AIM

Association for Information and Image Management

1100 Wayne Avenue, Suite 1100
Silver Spring, Maryland 20910
301/587-8202

Centimeter

Inches

MANUFACTURED TO AIM STANDARDS
BY APPLIED IMAGE, INC.

1 of 1

GA-A21329

TRITIUM IN THE DIII-D CARBON TILES

by
P.L. TAYLOR, A.G. KELLMAN, and R.L. LEE

This is a preprint of a paper to be presented at the International Atomic Energy Agency Technical Meeting on Development in Fusion Safety, June 7-11, 1993, Toronto, Canada and to be printed in the *Proceedings*.

Work supported by
U.S. Department of Energy
Contract DE-AC03-89ER51114

GENERAL ATOMICS PROJECT 3466
JUNE 1993

GENERAL ATOMICS

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

for

ABSTRACT

The amount of tritium in the carbon tiles used as a first wall in the DIII-D tokamak was measured recently when the tiles were removed and cleaned. The measurements were made as part of the task of developing the appropriate safety procedures for processing of the tiles. The surface tritium concentration on the carbon tiles was surveyed and the total tritium released from tile samples was measured in test bakes. The total tritium in all the carbon tiles at the time the tiles were removed for cleaning is estimated to be 15 mCi and the fraction of tritium retained in the tiles from DIII-D operations has a lower bound of 10%. The tritium was found to be concentrated in a narrow surface layer on the plasma facing side of the tile, was fully released when baked to 1000°C, and was released in the form of tritiated gas (DT) as opposed to tritiated water (DTO) when baked.

I. INTRODUCTION

The DIII-D research tokamak has been operating for approximately the last four years with deuterium as the working gas.⁽¹⁾ Deuterium operation generates a significant number of neutrons and a small amount of tritium; less than 0.3 Ci of tritium is produced per year.

The retention of tritium in graphite is of interest due to the tritium inventory monitoring requirements for future fusion devices where graphite is a candidate for the first wall. Tritium can be retained in graphite by four means:⁽²⁾ saturation of the implant area when energetic tritium atoms or ions impact the surface; co-deposition with carbon on surfaces when carbon atoms are sputtered from the graphite surface by plasma bombardment; absorption of gas on internal porosity; and transgranular diffusion with trapping. DIII-D uses only deuterium as a working gas and only a small amount of gas is pulsed into the machine for each shot. The sole source of tritium is the 1 MeV tritons produced by the deuterium-deuterium (D-D) fusion reactions in the plasma. Thus the first two tritium retention methods can be important mechanisms for depositing tritium in DIII-D.

During a vent starting in August 1992 all the carbon tiles used as a first wall inside the DIII-D tokamak were removed for cleaning prior to reinstallation, and the carbon tile coverage was increased from approximately 45% to 100% of the first wall. The remainder of the first wall before the vent consisted of the Inconel vessel wall and 113 Inconel tiles. Approximately 1544 carbon tiles out of a total of 1857 tiles were cleaned and reinstalled. The cleaning procedure consisted of removing 20 to 40 microns from all the tile surfaces by grit blasting with a boron-carbide grit to remove any metal deposits. The tiles were then rinsed in an ultrasonic alcohol bath and baked in a vacuum oven to 1000°C. Following the conclusion of plasma operations in

June, the vessel was initially baked to 350°C, vented to air for 10 days, and then pumped down and kept under vacuum until the August vent when the tiles were removed.

To determine the appropriate safety procedures for processing the tiles, a sample set of tiles (10% of the total) were tested for radioactivity immediately after removal from the machine. The tiles were found to have no neutron activation above background. Surface contamination wipes for removable alpha and removable beta showed only background levels. However, wipe samples for removable tritium contamination showed levels of up to 3400 DPM/100 cm². The applicable limits for tritium surface contamination below which items are releasable are the State of California limit of 4000 DPM/100 cm² and the U.S. Department of Energy (DOE) limit of 1000 DPM/100 cm². While most of the tiles were within the releasable surface contamination limits, the relation between the surface contamination and tritium retained in the bulk which might be released upon baking was unknown and required further measurements to determine the amount of tritium that would be released. Measurements were then made of the amount of tritium released during baking of small sections cut from the tiles. From this data the total tritium retained in the tiles was estimated. To determine the safe handling procedures required for the grit blasted and alcohol cleaned tiles the total tritium in the tiles after grit blasting and alcohol cleaning was also estimated. This estimate was made from measurements of the total tritium released in bakes of small sections of grit blasted tiles and in a bake of 24 grit blasted and alcohol cleaned tiles in a vacuum oven.

II. DIII-D CARBON TILES AND TRITIUM ANALYSIS

The DIII-D carbon tiles are made of Union Carbide ATJ graphite. While there is a large variety of different carbon tile sizes and shapes used in the tokamak a typical tile and the total carbon in DIII-D is described in Table I. The inner wall, floor, and ceiling had complete coverage with tiles while only selected regions of the outer Inconel vessel wall were tiled (Fig. 1).

TABLE I. Typical graphite tile and total graphite in DIII-D before the tile cleaning

Mass of average tile	~1.0 kg
Average plasma facing surface area	~187 cm ²
Total number of carbon tiles	~1857
Total mass of carbon	~1900 kg
Plasma facing surface area of carbon tiles on inner wall, ceiling, and floor	~27 m ²
Total plasma facing surface area of all carbon tiles (inner wall, ceiling, floor, port protection, outer wall, ADP ring, and limiters)	~34 m ²
Total surface area of graphite (plasma facing and non plasma facing)	~100 m ²

Three interesting features are seen in the poloidal distribution of the tritium surface contamination shown in Fig. 2. The highest levels are found on the limiter, the ring, and the lower inner corner tiles. The limiter and ring contamination may be large due to the fast tritons that are lost before they are thermalized.⁽³⁾ These fast ion losses have orbits that spiral outward and can intersect the ring and limiter. The tritium peak near the lower 45° tile corresponds with the position of a co-deposited layer of eroded material. A previously made measurement (Fig. 3) of the surface profile change in the carbon tiles on the vessel floor after a period of nine months of operation shows an erosion of floor material on the outboard side and a net deposition at the

FIG. 1. Location of the graphite tiles in the DIII-D tokamak with tile row number given. Full toroidal coverage is provided by the all the tiles shown except the limiter which is located at one toroidal angle. Shown for reference are the magnetic flux surfaces for a typical shot.

FIG. 2. Removable tritium surface contamination of the graphite tiles by poloidal position in the tokamak. Each measurement represents a toroidal average. Note the tile positions as shown in Fig. 1.

FIG. 3. Net change in the surface profile of the divertor floor from measurements before and after 9 months of plasma operation.

lower inside corner, which is the location of the inner divertor region.⁽⁴⁾ This occurs due to plasma bombarding the floor and sputtering the carbon and other material; tritium is then co-deposited with the carbon.

Tables II through V list the steps through which sample pieces of various tiles were processed in order to determine the amount of tritium they contained and where the tritium was located. The amount of tritium in these samples was measured by baking the tiles in a flowing gas mixture (~30 ml/min) of 4% H₂, 96% He, where the hydrogen acts as a catalyst to help exhaust the tritium. The exhaust gas passes first through a hot copper oxide bed that converts any

TABLE II. Characteristic data from processing a tile sample from the plasma facing surface of a lower inner divertor tile

Initial tritium on plasma facing surface	1970 DPM/100 cm ²
Grit blasted, removing	20 to 40 microns
Tritium on surface after grit blasting	80 DPM/100 cm ²
Mass, surface area of sample from plasma facing surface	92 g, 21 cm ²
Baked for 7 hr at 1000°C — tritium released	0.110 µCi
Burned in O ₂ for 8 days at 1000°C — tritium released	0.0085 µCi

TABLE III. Characteristic data from processing a tile sample from the plasma facing surface of a lower inner divertor tile

Initial tritium on plasma facing surface	549 DPM/100 cm ²
Not grit blasted	
Mass, surface area of sample from plasma facing surface	55 g, 28 cm ²
Baked for 7 hr at 350°C — tritium released	0.38 µCi
Baked for 7 hr at 1000°C — additional tritium released	0.85 µCi
Total tritium released	1.23 µCi
The amount of tritium released after 5 hr in each bake:	
% of 350°C total released after 5 hr	68%
% of 1000°C total released after 5 hr	97%

TABLE IV. Characteristic data from processing a tile sample from the plasma facing surface of an inner wall tile

Initial tritium on surface	200 DPM/100 cm ²
Tile cut in half perpendicular to plasma facing surface	
Half 1	
mass, surface area	34.6 g, 9.38 cm ²
baked for 7 hr at 1000°C — tritium released	0.37 µCi
Half 2	
mass, surface area surface area	30.3 g, 9.38 cm ²
Grit blasted, removing	~13–20 microns
Tritium on surface (~ background)	50 DPM/100 cm ²
Baked for 7 hr at 1000°C — tritium released	0.017 µCi

tritiated gas or tritiated hydrocarbons to tritiated water which is then collected in a bubbler using Ultima Gold as the fluid which is analyzed after the bake to measure the total tritium released.

A 1000°C bake for 7 hours releases 93% of the tritium in the graphite tile; the remaining tritium was measured by burning the tile after the bake (Table II). Baking at 350°C for 7 hours releases only 31% of the tritium and the tritium is released at a slower rate than when baked at 1000°C (Table III). Comparing the amount of tritium released from part of a tile surface that was grit blasted with a part not grit blasted shows that 95% of the tritium is in a thin layer (<20 microns) on the surface facing the plasma (Table IV). Similar size samples from tiles that had no line of sight to the plasma (Table V) when compared to these samples (Tables II, III, & IV) also confirm that the majority of the tritium is in a narrow layer on the plasma facing side of the tile.

The tritium surface contamination from wipe measurements does not adequately represent the total tritium in the tile which can be released upon baking as can be seen by examining the ratio of the amount of tritium released in the above samples to the amount calculated assuming

TABLE V. Characteristic data from processing tile samples with no line of sight to the plasma

Center post tile mounting foot	
Not grit blasted	
Mass	37.3 g
Baked for 7 hr at 1000°C --- tritium released	0.0019 μ Ci
Lower inner divertor region tile foot	
Not grit blasted	
Mass	44.6 g
Baked for 7 hr at 1000°C — tritium released	0.019 μ Ci

the tritium was all on the surface and given by the wipe measurement. This ratio is not proportional to the surface contamination and varies widely. The ratio is: 2×10^4 for a tile with no grit blasting and a surface contamination of 549 DPM/100 cm²; 6×10^4 for a tile with no grit blasting and a surface contamination of 200 DPM/100 cm²; and infinite for a tile after grit blasting whose surface contamination was reduced to background.

As a further check on the small test sample bakes a test vacuum bake of 24 grit blasted and alcohol cleaned tiles was performed at 1000°C and the total tritium released was measured including the tritium trapped in the diffusion pump and roughing pump oil and the tritium in the pump exhaust. The 24 tiles, which were grit blasted and alcohol cleaned, had removable tritium surface contamination levels before the bake ranging up to 800 DPM/100 cm². Tritium wipes of the inside of the oven before and after the bake showed a slight increase on the cold outer wall of the oven to approximately twice background. Tritium oil sample analysis of the pumping oils before and after the bake showed no contamination of the diffusion pump oil and some contamination of the roughing pump oil; a total of 0.22 μ Ci was trapped in the pump oils. (Note that in another larger oven where the majority of the tiles were later baked, the diffusion pump oil had a tritium contamination of 2.3 μ Ci, a factor of 10 higher than the roughing pump oil contamination of 0.26 μ Ci). The exhaust gas from the roughing pump was passed through a six-stage tritium collection bubbler where tritium in the form of DTO was trapped in the first three

stages and tritium in the form of DT was trapped in the last three stages. The total tritium exhausted included 0.004 μCi of DTO and 11.4 μCi of DT. Thus a total of 11.4 μCi of tritium passed through the pumps and a total of 0.22 μCi of tritium was trapped in the pump oil in this test bake.

III. ESTIMATES FOR THE TOTAL TRITIUM IN THE TILES

An estimate was made of the total tritium in all of the carbon tiles before any cleaning was done from which the tritium retention was derived, and for purposes of determining the safe handling procedures an estimate of the total tritium after the tiles were cleaned (but not baked) was also made (Table VI). The lowest estimate (#1) of 0.023 μCi was obtained by assuming that the tritium is given by the removable contamination, and a removable contamination level that was more than twice the largest measured was used. Estimate #2 scales the amount released in a bake of two non-grit blasted tiles (one that had a high initial surface contamination, and one that had a low surface contamination), and estimated total values are given for two assumptions: that the tritium was uniform though the volume of the graphite, or that the tritium was in a surface layer. The best estimate for the total tritium in all the tiles uses the conclusions of the previous section, that the tritium is in a surface layer and can not be represented by the surface contamination measurements. Thus the best estimate, obtained by using the value from the non grit blasted tiles (estimate #2) with the assumption that the tritium is concentrated in the surface layer, is that a total of 13 to 15 mCi of tritium was retained in the tiles from operation of DIII-D.

For purposes of determining the safe handling procedures of the grit blasted tiles estimates #3 and #4 were made. Estimate #3 scales to all the tiles the amount of tritium measured in two grit blasted tiles, while estimate #4 scales the amount released in the test bake of 24 grit blasted and alcohol cleaned tiles to all the tiles. Notice the good agreement of this 24 tile test bake result (0.87 mCi) with the values (0.62 and 1.8 mCi) obtained from the small sample measurements in estimate #3 with the surface layer assumption.

TABLE VI. Estimates of the total tritium in all the tiles

Estimate	Method Assumed	Total Tritium in All the Tiles (mCi)	
Before Tile Cleaning			
1	Tritium on surface only, with a surface contamination of 5000 DPM/100 cm ²	0.023	
2	All the tiles have the amount released in a bake of a non-grit blasted tile Tritium assumed to be uniform through volume Tritium assumed to be in surface layer	With surface contamination	
		Low	High
		20.0 13.0	43.0 15.0
After Tile Cleaning			
3	All the tiles have the amount released in a bake of a grit blasted tile Tritium assumed to be uniform through volume Tritium assumed to be in surface layer	With surface contamination	
		Low	High
		1.1 0.62	2.3 1.8
4	All the tiles have the amount proportional to that released in a test vacuum bake of 24 tiles that were grit blasted and alcohol cleaned	0.87	

The total tritium produced in DIII-D by calendar quarter is shown in Fig. 4. Tritium which is initially retained in the graphite can be released by future plasma shots that provide a high heat flux to the graphite surface and that churn the carbon surface and co-deposited layer, by vessel baking sessions, and by vents. The vessel is baked to 350°C frequently which as seen in Section II is sufficient to drive out some of the tritium. Over the last four years the vessel has been baked an average of 85 hours per quarter to 350°C. Tritium is also released from the graphite when it is exposed to air.⁽⁵⁾ From 1990 until the tiles were removed the vessel was vented to air for a total of 265 days. Using the best estimate of 15 mCi as the amount of tritium

FIG. 4. Tritium produced in DIII-D by calendar year quarter and the fraction of the total tritium from the given quarter to the end of 1992 that is retained.

in the tiles the fraction of the total tritium generated from the given calendar quarter to the second quarter of 1992 that is retained is also plotted in Fig 4. If the 15 mCi of tritium remaining at end of operations in 1992 was assumed to have been generated only in 1991 and 1992, i.e. the tritium generated prior to 1991 was removed by baking and vents, then the fraction retained in the tiles would be 20%. Including all the tritium generated since 1989 approximately 10% was retained in the graphite tiles.

IV. SUMMARY

The tritium contamination of the DIII-D carbon tiles was measured during the cleaning of the tiles. The tritium was found to be in a thin surface layer (<40 microns) facing the plasma. The highest surface concentration of tritium other than in the limiter was in the co-deposited layer on the divertor floor. Most of the tritium in the tiles is released when the tiles are baked to 1000°C. The tritium surface contamination measurements from wipes can not be used to infer the total tritium in the tile. A total of 15 mCi of tritium was estimated to be in all the tiles and approximately 10% of all the tritium generated during DIII-D operations has been retained in the tiles.

REFERENCES

- ¹T. C. Simonen, J. Fusion Energy **11**, 79 (1992).
- ²R. A. Causey, J. Nucl. Mater. **162-164** , 151 (1989).
- ³H. H. Duong and W. W. Heidbrink, Nucl. Fusion **33** , 211 (1993).
- ⁴D. N. Hill, et al., in Proceedings of the 13 International Conference on Plasma Physics and Controlled Nuclear Fusion, Vol. III, Washington, D.C., 487 (1990).
- ⁵J.P. Coad, et al., J. Nucl. Mater. **162-164**, 533 (1989).

ACKNOWLEDGMENT

This work was supported by the U.S. Department of Energy under Contract No. DE-AC03-89ER51114. The authors wish to thank A. Greenwood and G. Lain for their tritium analysis work and Dr. D. Hill for Fig. 3.

**DATE
FILMED**

9 / 10 / 93

END