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TITLE: FUSION REACTOR HIGH VACUUM PUMPING: CHARCOAL CRYOSORBER
TRITIUM EXPOSURE RESULTS

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Abstract

Recent experiments (1) (2), have shown the practicality of using activated charcoal (coconut charcoal) at 4°K to pump helium and hydrogen isotopes for a fusion reactor. Both speed and capacity for deuterium/helium and tritium/helium-3 mixtures were shown to be satisfactory.

The long term effects of tritium on the charcoal/cement system developed by Grumman and LLNL were not known and a program was undertaken to see what, if any, effect long term tritium exposure has on the cryosorber. Several charcoal on aluminum test samples were subjected to six months exposure of tritium at approximately 77°K. The tritium was scanned several times with a residual gas analyzer and the speed-capacity performance of the samples was measured before, approximately half way through, and after the exposure. Modest effects were noted which would not seriously restrict charcoal's use as a cryosorber for fusion reactor high vacuum pumping applications.

Introduction

The Tritium Systems Test Assembly (TSTA) at Los Alamos, N.M. has been the site for a number of experiments which relate to high vacuum pumping of an expected fusion reactor exhaust (4). Compound cryopumps were shown to be unnecessary in tests which demonstrated co-pumping of hydrogen isotopes and helium on a single, 4K, activated charcoal panel. Tritium and helium-3 were pumped in one of these tests but long duration operation was not practical in the large TSTA cryopumps. Instead, two small chambers were procured and installed in a glovebox at TSTA. In one of the chambers tritium can be maintained at desired pressure, temperature levels for long durations. The second chamber has a liquid helium cooled specimen mounting surface where helium pumping speed and capacity measurements may be made. The goal of the test program which utilized these chambers was to assess whether tritium would affect the speed/quantity characteristics of the charcoal or whether interactions between the tritium and charcoal would result in objectional by-products such as methane.

Test Apparatus

Two chambers were procured and added to a glovebox in which several devices necessary to the present program were operational from another experiment. These included metal getter beds for storage and generation of tritium, a residual gas analyzer, a turbopump connected to the facility's clean vacuum system, a Metal Bellows pump, instrumentation and various facility support items. Figure one is a schematic of the environmental chamber and the relevant piping and components used as support

equipment. Approximately 2500 Ci of tritium was stored on the zirconium-cobalt metal getter bed for the experiment. The chamber was evacuated through the turbopump, VP .01 to the facility clean vacuum system and then to the Tritium Waste Treatment, TWT, system. To load the environment chamber, the zirconium cobalt getter bed was heated until the desired tritium pressure was achieved. At the end of the exposure, tritium was circulated through the getter bed by metal bellows pump CP-01 in a loop which included the chamber. At low pressures helium was added to serve as a carrier gas. The progress in reducing tritium content to trace amounts was monitored with the residual gas analyzer, P1-05 which was also used to analyze tritium in the chamber at the beginning of and conclusion of the exposure.

The second chamber was not connected directly to the tritium plumbing described above but was located alongside the environmental chamber to facilitate transfer of the specimens. The chamber is shown schematically in figure 2; its purpose is to measure pumping speed and capacity of the specimens for helium. The specimen is mounted on the lower surface of a liquid helium reservoir. Attachment is made with a clamping ring which presses the outer 3mm specimen circumference to the lower surface. A center stud applies pressure in that area. Diodes for temperature measurement are located in washers which are placed beneath the center nut and one of the screws which hold the clamping ring. Surrounding the liquid helium reservoir is a liquid nitrogen cooled heat shield in the form of a closed ends cylinder. The head shield is split into upper and lower sections for easy access to the specimen. The lower portion of the chamber can be lowered by a screw jack arrangement without dismantling cryogenic feed or vent lines.

A mass flow controller with a range of 0-10 sccm is used to control the flow of test helium gas to the chamber formed by the specimen surface and the heat shield. Pressure in the volume is sensed by a Bayard Alpert ionization gauge.

Test Description

Methane generation has been observed in stainless steel containers used to store tritium. The empty environmental chamber was pressurized with tritium to 25 torr at near liquid nitrogen temperature ($\cong -175^{\circ}\text{C}$) for five weeks and the gas was analyzed with the residual gas analyzer at the beginning and end of the exposure. This served as a baseline for methane generation later when the specimens were installed.

Several specimens were prepared of activated charcoal bonded with an inorganic cement to a 3mm thick aluminum substrate according to procedures described in (3). The substrates were 108 mm (4.25 in.) diameter discs with an active surface area of 71 cm². Two specimens were exposed to tritium during the program. The third served as a control

and was tested at the same time as the other specimens but was only exposed to glovebox tritium levels of several m Ci/m³ during transfer to and from the measuring chamber. Prior to tritium exposure all three specimens were tested for helium cryosorbing capability in the liquid helium cooled cryostat.

Poor results were obtained with specimen #1 which were attributed to insufficient thermal contact between the liquid helium reservoir and the specimen substrate. Gold foil, 4 mils thick, was used as a thermal interface material and generally gave repeatable results except in this case. All speed tests were done with a feed rate of 1 SCCM helium gas. Indicated pressure was increased by a factor of seven according to manufacturer's recommendations for the ion gauge sensitivity for helium. All speed tests were preceded by a bakeout to 100°C with the turbopump maintaining vacuum. During liquid helium fill, the evacuation valve was closed as soon as a decrease in pressure was noted, usually around 100 K. Specimen hold-down screws were torqued during installation in an attempt to make the installation repeatable.

Two specimens were transferred to the environmental chamber and tritium was transferred to the chamber by heating the zirconium - cobalt getter bed until the desired pressure of 100 torr was achieved. The gas was sampled by the residual gas analyzer and the chamber was cooled with liquid nitrogen to an indicated temperature of -175°C. These conditions were maintained for 61 days and equal 3.7×10^4 torr-hours.

Tritium was sampled after warming the environmental chamber to room temperature, then transferred to the zirconium-cobalt getter bed. Helium was added as a carrier gas to permit circulation of the tritium through the getter bed. The added pressure of helium was 50 torr. When gas analysis showed the tritium content to be low, the helium was pumped away to the tritium clean up system. The chamber was pressurized with helium and the specimens removed and transferred to the cryostat for speed/loading tests.

The specimens were again mounted in the environmental chamber, tritium from the getter bed was sampled by the analyzer after the desired pressure was achieved. The chamber was chilled with liquid nitrogen and conditions of approximately 25 torr tritium pressure at an indicated temperature of -175°C were maintained for 119 days. In pressure duration terms this exposure equals 7.1×10^4 torr hours.

Tritium was analyzed, transferred to the getter bed and all specimens were tested for speed vs. quantity while pumping helium.

Results

• Tritium Effect on Pumping Speed

The specimens showed no degradation of pumping speed in the first exposure of 3.7×10^4 torr-hours and a marked loss in speed and capacity which occurred between 3.7×10^4 torr-hours and 10.8×10^4 torr-hours. Exactly why the pumping performance decreased is not known. The bond between the inorganic cement and the charcoal may have been weakened so that the amount of charcoal lost during specimen handling had an effect or the tritium exposure may have changed the charcoal. The pumping speed curves for the three specimens are shown in figures 3-5. Pre-exposure results for specimen #1 were not consistent with other results and are not presented.

• Charcoal Effect on Tritium

Gas analysis of the tritium after each of the three exposures; empty chamber, first exposure of 61 days and second exposure of 119 days, showed the percent of gases in the amu range from 18-24 to be 7-9% of the total. Any contribution by the charcoal specimens was not significant enough to be measured in the present apparatus. Gases in the 18-24 amu range may be water, tritiated water or tritiated methane. In the range observed, 10 to 40 amu, no other peaks were noted except 1-6 which may be combinations of the hydrogen isotopes and 28 which can be accounted for by the nitrogen atmosphere in the glovebox.

• Visual Examination

Charcoal granules are loosely bound to the cement which holds them to the substrate so there is considerable shedding of charcoal each time the specimen is handled. As the specimens were transferred within the glovebox, using thick gloves and working in tight quarters, the shedding experienced during this program is more severe than that to be expected in the normal application of this material. At the conclusion of the present program the specimens were noticeably brighter in appearance with much more of the substrate visible.

• Tritium Retention

It was not anticipated that tritium retention would be a problem with activated charcoal so no plans to measure tritium retention were included in this program. During the last specimen transfers from the environmental chamber to the cryostat, glovebox levels of several m Ci/m³ were noted. These levels are believed due to the specimens and it will be attempted to measure tritium off gassing from charcoal in the future.

• Conclusions

Both charcoal specimens performed normally after a 3.7×10^4 torr hour exposure to tritium at near liquid nitrogen temperature. No measurable effects on the tritium which

could be attributed to the charcoal were noted. Considering that charcoal is exposed to higher tritium pressure only during regeneration, it should be possible to design a cryopump system capable of years of operation without tritium degradation effects. Very long exposures on the order of 10^5 torr-hours may produce reduced pumping performance on the panels.

References

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FIGURE 1 SCHEMATIC OF ENVIRONMENTAL CHAMBER AND RELATED PIPING

FIGURE 2 CRYOSTAT FOR
MEASURING CRYOSORBER
PUMPING SPEED

SPECIMEN #1

SPEED VS QUANTITY

AFTER 2 MOS TRITIUM EXPOSURE

○ ~ 3/13/91

AFTER 6 MOS TRITIUM EXPOSURE

□ ~ 7/16/91

SPEED VS QUANTITY
CHARCOAL CRYLSORBER #2
PUMPING HELIUM

CHARCOAL CRYOSORBER #3 (NOT EXPOSED)
PUMPING HELIUM

TRITIUM WITHOUT SPECIMENS

AS LOADED

6-29-90

APPROX 56 TORR AT ROOM TEMP.

AFTER 5 WEEKS

8-6-90

FIGURE 5 - TRITIUM ANALYSIS BEFORE AND AFTER EMPTY CHAMBER EXPOSURE

FIRST TRITIUM LOADING WITH (2)
1/2/91 CHARCOAL SPECIMENS

ANALYSIS AFTER 2 MONTHS
3/4/91

FIG. 7. TRITIUM ANALYSIS BEFORE AND AFTER
61 DAY EXPOSURE WITH SPECIMENS
1 & 2 INSTALLED AND TRITIUM PRESSURE
OF 25 TORR, TEMPERATURE -175°C

FIGURE 8. TRITIUM ANALYSIS BEFORE AND AFTER SECOND EXPOSURE OF 119 DAYS, 25 TORR, -175°C