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VANADIUM ALLOYS

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SWELLING OF NEUTRON-IRRADIATED VANADIUM ALLOYS - B. A. Loomis and D. L. Smith (Argonne National Laboratory) and F. A. Garner (Pacific Northwest Laboratory)

OBJECTIVE

The objective of this research is to provide guidance on the applicability of vanadium-base alloys for structural components in a fusion reactor.

SUMMARY

The swelling of V-10.0Cr-0.1Al, V-14.1Cr-0.3Al, V-3.1Ti-0.3Si, V-4.9Ti, V-9.8Ti, V-14.4Ti, V-17.7Ti, V-20.0Ti, V-14.4Cr-0.3Ti-0.3Al, V-14.1Cr-1.0Ti-0.3Al, V-13.7Cr-4.8Ti, V-9.0Cr-3.3Fe-1.2Zr (Vanstar-7), V-14.5Ti-7.2Cr, V-8.6W, V-4.0Mo, and V-12.3Ni alloys and unalloyed V was determined after neutron irradiation at 420°C and 600°C to irradiation damage levels ranging from 17 to 77 dpa in the FFTF-MOTA reactor facility. The swelling of these alloys was obtained from a determination of the density for the unirradiated and irradiated alloys on immersion in CCl₄. The swelling of unalloyed V at 600°C was substantially increased by the addition of Cr. The addition of either Ni, W, or Mo to V had a relatively minor effect on the swelling of V. The swelling of the V-Cr-Ti alloys was strongly dependent on the Ti concentration. The swelling of the V-3.1Ti-0.3Si and V-14.4Ti alloys at 600°C was greater than that exhibited by the other binary V-Ti alloys. The Vanstar-7 alloy underwent larger swelling than the V-Ti and V-Cr-Ti alloys. For the binary V-Ti alloys and the ternary V-Cr-Ti alloys, the dependence of swelling on the amount of irradiation damage was <0.1% swelling per dpa.

PROGRESS AND STATUS

Introduction

The swelling of V-15Cr-5Ti, Vanstar-7, V-3Ti-1Si, V-20Ti, and V-15Ti-7.5Cr alloys 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 the V-15Cr-5Ti, V-3Ti-1Si, V-20Ti, and V-15Ti-7.5Cr alloys are resistant to swelling and typically exhibit swelling values of <0.3%. The results obtained from these investigations also show that the Vanstar-7 alloy can exhibit swelling values ranging up to 6%.

Ohnuki et al. have determined the swelling of V-3Cr, V-15Ti, and V-3Mo alloys 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 strongly 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 irradiation-induced precipitate formation and dissolution, thermal- and irradiation-induced phase change, thermal- and irradiation-induced solute segregation, and anisotropic dislocation glide and climb. The swelling values that have been previously reported for neutron-irradiated V alloys have been obtained from transmission electron microscopy (TEM) observations. Therefore, these TEM swelling evaluations have only taken into account the dimensional change (swelling) that can be attributed to the presence of voids and cavities. The TEM observations of irradiated V alloys have shown the presence of copious irradiation- and/or thermal-induced precipitates.¹⁻⁸ In this report, we present for neutron-irradiated V alloys and unalloyed V swelling values that have been derived from the determination of the density of specimens on immersion in CCl₄.

Materials and procedures

Specimens with approximate dimensions of 0.3-cm-diameter and 0.03-cm-thickness were obtained from 50% cold-worked sheets of the materials listed in Table 1. The chemical analyses of these materials were performed by the Analytical Department of the Teledyne Wah Chang Albany Company. The cold-worked specimens were annealed at 1125°C for 1 h in an ion-pumped vacuum system with a typical pressure of 8 x 10⁻⁹ mm Hg.

Table 1. Materials composition

ANL I.D.	Material	Melt Number	Concentration (wt ppm)				
			O	N	C	Si	Fe
BL-1	V-4.0Mo	ANL 1	230	73	90	110	<100
BL-2	V-8.6W ¹	ANL 2	300	150	120	59	<100
BL-3	V-12.3Ni	ANL 3	490	280	500	405	175
BL-4	V-10.0Cr-0.1Al	ANL 4	530	76	240	<50	530
BL-5	V-14.1Cr-0.3Al	ANL 5	330	69	200	<50	570
BL-10	V-7.2Cr-14.5Ti	ANL 94	1110	250	400	400	910
BL-11	V-4.9Ti	ANL 11	1820	530	470	220	7800
BL-12	V-9.8Ti	ANL 12	1670	390	450	245	6300
BL-13	V-14.4Ti	ANL 13	1580	370	440	205	6300
BL-15	V-17.7Ti	CAM 832	830	160	380	480	390
BL-16	V-20.0Ti	CAM 833	390	530	210	480	390
BL-20	V	ANL 20	570	110	120	325	<100
BL-21	V-13.7Cr-4.8Ti	CAM 835	340	510	180	1150	300
BL-22	V-13.4Cr-5.1Ti	ANL 114	300	52	150	56	140
BL-23	V-12.9Cr-5.9Ti	CAM 834	400	490	280	1230	420
BL-24	V-13.5Cr-5.2Ti	ANL 101	1190	360	500	390	520
BL-25	V-14.4Cr-0.3Ti-0.3Al	ANL 25	390	64	120	<50	680
BL-26	V-14.1Cr-1.0Ti-0.3Al	ANL 26	560	86	140	<50	650
BL-27	V-3.1Ti-0.3Si ²	ORNL 10837	210	310	310	2500	380
BL-28	V-9.0Cr-3.3Fe-1.2Zr ³	CAM 837	275	540	740	—	—
BL-34	V-8.6Ti	ANL 34	990	180	420	290	150
BL-35	V-9.5Cr-0.2Al	ANL 35	340	45	120	<50	410
BL-35	V	ANL 36	810	86	250	<50	<100
BL-48	BL-23 + 20% CW	CAM 834	400	490	280	1230	420

¹Alloy BL-2 contained 365 ppm Nb; all other alloys contained <100 ppm Nb.²Alloy BL-27 contained 250 ppm Ta.³Vanstar-7 alloy.

The annealed specimens were irradiated at 420°C and 600°C in lithium-filled TZM capsules during cycles 7, 8, and 9 of the FFTF-MOTA reactor facility to neutron fluences ($E > 0.1$ eV) ranging from 2.8×10^{22} n/cm² (17 dpa) to 13.1×10^{22} n/cm² (77 dpa). The specimens that were irradiated at 600°C experienced a temperature excursion of 249°C for 50 min during cycle 7 of the FFTF-MOTA. The irradiated specimens were removed from the lithium-filled TZM capsules by immersion of the opened capsules in liquid NH₃ and subsequent immersion in a 50/50 mixture of ethanol and methanol.

The swelling (S) of an irradiated specimen was obtained from a determination of the density of an annealed, unirradiated (D_{ann}) specimen and an irradiated (D_{irr}) specimen on immersion in CCl₄, i.e.,

$$S = (D_{ann} - D_{irr}) / D_{ann}.$$

The reported density for a specimen was determined with a precision of 0.2% from 6-10 separate determinations on a specimen. In the case of specimens irradiated at 600°C, some specimens of an alloy (with ostensibly the same irradiation conditions) had significantly different (2-3%) swelling values, and these values are listed separately in Table 2.

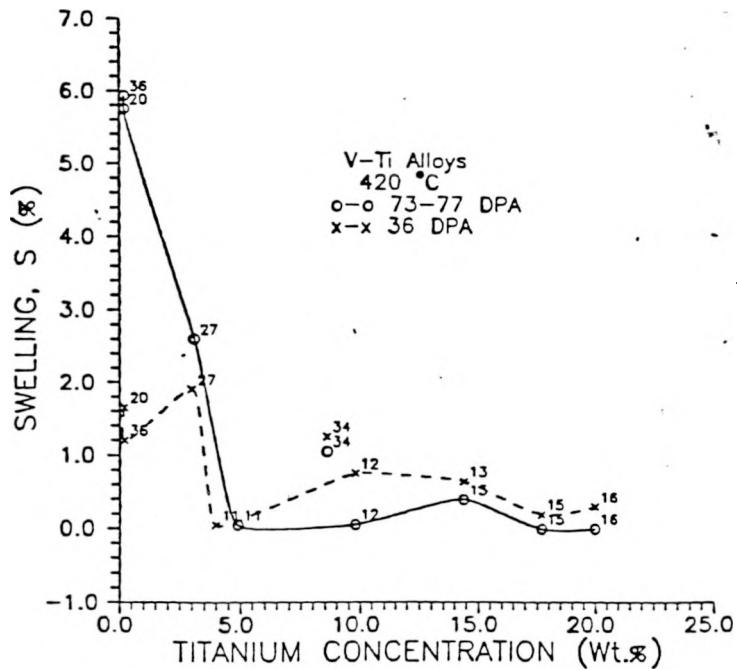
Experimental results

The swelling values for neutron-irradiated specimens of the V alloys and unalloyed V (Table 1) are presented in Table 2. The density (D_{ann}) values for the annealed alloys and unalloyed V are also listed in Table 2. The data in Table 2 suggest that the addition of either Ni, W, or Mo to V had a relatively minor effect on the swelling of V.

Table 2. Swelling of neutron-irradiated V alloys and unalloyed V

ANL I.D.	Material	D _{ann} (g/cc)	Irradiation Temperature			
			420°C	500°C	600°C	600°C
BL-1	V-4.0Mo	6.227	36	1.12	17	0.60
BL-2	V-8.6W	6.448	36	2.30	17	0.51
BL-3	V-12.3Ni	6.336	36	1.06	17	0.60
BL-4	V-10.0Cr-0.1Al	6.204	36	2.39	17	1.64
BL-4	V-10.0Cr-0.1Al	6.204	77	0.53	77	41.60
BL-5	V-14.1Cr-0.3Al	6.277	36	3.76	17	1.18
BL-5	V-14.1Cr-0.3Al	6.277	—	—	77	38.85
BL-10	V-7.2Cr-14.5Ti	5.887	36	0.61	21	0.75
BL-10	V-7.2Cr-14.5Ti	5.887	46	0.97	—	—
BL-10	V-7.2Cr-14.5Ti	5.887	77	0.18	—	—
BL-11	V-4.9Ti	6.007	36	0.03	17	-0.33
BL-11	V-4.9Ti	6.007	77	0.01	77	1.39
BL-11	V-4.9Ti	6.007	—	—	77	3.47
BL-12	V-9.8Ti	5.912	36	0.74	17	-0.10
BL-12	V-9.8Ti	5.912	77	0.22	77	1.41
BL-13	V-14.4Ti	5.835	36	0.63	21	0.60
BL-13	V-14.4Ti	5.835	77	0.39	77	2.86
BL-13	V-14.4Ti	5.835	—	—	77	7.71
BL-15	V-17.7Ti	5.704	36	0.12	21	-0.04
BL-15	V-17.7Ti	5.704	46	—	77	0.96
BL-15	V-17.7Ti	5.704	77	0.20	77	3.42
BL-16	V-20.0Ti	5.711	36	0.29	17	0.09
BL-16	V-20.0Ti	5.711	77	0.15	77	0.98
BL-20	V	6.099	36	1.67	21	0.26
BL-20	V	6.099	73	5.75	77	1.53
BL-21	V-13.7Cr-4.8Ti	6.173	36	0.44	21	0.42
BL-21	V-13.7Cr-4.8Ti	6.173	73	1.99	77	4.46
BL-21	V-13.7Cr-4.8Ti	6.173	—	—	77	6.12
BL-22	V-13.4Cr-5.1Ti	6.151	36	1.44	17	0.33
BL-22	V-13.4Cr-5.1Ti	6.151	73	0.89	77	5.37
BL-22	V-13.4Cr-5.1Ti	6.151	—	—	77	6.67
BL-23	V-12.9Cr-5.9Ti	6.145	36	1.28	21	0.28
BL-23	V-12.9Cr-5.9Ti	6.145	73	1.95	77	3.83
BL-23	V-12.9Cr-5.9Ti	6.145	—	—	77	4.26
BL-24	V-13.5Cr-5.2Ti	6.151	36	0.08	17	0.11
BL-24	V-13.5Cr-5.2Ti	6.151	46	1.17	77	3.83
BL-24	V-13.5Cr-5.2Ti	6.151	73	3.11	77	6.32
BL-25	V-14.4Cr-0.3Ti-0.3Al	6.226	36	0.46	17	0.26
BL-25	V-14.4Cr-0.3Ti-0.3Al	6.226	—	—	77	14.88
BL-26	V-14.1Cr-1.0Ti-0.3Al	6.235	36	0.85	17	0.77
BL-26	V-14.1Cr-1.0Ti-0.3Al	6.235	73	3.40	77	5.78
BL-27	V-3.1Ti-0.3Si	6.039	36	1.97	17	1.79
BL-27	V-3.1Ti-0.3Si	6.039	73	2.59	77	8.12
BL-27	V-3.1Ti-0.3Si	6.039	—	—	77	2.42
BL-28	V-9.0Cr-3.3Fe-1.2Zr	6.261	36	1.18	17	1.24
BL-28	V-9.0Cr-3.3Fe-1.2Zr	6.261	73	2.75	77	6.81
BL-28	V-9.0Cr-3.3Fe-1.2Zr	6.261	—	—	77	9.67
BL-34	V-8.6Ti	5.933	36	1.24	17	0.72
BL-34	V-8.6Ti	5.933	73	1.06	77	3.63
BL-34	V-8.6Ti	5.933	—	—	77	5.42
BL-35	V-9.5Cr-0.2Al	6.214	36	0.45	17	2.72
BL-35	V-9.5Cr-0.2Al	6.214	73	0.94	77	42.40
BL-36	V	6.110	36	1.21	17	0.10
BL-36	V	6.110	73	5.94	77	2.73
BL-48	BL-23 + 20% CW	6.125	36	0.88	17	1.47

The dependence of swelling on Ti concentration for the binary V-Ti alloys on neutron irradiation at 420°C to 36 dpa and 73-77 dpa is shown in Fig. 1. For the purpose of Fig. 1, we consider that the Si content in the V-3Ti-0.3Si alloy (BL-27) did not significantly affect the swelling of binary V-Ti alloys. The swelling of V decreased with the addition of Ti to a concentration of approximately 5 wt %, and was nearly independent of Ti concentration in the range of 5 to 20 wt % Ti.



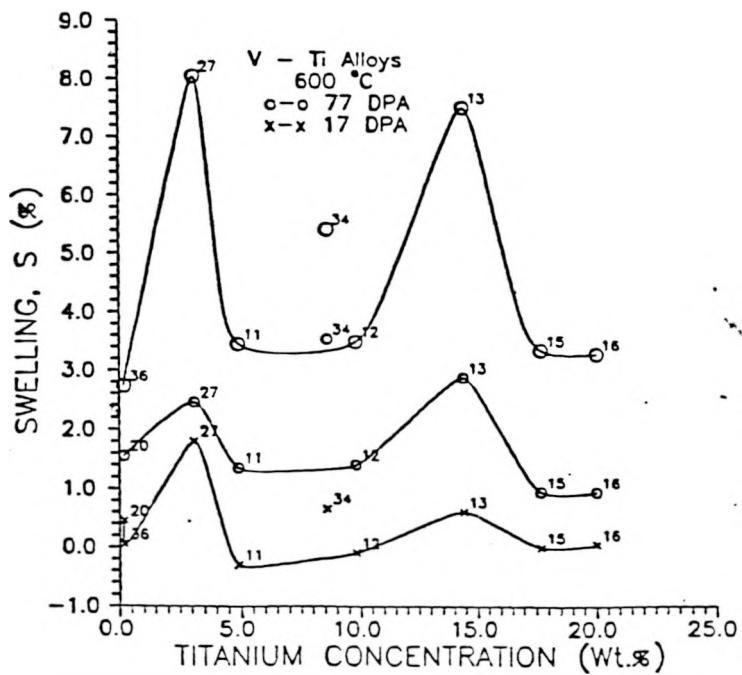


Fig. 2. Swelling of V-Ti alloys on neutron irradiation at 600°C to 17 dpa and 77 dpa (data point numbers refer to ANL I.D. in Table 1).

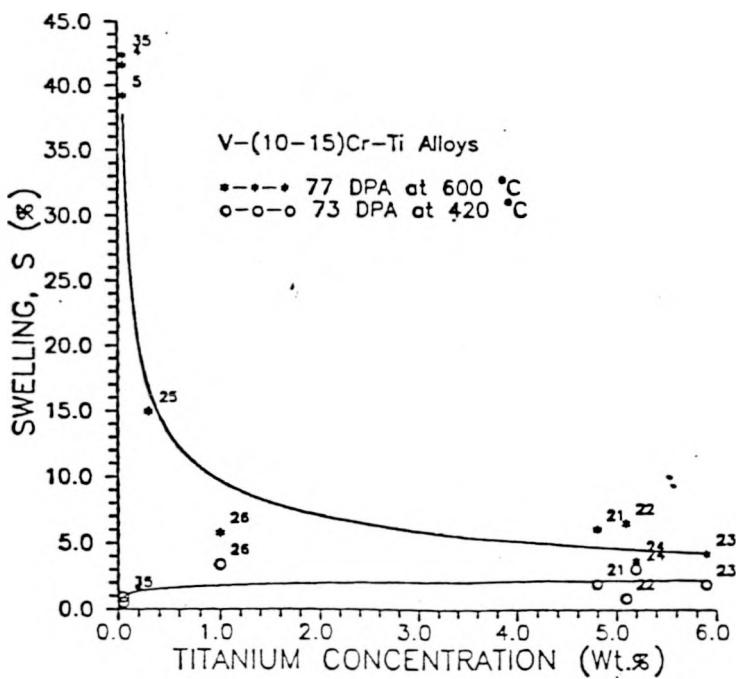


Fig. 3. Swelling of V-(10-15)Cr-Ti alloys on neutron irradiation at 420°C to 73 dpa and at 600°C to 77 dpa (data point numbers refer to ANL I.D. in Table 1).

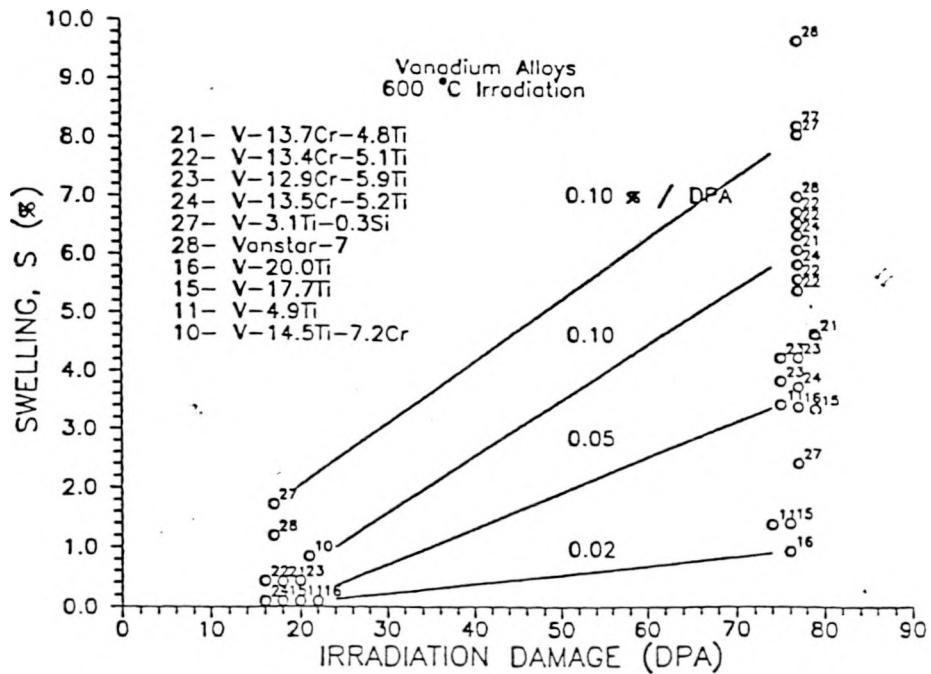


Fig. 4. Swelling of vanadium alloys on neutron irradiation at 600°C to 17-77 dpa.

Discussion of results

The swelling values presented in this report were obtained from density determinations on annealed specimens of unirradiated and irradiated alloys. It has been previously reported that V-(1.5-10.0)Ti alloys containing 1400-5000 ppm oxygen undergo aging on annealing at 550-750°C for 4-7 h.⁹ Since the FFTF-MOTA reactor facility did not achieve full power within 10 h, the irradiated alloy specimens may have undergone significant aging. Therefore, the reference density for the swelling evaluations should have been the density for aged (unirradiated) specimens rather than the density for annealed (unirradiated) specimens. In addition to the aging effects that may have occurred for the specimens that were irradiated at 420 and 600°C, the specimens irradiated at 600°C underwent a temperature excursion of 249°C for 50 min. At the present time, we have no information on the impact of the aging and temperature excursion effects on the swelling of the irradiated alloys. Either or both of these effects may have contributed to the wide range (2-3%) of swelling that was determined for the alloys irradiated at 600°C.

The swelling for V-Ti alloys on irradiation at 600°C showed swelling maxima for alloys containing 3.1 and 14.4 wt % Ti (Fig. 2). The swelling maxima may have been due to the separate and/or combined effects of (1) the temperature excursion, (2) aging of the alloys, (3) phase changes, and (4) phases initially present in the alloys.^{9,10}

CONCLUSIONS

1. The swelling of V-4.9Ti, V-9.8Ti, V-14.4Ti, V-17.7Ti, V-20.0Ti, V-3.1Ti- 0.3Si, Vanstar-7, V-14.5Ti-7.2Cr, and V-13.5Cr-5.2Ti alloys is in the range of 0.01-0.14% per dpa on neutron irradiation at 420°C and 600°C to 77 dpa.
2. The swelling of V at 600°C is increased substantially by the addition of Cr.
3. The swelling of V-Cr alloys at 600°C is significantly reduced by the addition of Ti.

FUTURE WORK

1. The density of V-base alloys will be determined after receiving an aging treatment that is similar to the time-temperature schedule for the start-up of the FFTF-MOTA reactor facility.
2. The swelling of V-base alloys that were irradiated during cycle 10 of the FFTF-MOTA will be determined for the purpose of evaluating the effect of the temperature excursion during cycles 7 and 8 on the swelling of V-base alloys.

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