

## EFFECT OF PREIRRADIATION HEAT TREATMENT ON SWELLING OF NEUTRON-IRRADIATED VANADIUM-BASE ALLOYS\* - B. Loomis, L. J. Nowicki, and D. L. Smith (Argonne National Laboratory)

### OBJECTIVE

The objective of this research is to determine the effect of preirradiation heat treatment on the physical and mechanical properties of neutron-irradiated vanadium-base alloys. These results are expected to show the importance of selecting appropriate preirradiation heat treatment for prime-candidate vanadium alloys that will result in the optimal combination of physical and mechanical properties of the alloy for use as structural material in a fusion reactor.

### SUMMARY

The dependence of swelling of neutron-irradiated V-14Cr-5Ti, V-7Cr-15Ti, V-3Ti-0.3Si, and V-18Ti alloys on preirradiation heat treatment (which consisted of a 1-h annealing at either 850, 950, 1100, 1125, or 1200°C) was determined from density measurements of the alloys after irradiation at either 420, 520, or 600°C to 21-88 dpa. Swelling of the V-14Cr-5Ti alloy was minimal after 1-h annealing at 1125°C, whereas swelling of the V-7Cr-15Ti alloy was minimal after 1-h annealing at 1125 and 1200°C. Swelling of the V-3Ti-0.3Si alloy increased when the annealing temperature was increased from 850°C to 1200°C, and swelling of the V-18Ti alloy was minimal after preirradiation annealing at 1125°C.

### INTRODUCTION

Vanadium-base alloys have been identified as leading candidates for application as first-wall/blanket structural material in a fusion reactor [1-5]. The advantages of the physical and mechanical properties of vanadium-base alloys over other structural materials for a fusion reactor, in addition to their excellent safety and environmental features, have been evaluated in several design studies [1-5]. However, the effects of preirradiation heat treatment on the physical and mechanical properties of irradiated vanadium-base alloys have not been determined for selection of the heat treatment that will result in optimal combination of properties in the anticipated, hostile environment of a fusion reactor.

In a previous report [6], it was shown that the recovery of the microhardness of cold-worked (85%) vanadium-base alloys significantly depends on annealing temperature. Here, we present experimental data on the dependence of swelling of irradiated V-14Cr-5Ti, V-7Cr-15Ti, V-3Ti-0.3Si, and V-18Ti alloys on preirradiation heat treatment. In a future report, we will present experimental data on the dependence of the tensile properties of these irradiated alloys on preirradiation heat treatment.

### MATERIALS AND PROCEDURES

Specimens  $\approx$  3 mm in diameter and 0.3 mm thick were obtained from 50% cold-worked sheets of V-14Cr-5Ti, V-7Cr-15Ti, V-3Ti-0.3Si, and V-18Ti alloys with the compositions listed in Table 1. The cold-worked specimens were annealed at either 850, 950, 1100, 1125, or 1200°C ( $\pm$ 10°C) for 1 h in an ion-pumped vacuum system with a typical pressure of  $1.3 \times 10^{-6}$  Pa. The annealed specimens, irradiated in the Materials Open Test Assembly (MOTA) in the Fast Flux Test Facility (FFTF) reactor, were contained in sealed, Li<sup>7</sup>-filled, TZM molybdenum capsules to prevent contamination from oxygen, nitrogen, and carbon impurities in the sodium coolant of the FFTF. The specimens were irradiated at 420, 520, and 600°C to neutron fluences ( $E > 0.1$  MeV) ranging from  $3.9 \times 10^{22}$  n/cm<sup>2</sup> (21 dpa) to  $1.6 \times 10^{23}$  n/cm<sup>2</sup> (88 dpa) during Cycles 9, 10, and 11 of the FFTF-MOTA facility. 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

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50% ethanol and 50% methanol.

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

Table 1. Composition of Vanadium-Base Alloys.

Alloy	ANL ID	Cr, wt. %	Ti, wt. %	O, wt. ppm	N, wt. ppm	C, wt. ppm	Si, wt. ppm
V-14Cr-5Ti	BL 24	13.5	5.2	1190	360	500	390
V-7Cr-15Ti	BL 10	7.2	14.5	1110	250	400	400
V-3Ti-0.3Si	BL 27	-	3.1	210	310	310	2500
V-18Ti	BL 15	-	17.7	830	160	380	480

## EXPERIMENTAL RESULTS

The dependence of swelling on preirradiation heat treatment temperature of V-14Cr-5Ti alloy after irradiation at 420, 520, and 600°C is shown in Fig. 1. The data show that the swelling of this alloy was substantially reduced by increasing the preirradiation anneal temperature from 850 to 1100-1125°C. Increase of the annealing temperature from 1125 to 1200°C resulted in significant increase of swelling.

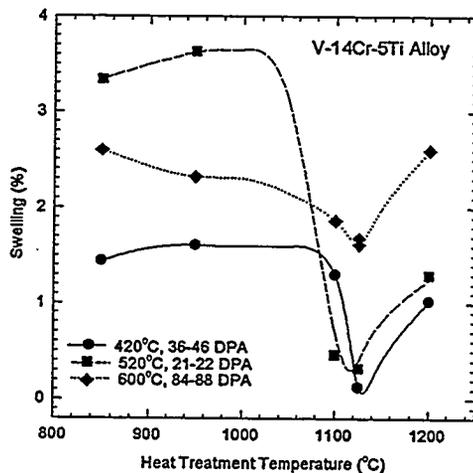


Fig. 1. Dependence of swelling of V-14Cr-5Ti alloy on temperature of pre-irradiation heat treatment.

Figure 2 shows the dependence of swelling on preirradiation heat treatment temperature of V-7Cr-15Ti alloy after irradiation at 420 and 600°C. The swelling of this alloy increased when the heat treatment temperature was increased from 850°C to 1100°C; it then decreased to a minimum when the heat treatment temperature was increased from 1125 to 1200°C.

The extent to which the swelling of V-3Ti-0.3Si alloy after irradiation at 420, 520, and 600°C depended on preirradiation heat treatment temperature is shown in Fig. 3. The swelling of this alloy generally increased with increase of heat treatment temperature from 850°C to 1200°C.

Figure 4 shows the dependence of the swelling on preirradiation heat treatment temperature of the V-18Ti alloy after irradiation at 420, 520, and 600°C. Although, the swelling of this alloy after irradiation at 420 and 520°C decreased significantly when the heat treatment temperature was increased from 850°C to 1125°C, on irradiation at 600°C, it did not strongly depend on preirradiation heat treatment temperature.

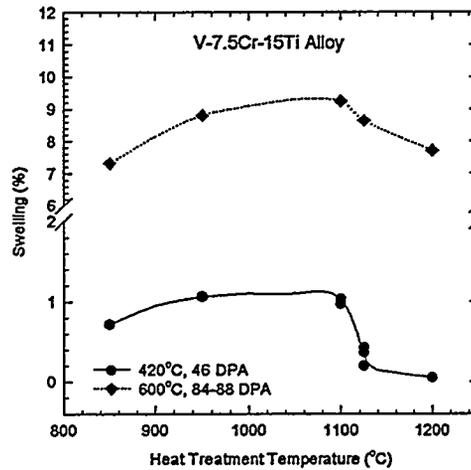


Fig. 2. Dependence of swelling of V-7Cr-15Ti alloy on preirradiation heat treatment temperature.

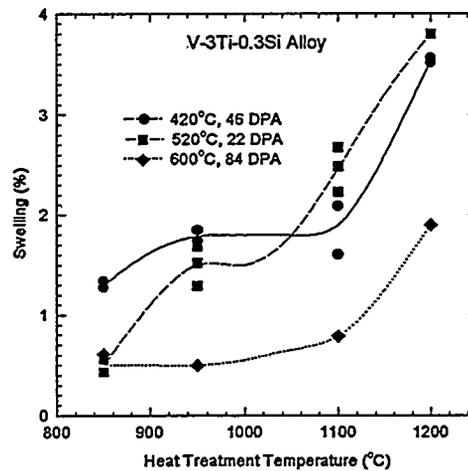


Fig. 3. Dependence of swelling of V-3Ti-0.3Si alloy on preirradiation heat treatment temperature.

## DISCUSSION OF RESULTS

Experimental data presented in Figs. 1-4 show that the swelling of V-14Cr-5Ti, V-7Cr-15Ti, V-3Ti-0.3Si, and V-18Ti alloys strongly depends on preirradiation heat treatment. Therefore, it may be inferred that the swelling of other vanadium-base alloys, e.g., V-(3-5)Cr-(3-5)Ti alloys, which are the current focus of experimental effort, will exhibit a similar strong dependence of swelling on preirradiation heat treatment [7-10].

The microstructures of 50% cold-worked V-14Cr-5Ti, V-7Cr-15Ti, V-3Ti-1Si, and V-18Ti alloys after annealing at 1050 and 1125°C for 1 h have been observed by optical and transmission electron microscopy by Gazda et al. [11]. The annealing conditions resulted in an average recrystallized-grain diameter of 0.02-0.04  $\mu\text{m}$  and a dislocation density of  $\approx 10^{13} \text{ m}^{-2}$ . It may be inferred from these results that a recrystallized microstructure contributed, in part, to the minimal swelling of the irradiated V-14Cr-5Ti, V-7Cr-15Ti, and V-18Ti alloys after a preirradiation heat treatment at 1125°C.

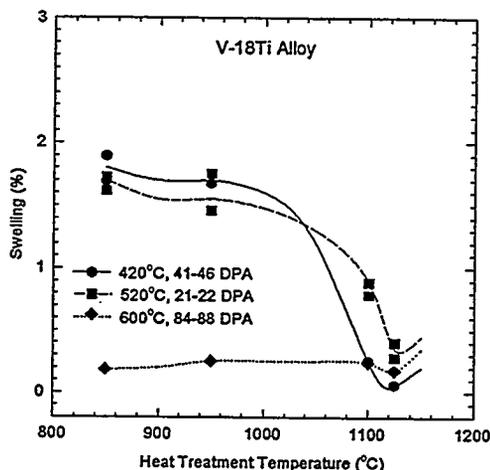


Fig. 4. Dependence of swelling of V-18Ti alloy on preirradiation heat treatment temperature.

However, other factors, e.g., grain growth, precipitate formation and growth, and dislocation structure, also must have contributed to the observed dependence of swelling on preirradiation heat treatment, because swelling of the V-14Cr-5Ti alloy increased when the preirradiation heat treatment temperature was increased to 1200°C, swelling of the V-7Cr-15Ti alloy decreased when the heat treatment temperature was increased to 1200°C, and swelling of the V-3Ti-0.3Si alloys generally increased when the heat treatment temperature was increased from 850°C to 1200°C. Further interpretation of the dependence of swelling of these alloys on preirradiation heat treatment would be suppositious in the absence of a comprehensive series of microstructural observations by optical and transmission electron microscopy.

## FUTURE EFFORT

1. The tensile properties of irradiated V-14Cr-5Ti, V-7Cr-15Ti, V-3Ti-0.3Si, and V-18Ti alloys subjected to different preirradiation heat treatments will be determined.
2. Swelling, tensile properties, and microhardness of the irradiated alloys will be correlated with the microstructures of alloys subjected to different preirradiation heat treatments.

## REFERENCES

- [1]. D. L. Smith, et al., *Fusion Technology*, 8, 10 (1985).
- [2]. D. Ehst, et al., *Tokamak Power System Studies*, Argonne National Laboratory, Report ANL/FPP/86-1 (1986).
- [3]. The ARIES Team, *The ARIES-II Tokamak Reactor Study*, University of California at Los Angeles, Report UCLA-PPG-1461.
- [4]. J. Holdren, et al., "Report of the Senior Committee on Environmental, Safety, and Economic Aspects of Magnetic Fusion Energy," Lawrence Livermore National Laboratory, Report UCRL-53776 (1989).
- [5]. H. K. Birnbaum, et al., "Technical Evaluation of the Technology of Vanadium Alloys for Use as Blanket Structural Materials in Fusion Power Systems," Report DOE/ER-0313/100, August 4, 1993.
- [6]. B. A. Loomis, L. J. Nowicki, and D. L. Smith, "Hardness Recovery of 85% Cold-Worked V-Ti and V-Cr-Ti Alloys upon Annealing at 180°C to 1200°C," *Fusion Reactor Materials Semiannual Progress Report for Period Ending September 30, 1994*, Report DOE/ER-0313/17 (1995).
- [7]. B. A. Loomis, L. J. Nowicki, and D. L. Smith, "Effect of Neutron Irradiation on Tensile Properties of V-Cr-Ti Alloys," *J. Nucl. Mater.* 212-215 (1994) 790-793.
- [8]. B. A. Loomis, H. M. Chung, L. J. Nowicki, and D. L. Smith, "Effects of Neutron Irradiation and Hydrogen on Ductile-Brittle Transition Temperatures of V-Cr-Ti Alloys," *J. Nucl. Mater.* 212-215 (1994) 799-803.
- [9]. H. M. Chung, B. A. Loomis, and D. L. Smith, "Effect of Irradiation Damage and Helium on Swelling and Structure of Vanadium-Base Alloys," *J. Nucl. Mater.* 212-215 (1994) 804-812.
- [10]. H. M. Chung, B. A. Loomis, and D. L. Smith, "Creep Properties of Vanadium-Base Alloys," *J. Nucl. Mater.* 212-215 (1994) 772-777.
- [11]. J. Gazda, B. A. Loomis, L. J. Nowicki, D. L. Smith, and S. Danyluk, "Relationship of Microstructure and Mechanical Properties of V-Cr-Ti Alloys," in: *Fusion Reactor Materials Semiannual Progress Report for Period Ending September 30, 1993*, pp. 232-239 (1994).