

## **PERFORMANCE OF V-4Cr-4Ti MATERIAL EXPOSED TO DIII-D TOKAMAK ENVIRONMENT -- H. Tsai, H. M. Chung, and D. L. Smith (Argonne National Laboratory), W. R. Johnson and J. P. Smith (General Atomics)**

### **SUMMARY**

Test specimens made with the 832665 heat of V-4Cr-4Ti alloy were exposed in the DIII-D tokamak environment to support the installation of components made of a V-4Cr-4Ti alloy in the radiative divertor of the DIII-D. Some of the tests were conducted with the Divertor Materials Evaluation System (DiMES) to study the short-term effects of postvent bakeout, when concentrations of gaseous impurities in the DIII-D chamber are the highest. Other specimens were mounted next to the chamber wall behind the divertor baffle plate, to study the effects of longer-term exposures. By design, none of the specimens directly interacted with the plasma. Preliminary results from testing the exposed specimens indicate only minor degradation of mechanical properties. Additional testing and microstructural characterization are in progress.

### **OBJECTIVE**

In service, V-4Cr-4Ti divertor components in the DIII-D vessel will be exposed to a range of temperature and impurity conditions and to alternating vacuum/low-pressure hydrogen plasma operation, thermal cycles, periodic bakeout, glow-discharge cleaning with helium, periodic boron coating, and occasional re-exposure to air during vents for maintenance. The objective of this task is to determine the effects of these environmental exposures on the performance of the vanadium alloy material. It is particularly important to determine whether interstitial impurities are absorbed sufficiently to lead to material embrittlement.

### **TEST RESULTS**

To date, three experiments (D1, D2, and W1) have been completed. Another experiment, W2, is still ongoing. The D1 and D2 experiments were conducted with the DiMES,<sup>1</sup> a device located below the vessel floor that, when extended, allows the specimen mounted on it to be inserted into the plasma chamber without disturbing the system vacuum. The specimens for experiments W1 and W2, on the other hand, were mounted on a bracket next to the vessel wall behind the divertor baffle plate (i.e., nonplasma-facing). Because loading and unloading the specimens require a manned entry, which is possible only during an air vent, their exposures encompass full DIII-D operating cycles.

#### **D1 Experiment**

The specimen for the D1 experiment, a polished disk 33.0 mm in diam. and 3.8 mm thick, was exposed to the vessel atmosphere during bakeouts on April 1, 9, 15, and 16, 1995, following air (dirty) vents on March 28 and April 5, 1995 and a nitrogen (clean) vent on April 11, 1995. The exposure conditions, as determined by infrared camera and residual gas analyzer (RGA), are summarized in Table 1. In each of the four bakeouts, the duration at temperature was  $\approx$ 5-10 h. Temperatures measured by the infrared camera tracked with the average vessel temperature (the mean of the inner vessel and outer vessel wall temperatures). During the experiment, the sample surface was recessed  $\approx$ 3.8 mm below the DIII-D graphite tile floor.

The exposed area of the disk exhibited a slight loss of luster (dulling). Surface hydrogen analyses were conducted by following an elastic recoil diffraction technique with a 2.6-

MeV  $^4\text{He}$  beam for both the front (i.e., exposed) and back sides of the disk. The measurements showed pronounced surface peaks of hydrogen:  $\approx 25,000$  appm for the front and  $\approx 65,000$  appm for the back. The concentration profiles for both sides, however, decreased rapidly with depth within the first  $\approx 0.1\mu\text{m}$ . At a depth of  $\approx 0.45\mu\text{m}$ , the approximate limit of beam penetration, the concentration profiles appear to have reached an asymptote of  $\approx 7,000$  appm for both sides. The fact that hydrogen concentration is greater on the back side suggests that the high surface readings may be a storage or handling artifact unrelated to the actual DIII-D exposure. Additional analyses, including bulk hydrogen analysis (by outgassing) and microhardness measurements, are planned to evaluate the extent and effect of bulk hydrogen uptake. Also to be conducted are Auger microprobe analyses for oxygen, carbon, and boron impurities to a depth of  $\approx 5\mu\text{m}$  for comparison with nonexposed sibling data.

Table 1. Partial pressure of gaseous impurities during the bakeouts in the D1 experiment. All values are peak readings.

Bakeout Date	Temp. ( $^{\circ}\text{C}$ )	Pressure (torr)	Partial Pressure ( $10^{-9}$ atm)			
			$\text{H}_2\text{O}$	$\text{N}_2$	CO	$\text{O}_2$
4/1/95	250	$4.6 \times 10^{-4}$	490	0.016	80	3.1
4/9/95	150	$1.1 \times 10^{-3}$	1400	0.018	30	3.5
4/15/95	325	$2.6 \times 10^{-4}$	240	0.013	64	2.6
4/16/95	325	$6.2 \times 10^{-5}$	67	0.003	11	0.63

## D2 Experiment

In the D2 experiment, five miniature Charpy impact specimens were exposed to the DIII-D tokamak environment with the DiMES during a single bakeout that followed a clean vent on June 6, 1995. The vent, for diagnostics repairs, lasted 35 days. The exposure conditions, as determined from measured inner and outer vessel wall temperatures and RGA measurements, are summarized in Table 2. The specimens were 1/3-size ( $3.3 \times 3.3 \times 25.4$  mm) and contained a 0.6 mm-deep,  $30^{\circ}$  blunt notch with a root radius of 0.08 mm. The crack plane direction was perpendicular to the rolling direction and through the thickness of the plate from which the specimens were prepared. After machining, the specimens were vacuum-annealed at  $1000^{\circ}\text{C}$  for 1.0 h in a pure Ti foil wrap, which acted as an impurity getter. The vacuum, produced by a diffusion pump with a cold trap, was  $\approx 10^{-6}$  torr during the annealing.

Table 2. Partial pressure of gaseous impurities during the bakeout in the D2 experiment. All values are peak readings.

Bakeout Date	Temp. ( $^{\circ}\text{C}$ )	Pressure (torr)	Partial Pressure ( $10^{-9}$ atm)			
			$\text{H}_2\text{O}$	$\text{N}_2$	CO	$\text{O}_2$
7/13/95	250	$5.5 \times 10^{-5}$	31	0.33	2.4	0.08

Four of the specimens were impact-tested, one each at  $-190$ ,  $-150$ ,  $23$  and  $150^{\circ}\text{C}$ . Preliminary results, shown in Fig. 1, reveal that the exposed specimens were ductile over

the entire test temperature range and that the ductile-brittle transition temperature (DBTT) was below the lowest test temperature, which was  $-190^{\circ}\text{C}$ . In the lower temperature range, the measured upper-shelf energies agree closely with available nonexposed sibling data, also shown Fig. 1. (The sibling data were obtained with specimens prepared and annealed under conditions that were identical as to those used for the test specimens.) In the above-room-temperature range, however, there appears to be a decrease in the upper-shelf energy of the test specimens. The cause of this decrease is currently not known, although data scatter cannot be ruled out. To better define the upper-shelf energy curves, additional tests with sibling specimens and with the remaining exposed specimen are planned during the next reporting period. Also to be completed are bulk hydrogen analysis and microstructural/chemical characterization of both the exposed and fracture surfaces.

### W1 Experiment

In the W1 experiment, five Charpy and five tensile specimens were exposed to the DIII-D environment for an entire DIII-D operation cycle, from February through December 1995. The configuration of the Charpy specimens was the same as the configuration of the specimens in the D2 test. The tensile specimens were 25.4 mm long and had a gauge section of 7.62 mm(length) x 1.52 mm(width) x 0.76 mm(thickness). The long direction of the gauge section was parallel to the final rolling direction of the plate from which the tensile specimens were machined. All specimens were annealed in an ion-pumped vacuum at  $1000^{\circ}\text{C}$  for 1.0 h prior to the test. For the exposure, the specimens were held in a frame mounted on the vessel wall behind the divertor baffle plate. The temperatures of the specimens were measured with four thermocouples attached to the ends of the specimens.

The specimens were installed in DIII-D after a dirty vent which was initiated on February 14, 1995, and were removed after a dirty vent on December 15, 1995. During this time period the specimens were exposed for a total of 265 days to a complete range of DIII-D operating conditions including dirty and clean vessel vents, elevated-temperature bakeouts, helium glow discharge cleanings, boronizations of the first wall (graphite tiles), and plasma operations. In their positions behind the DIII-D divertor, the specimens would be expected to have been most affected only by those DIII-D operations for which the specimens would have been heated to elevated temperatures and at which the greatest interactions with gaseous impurities in the DIII-D environment might be expected. These operations include the elevated-temperature bakeouts and boronizations. Based on the specimen thermocouple data, the temperatures of the specimens tracked with the measured outside vessel wall temperature, and reached values in the range of  $\sim 150$  to  $330^{\circ}\text{C}$  during bakeouts and  $\sim 250$  to  $280^{\circ}\text{C}$  during boronizations. Analysis of the RGA monitoring data obtained during the exposure period indicated that the specimens were exposed to impurities in the DIII-D environment which generally ranged, depending on the type of vent, between those indicated in Tables 1 and 2. A summary of the various vents and bakeouts experienced by the specimens is presented in Table 3.

Table 3. DIII-D environment history for the W1 experiment - February 14 to December 15, 1995.

Air Vents	N <sub>2</sub> Vents	150°C Bakeouts	200-250°C Bakeouts	280-350°C Bakeouts	280°C Boronizations
		4-6 h	2-7 h	5-20 h	5.5 h
5	5	2	3	20	2

Tensile tests have been conducted on two of the five specimens: one at room temperature in air and the other at 350°C in argon. Strain rate for both tests was  $1.1 \times 10^{-3}$ /s. The results, summarized in Table 4 along with the nonexposed sibling data, indicate that DIII-D exposure produced no appreciable changes in either strength or ductility.

Four of the five Charpy specimens have been tested, one each at -190, -150, 23, and 150°C. The results are shown in Fig. 2, along with sibling data obtained from specimens prepared and annealed under identical conditions.<sup>2</sup> All specimens were ductile over the entire temperature range. But again, their upper-shelf energies seem to be lower than those of the nonexposed siblings.

Table 4. Tensile properties of specimens exposed in the W1 experiment in DIII-D (YS:0.2% offset yield stress; UTS: engineering ultimate tensile stress; UE: uniform elongation; TE: total elongation.)

Test Temp. (°C)	25°		350°	
	DIII-D	Control	DIII-D	Control
YS (Mpa)	334	357	421	205
UTS (Mpa)	449	428	377	359
UE (%)	19.0	19.1	14.8	17.6
TE (%)	27.0	29.2	22.0	25.4

## FUTURE ACTIVITIES

Additional testing and examination of the discharged specimens from the D1, D2, and W1 experiments will be completed to elucidate the possible effects of impurity uptake, particularly the effects of hydrogen. Testing will commence for the W2 specimens once the exposure is completed.

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## REFERENCES

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Fig. 1. Charpy impact data for D2 experiment specimens exposed in DIII-D, showing ductile behavior even at  $\approx -190^{\circ}\text{C}$ . Triangles denote nonexposed sibling data.

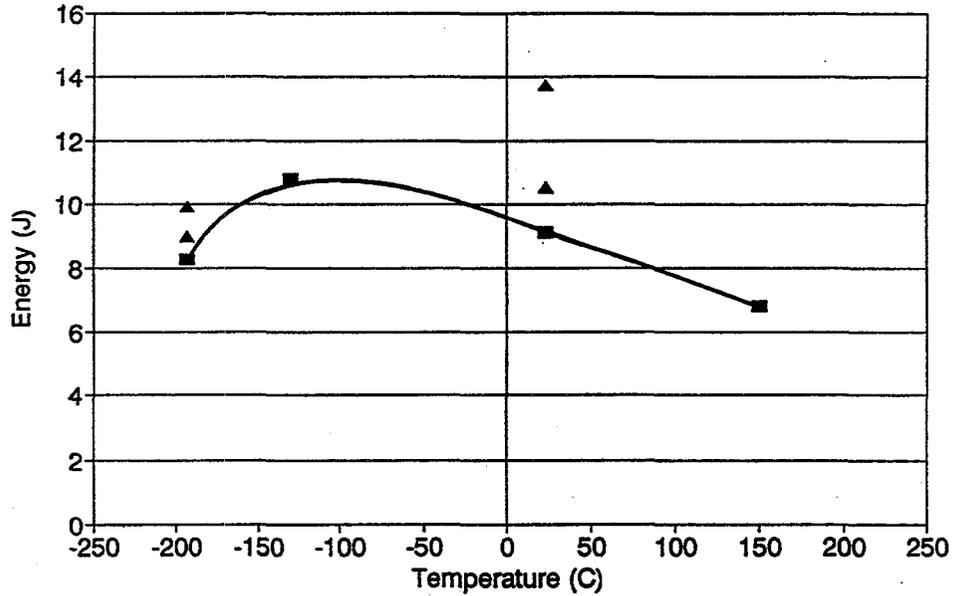


Fig. 2. Charpy impact data for W1 experiment specimens exposed in the DIII-D, showing ductile behavior even at  $\approx -190^{\circ}\text{C}$ . Shelf energies, however, are not as high as those of nonexposed siblings (triangles).

