# EFFECTS OF NEUTRON IRRADIATION AND HYDROGEN ON DUCTILE–BRITTLE TRANSITION TEMPERATURES OF V–Cr–Ti ALLOYS*

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**Abstract**

The effects of neutron irradiation and hydrogen on the ductile-brittle transition temperatures (DBTTs) of unalloyed vanadium and V-Cr-Ti alloys were determined from Charpy-impact tests on 1/3 ASTM standard size specimens and from impact tests on 3-mm diameter discs. The tests were conducted on specimens containing <30 appm hydrogen and 600-1200 appm hydrogen and on specimens after neutron irradiation to 28-46 dpa at 420, 520, and 600°C. The DBTTs were minimum (<-220°C) for V-(1-5)Ti alloys and for V-4Cr-4Ti alloy with <30 appm hydrogen. The effect of 600-1200 appm hydrogen in the specimens was to raise the DBTTs by 100-150°C. The DBTTs were minimum (<-220°C) for V-(1-5)Ti alloys and V-4Cr-4Ti alloys after neutron irradiation.

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## 1. Introduction

Vanadium-base alloys have significant advantages over other candidate alloys, e.g., austenitic and ferritic steels, for use as structural material in the International Thermonuclear Experimental Reactor (ITER) and in fusion reactors that may be constructed in the future [1]. These advantages include intrinsically lower levels of long-term neutron activation, neutron irradiation after-heat, neutron-induced helium and hydrogen transmutation rates, biological hazard potential, thermal-stress factor, high temperature strength, and resistance to irradiation damage [1-6]. At the present time, the V-4Cr-4Ti alloy is recommended for the near-optimum combination of these desirable neutronic, physical, and mechanical properties for a vanadium alloy structural material [1]. However, to make use of these favorable neutronic, physical, and mechanical properties in structural material in fusion systems, an alloy should have a low DBTT. For confirmation of the selection of the V-4Cr-4Ti alloy as prime-candidate vanadium alloy, we present in this paper the results of experimental investigations to determine the DBTTs (on impact loading) of vanadium alloys after neutron irradiation at 420-600°C to 21-41 atom diaplacements per atom (dpa).

## 2. Materials and procedures

Unalloyed vanadium and V-Cr-Ti alloys with the nominal compositions listed in Table 1 were obtained in sheet form with a 50% thickness-reduction resulting in a nominal

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thickness of 3.8 mm. Miniature Charpy-impact specimens (1/3 ASTM standard size) with overall dimensions of 3.30 x 3.30 x 25.4 mm and a notch depth of 0.61 mm were prepared from these materials. Discs with 3-mm diameter and 0.25-mm thickness, i.e., transmission electron microscopy (TEM) discs, were also prepared for impact tests on these materials.

Dehydrogenated Charpy and TEM impact-test specimens in the fully recrystallized condition were prepared from the 50% cold-worked materials by annealing for 1 h in a vacuum of $2 \times 10^{-5}$ Pa. The V and V-1Ti alloy specimens were annealed at 1025°C; and the V-3Ti-1Si, V-(3-20)Ti alloy specimens were annealed at 1100°C. The V-Cr-Ti alloys (Table 1) were annealed at 1125°C. These annealing conditions for the cold-worked materials resulted in an average recrystallized-grain diameter of 0.02-0.04 mm and a dislocation density of $\approx 10^{13}/m^2$ in the specimens. Hydrogen was introduced into Charpy specimens of the alloys by annealing the cold-worked specimens in argon-filled quartz tubes for 1 h and subsequently quenching the tubes and their contents, without rupture, in water. The hydrogen concentration in the specimens was adjusted by surface-finishing of the specimens in contact with water prior to the anneal.

The Charpy and TEM specimens were irradiated in $Li^7$-filled TZM molybdenum capsules at 420, 520, and 600°C in the Materials Open Test Assembly (MOTA) in the Fast Flux Test Facility (FFTF) during Cycles 9-11 of the reactor.

The Charpy-impact tests were performed with an instrumented Dynatup Drop-Weight Test machine. The impact velocity and load for these tests were 2.56 m/s and 14.995 kg, respectively. Energy absorption during impact was determined from applied load-time data that were acquired during the impact. The specimen temperature at the instant of impact was determined from a thermocouple arc-welded near the notch. The DBTT of an alloy was determined from the energy-absorbed versus test-temperature curve. In the case of impact tests on TEM discs, a specimen was mounted for impact by a swinging pendulum on 1/2 of the specimen. The temperature of the disc was determined from a thermocouple arc-welded to the TEM disc. The DBTT of an alloy was determined from optical and scanning-electron-microscope (SEM) observations of the fractured surfaces of disc specimens.

**3. Experimental results**

The temperature dependence of energy absorption during impact on dehydrogenated (unirradiated), hydrogenated (unirradiated), and irradiated Charpy specimens of V-14Cr-5Ti, V-9Cr-5Ti, V-5Ti, and V-4Cr-4Ti alloys is shown in Figs. 1a, 1b, 1c, and 1d, respectively. The DBTTs for dehydrogenated (<30 appm H), hydrogenated (590 appm H), and irradiated (21-44 dpa at 420-600°C) V-14Cr-5Ti alloy are -10, 130, and >400°C, respectively. The DBTTs for dehydrogenated (<30 appm H), hydrogenated (670 appm H), and irradiated (41-50 dpa at 420-600°C) V-9Cr-5Ti alloy are -55, 45, and 150°C, respectively. The DBTTs for dehydrogenated, hydrogenated (860 appm H), and irradiated (24-34 dpa at 427-599°C) V-5Ti alloy are <-220, -160, and <-220°C, respectively; and the DBTTs for dehydrogenated, hydrogenated (1200 appm H), and irradiated (24-34 dpa at 427-599°C) V-4Cr-4Ti alloy are <-220, -150, and <-220°C, respectively.

The dependence of DBTT for V-(0-18)Ti alloys on Ti concentration in the alloy is shown in Fig. 2. The DBTTs are minimum (<-220°C) for V-(1-5)Ti alloys with <30 appm H; and the DBTTs for hydrogenated alloys (600-1000 appm H) are minimum for the V-5Ti alloy. The DBTTs for V-(0-18)Ti alloys (<30 appm H) after irradiation at 420°C to 34-44 dpa are minimum (<-220°C) for V-(1-5)Ti alloys. DBTT data for alloys irradiated at 520 and 600°C are not shown in Fig. 2, but the DBTTs for V-(1-5)Ti alloys are also <-220°C.

The dependence of DBTT for dehydrogenated, hydrogenated, and irradiated V-Cr-(4-5)Ti alloys on Cr concentration in the alloy is shown in Fig. 3. The DBTT of dehydrogenated, hydrogenated, and irradiated V-Cr-(4-5)Ti alloys increased significantly for >4.1 wt.% Cr in an alloy.

The dependence of DBTTs for vanadium alloys on the combined (Cr + Ti) concentration in an alloy is shown in Fig. 4. These results suggest that the minimum DBTT for either unirradiated, hydrogenated, or neutron-irradiated V-Cr-Ti alloys is obtained for a vanadium alloy containing 0-4 wt.% Cr and 1-4 wt.% Ti and a combined (Cr + Ti) concentration of 4.6 to 8.4 wt.%.

**4. Discussion**

The experimental results presented in this paper tend to confirm the selection of the V-4Cr-4Ti alloy as the vanadium alloy with the near-optimum combination of physical and mechanical properties for use as structural material in a fusion reactor. The DBTT for the unirradiated and irradiated (24-34 dpa at 427-599°C) V-4Cr-4Ti alloy is <-220°C; the ductility of this alloy at 25°C is >8% after irradiation at 420°C to 34 dpa [7]; and the swelling of this alloy is low (<0.5%) after neutron irradiation at 420-600°C to 40 dpa [8].

The number-density and average diameter of precipitates in V-Cr-Ti alloys has been determined by Loomis, et al [9]. The low DBTT for V-(1-5)Ti alloys may be related to a minimum volume-fraction of non-coherent precipitates ($Ti(N_{1-x-y}C_xO_y)$, $Ti_{17}P_{10}$, $Ti_8S_3$) and coherent precipitates ($V_4C_3$, TiO) in these V-Ti alloys. The low DBTT for the V-4Cr-4Ti alloy may be related to a high volume-fraction (relatively large average-diameter) of non-coherent precipitates ($Ti(N_{1-x-y}C_xO_y)$) and absence of coherent precipitates.

**5. Conclusions**

1. The DBTTs for unirradiated V-(1-5)Ti and V-4Cr-4Ti alloys are <-220°C .
2. The DBTTs for V-Cr-Ti alloys are increased 100-150°C by the presence of 600-1200 appm hydrogen in the alloys.
3. The DBTTs of V-Cr-Ti alloys are minimum (<-220°C) for V-(3-5)Ti and V-4Cr-4Ti alloys after neutron irradiation at 420-600°C to 28-46 dpa.
4. The DBTTs for either unirradiated, irradiated, or hydrogenated V-Cr-(4-5)Ti alloys increase significantly for >4 wt.% Cr in an alloy.
5. The DBTTs for either irradiated or dehydrogenated V-Ti alloys are minimum for 1-5 wt.% Ti in an alloy.
6. The minimum DBTTs for V-(3-5)Ti alloys may be related to a low volume-fraction of precipitates (high number-density, small diameter $Ti(N_{1-x-y}C_xO_y)$, $Ti_{17}P_{10}$, $Ti_8S_3$) and the presence of coherent precipitates (fcc $V_4C_3$ and hcp TiO) in these alloys.
7. The minimum DBTT for V-4Cr-4Ti alloy may be related to a high volume-fraction of precipitates (low number-density, large diameter, fcc $Ti(N_{1-x-y}C_xO_y)$) and absence of coherent precipitates in this alloy.

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Table 1. Compositions of V and V-Cr-Ti alloys.

| Nominal Composition | ANL I.D. | Concentration (wt.%) | | | Concentration (ppm) | | | |
|---|---|---|---|---|---|---|---|---|
| | | Cr | Ti | Fe | O | N | C | Si |
| V | BL 51 | - | - | - | 570 | 49 | 56 | 370 |
| V-1Ti | BL 50 | - | 1.0 | - | 230 | 130 | 235 | 1050 |
| V-3Ti | BL 62 | - | 3.1 | - | 320 | 86 | 109 | 660 |
| V-3Ti-1Si | BL 45 | - | 2.5 | 0.01 | 345 | 125 | 90 | 9900 |
| V-5Ti | BL 46 | - | 4.6 | - | 300 | 53 | 85 | 160 |
| V-10Ti | BL 12 | - | 9.8 | 0.63 | 1670 | 390 | 450 | 245 |
| V-18Ti | BL 15 | - | 17.7 | 0.04 | 830 | 160 | 380 | 480 |
| V-14Cr | BL 5 | 14.1 | - | 0.06 | 330 | 69 | 200 | 50 |
| V-14Cr-1Ti | BL 26 | 14.1 | 1.0 | 0.06 | 560 | 86 | 140 | 50 |
| V-5Cr-3Ti | BL 54 | 5.1 | 3.0 | - | 480 | 82 | 133 | 655 |
| V-4Cr-4Ti | BL 47 | 4.1 | 4.3 | - | 350 | 220 | 200 | 870 |
| V-5Cr-5Ti | BL 63 | 4.6 | 5.1 | - | 440 | 28 | 73 | 310 |
| V-8Cr-6Ti | BL 49 | 7.9 | 5.7 | - | 400 | 150 | 127 | 360 |
| V-9Cr-5Ti | BL 43 | 9.2 | 4.9 | 0.02 | 230 | 31 | 100 | 340 |
| V-14Cr-5Ti | BL 24 | 13.5 | 5.2 | 0.05 | 1190 | 360 | 500 | 390 |
| V-15Cr-5Ti | BL 41 | 14.5 | 5.0 | 0.02 | 330 | 96 | 120 | 400 |
| V-10Cr-9Ti | BL 44 | 9.9 | 9.2 | 0.04 | 300 | 87 | 150 | 270 |
| V-7Cr-15Ti | BL 10 | 7.2 | 14.5 | 0.09 | 1110 | 250 | 400 | 400 |

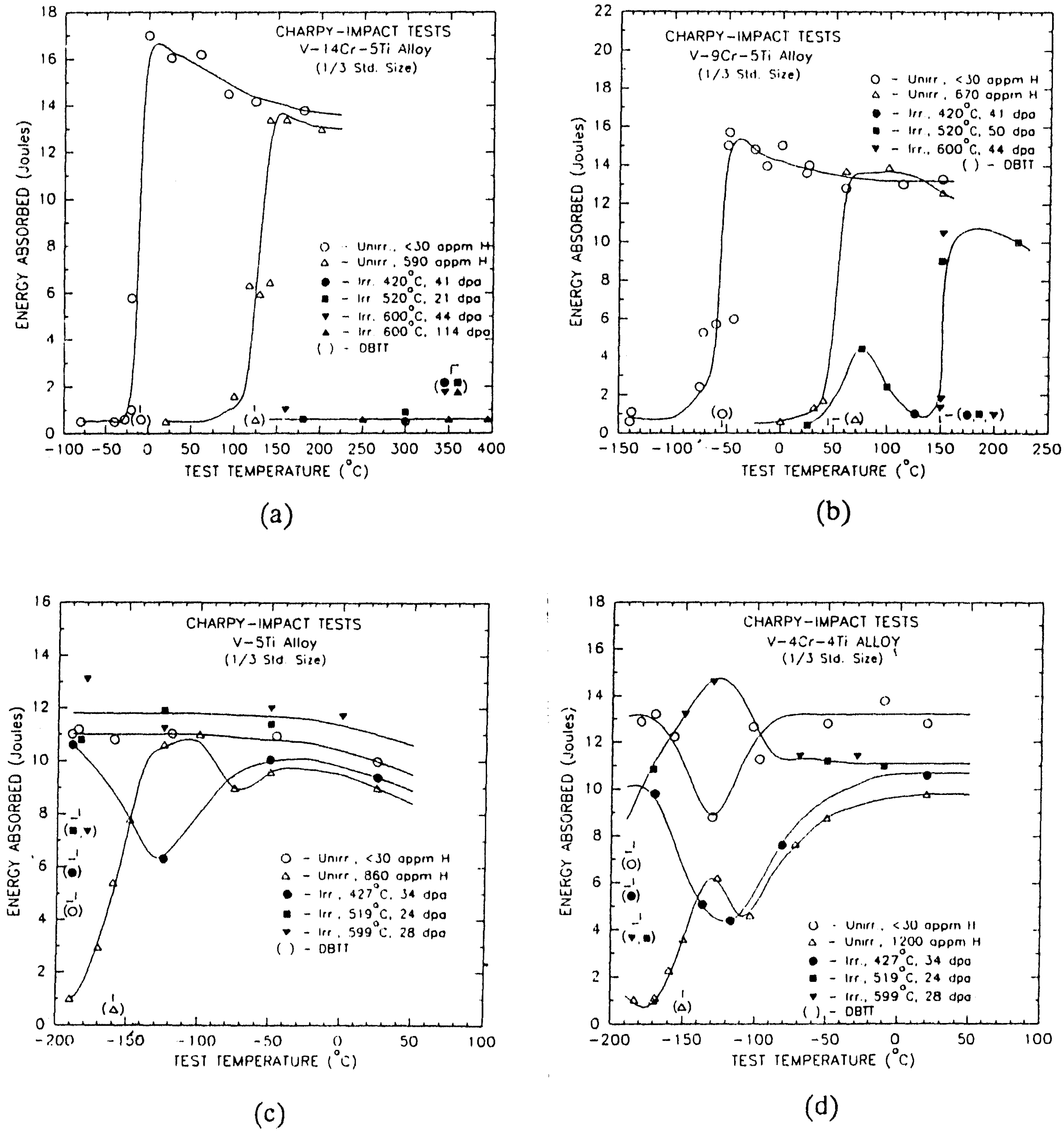
Figure titles:

Fig. 1. Dependence on test temperature of energy absorbed in Charpy-impact tests on (a) V-14Cr-5Ti, (b) V-9Cr-5Ti, (c) V-5Ti, and (d) V-4Cr-4Ti alloys.

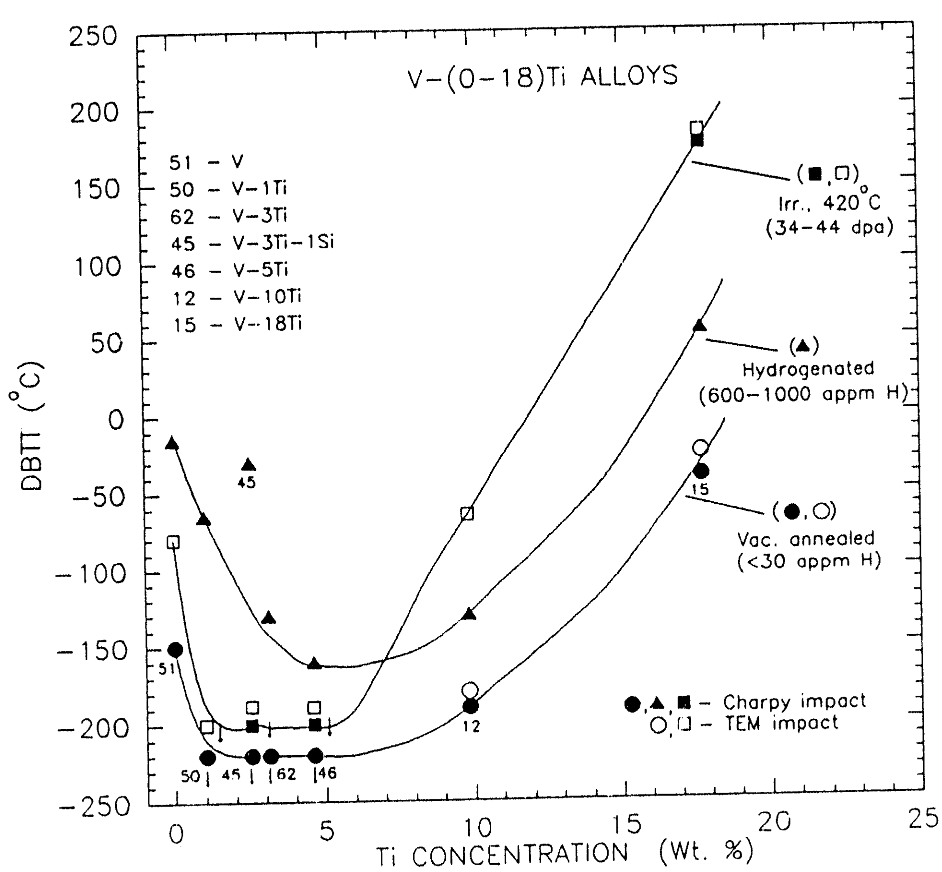
Fig. 2. Dependence of DBTT for V-(0-18)Ti alloys on Ti concentration.

Fig. 3. Dependence of DBTT for V-Cr-(3-5)Ti alloys on Cr concentration.

Fig. 4. Dependence of DBTT for vanadium alloys on (Cr + Ti) concentration.

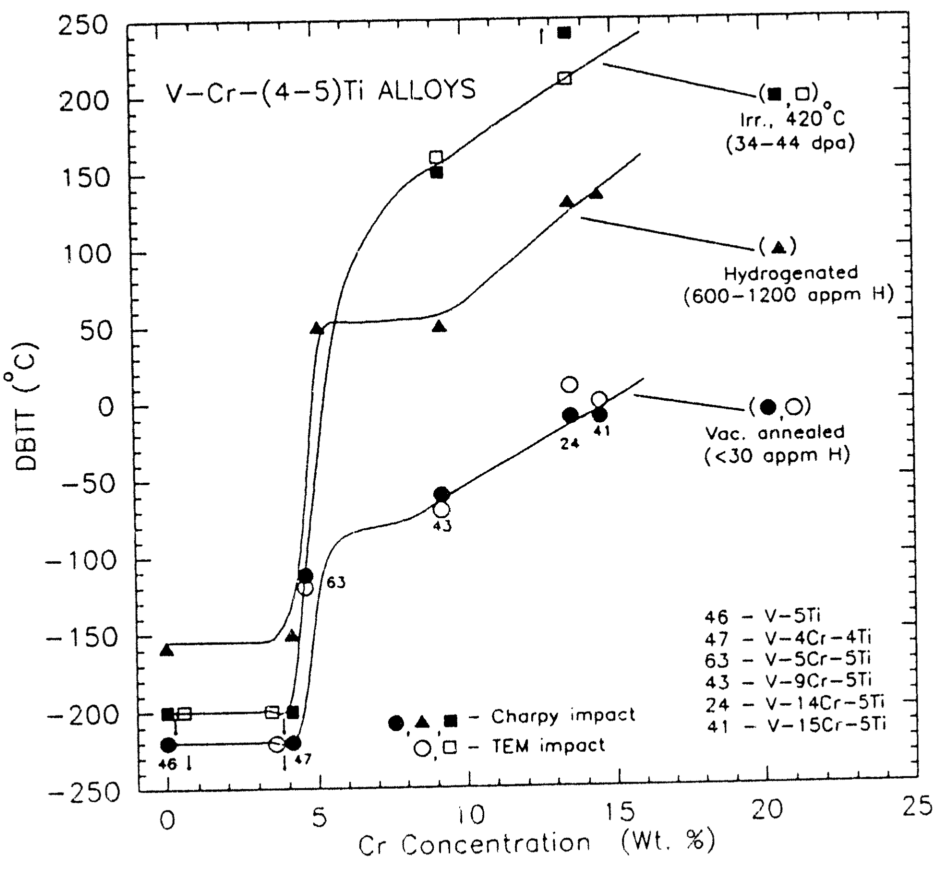

CHARPY-IMPACT TESTS
V-14Cr-5Ti Alloy
(1/3 Std. Size)
O - Unirr., <30 appm H
Δ - Unirr., 590 appm H
● - Irr. 420°C, 41 dpa
■ - Irr. 520°C, 21 dpa
▼ - Irr. 600°C, 44 dpa
▲ - Irr. 600°C, 114 dpa
( ) - DBTT
ENERGY ABSORBED (Joules)
TEST TEMPERATURE (°C)
(a)
CHARPY-IMPACT TESTS
V-9Cr-5Ti Alloy
(1/3 Std. Size)
O - Unirr., <30 appm H
Δ - Unirr., 670 appm H
● - Irr., 420°C, 41 dpa
■ - Irr., 520°C, 50 dpa
▼ - Irr., 600°C, 44 dpa
( ) - DBTT
ENERGY ABSORBED (Joules)
TEST TEMPERATURE (°C)
(b)
CHARPY-IMPACT TESTS
V-5Ti Alloy
(1/3 Std. Size)
O - Unirr., <30 appm H
Δ - Unirr., 860 appm H
● - Irr., 427°C, 34 dpa
■ - Irr., 519°C, 24 dpa
▼ - Irr., 599°C, 28 dpa
( ) - DBTT
ENERGY ABSORBED (Joules)
TEST TEMPERATURE (°C)
(c)
CHARPY-IMPACT TESTS
V-4Cr-4Ti ALLOY
(1/3 Std. Size)
O - Unirr., <30 appm H
Δ - Unirr., 1200 appm H
● - Irr., 427°C, 34 dpa
■ - Irr., 519°C, 24 dpa
▼ - Irr., 599°C, 28 dpa
( ) - DBTT
ENERGY ABSORBED (Joules)
TEST TEMPERATURE (°C)
(d)

Fig, 1



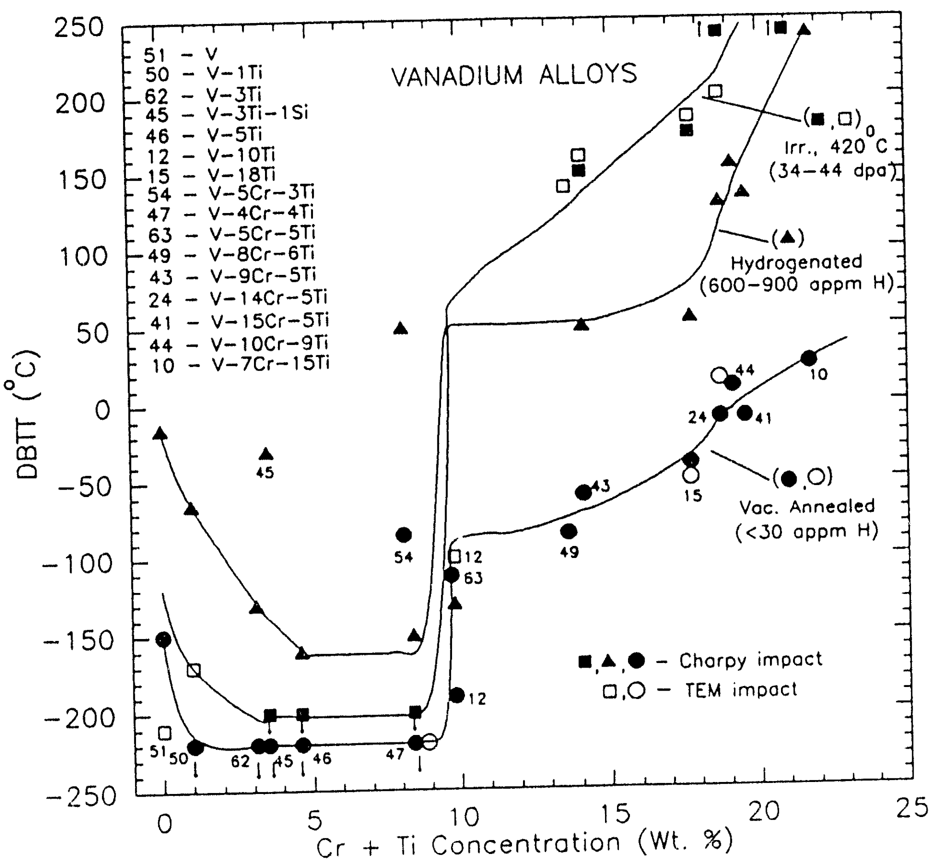
V–(0–18)Ti ALLOYS
51 – V
50 – V–1Ti
62 – V–3Ti
45 – V–3Ti–1Si
46 – V–5Ti
12 – V–10Ti
15 – V–18Ti
(■,□) Irr., 420°C (34–44 dpa)
(▲) Hydrogenated (600–1000 appm H)
(●,○) Vac. annealed (<30 appm H)
●,▲,■ – Charpy impact
○,□ – TEM impact
DBTT (°C)
Ti CONCENTRATION (Wt. %)


Fig. 2



V-Cr-(4-5)Ti ALLOYS
(■,□) Irr., 420°C (34-44 dpa)
(▲) Hydrogenated (600-1200 appm H)
(●,○) Vac. annealed (<30 appm H)
46 - V-5Ti
47 - V-4Cr-4Ti
63 - V-5Cr-5Ti
43 - V-9Cr-5Ti
24 - V-14Cr-5Ti
41 - V-15Cr-5Ti
●, ▲, ■ - Charpy impact
○, □ - TEM impact
DBTT (°C)
Cr Concentration (Wt. %)


Fig. 3



VANADIUM ALLOYS
51 – V
50 – V-1Ti
62 – V-3Ti
45 – V-3Ti-1Si
46 – V-5Ti
12 – V-10Ti
15 – V-18Ti
54 – V-5Cr-3Ti
47 – V-4Cr-4Ti
63 – V-5Cr-5Ti
49 – V-8Cr-6Ti
43 – V-9Cr-5Ti
24 – V-14Cr-5Ti
41 – V-15Cr-5Ti
44 – V-10Cr-9Ti
10 – V-7Cr-15Ti
Irr., 420°C
(34–44 dpa)
Hydrogenated
(600–900 appm H)
Vac. Annealed
(<30 appm H)
– Charpy impact
– TEM impact
DBTT (°C)
Cr + Ti Concentration (Wt. %)


Fig. 4

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