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# Tensile properties and fracture behaviour of V–Cr–Ti alloys after neutron irradiation at 330°C

V.A. Kazakov *, V.P. Chakin, Yu.D. Goncharenko

*SSC RF, Research Institute of Atomic Reactors, 433510 Dimitrovgrad, Ulyanovsk Region, Russian Federation*

**Abstract**

Results of irradiation effects in the fast neutron spectrum of the BOR-60 reactor on the mechanical properties and fracture behaviour of vanadium as well as on binary and ternary alloys (including V–4Cr–4Ti and V–5Cr–10Ti) are presented. The irradiation was carried out at 330°C in a $^{7}$Li isotope environment to a damage dose of 18 dpa. It is shown that all alloys without exception experienced severe radiation embrittlement. Most specimens failed without discernible traces of ductile strain at room temperature. At a test temperature of 350°C, ductility was observed but the total elongation, as a rule, did not exceed 1%. The fracture was mixed: transgranular brittle cleavage and ductile dimple rupture. As the test temperature was increased from 20°C to 350°C, the brittle fracture fraction decreased. At 600°C only ductile fracture was observed. The behaviour of V–4Cr–4Ti alloys of American and Russian production is analyzed in the 275–530°C temperature range, where a shift in behaviour from brittle to ductile failure is observed. 

## 1. Introduction

Vanadium base V–Cr–Ti alloys are considered as candidate materials for first wall and blanket applications of fusion reactors using lithium coolants. An optimum combination of the technological availability, strength, ductility, weldability and radiation stability has been considered to be for the alloys of V–(4–5)Cr–(4–5)Ti [1–10]. However, the results following irradiation in the fast reactor, showing extremely small increases in ductile to brittle transition temperatures (DBTT) of −150°C, were obtained to irradiation temperatures between 420°C and 600°C [11–13]. It is, however, expected that irradiation hardening and embrittlement at lower irradiation temperatures will be more substantial. More recently it has been shown that ductile instability occurs in a V–4Cr–4Ti alloy at the room temperature after irradiation at 231–275°C to only 0.5 dpa where DBTT is increased to +150°C [14].

Irradiation of V-base alloys in a fast neutron spectrum is similar to the operating conditions of thermonuclear reactors. In a mixed neutron spectrum, rapid chromium accumulation takes place from the V(n,γ)Cr reaction with thermal neutrons, and the alloy goes out its optimum composition range [15,16].

The objective of the present work was to obtain new data on the influence of irradiation in the fast neutron spectrum of the BOR-60 reactor at the lowest possible temperature of 330°C in a $^{7}$Li environment on changes of the mechanical properties and microstructure of V-base alloys, including the reference alloy V–4Cr–4Ti.

## 2. Experimental

### 2.1. Materials and specimens

Pure vanadium and seven V-base alloys were irradiated. The chemical composition and initial heat treatments of these alloys are presented in Table 1. Besides the principal alloying elements, the alloys contained the following impurities: iron – 0.02–0.1 wt%, silicon – 0.03–0.07%, aluminium – 0.025–0.17%, carbon (0.0005–0.03 wt%), nitrogen (0.0025–0.007%), oxygen (0.02–0.058%), and hydrogen ⩽ 0.001%. The alloys were manufactured as sheets of 0.5 and 0.7 mm thickness. Specimens for

* Corresponding author. Tel.: +7 84235 32021; fax: +7 84235 35648; e-mail: fae@omv.niiar.simbirsk.su.

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Table 1
Alloy chemical compositions and initial heat treatments

| Chemical composition, wt% | Temperature for 1h anneal, °C |
|---|---|
| V | 900 |
| V–3.0Ti | 900 |
| V–3.1Fe | 900 |
| V–6.1Cr | 1100 |
| V–4.4Cr–3.9Ti | 1000 |
| V–6.3Cr–5.2Ti | 1000 |
| V–5Cr–10Ti | 1000 |
| V–5.4Cr–1.1Zr–0.09C | 1100 |

tensile tests with gauge sections of 2 × 15 mm were punched out. The sheets of V–4.4Cr–3.9Ti and V–5Cr–10Ti alloys were produced by SSC RF RIIM (Bochvar Institute).

### 2.2. Irradiation test conditions

Neutron irradiation was carried out in the sealed FUSION-1 capsule in the G-23 position of the 5th Row of the BOR-60 reactor at an averaged temperature of 330°C in a $^{7}$Li environment. The isotopic purity of the $^{7}$Li was 99.86%. Before filling, $^{7}$Li was distilled on a special stand, passing through a porous stainless steel filter with 5–10 μm pores and through a getter trap of Zr-foil. After purification, the nitrogen content in the lithium decreased from 200 to <3 wppm. The carbon content was 30 wppm. In order to reduce the cavity formation during lithium filling, a mechanical vibrator was used and vibration continued for 15 min. The filling process was carried out in a vacuum better than $1 \times 10^{-4}$ Torr at 250–300°C.

The FUSION-1 capsule was irradiated from 27 June 1995 to 7 June 1996 for two cycles. The reactor total energy output was 271 700 MW-h which corresponded to a maximum neutron fluence of $2.7 \times 10^{22}$ n/cm$^2$ ($E$ > 0.1 MeV), or a radiation damage dose of 18 dpa. It must be noted that there were nine reactor shut downs where the sodium temperature was lowered to 210–230°C. Our estimate of the accumulated neutron fluence is $\sim 1 \times 10^{20}$ n/cm$^2$ ($E$ > 0.1 MeV) at 220–250°C.

After the capsule was discharged from the reactor, lithium was melted under diffusion oil in a pan with a heater at 250°C. After cooling to room temperature, the hanger holding the specimens was transferred to an alcohol pan. Dissolution was carried out by periodically changing the alcohol and using an ultrasonic cleaner. The irradiation conditions, including the transmutant accumulation, are presented in more detail in [17].

Tensile tests were performed by means of 1931U machine with displacement control at cross head speed of 1 mm/min (strain rate is $\sim 1 \times 10^{-3}$ s$^{-1}$) in vacuum better than $1 \times 10^{-4}$ Torr. Investigations of the fracture surfaces after tensile tests at 20°C, 350°C and 600°C were made using an REM-101 scanning electron microscope.

## 3. Results

### 3.1. Mechanical properties

Based on mechanical tensile tests at room temperature, it is shown that all alloys, except for V–5Cr–10Ti (where $\delta_{un} = \delta_{tot} = 0.3\%$) and pure vanadium (where $\delta_{un} = \delta_{tot} = 1.3\%$), fracture was completely brittle on the elastic portion of the tensile curve. At a test temperature of 350°C, all irradiated specimens had insignificant ductile strains in the range $\delta_{un} = \delta_{tot} = 0.3$–$1.0\%$, excluding the V–5Cr–10Ti and V–3.1Fe alloys which had $\delta_{tot} = 4.3$–$5.3\%$. At test temperatures of 600°C and 800°C, the ductility of the alloys ranged from 0.8% to 26% depending on chemical composition.

Comparing the alloys as a function of radiation hardening at 350°C (Fig. 1), it can be shown that the value is maximum for V–4.4Cr–3.9Ti and is 310%. Minimum values are for binary V-alloys with chromium, iron and titanium. Comparing V–4.4Cr–3.9Ti to V–5Cr–10Ti, it may be noted that V–5Cr–10Ti is embrittled to a lesser extent under irradiation. It does not become absolutely brittle at room temperature and at 350°C, it has substantial necking ($\delta_{tot} = 4.3\%$) although the uniform elongation differs little from V–4.4Cr–3.9Ti (1.6% against 0.3%). In the initial state, the V–5Cr–10Ti alloy was more strengthened and is less ductile in comparison with the V–4.4Cr–3.9Ti alloy.

In comparison, radiation strengthening of V–4Cr–4Ti alloy at $T_{test} = 350$°C, the relative radiation hardening of heat BL-47 of this alloy is 141% after irradiation in $^{7}$Li environment at a higher temperature of 420°C but to a higher dose of 28–34 dpa [11]. Possibly, the value of radiation hardening for the 500 kg heat 832 665 V–4Cr–4Ti will be higher because in the initial state this material has slightly lower strength in comparison with heat BL-47. This is most likely due to differences in heat treatment. As a rule, radiation hardening is characterized by the density, average size and type of radiation defects. Probably, after irradiation these parameters differ significantly for the Russian and the American alloys of V–4Cr–4Ti type.

### 3.2. Fracture character

The results of fractographic investigations of the fracture surfaces are in good agreement with the tensile response and ductility characteristics.

At room temperature, the fracture surface of V–4.4Cr–3.9Ti and V–5Cr–10Ti corresponds to brittle transgranular failure. The cleavage steps clearly show

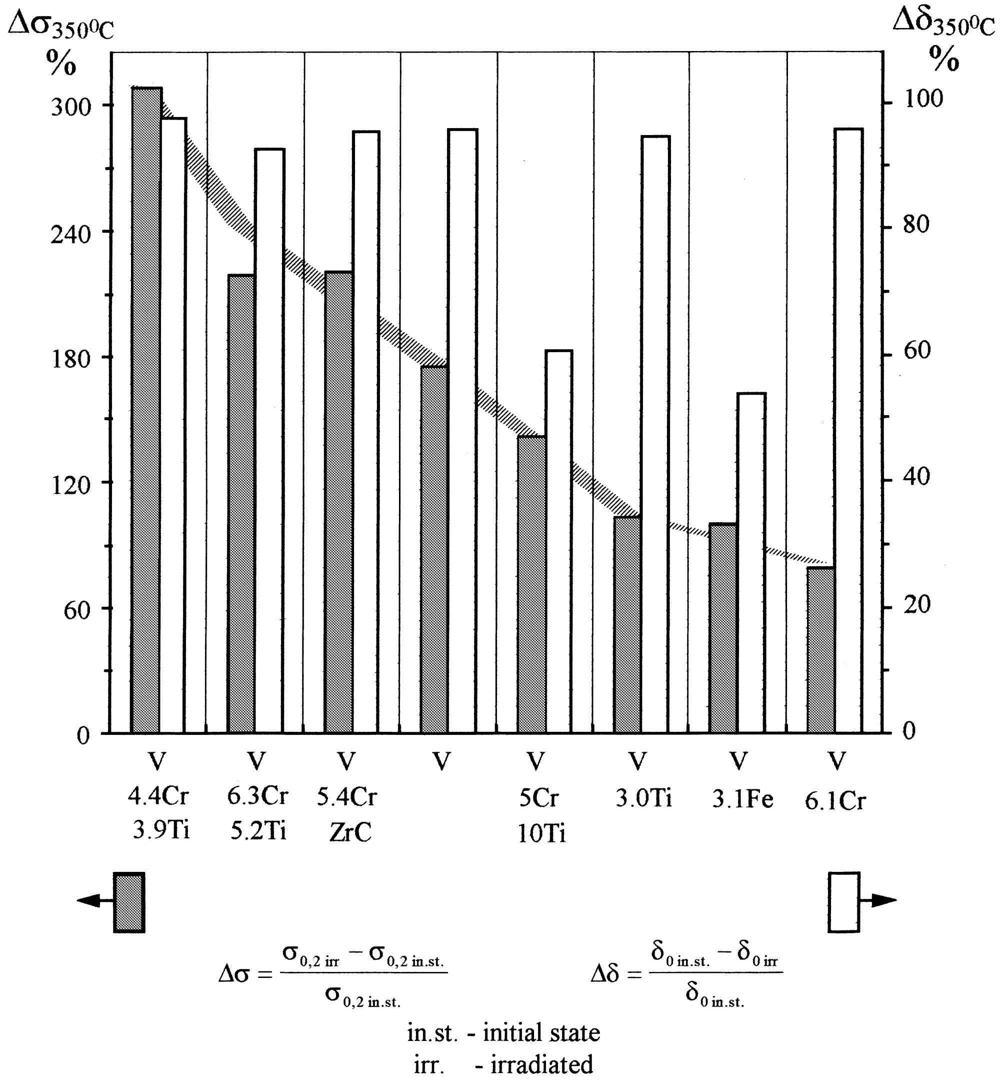


Fig. 1. Radiation hardening and embrittlement of vanadium and some of its alloys following irradiation at 330°C to 18 dpa.

river pattern steps. Between the cleavage facets, there are steps of ductile rupture. The difference between alloys is due to the fact that in the V–4.4Cr–3.9Ti alloy, cleavage steps are seen in facets as river pattern. This shows that cracking can be in a direction perpendicular to the rupture surface. Opened secondary cracks are sometimes seen on grain boundaries.

In contrast to the V–Cr–Ti alloys, the fracture surfaces of vanadium and V–3.0Ti alloys correspond to dimple rupture with low strain capacity. The dimples have different sizes. The rupture of V–3.1Fe and V–6.1Cr alloys show brittle transgranular character.

At a test temperature of 350°C, failure of V–4.4Cr–3.9Ti alloy has a mixed character. The central part of the specimen corresponds to brittle transgranular fracture, but corresponds to ductile dimple rupture with a low strain capacity near surfaces. In the central part of the specimen, besides the cleavage facets with the river pattern, open secondary cracks are found on grain boundaries.

For V–5Cr–10Ti, dimple intergranular fracture is observed before separate grains begin to be strongly deformed. Near grain boundaries are probably more ductile than grain interiors. As a result, rupture occurs in regions of the specimen adjacent to grain boundaries. In pure vanadium, and V–3.0Ti alloy ductile fracture is observed, but as before with low strain capacity.

The rupture character of V–3.1Fe is more intricate. Apparently, this is brittle transgranular fracture with very extensive cracking. The failure of V–6.1Cr alloy is brittle transgranular with well developed river patterns and cracking along grain boundaries. Occasionally, between the cleavage facets, sections of ductile dimple failure are found.

At the test temperature of 600°C, fracture surfaces of all alloys correspond to ductile dimple rupture exclusively. No regions with brittle transgranular fracture were detected.

## 4. Discussion

The presented results are not complete. In the future, microstructural investigations will be performed, including specimens of V–4.4Cr–3.9Ti and V–5Cr–10Ti cut from welds made using electron beam and argon-arc methods.

In Fig. 2, the influence of irradiation on the temperature dependence of mechanical properties for the Russian production of V–4.4Cr–3.9Ti after irradiation at 65°C, 95°C and 330°C is presented. Also, the influence of irradiation under similar conditions for V–2.5Zr–0.35C is shown for comparison. The main conclusion, which follows from this figure, is that V–4.4Cr–3.9Ti after irradiation at 330°C undergoes strong radiation embrittlement, at least up to $T_{test} = 400$°C. The total elongation in the temperature range 20–400°C is less than 1%.

After irradiation at low temperatures, the V–4.4Cr–3.9Ti alloy, as well as the V–4Cr–4Ti from American production, exhibits plastic instability. The only difference is that in the V–4.4Cr–3.9Ti alloy, necking is two times smaller: 3.5–4.5% in comparison with 10% for the V–4Cr–4Ti alloy [16,18,19]. In both cases, the uniform elongation is probably 0.2–0.3% for test temperatures up to 500°C [20].

In the same figure, the temperature dependence of the mechanical properties of V–2.5Zr–0.35C after irradiation in a $^7$Li environment at 410°C to 8 dpa and after irradiation at 150°C in a helium environment to 3.6 dpa are shown [21]. The behaviour of this alloy is analogous. The difference is that the V–4.4Cr–3.9Ti alloy is somewhat stronger in the unirradiated state for $T_{test} = 20$–200°C, and has different temperature dependence for total elongation. After irradiation at 330°C, it is strengthened slightly more, but its high-temperature ductility at $T_{test} \geqslant 600$°C remains below the ductility of the V–Zr–C alloy. Probably, it remains brittle to $T_{test} \geqslant 200$°C.

Comparing the alloys of V–4Cr–4Ti type from Russian and American production, the following can be noted. In both cases, the chemical compositions are not very different and the alloys contain about equal quantities of alloyed and impurity elements (Cr, Ti, O, N, Si). In the unirradiated state, given similar heat treatments of 1000°C for 1 h, the American alloy is 1.5 times stronger at room temperature, and ductility is about 1.5 times greater over the temperature range 20–600°C. It is

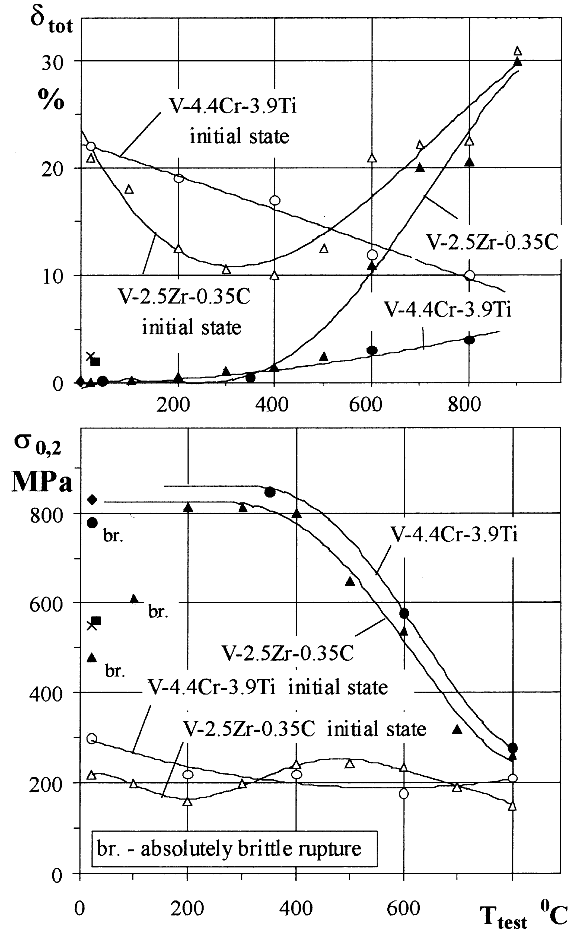


Fig. 2. Irradiation effects on the temperature dependence of yield stress and total elongation of V–4.4Cr–3.9Ti and V–2.5Zr–0.35C alloys.(○) V–4.4Cr–3.9Ti: Initial state, At 1000°C for 1 h. (●) V–4.4Cr–3.9Ti: Irradiation at 330°C in BOR-60 to 18 dpa in $^7$Li. (×) V–4.4Cr–3.9Ti: Irradiation at 65°C in SM to 3.8 dpa in water. (■) V–4.4Cr–3.9Ti: Irradiation at 95°C in SM to 4.0 dpa in He. (△) V–2.5Zr–0.35C: Initial state. At 1600°C for 1 h + 1100°C for 1 h. (▲) V–2.5Zr–0.35C: Irradiation at 410°C in BOR-60 to 8 dpa in $^7$Li. (♦) V–2.5Zr–0.35C: Irradiation at 150°C in SM to 3.6 dpa in He.

not yet clear to what extent this leads to good properties after irradiation in the temperature range 420–600°C. However, after irradiation at 231–275°C, it can be anticipated that V-alloys will undergo embrittlement even after 0.5 dpa.

In the future, it would be very useful to compare the defects produced in V–4Cr–4Ti alloys during irradiation at 420°C and 320–330°C. In the irradiation temperature range of 275–420°C, major changes probably occur in the radiation damage structure to the alloy, causing embrittlement. In V–Zr–C alloy, embrittlement occurs at higher temperatures of 410–530°C. In the temperature range of 530–920°C, the mechanical properties of this alloy depend weakly on the irradiation temperature and damage dose, and the total elongation does not drop below than 8%.

## 5. Conclusions

1. All investigated V-alloys, including V–Cr–Ti alloys, undergo strong radiation embrittlement as a result of irradiation in the BOR-60 reactor at 330°C in a $^{7}$Li environment to 18 dpa. The majority of fracture conditions show absolutely brittle behaviour at room temperature. At $T_{test} = 350$°C, all of the alloys possess some ductility; however the total elongation, as a rule, does not exceed 1%.
2. Scanning electron-microscopic investigations showed that at $T_{test} = 20$°C and 350°C, the fracture appearance of all alloys has intermixed character: brittle transgranular and dimple rupture. At 600°C, failure is entirely by dimple rupture.

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