electrolyzed with the oxygen transported through the ceramic tube and the hydrogen isotopes flowing out the top of the cell. The cell was heated with "clam shell" heaters outside the primary containment. This method of heating the cell resulted in a high heat load to the glovebox.

The first of the model 1 cells was tested in three phases at TSTA. First tests were performed to confirm the operation with normal water obtained at JAERI. Phase 2 tests were done with low level tritiated water and the cell operating in a once-through mode. The third tests were long term operation with tritiated water in a circulating mode. The CEC was placed in a closed loop with a metal bellows pump used to circulate the gas (38 curies tritium and helium as a carrier, 3.0% tritiated water, tritium processing rate of 20 000 Ci/day). The cell decomposed the tritiated water, the two output streams (tritium/helium and oxygen) from the cell were mixed and passed through a hopcalite catalyst bed, and then passed back to the cell. This cell was operated in the circulating mode for five months. At that time the electrolysis current went to zero indicating that there was no longer any water in the loop or the cell had failed. The cell could not be made operational by adding more water or regeneration of the catalyst bed. The cell was dismantled and the ceramic tube was found to be fractured. This failure initiated the testing of an identical CEC.

This second cell was tested in a closed circulating loop almost identical to the test configuration of the first cell. Figure 1 shows the configuration. A pressure gauge, ion chamber and throttling valve were added to the oxygen side of the cell. The ion chamber allows for the monitoring of the tritium on the oxygen side of the cell. This permits monitoring the integrity of the zirconia tube during operation. Any cracks in the cell will cause an increase in tritium concentration in the oxygen side of the cell. The throttling valve allows a higher pressure (500 torr) so that the back diffusion of tritium into the oxygen side will be reduced. In addition an electronic circuit was added to the potentiostat to prevent overvoltage of the cell.

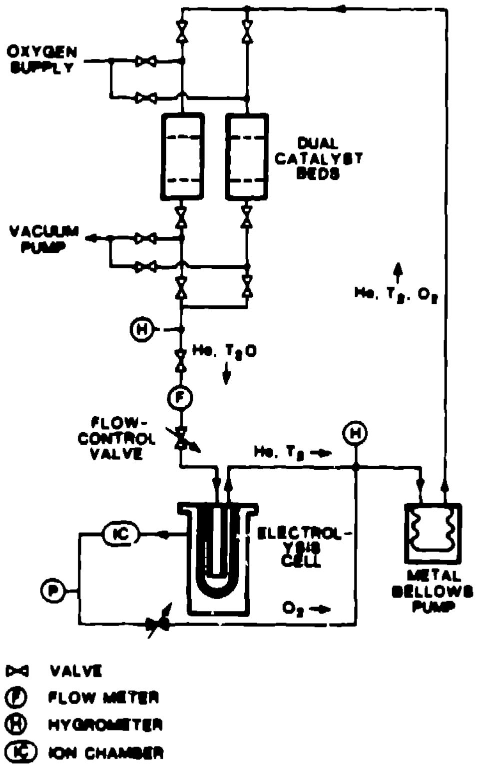

Figure 1. Flow schematic for CEC tests

This system was continuously operated for one year with 20 curies of tritium and helium as the carrier gas, then a series of tests were performed to induce failure:

- Temperature cycling of the cell with the cell at approximately 1 torr and no electrolysis current.
- Temperature cycling of cell in the presence of hydrogen.
- Loop operation with no water, no oxidation of electrolyzed hydrogen.
- Operation of the cell at higher voltage, overvoltage protection removed.

During these tests the pressure difference between the input side of the cell and the oxygen size of the cell, and the ion chamber reading were used to determine if the cell had failed.

Model 2: This cell contains ten zirconia tubes, each 10 mm diameter[8]. Figure 2 shows a cross section of a single tube of this CEC. Again water vapor flows to the cell through a stainless steel tube (coated with ceramic for insulation) to the bottom of the ceramic tube. The cell is contained in a vacuum jacket which reduces the heater power requirements and the heat load to the glovebox. The heater for the cell is placed in a copper block surrounding the ten electrolysis cells.

The cell is being tested to determine the integrity of operation with tritium and the tritiated water conversion efficiency. The CEC is in a continuous flow loop equivalent to the loop used to test the first cell.

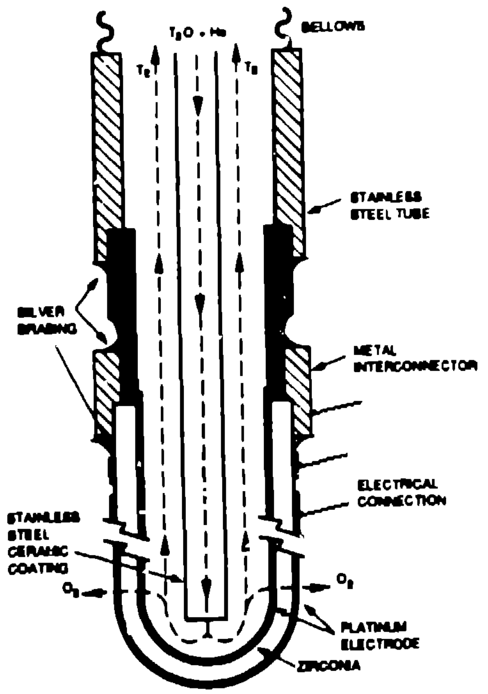

Figure 2. Single cell of ten-tube CEC

Results of Ceramic Electrolysis Cell Experiments

Ref. 1 presents the initial results of the operation of the model 1 cell during the three phases of tests. The conversion ratio X of water vapor to hydrogen is determined from the equation

$$E-Ir = E_0+(RT/(2F))\ln(X/(1-X))$$

where E is the voltage, I is the current, r is the cell resistance, R is the gas constant, T is the temperature, and F is the Faraday constant. The standard voltage for water decomposition E_0 is a function of temperature, at 600°C it is 1.041 V.

Phase 1 tests confirmed the nontritium results obtained at JAERI and demonstrated the feasibility of using hydrogen as a carrier gas during electrolysis. This is important since one of the methods to recover the tritium from impurities involves regeneration of tritiated water using deuterium as a carrier gas. Phase 2 tests demonstrated the operation of the cell with tritium and showed a small isotope effect between HTO and H_2O in E_0 of 0.04 V. The high operating temperature of the cell did not result in loss of tritium to the glovebox through diffusion or leakage. Phase 3 experiments measured the CEC characteristics for T_2O. There is a small effect on the open circuit voltage E-Ir) for the decomposition of H_2O and T_2O of 0.08 volts, and 1.38 V was required to achieve 99.9% conversion of tritiated water.

The phase 3 tests involving continuous operation of the cell in a closed loop, resulted in fracture of the ceramic after five months of operation. The fractures occurred only in the area the platinum electrodes. The second cell operated for one year successfully. After the tests to induce failure were completed the CEC was dismantled the ceramic was found to again have fractured.

The cause of the fracture of the cell ceramic in both cases was most likely a result of an overvoltage across the ceramic during operation. For voltages greater than 2.6 V (energy of formation of ZrO_2), the ZrO_2 is electrically reduced. When operated in a reducing atmosphere, such as hydrogen, this voltage is lowered from 2.6 V. A solid solution of zirconium metal and zirconium oxide can form. When the content of zirconium metal becomes high enough, a phase transition in the ceramic will occur thus causing the ceramic to fail. As evidence that this was the cause of failure, the broken ceramic samples were darkened indicating the presence of zirconium metal and the ceramic was only brittle in the area of the platinum electrodes. Also Raman spectra done at JAERI indicated the existence of a different phase in the broken pieces. The cell was operated at 2.5 V, however, the open circuit voltage was maintained at 1.38 V because of the ohmic drop across the ceramic. A loss of tritiated water in the system would cause the open circuit voltage to rise to 2.5 V since the current and thus the ohmic drop decreased. In addition the ceramic was probably in a hydrogen atmosphere as noted by the reduced copper wires (used to make electrical contact to the electrodes) by the electrodes. The reducing voltage of the ZrO_2 would then be reduced, leading to the failure of the cell. The first cell was operated with no overvoltage protection and when the water was lost from the system the cell failed. The second cell (operated with overvoltage protection) did not fail until an overvoltage was purposely applied to the cell.

The testing of the model 2 ceramic electrolysis cell is ongoing at TSTA. Initial tests have been done with tritium. The vacuum jacket concept for the cell has worked well. The tests of the CEC have been performed under the simulated conditions of the TSTA FCU. Tritiated water was synthesized by the oxidation of 15 Ci of tritium in a hopcalite bed and the trapped in a liquid nitrogen freezer. The freezer is regenerated with argon gas, passing through the CEC where the tritiated water vapor was decomposed. The open circuit voltage was controlled at 1.38 V. Oxygen from the cell was sent to the TSTA waste treatment system. Tritium gas was collected on a ZrCo bed.

Palladium Diffuser Experiments

Description of the Diffusers and Tests

Two models of palladium diffusers have been tested at TSTA with tritium. The tubes in both diffusers were made of the same alloy. Nominally the tubes are palladium with 20 to 25 weight percent silver and much lesser amounts of gold and ruthenium. The diffusers differed in the number of tubes (12 and 35) and the construction of the containment. The tubes were also of the same dimension, 1.6 mm OD and 130 mm in length.

The first diffuser (model 1) did not have secondary containment and was heated by "clam shell" heaters mounted outside the stainless steel primary containment. This did not affect the operation of the diffuser for permeation of hydrogen isotopes through the alloy, however it did result in a rather high rate of diffusion of tritium through the primary containment into the glovebox. The diffuser was operated at 280°C to reduce this tritium diffusion. Even at the lower temperature the system lost approximately 0.5 Ci/day through the primary containment.

The second diffuser (model 2) is a prototype diffuser for operation in a FCU. It is equipped with heat shields and a secondary containment jacket around the primary containment. The secondary containment can be operated either with a vacuum or an inert gas. The thermal shields reduce the power necessary for heating the diffuser and also reduce the tritium permeation through the secondary containment jacket into the glovebox. Tritium which permeates through the primary containment is collected and can be recovered by using a hydrogen getter bed. The primary difference between this diffuser and one for application in a FCU is the length of the permeation tubes. AERI analysis predicts that the tubes should be 66 cm long to achieve the desired low level of hydrogen isotopes in the bleed stream.

Both diffusers were installed in similar circulating flow loops. Figure 3 is a schematic of the plumbing for the tests on the second diffuser. The arrangement for the first had only one pump, fewer sampling points and used a uranium bed for storage rather than the ZrCo bed.

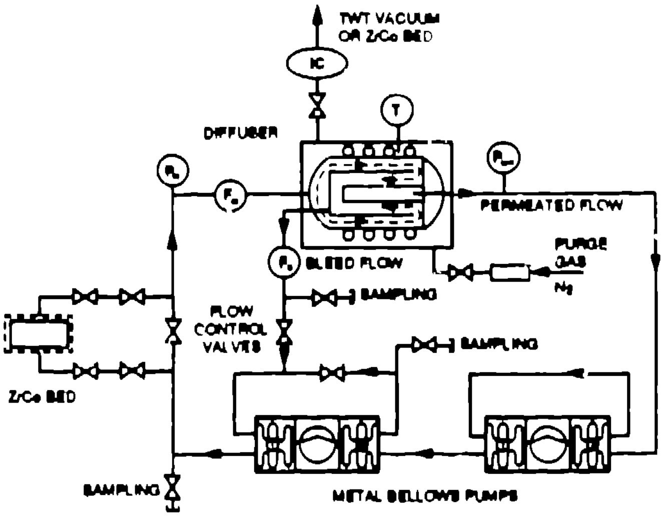

Figure 3. Schematic of plumbing for diffuser measurements

Model 1 was operated for approximately 450 days with either tritium or tritium plus impurities. Initial tests were done to measure the permeability with hydrogen, deuterium and tritium. After completion of the permeation tests the diffuser was operated with pure tritium for five months. The temperature was 280°C, permeation flow at 280 sccm, and input pressure at 2500 torr. Impurities were then added to the tritium to determine the effect on the operation of the diffuser with impurities present. Ammonia, methane and helium were used independently. "Grab" samples were periodically taken for offline Raman analysis. After completion of the impurity tests, the permeability of the diffuser was measured with hydrogen and deuterium and tritium to determine if changes in the permeability had occurred after long term tritium operation.

Metallurgical studies were then performed on the palladium alloy tubes. The diffuser was carefully cut apart so that the tubes could be removed. The tests performed and the results are given in the section on metallurgical studies.

Permeation measurements at several temperatures, with hydrogen, deuterium and tritium have been made on the model 2 diffuser. The diffuser has been operated with hydrogen and methane to measure the separation characteristics. In addition, initial measurements of wall permeation of tritium have been made.

Results of Palladium Diffuser Experiments

Figure 4 is a comparison of the pressure dependence of the permeability of the model 1 diffuser for H_2, D_2, and T_2, between measurements made initially and measurements after approximately 450 days of operation with tritium or tritium and impurities. As expected, the permeation depends on the difference of the square-root of the pressures across the palladium membrane. There was an increase (approximately 8%) in the permeation flow rate of the diffuser for hydrogen and deuterium. This may be attributed to the effects of the helium in the palladium tubes. This increase in the permeation did not affect the operating characteristics of the system and would not influence the operation of a diffuser in a FCU for a fusion reactor.

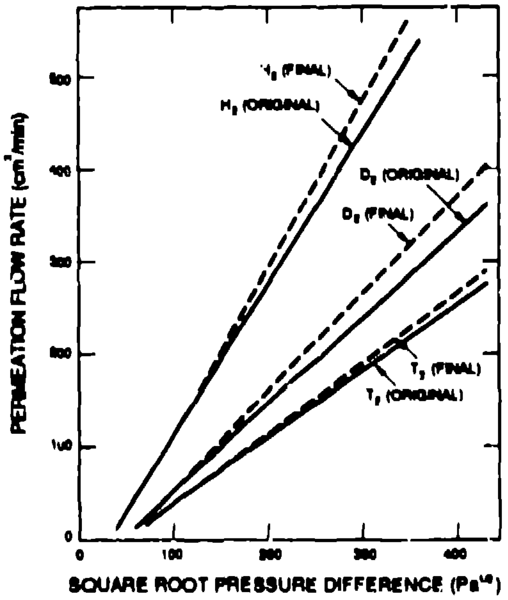

Figure 4. Shaft in permeation with 450 days of exposure to tritium.

The impurity studies demonstrated that the diffuser operated acceptably with impurity levels as high as 20%. At the higher impurity levels, it was necessary to increase the bleed flow rate to prevent blanketing of the diffuser tubes. The blanketing stopped the diffusion of hydrogen isotopes through the palladium. The bleed flow was normally set at 10% of the feed flow, however it was increased to 20% at the higher impurity levels. The blanketing effect is caused by the formation of a boundary layer of fluid having a high concentration of impurities on the surface of the Pd tubes. This phenomenon will strongly depend on the hydrodynamic conditions such as Reynolds Number of the fluid in the vicinity of the Pd-tube surface and the mutual diffusivity of the impurities. Therefore the study of optimum operating conditions will be continued.

Long term operation of the diffuser with ammonia or methane was not possible. When methane (CH_4 added) was introduced, it was decomposed (to carbon and hydrogen isotopes) as a result radiological reaction induced by the beta from the tritium decay. The decomposition reaction, was found to be first order and independent of the presence of the palladium diffuser and independent of temperature. The methane concentration circulating in the diffuser loop decreased by a factor of ten in approximately 20 days. This was the same time constant as for the rate of decay measured for methane in tritium contained in a stainless steel bottle at room temperature. Carbon was most likely deposited uniformly throughout the system. When the diffuser was cut apart there was no evidence of carbon deposits on the palladium tubes.

In the ammonia experiments (NH_3 added), the palladium diffuser itself catalyzed the decomposition of ammonia to hydrogen isotopes and nitrogen. In these experiments, no evidence of nitrogen was found in the Raman analysis of the circulating gas, however the increase in hydrogen in the system was seen.

Figure 5 shows the results of the measurements of the hydrogen isotope permeability of the model 2 diffuser. Measurements were made as a function of temperature and pressure difference across the palladium tubes. The results for all three hydrogen isotopes show a linear relationship between the permeability flow rate and difference in the square-root of the pressures across the diffuser. There is a deviation from linearity at the high and low flows as was seen in the experimental results for the first diffuser tested.

Initial impurity tests were performed using H_2 and methane. Calculations done at JAERI predicted for a 35 tube diffuser with tubes 33.0 cm long, a 10% hydrogen concentration in the bleed stream. The measured concentration (by Raman analysis) was approximately 50% hydrogen/50% methane, independent of bleed flow rate. The discrepancy could be a sampling problem. Further tests are planned to determine the separation characteristics.

The tritium diffusion into the vacuum jacket at 300°C is 0.8 Ci/day when the process pressure is 600 torr. These studies will continue.

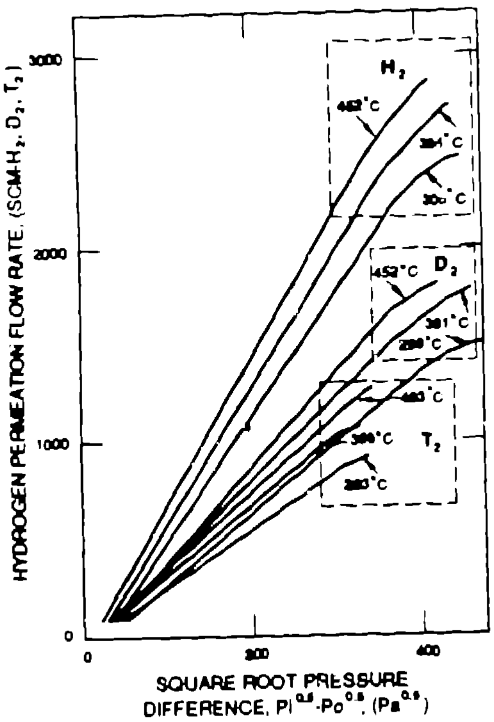

Figure 5. Permeation for 35 tube diffuser

Metallurgical Studies of Diffuser Palladium Tubes

In addition to the loop tests on the palladium diffuser, tensile tests were done on the palladium alloy tubes after exposure to tritium. Two types of tests were performed. The first tests involved static soaking of palladium alloy tubes in tritium at 300°C for varying lengths of time and subsequent tensile testing of the samples. The second tests involved tensile tests on the tubes from the model 1 diffuser after completion of the loop test. The later tests were inherently different in that the tritium was driven through the palladium by the pressure difference across the tubes (1200 to 1500 torr, high pressure side; 50 torr low pressure side). The tubes used in both tests were nominally the same alloy, made by the same manufacturer and cut from the same model diffusers. However, initial tests on tubes that had not been exposed to tritium showed that there were two set of tubes with different yield strength (40.5 ksi and 28.5 ksi). This did not affect the results with tritium. Tritium in the metal was removed from the samples before testing.

The results of these tests demonstrated that with increasing exposure to tritium (time of exposure varied from 56 days to 450 days);

- The 0.2% yield strength and the tensile strength of the palladium alloy tubes increased (maximum value 74 ksi and 82 ksi respectively).
- The ductility decreased by a factor of 2.3 from the value measured for no tritium exposure.
- The ductility was acceptable after 450 days of exposure to tritium.
- No helium bubbles were detected in the matrix or at the grain boundaries.
- Samples which contained residual tritium during the tensile tests exhibited a very low ductility.
- The hardness (diamond pyramid number) increased with exposure to tritium.

The above effects may be attributed to the buildup of helium-3 atoms in the palladium alloy metal from the decay of tritium. Although there was a loss of ductility, the measured value is still acceptable for operation of a diffuser under these conditions for periods up to one year.

ZrCo Tritium storage bed

Zirconium-cobalt has been suggested as one of the alternative getter materials for recovering, storing and supplying gaseous tritium in fusion reactor systems. This material has the advantage of being a nonnuclear fuel and thus is more easily available than uranium. Initial testing with hydrogen at JAERI have demonstrated that when hydrided it will not burn when exposed to air, as activated uranium will.

Two ZrCo beds were supplied to TSTA for use in the diffuser and CEC experiments. The beds are vacuum insulated and contain approximately 35 grams of ZrCo. They have been used to supply tritium to both experiments. Three thousand curies has been loaded to and unloaded from the bed in the diffuser experiment several times. Since the pressure-composition isotherms for the T_2-ZrCo system do not exhibit the rectangular shape as in uranium but rather are sloped, a transfer pump must be used to recover tritium from the bed. The beds operate well and further tests at TSTA to measure the properties with tritium are planned.

Conclusions and Future Plans

The tritium tests of the ceramic electrolysis cells and the palladium alloy diffusers at TSTA have demonstrated that these systems are possible components in a fuel cleanup system for a fusion reactor. Testing of the model 2 CEC will continue to measure the effects of impurities (CO_2 and CH_4) on the cell performance, cell efficiency for water decomposition and effect of long term operation. Continuing diffuser tests will measure wall permeation under various conditions, impurity separation characteristics, surface poisoning effects, long term operation, and mechanical tensile properties after completion of loop testing.

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