MICROSTRUCTURAL EXAMINATION OF IRRADIATED V-4CR-4TI PRESSURIZED CREEP TUBES - D. S. Gelles (Pacific Northwest National Laboratory)*

OBJECTIVE

The objective of this effort is to provide further understanding of processes controlling irradiation creep in vanadium alloys.

SUMMARY

Three pressurized tubes of V-4Cr-4Ti have been examined to determine microstructural development due to irradiation creep following irradiation in ATR at 300ºC.

PROGRESS AND STATUS

Introduction

Application of vanadium alloys such as V-4Cr-4Ti for fusion requires understanding of irradiation creep response in order to optimize reactor design. A number of experiments are underway in order to provide that data,[1] but the nature of irradiation creep experiments limits the number of data points that can be accumulated. Generally, this is done by irradiating pressurized tubes and determining the change in tube diameter as a function of irradiation dose. However, recent experiments do not allow reirradiation of specimens and therefore a diameter change measurement includes effects of primary creep, steady state creep, and perhaps, tertiary creep, as well as effects of swelling and densification due to precipitation. It is therefore beneficial to characterize the microstructure and to measure densification and swelling response in order to better assess pressurized tube response. This report is intended to provide microstructural examinations of pressurized tubes of V-4Cr-4Ti heat 832665 following irradiation in the Advanced Test Reactor (ATR), Idaho Falls, at ~300ºC to ~5 dpa. The creep response of pressurized tubes in the ATR experiment is shown in Figure 1. In order to optimize microstructural information obtained from this study, specimens irradiated at the higher temperature were selected for examination. The present study continues work on similar tubes following thermal creep deformation.[2]

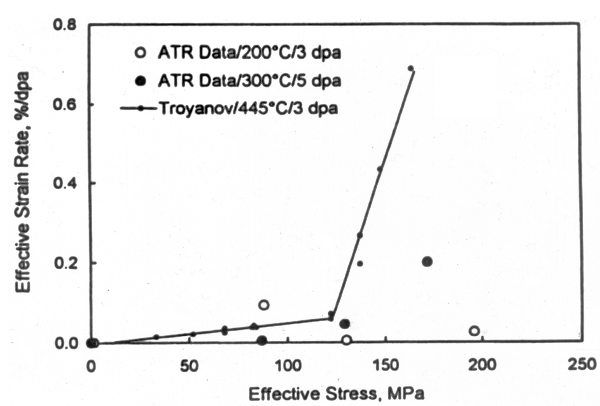

Figure 1. Effective strain rate for ATR irradiated specimens in comparison with torsional creep tests by Troyanov.[1]

Experimental Procedure

Details for the pressurized tubes examined in this study are provided in Table 1. Ring sections of tubing were sectioned and punched to produce 3 mm curved disks. Disks were thinned by grinding but evidence of the curvature was retained in order to be able to orient the microstructure relative to the stress state. Disks were then electropolished

*Pacific Northwest National Laboratory (PNNL) is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO-1830.

at low temperature using standard techniques to produce thin foils. Each disk was mounted in the microscope so that images could be related to the tube orientation and therefore the state of stress could be related to the microstructure. Dislocation imaging involved procedures that allowed identification of all $\frac{a}{2}$<111> Burgers vectors present.

Table 1. Conditions for specimens examined.

ID	Hoop Stress (MPa)	Temp. (ºC)	Dose	Strain (%)
A1	0	286	4.3 dpa*	0.11
A10	87	302	4.6 dpa*	0.10
A7	129	300	4.7 dpa*	0.28

* 133 EFPD or 3192 h

<u>Results</u>

The microstructure of the unstressed condition A1 is shown in Figure 2. This figure and the two that follow have been prepared to allow identification of all $\frac{a}{2}$<111> Burgers vectors present, so three views of the same area are given, one in 01$\bar{1}$ contrast and one in 200 contrast both taken near a (011) orientation and the third in $\bar{1}$01 contrast taken after a large tilt of the foil. A large TiO_2 particle is located on the lower right in order to reveal any changes in behavior near such particles.

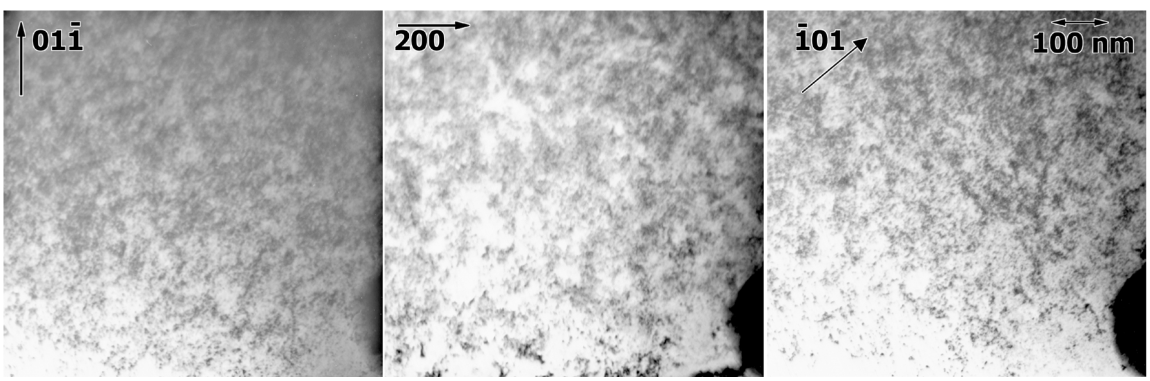

Figure 2. Condition A1 at 300ºC, 0 MPa to 4.3 dpa and 0.11% strain.

Similar images are also provided for conditions A10 and A7 in Figures 3 and 4, respectively. Examination and comparison of Figures 2, 3 and 4 reveals that dislocation images are difficult to identify due to extensive precipitation that is present. Diffraction only revealed additional intensity in the vicinity of $\frac{2}{3}$ <222>. The dislocations appear to be only of type $\frac{a}{2}$<111>, based on imaging under 200 strain contrast, as is usually the case in vanadium alloys. No evidence for void swelling could be identified.

In the course of examination of condition A7, it became apparent that several examples of grain boundary migration could be identified. Two examples are provided in Figure 5. In the first case, the boundary has moved but was pinned by a large TiO_2 particle, so migration probably is on the

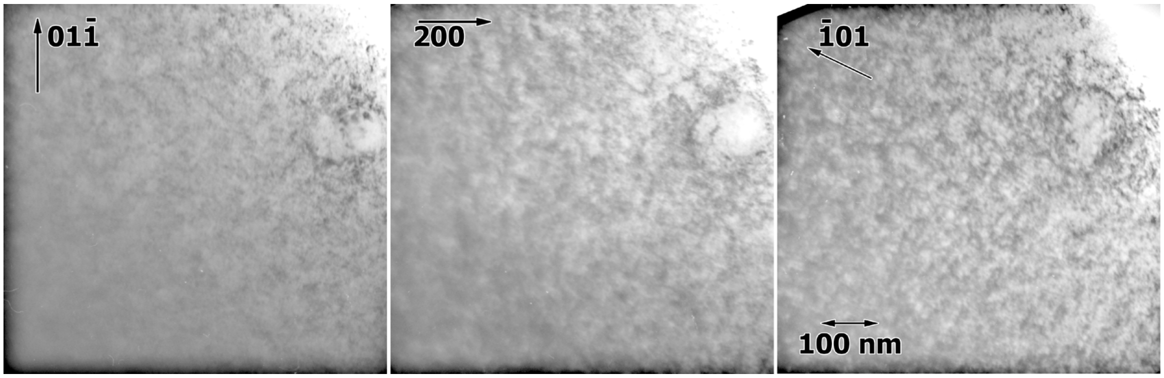

Figure 3. Condition A10 at 300ºC, 87 MPa to 4.6 dpa and 0.10% strain.

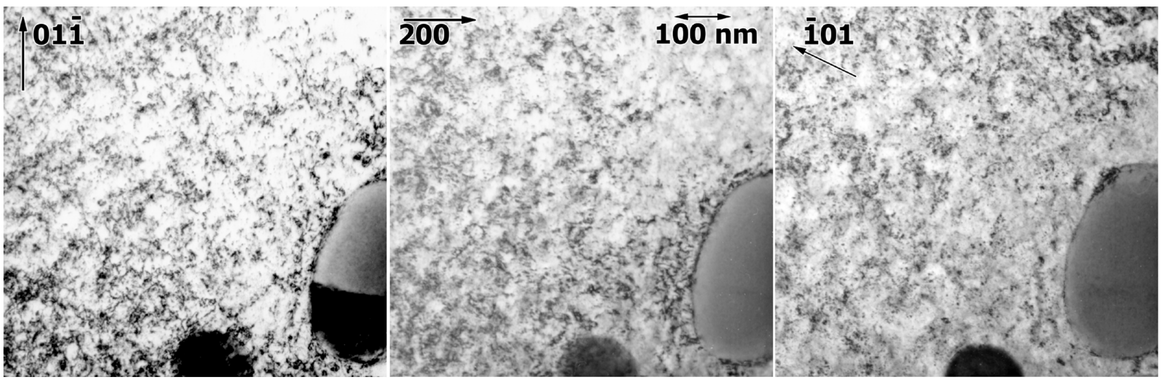

Figure 4. Condition A7 at 300ºC, 129 MPa to 4.7 dpa and 0.28% strain.

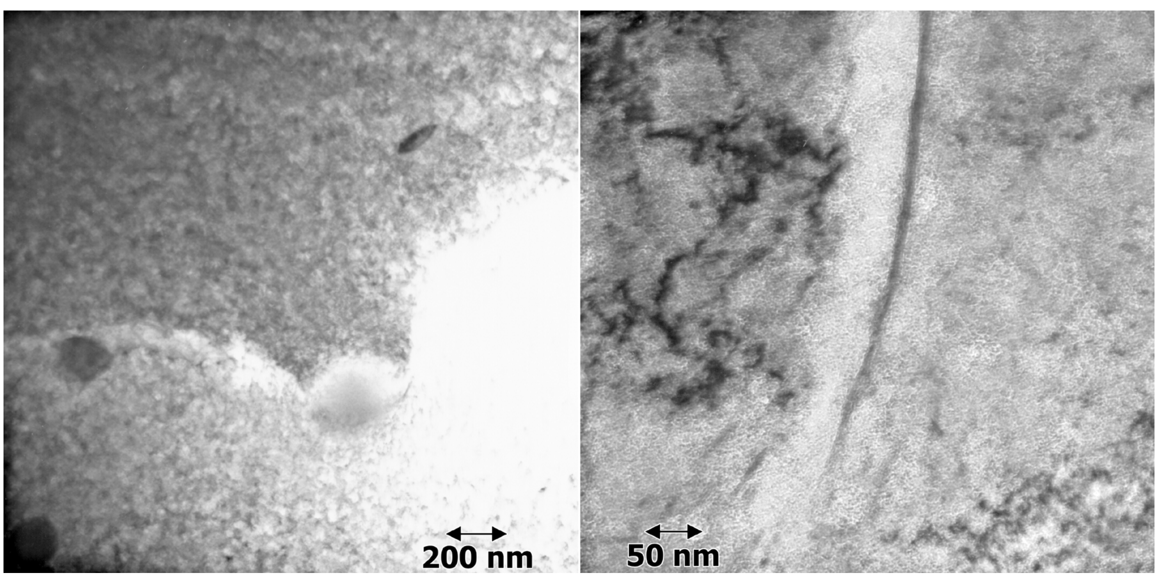

Figure 5. Examples of grain boundary migration in condition A7 at 300ºC, 129 MPa to 4.3 dpa.

order of 800 nm, or close to 1 µm. In the second case, the boundary has polished differently than the surrounding matrix with what appears to be a denuded zone (without precipitation, perhaps) adjacent. Specimen A7 was further examined using field emission gun analytical electron microscopy in order to identify chemistry changes near migrating boundaries. Results are given in Figure 6 showing plots of composition as a function of position across the boundary. A digital image is inset for each plot in order to show the positions of the points analyzed. Scan "gb 1" indicates enhancement of Cr at the boundary and depletion to the right with accompanying enhancement of Si at the boundary. Scans "gb 6" and "gb 7" of the same boundary but at different locations confirm Cr enrichment at the boundary. Scan "gb 3 repeat" demonstrates extremely high Si levels. Therefore, enrichment due to Cr and Si is indicated in coordination with grain boundary migration but Ti shows negligible evidence of enrichment.

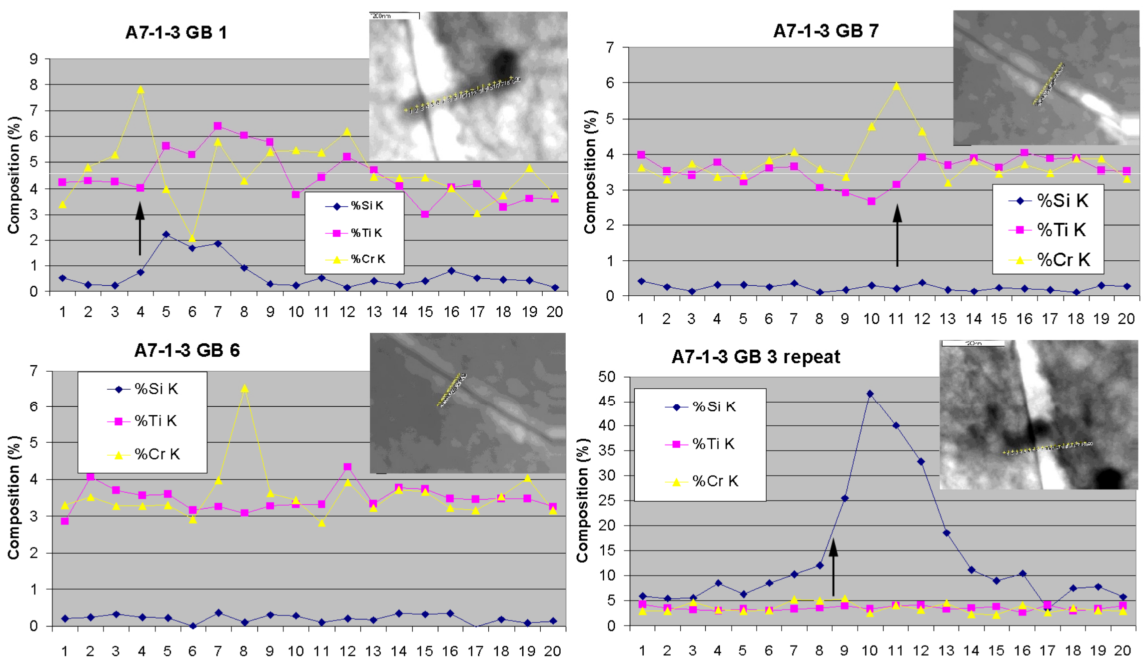

Figure 6. Grain boundary composition profiles in condition A7 at 300ºC, 129 MPa to 4.3 dpa.

Discussion

The results of microstructural examinations of irradiated V-4Cr-4Ti pressurized tubes may be summarized as follows:

> Extensive fine precipitation was found with diffraction intensity at ⅔ <222>. Precipitates may be nucleating near dislocation structure.
>
> The dislocation structure consisted of perfect dislocation line segments with few loops identified.
>
> Stress appears to encourage grain boundary migration

Evidence for segregation of Cr to boundaries and Si behind boundaries was found to accompany grain boundary migration.

Therefore, irradiation creep of V-4Cr-4Ti is probably controlled by climb of $\frac{a}{2}\langle 111 \rangle$ dislocations. Grain boundary migration is observed, probably enhanced by stress, but it is not anticipated that grain boundary sliding plays an important role in deformation. As with unstressed conditions, precipitation at low temperatures is extensive, obscuring other microstructural features and leading to irradiation hardening. Identification of the precipitate remains poorly defined. No evidence for non-steady state creep could be found.

It can be noted that this work was hindered by poor foil preparation leading to examinations of thicker specimens than optimum (see for example Figure 3) with a resultant need for digital dodging of the image. Dodging remnants appear at the edge of each image and are retained to emphasize that extensive dodging was used.

Conclusions

Irradiation creep at 300ºC is associated with dislocation development and may be affected by precipitation. Stress appears to encourage grain boundary migration during irradiation creep. Segregation of Cr to boundaries and Si behind boundaries was found to accompany the grain boundary migration.

FUTURE WORK

This work will be continued when more specimens are available for testing. Specimens irradiated in HFIR at 500°C would be appropriate.

ACKNOWLEDGEMENTS

This work would not have been possible without the cooperation and participation of H. Tsai and coworkers at the Argonne National Laboratory.

REFERENCES

[1] H. Tsai, H. Matsui, M. C. Billone, R. V. Strain and D. L. Smith, J. Nucl. Mater. 258-63 (1998) 1471.

[2] D. S. Gelles, M. L. Hamilton and R. J. Kurtz, DOE/ER-0313/26 (1999) 11.