# EFFECT OF IRRADIATION TEMPERATURE AND STRAIN RATE ON THE MECHANICAL PROPERTIES OF V-4Cr-4Ti IRRADIATED TO LOW DOSES IN FISSION REACTORS

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## OBJECTIVE

The objective of this report is to summarize recent data on the temperature-dependent tensile and fracture toughness properties of V-Cr-Ti alloys irradiated in the HFBR, ATR and BOR-60 fission reactors.

## SUMMARY

Tensile tests performed on irradiated V-(3-6%)Cr-(3-6%)Ti alloys indicate that pronounced hardening and loss of strain hardening capacity occurs for doses of 0.1-20 dpa at irradiation temperatures below ~330°C. The amount of radiation hardening decreases rapidly for irradiation temperatures above 400°C, with a concomitant increase in strain hardening capacity. Low-dose (0.1-0.5 dpa) irradiation shifts the dynamic strain aging regime to higher temperatures and lower strain rates compared to unirradiated specimens. Very low fracture toughness values were observed in miniature disk compact specimens irradiated at 200-320°C to ~1.5-15 dpa and tested at 200°C.

## PROGRESS AND STATUS

### Introduction

Several recent studies have shown that V-4Cr-4Ti alloys exhibit significant radiation hardening and a dramatic decrease in strain hardening capacity following neutron irradiation at temperatures up to ~400°C [1-6]. The radiation hardening produces large increases in the ductile to brittle transition temperature, as measured by Charpy impact testing [1,2,6]. Further work is needed to accurately determine the minimum acceptable operating temperature for vanadium alloys in a fusion reactor environment (currently estimated to be 400°C), which will be determined by radiation hardening/embrittlement considerations. In order to provide additional mechanical properties data, we have recently measured the tensile properties of V-(3-6%)Cr-(3-6%)Ti alloys irradiated in the HFBR, ATR, and BOR-60 reactors at relatively low irradiation temperatures ($\leq$505°C). In addition, a limited number of fracture toughness tests were performed on irradiated V-4Cr-4Ti.

### Experimental Procedure

Tensile measurements were performed on type SS-3 sheet tensile specimens (nominal gage dimensions 0.76 x 1.52 x 7.6 mm) from three recent neutron irradiation experiments. The first set of specimens were fabricated from the 500 kg US fusion program heat of V-4Cr-4Ti (heat 832665) and were irradiated at 160-504°C to doses of 0.1-0.5 dpa in the High Flux Beam Reactor (HFBR) [2]. Most of the HFBR specimens were vacuum annealed at 1000°C for 2 h prior to irradiation. Several specimens were given an alternative heat treatment of 900°C for 2 h prior to the HFBR irradiation. The second set of specimens consisted of V-4Cr-4Ti (heat 832665) and small (14 kg) heats [7] of V-3Cr-3Ti (heat T91), V-6Cr-3Ti (heat T92), and V-6Cr-6Ti (heat T90), all of which were irradiated in the Advanced Test Reactor (ATR) as part of the Gd-shielded ATR-A1 irradiation experiment [6]. All of the tested ATR specimens were vacuum annealed at 1000°C for

2 h prior to irradiation. The specimens were irradiated in ATR-A1 subcapsules AS5, AS6, AS11, or AS12 to doses of 3.0-3.5dpa at calculated temperatures of 205-230 and 4.5-4.7 dpa at 290-295°C. The third set of specimens included V-4Cr-4Ti (heats 832665 and T89), V-3Cr-3Ti (heat T91), V-6Cr-3Ti (heat T92), and V-6Cr-6Ti (heat T90), all of which were irradiated in the Li-bonded BOR-60 Fusion 1 irradiation experiment [8]. The V-4Cr-4Ti tensile specimens were annealed at either 1000°C for 2 h or 1050°C for 1 h prior to irradiation. The small heat tensile specimens (heats T89, T90, T91, T92) were annealed at 1050°C for 2 h prior to irradiation. Tensile specimens fabricated from gas tungsten arc and electron beam welds (fusion zone in the center of the gage length) in the as-welded and vacuum annealed (950°C, 2h) condition were also included in the BOR-60 capsule. The BOR-60 specimens tested in the present study were irradiated at tiers 1-5 of the Fusion 1 capsule at 316-325°C to doses of 15-20 dpa ($2.2\text{-}2.9 \times 10^{26}$ n/m$^2$, E>0.1 MeV). Further details of the specimen preparation and irradiation conditions for the HFIR [2], ATR [6], and BOR-60 [8] capsules are given elsewhere.

Miniature disk compact tension specimens with dimensions 12.5 mm diameter by 4.6 mm thickness (DCT) or 9.6 mm diameter by 3.56 mm thickness (DCT-A) were fabricated from the 500 kg heat of V-4Cr-4Ti in the T-L orientation. The specimens were annealed at 1000°C for 2 h and then fatigue pre-cracked at room temperature and side-grooved 10% of their thickness on each side prior to irradiation in ATR-A1 [6] and BOR-60 Fusion 1 [8] capsules. The ATR-A1 specimens (DCT-A geometry) were irradiated in subcapsules AS3 and AS13 at ~190°C,1.5 dpa and ~250°C, 2.3 dpa, respectively. The BOR-60 specimens (DCT geometry) were irradiated in tier 1 of the Fusion 1 capsule at 316°C to a dose of 15 dpa ($2.2 \times 10^{26}$ n/m$^2$, E>0.1 MeV).

Following irradiation, the tensile specimens were tested in vacuum (<$2.5 \times 10^{-6}$ torr) at the irradiation temperature at constant crosshead speeds ranging from 0.025 to 12.7 mm/minute, which corresponds to initial strain rates of $5.6 \times 10^{-5}$ to 0.028 s$^{-1}$. The lower yield point was recorded as the yield strength in cases where a yield drop was observed. In specimens with very low uniform elongation (<0.5%), the lower yield point was determined by graphical analysis of the load-elongation curve . The irradiated DCT specimens were tested in air under stroke control on a 445 kN capacity servohydraulic test machine in general accordance with ASTM E813-89, Standard Test Method for $J_{1C}$ and ASTM 1152-87, Standard Test Method for Determining J-R Curves. Further details of the DCT test technique are given elsewhere [9].

## Results and Discussion

The tensile properties of the irradiated V-4Cr-4Ti specimens are listed in Tables 1-3. The relatively low irradiation temperatures used for this study caused considerable radiation hardening with an accompanying reduction in strain hardening capacity. The uniform elongation ranged from ~0.1-0.5% in the specimens irradiated at temperatures below 325°C, even for irradiation doses as low as 0.1 dpa. High uniform elongation (~7-11%) was observed in V-4Cr-4Ti irradiated to 0.1 dpa at 390 and 505°C and to 0.5 dpa at 415°C (Table 1). The unirradiated uniform elongations in V-4Cr-4Ti is15-20% at test temperatures of 200-400°C It is worth noting that the fine-grained specimens (900°C/2 h anneal) had 30 to 80 MPa higher irradiated strengths than the coarse-grained (1000°C/2 h) HFBR specimens for irradiation temperatures up to 300°C. As an aside, there was a typing mistake in the HFBR irradiated reduction in area measurements reported in Table 3 of ref. [2]. The reduction in area listed for specimen WH25 (87%) should be deleted, and a value of 78% should be inserted for specimen WH23.

The tensile curves in the specimens irradiated at temperatures below 325°C were characterized by a yield peak at elongations <0.2%, followed by a monotonically decreasing engineering stress with increasing elongation. As shown in Fig. 1 and in previous studies [2], a significant change in the slope of the irradiated load-elongation curve typically occurred at elongations of ~0.5%. The stress at the transition point was recorded as the yield stress (lower yield point), since the 0.2% plastic offset stress was typically equal to the ultimate stress. The V-3Cr-3Ti specimen XC01 irradiated in BOR 60 (Table 3) did not exhibit an observable yield drop/ macroscopic plastic instability, although the strain hardening capacity was very low. The 0.2% plastic offset stress was used as the yield stress in Table 3 for this specimen.

Figure 2 shows the dose dependence of the yield strength in V-4Cr-4Ti irradiated at low temperatures, based on the present study and previous studies by several investigators. The data suggest that the radiation hardening approaches a saturation level for doses of ~5 dpa for irradiation temperatures of ~300 and ~400°C. There are no data available at doses between ~0.5 and 4 dpa to determine if saturation occurs at doses lower than 5 dpa. The data in Fig. 2 suggest that the yield strength decreases slightly with increasing dose above 5 dpa for irradiation temperatures of 400-430°C. However, further studies (particularly at doses of 1-10 dpa) are needed to determine the detailed dose dependence of the radiation hardening. It is possible that the apparent "radiation softening" that occurs at doses above 5 dpa at 400-430°C may be due to the higher irradiation temperatures (425-430°C)of the available data at doses >10 dpa compared to that at 4-10 dpa (400°C). Alternatively, coarsening of the microstructure may be occurring at doses above ~5 dpa which may lead to a reduction in the amount of radiation hardening at high doses.

Table 1. Summary of recent tensile data on V-4Cr-4Ti specimens irradiated in HFBR. The yield strength was determined at 0.2% plastic offset for the specimens irradiated at ≥325°C, whereas the lower yield point (typically occurring at ~0.5% plastic deformation) was used for the low irradiation temperature (<325°C) data marked with an asterisk. The data in italics refer to specimens which were given the alternate heat treatment of 900°C/2h prior to irradiation.

| Specimen ID, dpa, $T_{irr}$ | Test conditions | Yield strength | Ultimate strength | Uniform elongation | Total elongation |
|---|---|---|---|---|---|
| WH28, 0.5dpa, 160°C | 160°C, $5.6x10^{-5}$ $s^{-1}$ | 700 MPa* | 739 MPa[a] | 0.1% | 10.0% |
| WH29, 0.5dpa, 160°C | 160°C, 0.028 $s^{-1}$ | 735 MPa* | 766 MPa[a] | 0.2% | 9.8% |
| WH30, 0.5dpa, 160°C | 160°C, 0.028 $s^{-1}$ | 750 MPa* | 777 MPa[a] | 0.2% | 9.3% |
| ST14, 0.5dpa, 160°C | 160°C, $1.1x10^{-3}$ $s^{-1}$ | 684 MPa* | 752 MPa[a] | 0.1% | 10.0% |
| ST15, 0.5dpa, 160°C | 160°C, $1.1x10^{-3}$ $s^{-1}$ | 672 MPa* | 770 MPa[a] | 0.1% | 9.3% |
| *ST45, 0.5dpa, 160°C* | *160°C,* $1.1x10^{-3}$ $s^{-1}$ | *764 MPa** | *840 MPa*[a] | *0.2%* | *8.7%* |
| ST21, 0.5dpa, 268°C | 270°C, $1.1x10^{-3}$ $s^{-1}$ | 639 MPa* | 706 MPa[a] | 0.1% | 9.3% |
| *ST41, 0.5dpa, 268°C* | *270°C,* $1.1x10^{-3}$ $s^{-1}$ | *707 MPa** | *752 MPa*[a] | *0.1%* | *8.7%* |
| ST23, 0.5dpa, 260°C | 260°C, $1.1x10^{-3}$ $s^{-1}$ | 647 MPa* | 712 MPa[a] | 0.2% | 9.5% |
| *ST42, 0.5dpa, 260°C* | *260°C,* $1.1x10^{-3}$ $s^{-1}$ | *702MPa** | *773 MPa*[a] | *0.1%* | *7.7%* |
| ST27, 0.5dpa, 324°C | 325°C, $1.1x10^{-3}$ $s^{-1}$ | 592 MPa* | 635 MPa[a] | 0.2% | 9.2% |
| *ST43, 0.5dpa, 324°C* | *325°C,* $1.1x10^{-3}$ $s^{-1}$ | *605 MPa* | *605 MPa* | *0.5%* | *9.0%* |
| *ST44, 0.5dpa, 307°C* | *305°C,* $1.1x10^{-3}$ $s^{-1}$ | *597 MPa* | *601 MPa* | *0.7%* | *9.2%* |
| ST33, 0.5dpa, 414°C | 415°C, $1.1x10^{-3}$ $s^{-1}$ | 354 MPa | 449 MPa | 8.2% | 17.8% |
| ST34, 0.5dpa, 414°C | 415°C, $1.1x10^{-3}$ $s^{-1}$ | 352 MPa | 443 MPa | 6.6% | 16.7% |
| *ST49, 0.1dpa, 105°C* | *105°C,* $1.1x10^{-3}$ $s^{-1}$ | *632 MPa** | *669 MPa* | *0.2%* | *9.3%* |
| ST61, 0.1dpa, 256°C | 255°C, $1.1x10^{-3}$ $s^{-1}$ | 491 MPa* | 504 MPa | 0.2% | 11.8% |
| *ST51, 0.1dpa, 256°C* | *255°C,* $1.1x10^{-3}$ $s^{-1}$ | *526 MPa** | *533 MPa* | *0.2%* | *11.3%* |
| ST68, 0.1dpa, 391°C | 390°C, $5.6x10^{-5}$ $s^{-1}$ | 348 MPa | 425 MPa | 11.0% | 22.3% |
| ST69, 0.1dpa, 391°C | 390°C, 0.028 $s^{-1}$ | 348 MPa | 415 MPa | 9.0% | 18.0% |
| ST58, 0.1dpa, 504°C | 505°C, $5.6x10^{-5}$ $s^{-1}$ | 271 MPa | 434 MPa | 9.5% | 19.2% |
| ST75, 0.1dpa, 504°C | 505°C, 0.028 $s^{-1}$ | 281 MPa | 407 MPa | 12.1% | 21.7% |

*lower yield point

[a]upper yield point

Table 2. Summary of tensile test results on vanadium alloys irradiated in the ATR-A1 capsule. All tests were performed at a strain rate of $1.1x10^{-3}$ $s^{-1}$. The yield strength was determined at the lower yield point and the ultimate strength was measured at the upper yield point in all specimens.

| Alloy, ID number | Irradiation conditions | Test temperature (°C) | Yield Strength (MPa) | Ultimate Strength (MPa) | Uniform Elongation (%) | Total Elongation (%) |
|---|---|---|---|---|---|---|
| V-3Cr-3Ti , XC08 | 3.0 dpa, 205°C | 200 | 857 | 894 | 0.1 | 7.1 |
| V-6Cr-3Ti , UC08 | 3.5 dpa, 230°C | 200 | 863 | 906 | 0.15 | 7.0 |
| V-6Cr-6Ti , ZC08 | 3.0 dpa, 205°C | 200 | 943 | 957 | 0.3 | 7.3 |
| V-3Cr-3Ti , XC06 | 4.5 dpa, 290°C | 300 | 775 | 777 | 0.1 | 6.1 |
| V-6Cr-3Ti , UC07 | 4.7 dpa, 295°C | 300 | 882 | 897 | 0.1 | 5.7 |
| V-6Cr-6Ti , ZC06 | 4.7 dpa, 295°C | 300 | 869 | 876 | 0.2 | 8.2 |
| V-6Cr-3Ti , UC06 | 4.7 dpa, 295°C | 200 | 903 | 944 | 0.2 | 5.7 |

Table 3. Summary of tensile test results on vanadium alloys irradiated in BOR-60 ($1.1x10^{-3}$ $s^{-1}$ strain rate). The yield strength was determined at the lower yield point and the ultimate strength was measured at the upper yield point in all base-metal specimens except XC01.

| Alloy, ID number | Irradiation conditions | Test temperature (°C) | Yield Strength (MPa) | Ultimate Strength (MPa) | Uniform Elongation (%) | Total Elongation (%) |
|---|---|---|---|---|---|---|
| V-4Cr-4Ti (1000°C,2h), WE01 | 15 dpa, 316°C | 320 | 949 | 979 | 0.1 | 5.1 |
| V-4Cr-4Ti (1050°C,1h), WE21 | 19 dpa, 323°C | 320 | 962 | 975 | 0.2 | 4.3 |
| V-4Cr-4Ti-.1Si (T89), YC01 | 19 dpa, 323°C | 320 | 977 | 993 | 0.15 | 5.7 |
| V-6Cr-3Ti, UC01* | 19 dpa, 323°C | 320 | 960 | 980 | 0.1 | 6.0 |
| V-3Cr-3Ti, XC01 | 20 dpa, 325°C | 320 | 931 | 935 | 0.3 | 4.0 |
| V-6Cr-6Ti, ZC01* | 20 dpa, 325°C | 320 | 940 | 942 | 0.2 | 5.4 |
| V-4Cr-4Ti (GTA as-welded), WF08 | 18 dpa, 320°C | 320 | 976 | 976 | 0.1 | 0.3 |
| V-4Cr-4Ti (GTA+950°C,2h), WF01 | 18 dpa, 320°C | 320 | (882) | (882) | 0.0 | 0.0 |
| V-4Cr-4Ti (EB+950°C,2h), WF16 | 18 dpa, 320°C | 320 | (329) | (329) | 0.0 | 0.0 |

*the UC and ZC specimens were mislabeled in earlier loading lists [8]; the UC tensile series is V-6Cr-3Ti (heat T-92) and the ZC series is V-6Cr-6Ti (heat T-90)

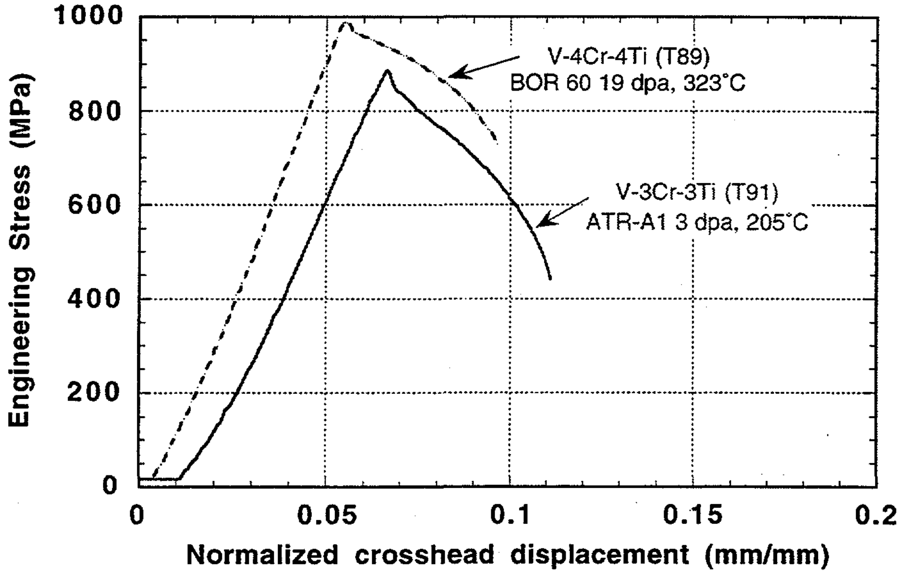


Fig. 1. Typical load-elongation tensile curves for V-Cr-Ti alloys irradiated in the ATR-A1 and BOR-60 Fusion 1 irradiation experiments. The tensile curves have been horizontally offset for clarity.

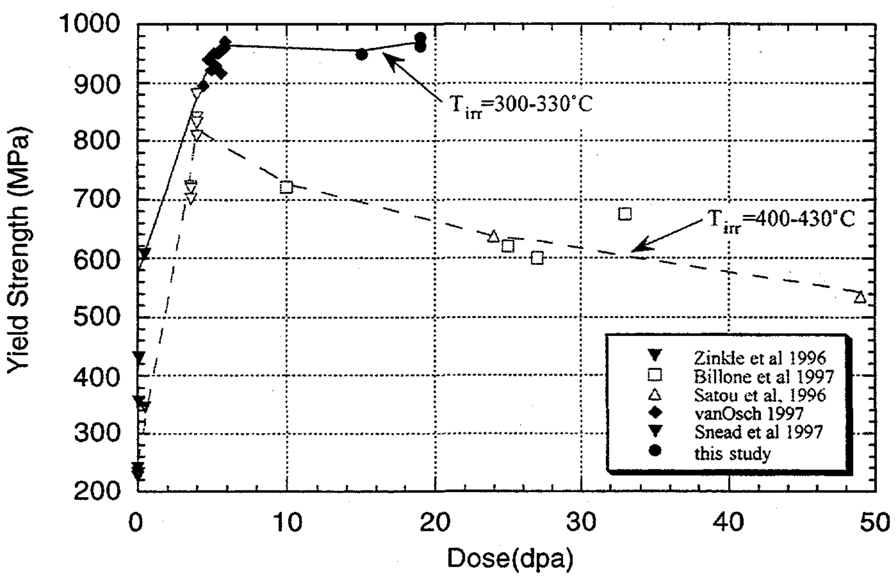


Fig. 2. Dose dependence of radiation hardening in V-(4-5%)Cr-(4-5%)Ti irradiated at low temperatures [1-3,10,11].

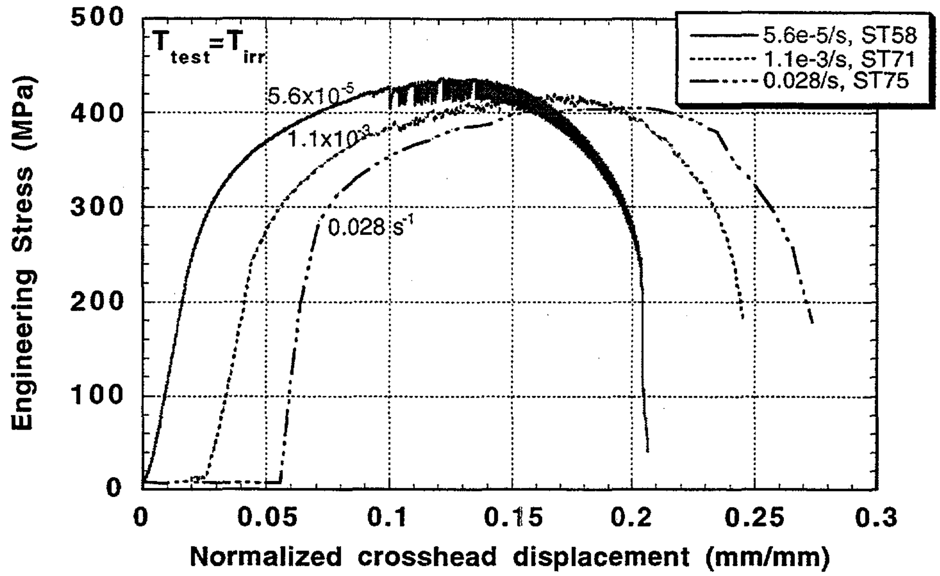


Fig. 3. Load-elongation curves for V-4Cr-4Ti tensile specimens irradiated in HFBR to 0.1 dpa at 504°C and tensile tested at 505°C. The tensile curves have been horizontally offset for clarity. The tensile data for specimen ST71 are tabulated elsewhere [2].

Somewhat lower yield strengths were reported by Kazakov and coworkers [4] for V-4Cr-4Ti irradiated to ~18 dpa at ~330°C in the same Fusion 1 capsule as the present BOR-60 specimens listed in Table 3. Since the total elongation was reported to be nearly zero (<1%, with brittle transgranular or mixed-mode fracture surfaces), the lower strength in their study may be attributable to embrittlement effects. Differences in the impurity content or differences in experimental technique (shoulder loaded Russian tests vs. pin-loaded ORNL tests, etc.) are possible explanations for the difference in tensile behavior. Recently reported tensile data on pin-loaded type SS-3 tensile specimens of V-4Cr-4Ti (heats 832665 and BL47) irradiated in the ATR-A1 capsule [6] are in agreement with the yield strength data shown in Fig. 2.

Serrations in the stress-strain tensile curves were only observed in specimens irradiated and tested at relatively high temperatures (>400°C). Figure 3 shows stress-strain curves for V-4Cr-4Ti irradiated to a dose of 0.1 dpa at 504°C in HFBR. The Portevin-Le Chatelier serrations fell below the general level of the stress-strain curve at the lowest strain rate ($5.6 \times 10^{-5}$ $s^{-1}$) investigated (Type C behavior [12]), whereas the serrations oscillated about the mean level of the stress-strain curve at higher strain rates (Type B behavior [12]). A similar transition from Type C to Type B serrations was observed in unirradiated V-4Cr-4Ti tested at 500°C at the same strain rates [12-14]. The amplitude of the oscillations decreased with increasing strain rate in both the unirradiated and irradiated specimens.

Previous work on unirradiated [12-14] and irradiated [14] V-4Cr-4Ti has found that the tensile strength increases with increasing strain rate at low temperatures and decreases with increasing strain rate at high temperatures (where dynamic strain aging is observed). The transition from a negative to a positive strain rate dependence occurs at 300°C in unirradiated V-4Cr-4Ti, with negative strain rate exponents observed at strain rates below $1 \times 10^{-3}\ s^{-1}$ and positive strain rate exponents observed at strain rates above $1 \times 10^{-3}\ s^{-1}$. The measured value of the hardening strain rate exponent in the HFBR specimens irradiated to 0.1 dpa at 504°C (Fig. 3) is m=-0.010, where m is defined by [15]

$$m = \frac{1}{\sigma} \frac{\partial \sigma}{\partial \ln \dot{\varepsilon}} \qquad (1)$$

where $\sigma$ is the stress and $\dot{\varepsilon}$ is the strain rate. This strain rate exponent is of slightly lower magnitude than that observed in unirradiated V-4Cr-4Ti specimens (m~-0.014 at 500°C). Previous work found that the strain rate exponent was reduced to m= -0.004 at 400°C following neutron irradiation to 4 dpa at 400°C, compared to the unirradiated value of m=-0.013 at 400°C [14]. The presence of a negative strain rate exponent is an indication of a significant interstitial solute concentration in the matrix which is free to migrate to dislocations during tensile testing. This implies that some of the C, O, N solute in the irradiated V-Cr-Ti alloys remains dissolved in the matrix and is not contained in titanium oxycarbonitride precipitates or solute-point defect clusters produced during irradiation [16], although the concentration of free interstitial solute is lower in irradiated specimens compared to unirradiated specimens. Since defect clusters introduce a weakly positive component to the strain rate exponent [17], the presence of defect clusters may be partially responsible for the reduced magnitude of the negative strain rate exponent in irradiated V-4Cr-4Ti specimens compared to unirradiated material. Interstitial solute bound to defect clusters or small precipitates would also decrease the magnitude of the irradiated strain rate exponent at 400-500°C compared to unirradiated values.

Table 4 summarizes the fracture toughness data obtained on V-4Cr-4Ti disk compact tension specimens irradiated in the ATR-A1 and BOR-60 Fusion 1 capsules. The result from an unirradiated BOR-60 control specimen is also included in Table 1 for purposes of comparison. Although the fracture toughness measured on the unirradiated control specimen does not meet the ASTM validity criteria, the high value is indicative of a high toughness. Similar high toughness values have been reported on larger unirradiated V-4Cr-4Ti specimens [18]. All of the irradiated specimens exhibited very low fracture toughness compared to unirradiated control specimens. Sample QA05 broke during preloading at an applied load of 14 pounds. The other two irradiated samples broke during the first three cycles of testing.

Table 4. Fracture toughness measured on irradiated and unirradiated disk compact tension specimens of V-4Cr-4Ti.

| Sample ID | Irradiation conditions | Test temperature (°C) | $K_J$ (MPa-m$^{1/2}$) |
|---|---|---|---|
| QA05 (ATR-A1) | 190°C, 1.5 dpa | 200 | (~3) |
| QA01(ATR-A1) | 250°C, 2.3 dpa | 200 | 30 |
| WC01(BOR-60) | 316°C, 15 dpa | 200 | 32 |
| WC12 | unirradiated | 20 | 233 |

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