COMPRESSION TESTING OF V-4Cr-4Ti - M. B. Toloczko (Pacific Northwest National Laboratory)[1], R. M. Ermi[1], D. S. Gelles[1], R. J. Kurtz[1]

OBJECTIVE

The objective of this effort is to better understand the deformation behavior of vanadium alloys after irradiation.

ABSTRACT

The NIFS-1 heat and heat 832665 of V-4Cr-4Ti, irradiated to 3.7 dpa at 425°C and tested in compression at temperatures of 25°C and ~420°C, were compared to compression tests at similar temperatures on unirradiated material. The yield strength increased by a factor of two, and the upper/lower yield point that was observed in the unirradiated material was not present in the irradiated material test traces. The strain hardening exponent of the irradiated material was 40-80% less than the unirradiated material. Transmission electron microscopy observations indicate the formation of a fine distribution of small defects and no dislocation channeling.

PROGRESS AND STATUS

Introduction

Vanadium alloys are of interest as potential first wall structural materials because of their good thermal conductivity, good elevated temperature tensile strength, good high temperature creep resistance, and relative resilience to becoming radioactive [1-12]. At low irradiation temperatures vanadium alloys experience very high hardening caused by a high density of small, but shearable defect clusters, that results in a type of deformation called "channel deformation" [3,4,9,12-15]. At the onset of yield in a tensile test, a dislocation may move through a grain shearing the obstacles and clearing out a channel. Subsequent dislocations may easily pass through this channel. As the test progresses, more channels form. Up to the point of tensile instability plastic deformation is confined to these channels. One important macroscopic result of this deformation behavior is the rapid onset of necking in a tensile test and very low uniform elongation. As a means to help understand the range of stress states where localized deformation may adversely affect macroscopic ductility in vanadium alloys, compression test specimens fabricated from two heats of V-4Cr-4Ti were irradiated in the High Flux Isotope Reactor (HFIR). The results of 25°C, ~250°C, and ~420°C compression tests on the unirradiated control materials and the irradiated materials are presented here and compared with uniaxial tensile values as possible.

Experimental Procedure

Cylindrical compression specimens were fabricated from V-4Cr-4Ti heats 832665 and NIFS-1. Heat 832665 is reported to have an oxygen content of 330 wppm, and the NIFS-1 heat is reported to have an oxygen content of 181 wppm [2]. The cylindrical specimens are 3 mm in diameter and 3.5 mm tall. Heat 832665 was received in a 40% cold-worked condition, while the NIFS-1 heat was received in a 98% cold-worked condition. Before testing, individual specimens from both heats were wrapped in tantalum and titanium foil and annealed in a vacuum furnace for 2 hours at 1000^{o}C at 1x10^{-6} torr or better. Identification codes were laser engraved onto one end of each specimen.

Compression tests were performed in a 10,000 lb screw-driven Instron test frame with either a 1000 lb or 2000 lb load cell. A special compression test fixture, as shown in Figure 1, was constructed for the testing. The upper and lower loading surfaces of the fixture were made from a high modulus tungsten carbide composite with a polished surface. The upper loading surface is in the form of a piston that is guided by a cylinder made from machineable carbide composite. A tight tolerance was maintained between the piston and cylinder to limit axial misalignment between the upper and lower loading surfaces. Silicon powder was used as a lubricant on upper

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and lower loading surfaces. Specimen displacement was monitored with a capacitance-type displacement transducer with a resolution better than 0.0002 mm (better than 0.006% strain).

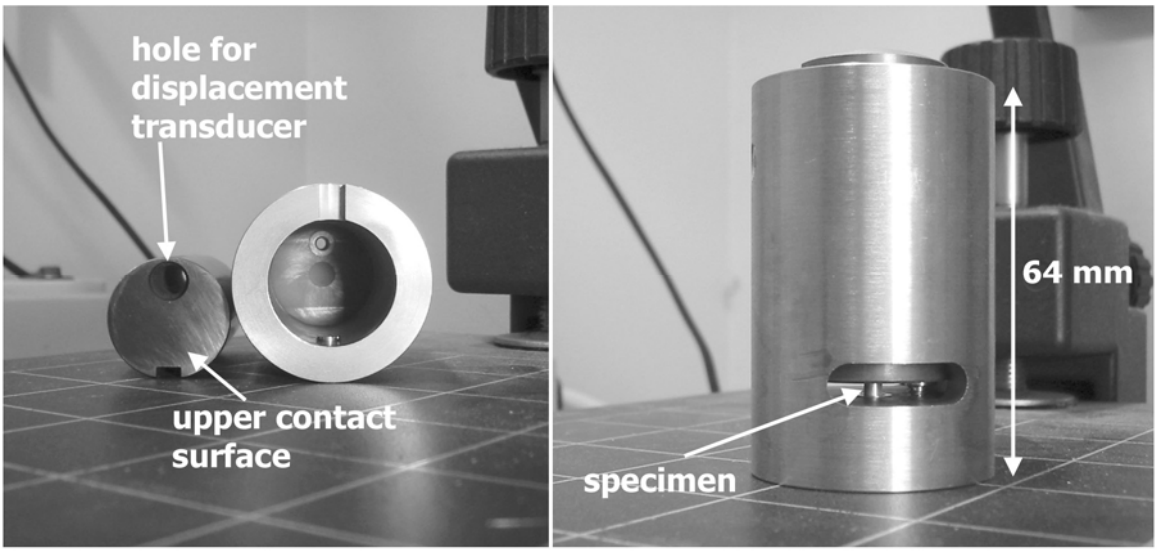

Figure 1. Pictures of the compression test fixture.

Compression tests were performed at constant crosshead speed and with an initial strain rate of $5x10^{-4}$ s^{-1}. The target test temperatures were room temperature (~25°C), 250°C, and 425°C. Heating was performed in a furnace capable of operation in an inert atmosphere or in a vacuum. Tests at 250°C were performed in 99.99% purity argon flowing at a rate of 2 L/min, and tests at 425°C were performed in a vacuum at 0.150 torr in an attempt to lower the partial pressure of oxygen during testing. Prior to testing, a thermocouple was placed below a dummy specimen in the fixture and a series of heating runs at different heating rates and target temperatures were performed to establish a correlation between specimen temperature and test chamber temperature. Preheating of the test chamber prior to the start of a test was done as quickly as possible (5.1°C/min for tests at 250°C and 9°C/min for tests at 425°C), and tests were performed while the temperature of the specimen was still changing, but at a rate where the increase in temperature during a test was limited to about 3°C. All data were monitored and recorded electronically. The 0.2% offset yield stress and, when present, the upper yield point were measured from engineering stress versus engineering strain plots. The power law strain hardening (PLSH) exponent was measured from true stress versus true plastic strain plots in the range of 1-3% true plastic strain.

Results

The actual test temperatures were 25°C, 250-255°C (unirradiated only), and 405-435°C. Engineering stress versus engineering strain curves for heat 832665 and the NIFS-1 heat in the unirradiated condition are shown in Figure 2. In most all of the tests, an upper and lower yield point was observed. The yield stress at 250°C was approximately 30% lower than at room temperature. The yield stress at ~415°C is essentially the same as at 250°C. Heat 832665 is consistently stronger than Heat NIFS-1 at all temperatures. All samples showed a continuous load increase during plastic deformation with a negative curvature up to the load limit of the load cell. Serrations from dynamic strain aging occurred only in CA06 (Heat 832665 tested at 430°C). As is common in a compression test, some barreling of the samples occurred. A typical amount of barreling for an unirradiated specimen is shown in Figure 3.

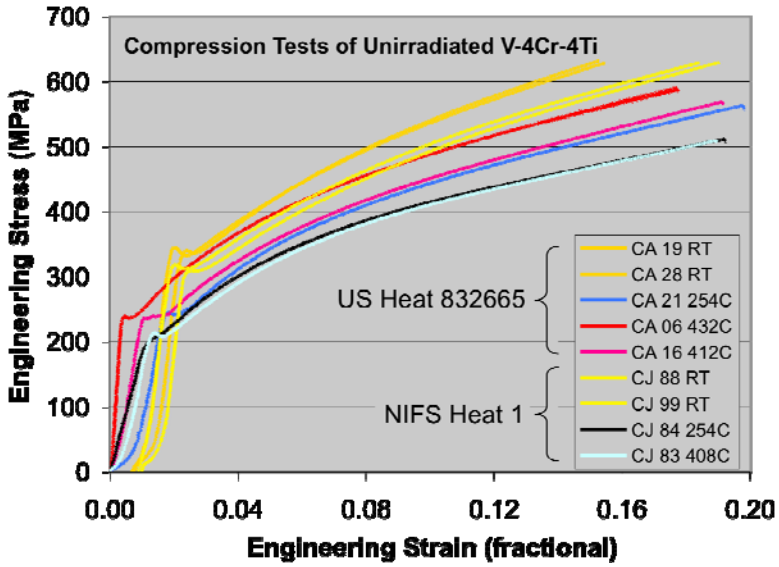

Figure 2. Engineering stress vs. engineering strain curves for unirradiated V-4Cr-4Ti at 25°C, 250°C, and ~420°C**.**

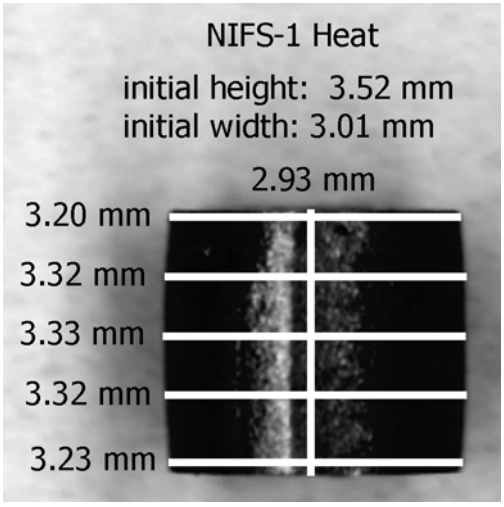

Figure 3. Typical amount of barreling in an unirradiated specimen tested to 20% engineering strain.

Engineering stress versus engineering strain curves for heat 832665 and the NIFS-1 heat in the irradiated condition are shown in Figure 4. The effects of irradiation are to raise the yield strength, eliminate the upper/lower yield point, and to change the strain hardening rate of curvature from negative to slightly positive. Strain serrations are apparent in both of the NIFS-1 specimens (CJ87, CJ89) tested at 425°C.

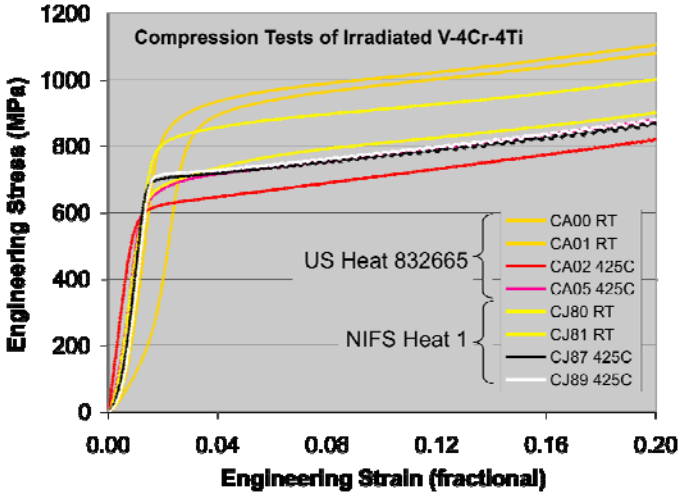

Figure 4. Engineering stress vs. engineering strain curves for irradiated V-4Cr-4Ti at 25°C and ~425°C**.**

Table 1 shows the average test temperature, 0.2% offset yield stress, upper yield point, and PLSH exponent. All values except temperature were calculated by hand. The upper yield point and 0.2% offset yield point were within 4 MPa for all tests where a yield point was observed. The 0.2% offset yield stress of the unirradiated heat 832665 samples remained approximately 22 MPa higher than the unirradiated NIFS-1 samples at all test temperatures as shown in Figure 5. The irradiated specimens have about twice the yield strength of their unirradiated counterparts. Strain hardening exponents were calculated from 0.01 to 0.03 true plastic strain following the reasoning from [16] and by assuming that V-4Cr-4Ti follows the power law strain hardening equation ($\sigma=k\varepsilon^n$). Figure 6 shows the strain hardening exponent versus temperature. Heat 832665 and the NIFS-1 heat both show similar deformation properties in either the unirradiated or irradiated condition with the effect of irradiation being to decrease the estimated strain hardening exponent by 40-80%.

Table 1. Compression test properties of unirradiated V-4Cr-4Ti at 25°C, 250°C, and ~420°C and irradiated V-4Cr-4Ti at 25°C and 425°C. CA = 832665 Heat and CJ = NIFS-1 Heat.

Specimen ID	Average Test Temp (°C)	0.2% Offset Yield Stress (MPa)	Upper Yield Point (MPa)	PLSH Exponent from $0.01 \leq \varepsilon_{pl} \leq 0.03$
Unirradiated				
CA·19	RT	340	340	0.17
CA·28	RT	344	344	0.16
CA·21	254	244	245	0.30
CA·06	432	236	240	0.25
CA·16	412	235	235	0.28
CJ·88	RT	310	310	0.16
CJ·99	RT	315	315	0.17
CJ·84	254	222	None	0.28
CJ·83	408	213	214	0.34
Irradiated·to·3.7·dpa·at~425°C				
CA00	RT	814	None	0.13
CA01	RT	820	None	0.09
CA02	425	570	None	0.06
CA05	425	623	None	0.08
CJ80	RT	632	None	0.11
CJ81	RT	771	None	0.07
CJ87	425	690	None	0.05
CJ89	425	705	None	0.05

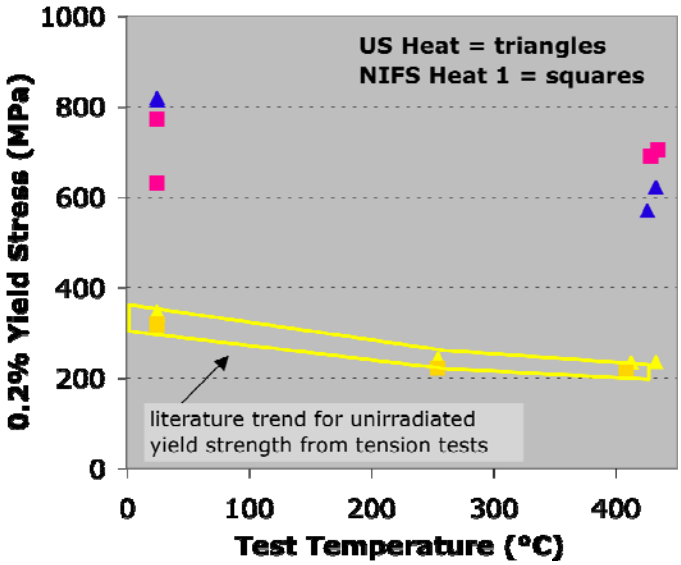

Figure 5. Temperature dependence at 25°C, 250°C, and ~425°C of 0.2% offset yield stress for unirradiated and irradiated V-4Cr-4Ti. Properties of the irradiated materials are blue and red symbols.

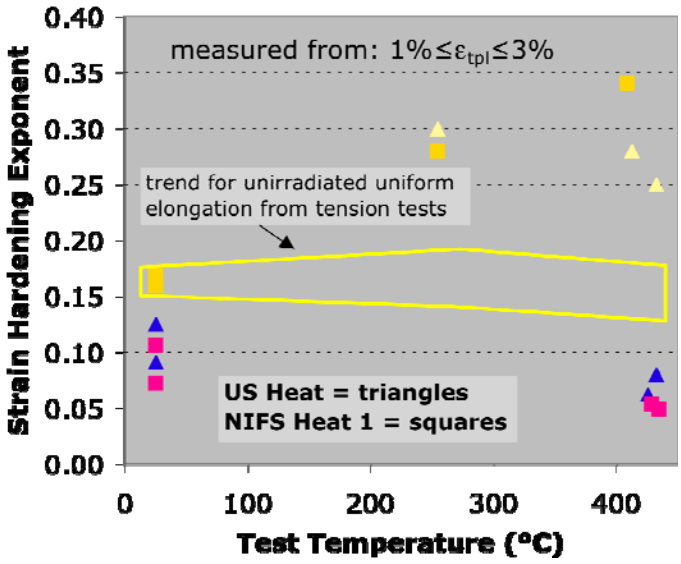

Figure 6. Temperature dependence at 25°C, 250°C, and ~425°C of the strain hardening exponent for unirradiated and irradiated V-4Cr-4Ti. Properties of the irradiated materials are blue and red symbols.

Discussion

Tests on Unirradiated Specimens

Yield stress values from the literature for uniaxial tensile tests on unirradiated heat 832665 are 315-355 MPa at 25°C, 220-260 MPa at 250°C, and 195-235 MPa at 400°C [8,9,11,12]. Unirradiated NIFS-1 yield stress values in tension were not found, but the NIFS-2 heat has a yield strength of around 300 MPa at 25°C [17]. Yield stress values from unirradiated heat 832665 and the NIFS-1 heat measured in compression fall within the range of values in the literature for these materials in tension as expected (Fig. 5) because polycrystalline vanadium with a random grain orientation should have isotropic deformation properties. Figure 5 shows that the elevated temperature yield stress in compression is about 30% lower than the room temperatureyield stress in

compression. Heat 832665 may be consistently stronger than the NIFS-1 heat due to heat 832665 having higher oxygen content [7, 18]. Strain serrations as exhibited in CA06 (heat 832665) are seen in uniaxial tensile data from 300-750°C [9], but were not seen in some other published V-4Cr-4Ti tensile traces [12]. Dynamic strain aging causes the strain serrations.

Necking does not occur in compression tests, so uniform elongation cannot be measured to compare with tensile data. The PLSH exponent, however, can be used to compare deformation behavior in a compression test to deformation behavior in a tensile test. If there is a sufficient amount of plastic strain during the test where the deformation along the length of the compression test specimen is uniform (i.e., minimal barreling for some part of the plastic deformation during the compression test), and if the true stress versus true plastic strain data fit well to the PLSH equation, then the PLSH exponent should be equal to the true uniform elongation (TUE) measured from an equivalent tensile test. The TUE from tensile data can be calculated from the engineering uniform elongation values in the literature using $\varepsilon_{UE}=\ln(e_{UE}+1)$ where ε_{UE} is the true uniform elongation and e_{UE} is the engineering uniform elongation. Literature values of tensile uniform elongation for heat 832665 are 0.14-0.20 at 25°C and 250°C, and 0.13-0.18 at 400°C [8,11,12]. Literature values for NIFS-1 are 0.18-0.2 for temperatures between 25°C and 400°C [19]. The TUE values are thus 0.13-0.18 at 250°C and 0.12-0.17 at 400°C for heat 832665 and approximately 0.18 for the NIFS-1 heat between 25°C and 400°C. A comparison of the measured PLSH exponent values from the compression tests with literature values of TUE from tensile tests are showing in Figure 6. For the 25°C compression tests, the PLSH exponent ranged from 0.16-0.18 which is in good agreement with the TUE values calculated from tensile data in the literature. For the compression tests at elevated temperature, the PLSH values were considerably higher than the TUE values calculated from tensile data in the literature. The reason for this is not clear and will be investigated further.

Tests on Irradiated Specimens

The goal of the research was to look at ductility response in compression for a material undergoing channel deformation. The irradiation temperature for this experiment is near the upper limit to create a microstructure that promotes channel deformation. As yet unreported tensile data from this same experiment show very high strength and low ductility which are strong indicators that channel deformation occurred during those tensile tests. A similar large increase in strength and concurrent reduction in strain hardening exponent in the compression tests are also suggestive of the possibility that the irradiation temperature may have been low enough to cause the formation of a microstructure in these compression specimens that allows channel deformation to occur.

It is interesting to use compression specimens to study channel deformation because it is thought that a specimen that undergoes channel deformation when tested under compression would have better measured ductility than a material undergoing channel deformation when tested in tension. The reason is that the induced increase in cross sectional area during a compression test (geometric hardening) in regions where channel deformation is occurring could make up for the softening that occurs in the channel deformation regions.Transmission electron microscopy was performed on selected untested, partially tested, and fully tested specimens in both the unirradiated and irradiated condition to observe the effect of irradiation on microstructure [20]. Unirradiated specimens were found to have a very low density of observable precipitates while the irradiated specimens had a high density of small defects as shown in Figures 7 and 8, respectively. The defects in the irradiated material may be a combination small dislocation loops, precipitates, and network dislocations, , but of greater interest is the lack of any clear dislocation channels. Figures 8 and 9 show the microstructure of two irradiated specimens tested to 2% and 20% plastic strain, respectively, at room temperature. Neither micrograph shows any signs of channel deformation. There are several possible reasons for a lack of channel deformation. The first is that it may be possible that the irradiation temperature was too high, resulting in a microstructure that does not allow channel deformation to occur. Alternatively, it may be possible that the deformation induced during a compression test may not allow channel deformation. More microscopy, compression tests, and examination of V-4Cr-4Ti tensile specimens from the same irradiation experiment will be needed to determine why no channel deformation was observed here.

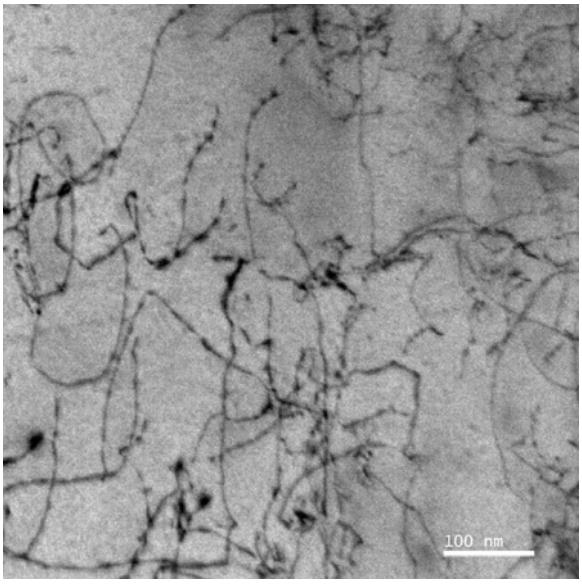

Figure 7. Microstructure observed in an unirradiated V-4Cr-4Ti compression specimen.

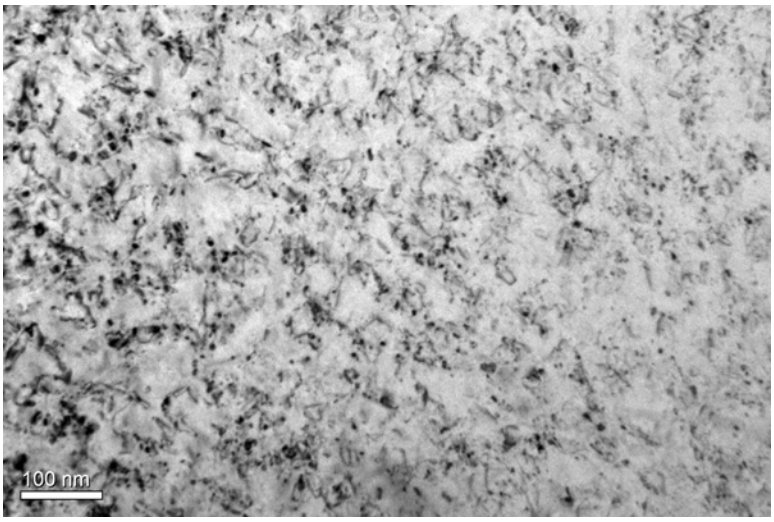

Figure 8. Dislocation and precipitate microstructure of a specimen tested in compression to 2% strain. No deformation channels are present.

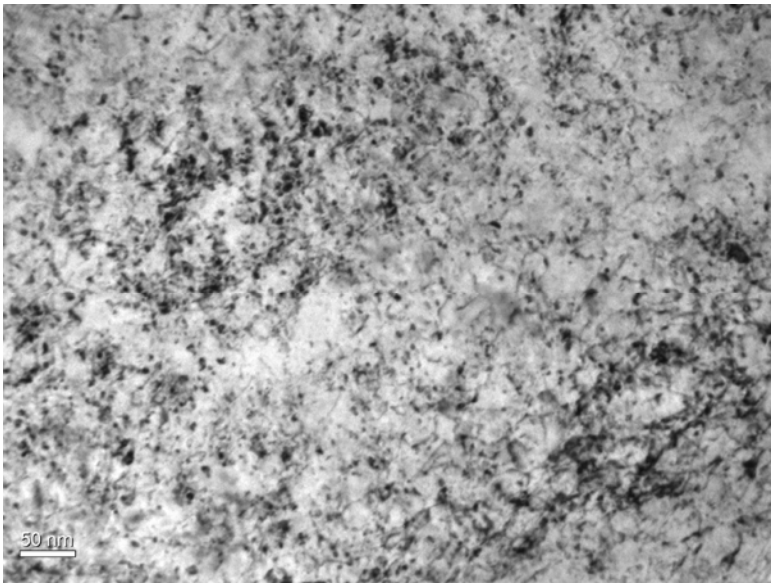

Figure 9. Dislocation and precipitate microstructure of a specimen tested in compression to 20% strain. No deformation channels are present.

Conclusions

Compression tests were performed on unirradiated V-4Cr-4Ti as part of a larger program to better understand the deformation behavior of irradiated V-4Cr-4Ti after irradiation. The effect of irradiation at 425°C on the compression test specimens was to cause a large increase in yield strength and concurrent strong reduction in strain hardening exponent which are both indicators of the possibility that channel deformation is occurring. However, microstructural examinations did not reveal the presence of channel deformation. Several reasons for this are possible, and further examination of compression and tensile specimens are needed to fully understand the response of the material under compression.

Future Work

To better understand the deformation response under compression, several actions are planned which include compression testing of irradiated specimens at 250°C, more microstructural studies of tested compression specimens and tested companion tensile specimens from the same irradiation experiment.

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