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SWELLING OF NEUTRON-IRRADIATED  
VANADIUM ALLOYS

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SWELLING OF NEUTRON-IRRADIATED VANADIUM ALLOYS\*

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Abstract

The swelling of unalloyed vanadium and vanadium alloys containing Cr, Ti, Si, Fe, Zr, W, Mo, and Ni additions was determined after neutron irradiation at 420 and 600°C to 17-84 dpa in the FFTF-MOTA reactor facility. The swelling of these materials was obtained from a determination of the immersion density of the unirradiated and irradiated materials. The swelling of V is substantially increased by the addition of Cr, and significantly reduced by the addition of Ti. The swelling of V-Cr-Ti alloys is strongly dependent on Ti concentration for Ti concentrations <3%. For resistance to irradiation-induced swelling, the Ti concentration in a V-Cr-Ti alloy that is to be used as a structural material in a magnetic fusion reactor should be greater than 3%. For these ranges of Ti concentration in a V-Cr-Ti alloy, Cr concentrations of <15% have minimal effect on the swelling of the alloy. The Vanstar-7 alloy exhibits high (7-10%) swelling on irradiation at 600°C to 77 dpa. Swelling of V-7.2Cr-14.5Ti, V-13.5Cr-5.2Ti, V-9.2Cr-4.9Ti, V-3.1Ti-(0.3-0.5)Si, and V-17.7Ti on neutron irradiation at 600°C to 87 dpa is (in % per dpa) 0.10, 0.03, 0.03, 0.01, and 0.01, respectively.

Key words: Vanadium, vanadium alloys, neutron irradiation, swelling.

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## SWELLING OF NEUTRON-IRRADIATED VANADIUM ALLOYS\*

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### 1. Introduction

The swelling of V-15Cr-5Ti, Vanstar-7, V-3Ti-1Si, V-20Ti, and V-15Ti-7.5Cr on neutron irradiation at temperatures ranging from 420 to 600°C and irradiation damage levels ranging up to 40 atom displacements per atom (dpa) have been reported by several investigators [1-7]. The results obtained from these investigations have shown that V-15Cr-5Ti, V-3Ti-1Si, V-20Ti, and V-15Ti-7.5Cr are resistant to swelling caused by the presence of voids or cavities and typically exhibit swelling <0.3% (40 dpa). The results obtained from these investigations also show that Vanstar-7 can exhibit swelling of up to 6%.

Ohnuki, et al. determined the swelling of V-3Cr, V-15Ti, and V-3Mo after neutron irradiation at 500-600°C to approximately 1 dpa [8]. The swelling values reported for these neutron-irradiated alloys suggest that (1) Ti suppresses swelling of V, (2) Cr exacerbates swelling of V, and (3) Mo has a relatively minor effect on the swelling of V.

A dimensional change (swelling) of materials on neutron irradiation can occur as a result of void formation, cavity formation, thermal- and irradi-

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ation-induced precipitate formation and dissolution, thermal- and irradiation-induced phase change, and thermal- and irradiation-induced solute segregation. The swelling values that have been previously reported for neutron-irradiated V alloys were obtained from observations by transmission electron microscopy (TEM) of irradiated materials [1-8]. Therefore, these evaluations have only taken into account swelling that is attributable to the presence of voids and cavities. The TEM observations of irradiated V alloys have shown the presence of irradiation- and/or thermal-induced precipitates in addition to voids [1-10]. In this paper, swelling data for neutron-irradiated V alloys and unalloyed V are presented that were obtained from determinations of the immersion density of unirradiated and neutron-irradiated specimens.

## 2. Materials and procedure

Specimens approximately 3.0 mm in diameter and 0.3 mm thick were obtained from 50% cold-worked sheets of the materials listed in Table 1. The cold-worked specimens were annealed at 1125°C for one hour in an ion-pumped vacuum system with a typical pressure of  $1.3 \times 10^{-6}$  Pa. The average recrystallized grain diameter in these annealed materials was 0.020 mm. The specimens were irradiated in the Materials Open Test Assembly (MOTA) in the Fast Flux Test Facility (FFTF) reactor. They were contained in sealed, Li<sup>7</sup>-filled, TZM molybdenum capsules during irradiation to prevent contamination from oxygen, nitrogen, and carbon impurities in the FFTF sodium coolant. The specimens were irradiated at 420 and 600°C to neutron fluences ( $E > 0.1$  MeV) ranging from  $3.4 \times 10^{22}$  n/cm<sup>2</sup> (17 dpa) to  $17.4 \times 10^{22}$

n/cm<sup>2</sup> (87 dpa) during Cycles 7,8,9,and 10 of the FFTF-MOTA facility. The specimens that were irradiated at 600°C during Cycles 7-8 experienced a temperature excursion of 249°C for 50 minutes during the Cycle 7 portion of the irradiation. The irradiated specimens were removed from the Li-filled TZM molybdenum capsules by immersion of the opened capsules in liquid NH<sub>3</sub> and subsequent immersion of the specimens in a mixture of 50% ethanol and 50% methanol.

The hydrogen concentration in irradiated specimens from each of the TZM capsules was determined with a quadrapole, partial-pressure gas analyzer mounted in an ion-pumped vacuum system [11]. The hydrogen concentration in specimens irradiated at 420°C during Cycles 7-8, 8, and 8-9 of the FFTF-MOTA was typically 5000-10000 appm, whereas the hydrogen concentration in specimens irradiated at 420°C during Cycles 9-10 and 10 was typically <500 appm. The hydrogen concentration in specimens irradiated at 600°C was typically <500 appm. The hydrogen concentration in specimens irradiated at 420°C was generally in the order of V-3.1Ti-0.3Si > V-13.5Cr-5.2Ti > V-7.2Cr-14.5Ti > V-17.7Ti, whereas the hydrogen concentration in specimens irradiated at 600°C was in the order of V-7.2Cr-14.5Ti > V-13.5Cr-5.2Ti > V-9.2Cr-4.9Ti > V-17.7Ti.

The specimens that were irradiated at 420°C and 600°C during Cycles 7-8, 8, and 8-9 of the FFTF-MOTA showed significant corrosion product on the surfaces. In contrast, the surfaces of specimens that were irradiated during Cycles 10 and 9-10 were conspicuously free of corrosion product. These observations, together with the results of the hydrogen analyses and the absence of a temperature excursion during irradiation, have led us to

consider that the swelling data obtained for specimens irradiated during Cycles 10 and 9-10 of the FFTF-MOTA are significantly more credible than those data obtained for specimens irradiated during Cycles 7-8, 8, and 8-9.

The swelling ( $S$ ) of an irradiated specimen was obtained from a determination of the density of an unirradiated specimen ( $D_{ann}$ ) and the density of an irradiated specimen ( $D_{irr}$ ) by immersion in  $CCl_4$ , i.e.,  $S = (D_{ann} - D_{irr})/D_{irr}$ . The reported density for a specimen was determined with a precision of 0.1% from 3-6 separate determinations on a specimen.

### 3. Experimental results

The dependence of swelling on Ti concentration for the V-Ti alloys after neutron irradiation at 420°C to 36 and 73-77 dpa is shown in Fig. 1 (data in Table 2, Ref. 12). The swelling of V on irradiation at 420°C to 36 and 77 dpa was significantly decreased by the addition of 4.9% Ti. With the exception of V-8.6Ti, the swelling of the binary V-Ti alloys on irradiation at 420°C was not strongly dependent on Ti concentration in the range of 5 to 20 wt.% Ti. The higher swelling of V-8.6Ti compared with that of V-4.9Ti, V-9.8Ti, and V-14.4Ti may be attributed to the relatively low oxygen concentration in V-8.6Ti. In general, the swelling of V-Ti alloys containing >3.1% Ti was slightly higher for those irradiated to 36 dpa than for those irradiated to 73-77 dpa.

The dependence on Ti concentration of swelling for the V-Cr-Ti and V-Ti-Si alloys on irradiation at 600°C to 77-84 dpa is shown in Fig. 2 (data from Table 2 of Ref. 12). The addition of 0.3% Ti to V-14.1Cr alloy was sufficient to reduce its swelling from 39% to 15%. An increase of the Ti concentration to 5% caused a further reduction of swelling of V-14.1Cr

alloy to approximately 2% on irradiation at 600°C to 84 dpa. The swelling of the V-Ti-Si alloys on irradiation at 600°C was not strongly dependent on the Ti concentration. The lower swelling of V on irradiation at 600°C compared with swelling at 420°C (Fig. 1) may be partially attributed to the irradiation temperature of 600°C being above the peak swelling temperature, which is ~550°C [13]. The swelling data in Fig. 2 for V-14.4Ti and V-7.2Cr-14.5Ti suggest that (1) the swelling of V-Ti and V-Cr-Ti alloys may be increased for Ti concentrations in the range of 10 to 18% Ti, and (2) the swelling of V-Cr-Ti alloys containing >5% Ti is not dependent on Cr concentration for Cr concentrations of <15%.

Figure 3 shows the dependence of the swelling of V-7.2Cr-14.5Ti, V-9.2Cr-4.9Ti, V-13.5Cr-5.2Ti, V-3.1Ti-(0.3-0.5)Si, and V-17.7Ti produced by irradiation at 600°C. These specimens were irradiated during Cycles 10 and 9-10 of the FFTF-MOTA. On the basis of these data, the swelling of V-7.2Cr-14.5Ti, V-13.5Cr-5.2Ti, V-9.2Cr-4.9Ti, V-3.1Ti-(0.3-0.5) Si, and V-17.7Ti on irradiation at 600°C to 84 dpa is (in % per dpa) 0.10, 0.03, 0.03, 0.01, and 0.01, respectively. It may be noted that the swelling of these alloys (Fig. 3) at the highest damage level (84 dpa) is inversely related to the tensile ductility of the irradiated alloys (600°C, 87 dpa), i.e., highest ductility for V-17.7Ti (26.2%), intermediate ductility for V-3.1Ti-0.3Si (13.0%) and V-13.5Cr-5.2Ti (11.0%), and lowest ductility for V-7.2Cr-14.5Ti (8.1%) [14].

The addition of 8.6% W to V increased the swelling of V on irradiation at 420°C to 36 dpa, whereas the addition of 4.0% Mo and 12.3% Ni had a minor effect on the swelling of V at 420°C. However, on irradiation at



600°C to 17 dpa, such additions of either Ni, W, or Mo to V increased the swelling of V by ~0.5% [12]. The Vanstar-7 exhibited high (7-10%) swelling on irradiation at 600°C to 77 dpa [12].

## 5. Conclusions

1. The swelling of V-Ti and V-Cr-Ti alloys on neutron irradiation at 420 and 600°C is strongly dependent on Ti concentration.
2. The V-Cr-Ti alloys with greater than 3% Ti exhibit greater resistance to irradiation-induced swelling. For these ranges of Ti concentration in a V-Cr-Ti alloy, Cr concentrations of <15% have minimal effect on the swelling.
3. The swelling of V-7.2Cr-14.5Ti, V-13.5Cr-5.2Ti, V-9.2Cr-4.9Ti, V-3.1Ti-(0.3-0.5)Si, and V-17.7Ti on neutron irradiation at 600°C to 84 dpa is (in % per dpa) 0.10, 0.03, 0.03, 0.01, and 0.01, respectively.

## References

- [1] D. N. Braski, in: Influence of Radiation on Material Properties: 13th International Symposium, ASTM STP 956, Eds. F. A. Garner, C. H. Henager, Jr., and N. Igata (American Society for Testing Materials, Philadelphia, 1986) p. 271.
- [2] J. Bentley and F. W. Wiffen, Nucl. Technol. 30 (1976) 376.
- [3] H. Bohm, in: Proceedings: International Conference on Defects and Defect Clusters in B.C.C. Metals and Their Alloys, Ed. R. J. Arsenault (M. P. Graphics Inc., Washington, D. C., 1973) p. 163.
- [4] M. L. Grossbeck and J. A. Horak, in: Influence of Radiation on Material Properties: 13th International Symposium, ASTM STP 956, Eds. F. A. Garner, C. H. Henager, Jr., and N. Igata (American Society for Testing Materials, Philadelphia, 1986) p. 291.

- [5] R. Carlander, S. D. Harkness, and A. T. Santhanum, in: Effects of Radiation on Substructure and Mechanical Properties of Metals and Alloys, ASTM STP 529 (American Society for Testing Materials, Philadelphia, 1973) p. 399.
- [6] M. P. Tanaka, E. E. Bloom, and J. A. Horak, J. Nucl. Mater., 103 & 104 (1981) 895.
- [7] D. N. Braski, J. Nucl. Mater., 141 & 143 (1986) 1125.
- [8] S. Ohnuki, H. Takahashi, H. Kinoshita, and R. Nagasaki, J. Nucl. Mater., 155-157 (1988) 935.
- [9] B. A. Loomis, B. J. Kestel, and D. L. Smith, J. Nucl. Mater., 155-157 (1988) 1305.
- [10] Y. Higashiguchi, H. Kayano, and S. Morozumi, J. Nucl. Mater., 133-134 (1985) 662.
- [11] B. A. Loomis, A. B. Hull, O. K. Chopra, and D. L. Smith, in: Fusion Reactor Materials, Semiannual Progress Report for Period Ending March 31, 1988, DOE/ER-0313/4, Oak Ridge National Laboratory, Oak Ridge, TN (September, 1988) p. 160.
- [12] B. A. Loomis and D. L. Smith, in: Fusion Reactor Materials, Semiannual Progress Report for Period Ending March 31, 1989, DOE/ER-0313/6, Oak Ridge National Laboratory, Oak Ridge, TN (September, 1989) p. 339.
- [13] A. F. Bartlett, in: Proceedings of Conference on Radiation Effects and Tritium Technology for Fusion Reactors, CONF-750989, Oct. 1-3, 1975, Gatlinburg, TN, p. I-122.
- [14] B. A. Loomis and D. L. Smith, in: Fusion Reactor Materials, Semiannual Progress Report for Period Ending September 30, 1989, DOE/ER-0313/7, Oak Ridge National Laboratory, Oak Ridge, TN (March, 1990) p.300.

Table 1.  
Vanadium and Vanadium Alloy Composition<sup>1</sup>

ANL I.D.	Material (Wt.%)	Melt Number	Concentration (ppm)				
			O	N	C	Si	Al
BL-1	V-4.0Mo	ANL 1	230	73	90	110	<100
BL-2	V-8.6W	ANL 2	300	150	120	59	<100
BL-3	V-12.3Ni	ANL 3	490	280	500	405	<100
BL-4	V-10.0Cr	ANL 4	530	76	240	<50	1190
BL-5	V-14.1Cr	ANL 5	330	69	200	<50	2740
BL-10	V-7.2Cr-14.5Ti	ANL 94	1110	250	400	400	30
BL-11	V-4.9Ti	ANL 11	1820	530	470	220	115
BL-12	V-9.8Ti	ANL 12	1670	390	450	245	<100
BL-13	V-14.4Ti	ANL 13	1580	370	440	205	<100
BL-15	V-17.7Ti	CAM 832	830	160	380	480	33
BL-16	V-20.0Ti	CAM 833	390	530	210	480	-
BL-20	V	ANL 20	570	110	120	325	<100
BL-24	V-13.5Cr-5.2Ti	ANL 101	1190	360	500	390	40
BL-25	V-14.4Cr-0.3Ti	ANL 25	390	64	120	<50	3270
BL-26	V-14.1Cr-1.0Ti	ANL 26	560	86	140	<50	3090
BL-27	V-3.1Ti-0.3Si	ORNL 10837	210	310	310	2500	160
BL-28	Vanstar-7 <sup>2</sup>	CAM 837	275	540	740	-	-
BL-34	V-8.6Ti	ANL 34	990	180	420	290	<100
BL-35	V-9.5Cr	ANL 35	340	45	120	<50	1450
BL-36	V	ANL 36	810	86	250	<50	<100
BL-42	V-3.1Ti-0.5Si	WI 8505	580	190	140	5400	290
BL-43	V-9.2Cr-4.9Ti	ANL 206	230	31	100	340	140

<sup>1</sup>The chemical analyses of these materials were performed by the Analytical Department of the Teledyne Wah Chang Albany Company.

<sup>2</sup>V-9.0Cr-3.2Fe-1.2Zr.

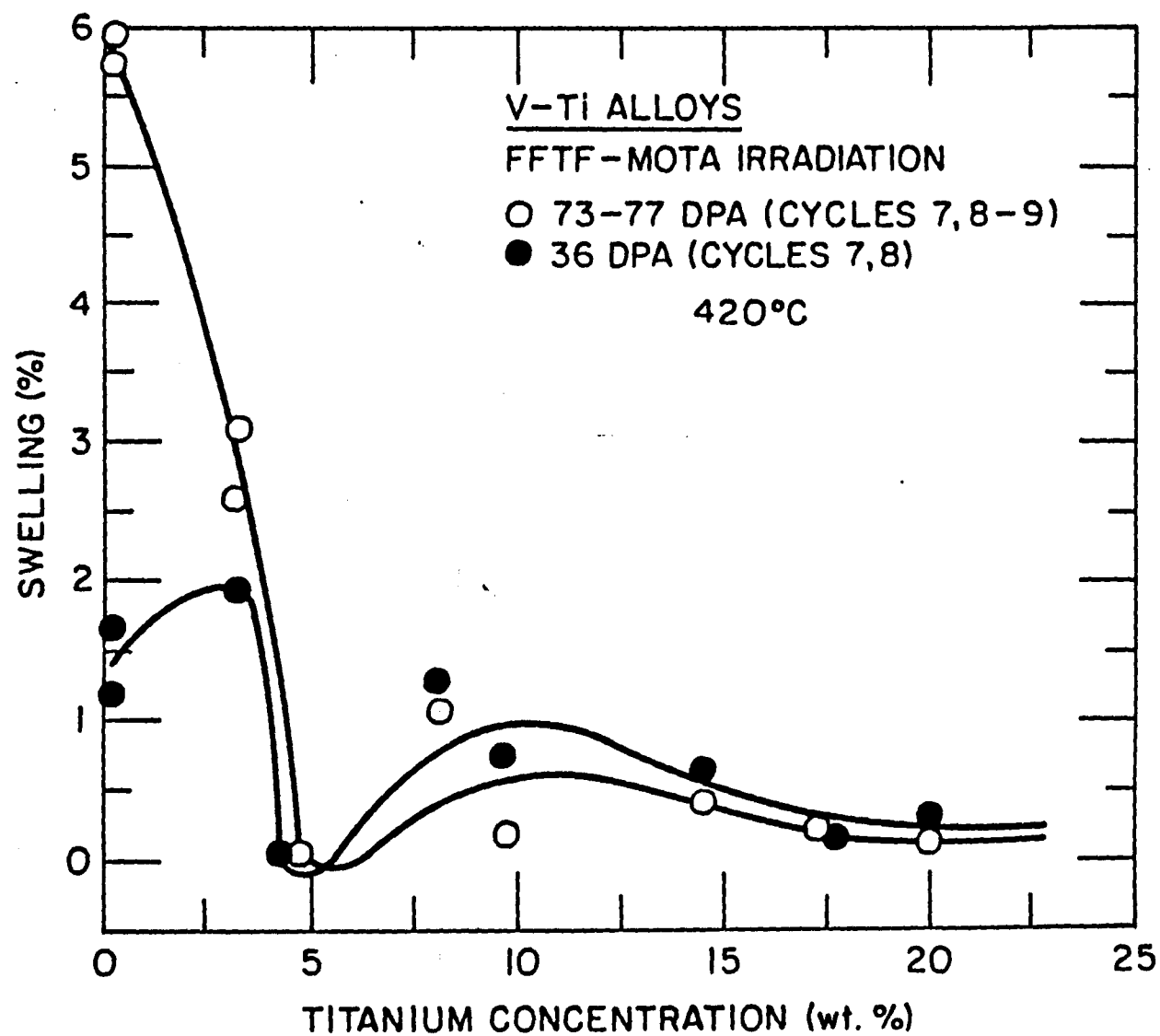
**List of Figures:**

**Fig. 1.** Swelling of V-Ti alloys on neutron irradiation at 420°C to 36 and 73-77 dpa.

**Fig. 2.** Swelling of V-Ti-Si and V-Cr-Ti alloys on neutron irradiation at 600°C to 77-84 dpa (open symbols for V-Cr-Ti alloys and filled symbols for V-Ti-Si alloys).

**Fig. 3.** Dependence of swelling of vanadium alloys at 600°C on irradiation damage (dpa).

Fig. 1



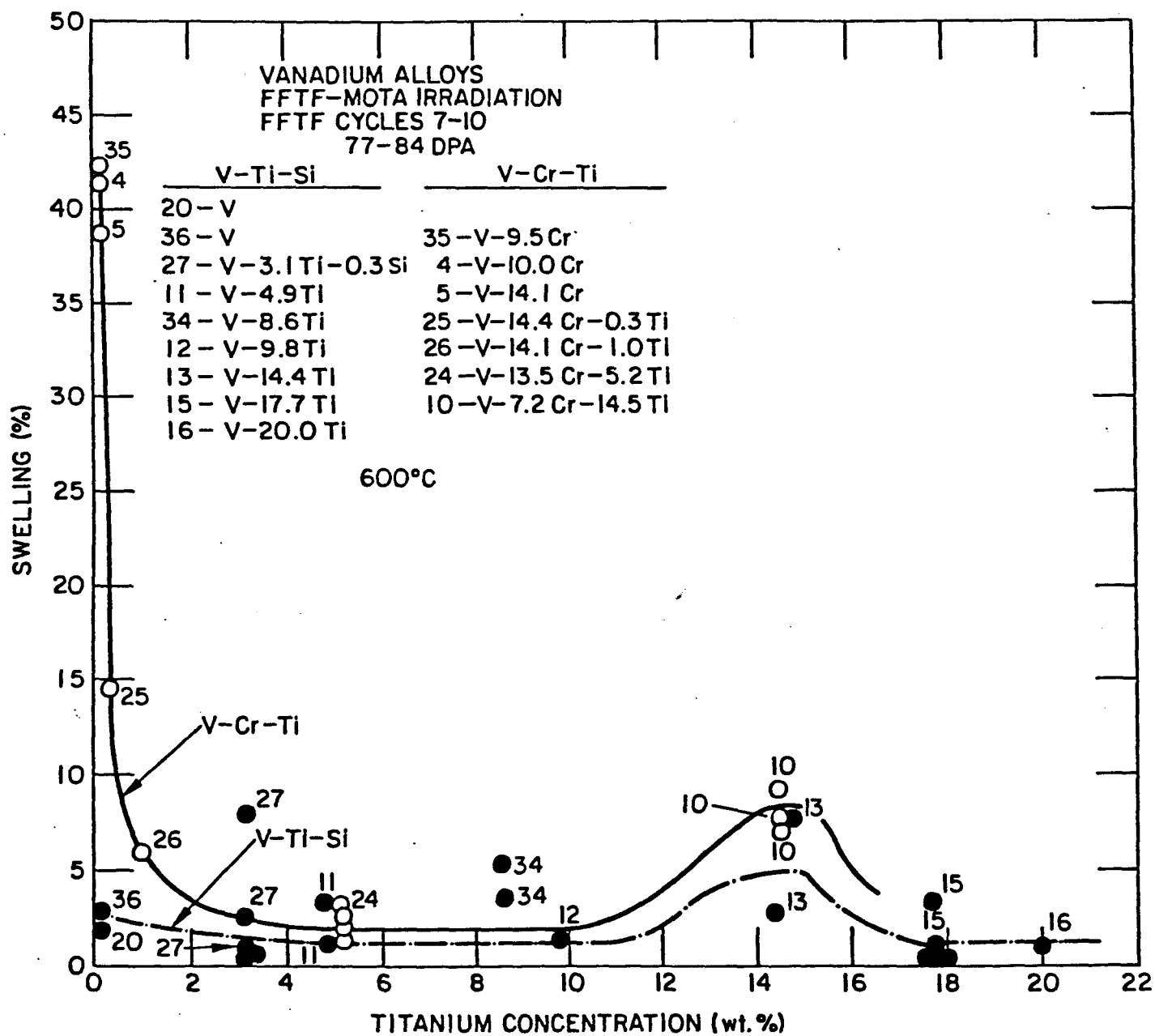


Fig. 2

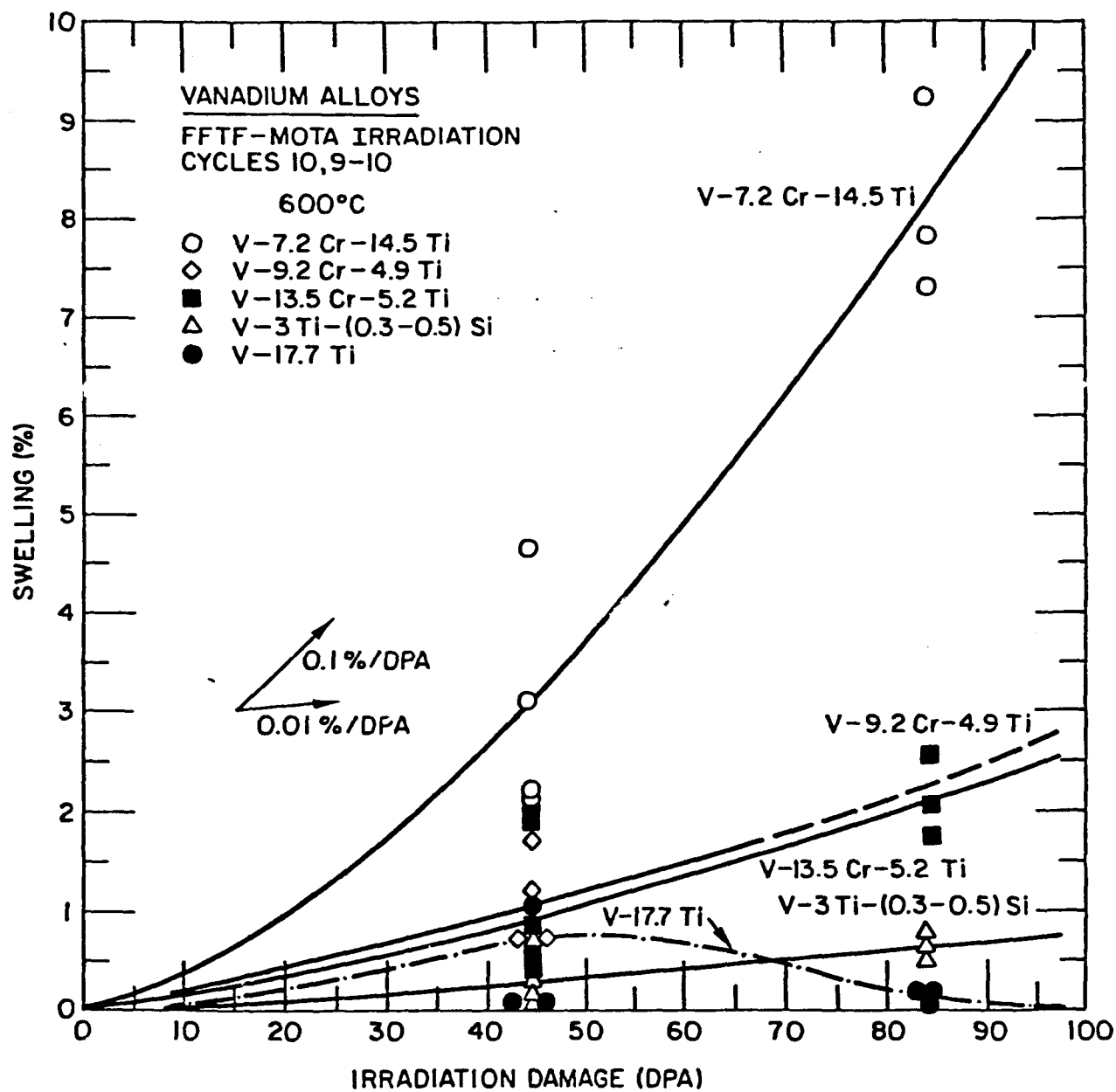


Fig. 3