GRAIN BOUNDARY MIGRATION INDUCED SEGREGATION IN V-Cr-Ti ALLOYS - D. S. Gelles (Pacific Northwest National Laboratory)[a] and S. Ohnuki and H. Takahashi (University of Hokkaido, Japan)

OBJECTIVE

The objective of this research is to assess effects of irradiation on microchemical stability in vanadium alloys for first wall applications in a fusion energy system.

SUMMARY

Analytical electron microscopy results are reported for a series of vanadium alloys irradiated in the HFIR JP23 experiment at 500°C. Alloys were V-5Cr-5Ti and pure vanadium which are expected to have transmuted to V-15Cr-5Ti and V-10Cr following irradiation. Analytical microscopy confirmed the expected transmutation occurred and showed redistribution of Cr and Ti resulting from grain boundary migration in V-5Cr-5Ti, but in pure V, segregation was reduced and no clear trends as a function of position near a boundary were identified.

PROGRESS AND STATUS

Introduction

The present work is part of an ongoing collaboration effort in the area of irradiation effects in vanadium alloys[1-4] and arose because of interest in using the JEOL 2010F analytical electron microscope at PNNL for high resolution microchemical segregation studies. Specimens of pure vanadium and V-5Cr-5Ti were included in the HFIR-JP23 experiment in order to study transmutation effects in vanadium alloys. It is of interest to note that the V-5Cr-5Ti alloy effectively transmuted to V-15Cr-5Ti, an early candidate composition previously studied.[1] Examination demonstrated that irradiation not only resulted in precipitation similar to that found in fast reactor irradiated V-15Cr-5Ti, but also led to radiation induced grain boundary migration. This report is intended to describe the segregation observed at these boundaries.

Experimental Procedure

Specimens UB06 of V-5Cr-5Ti and U006 of pure vanadium were examined following irradiation in the HFIR MFE-JP23 500°C Monbusho specimen holder.[5] The fluence accumulated was 4.23×10^{22} n/cm^2 (E total) corresponding to 10.02 dpa in pure vanadium.[6] It may be noted that irradiation of V-4Cr-4Ti in the HFIR core to 10 dpa will produce approximately 10% additional chromium.[7] Specimen preparation and examination followed standard procedures. Examinations were performed on a JEOL 2010F transmission electron microscope operating at 200 KeV with a field emission gun and compositional analysis used an Oxford Instruments ISIS system with a thin window detector.

Results

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Results

Specimen UB06 of V-5Cr-5Ti was found to be remarkably similar to specimens of V-15Cr-5Ti irradiated in FFTF.[1] Chromium levels had been increased to about 13% by transmutation and extensive precipitation consisting of both large and small rod shaped particles was found. Compositional analysis of precipitates contained in the foil only indicated enrichment of Ti.

However, in the course of observation, it became apparent that significant grain boundary migration had occurred, providing the possibility for grain boundary segregation measurements. Figure 1 gives examples of two areas along the same grain boundary in specimen UB06 chosen for chemical analysis. Figure 1a contains a section of boundary which did not appear to have moved due to irradiation whereas Figure 1b contains a very convoluted grain boundary which has moved from its original position. In each figure, fiducial numbers have been added to define areas used for compositional analysis. Figures 1c, d and e give those composition measurements as a function of distance from the position of the grain boundary prior to irradiation, so that Figure 1c represents the case for the grain boundary that did not migrate shown in Figure 1a and Figures 1d and 1e show behavior in Figure 1b for grain boundary migration of several hundred nm. Each composition plot is labelled to show the starting position, and where different, the final position of the grain boundary. From these figures, it can be concluded that compositional changes are small but there appears to be enrichment of Cr and depletion of Ti in the vicinity of the grain boundary path, with changes as large as 2%. It can also be noted that Si was measured in the specimen at levels between 0.3 and 1.3%, but no consistent trends for Si could be identified.

Specimen U006 of pure vanadium was similar except that titanium was not present. Chromium levels had been increased to about 8.5% by transmutation, and silicon levels ranged from 0.3 to 1.5%. However, rod shaped precipitates were not found, corroborating conclusions that such precipitates are titanium rich. Specimen U006 also contained examples of large scale grain boundary migration. Figure 2a and b show two grain boundaries where sections of the boundary had moved. These figures are also marked with fiducial numbers corresponding to areas for which compositional analysis was performed so that in each case one boundary section had not moved and a second section had moved 400 or 500 nm. The analyses as a function of distance from the original grain boundary position are given in Figures 2c, and d for Figure 2a and Figures 2e and f for Figure 2b. Figures 2e and f include duplicate measurements, for a second series of measurements performed after the first was completed. From these figures, it can be concluded that segregation following irradiation was small, that scatter in the data was as large as any segregation measured, and there were no clear trends as a function of position relative to a grain boundary.

Discussion

Definition of a candidate vanadium alloy for fusion applications appears to require fairly precise control of the levels of alloying elements used.[8] The present work appears to indicate that solute segregation accompanying irradiation induced grain boundary migration does not significantly alter local chemistries. For example, local chromium and titanium levels are altered only by about 2% following irradiation to 10 dpa at 500°C. Therefore, it can be anticipated that precise control of composition is not unreasonable in light of the level of segregation that can result during irradiation. However, this work does demonstrate the importance of radiation induced grain boundary migration and the consequent instability of the microstructure for grain growth during irradiation.

CONCLUSIONS

Radiation induced solute segregation in association with extensive grain boundary migration has been

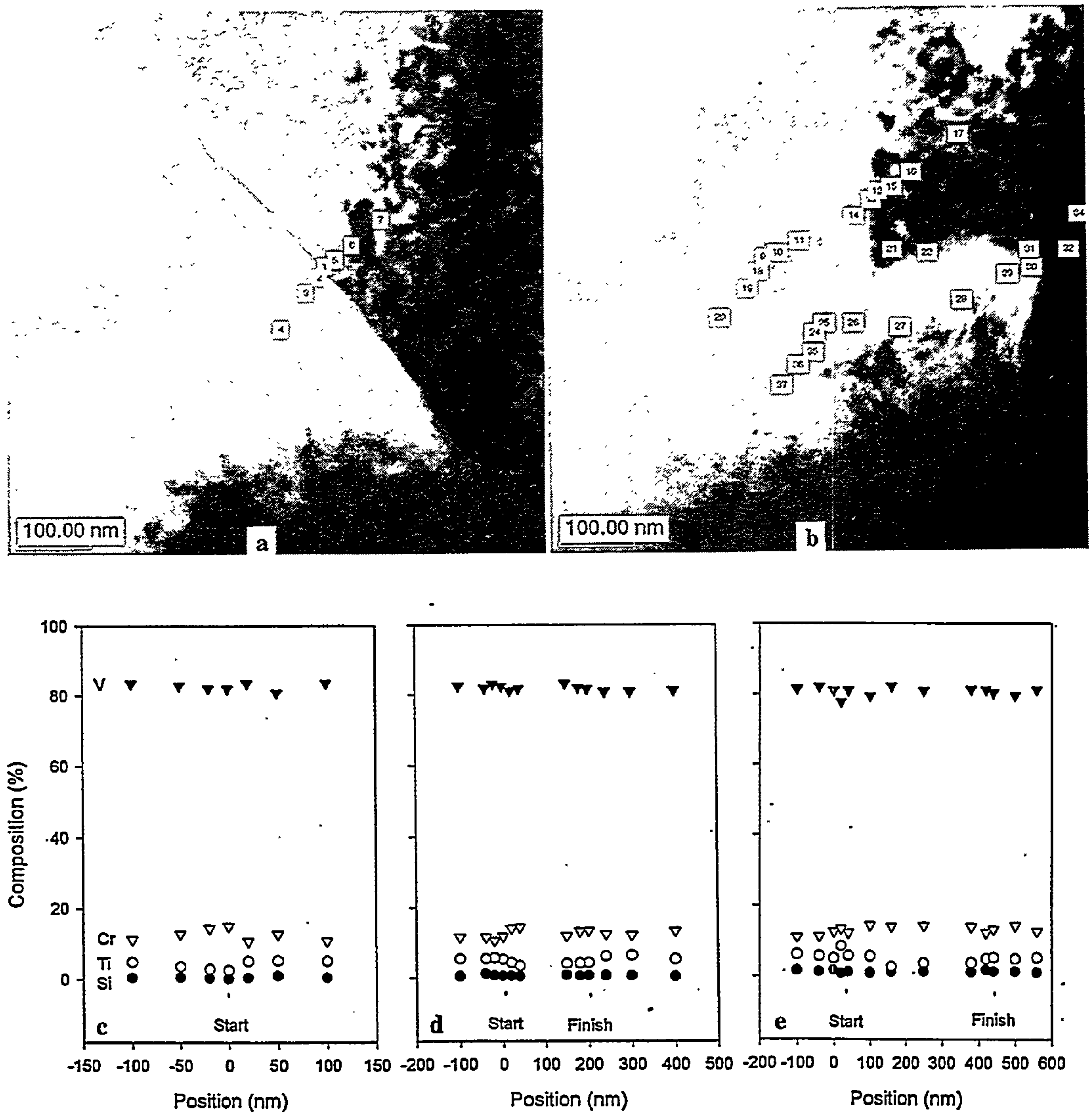

Figure 1 Two sections of a grain boundary in specimen UB06 of V-5Cr-5Ti following irradiation at 500°C to 10 dpa, a), and b), and compositional analysis for locations indicated c), d), and e).

measured in two experimental alloys, V-5Cr-5Ti and pure vanadium following irradiation at 500°C to 10 dpa in the HFIR JP23 experiment. Results indicate that Cr levels are increased by about 8% due to transmutation, and up to 2% enrichment in Cr and 2% depletion in Ti occurs at grain boundaries in V-5Cr-5Ti whereas in pure vanadium, segregation was reduced and no clear trends as a function of position near a boundary were identified.

Future work

This work will be resumed when suitable specimens are available.

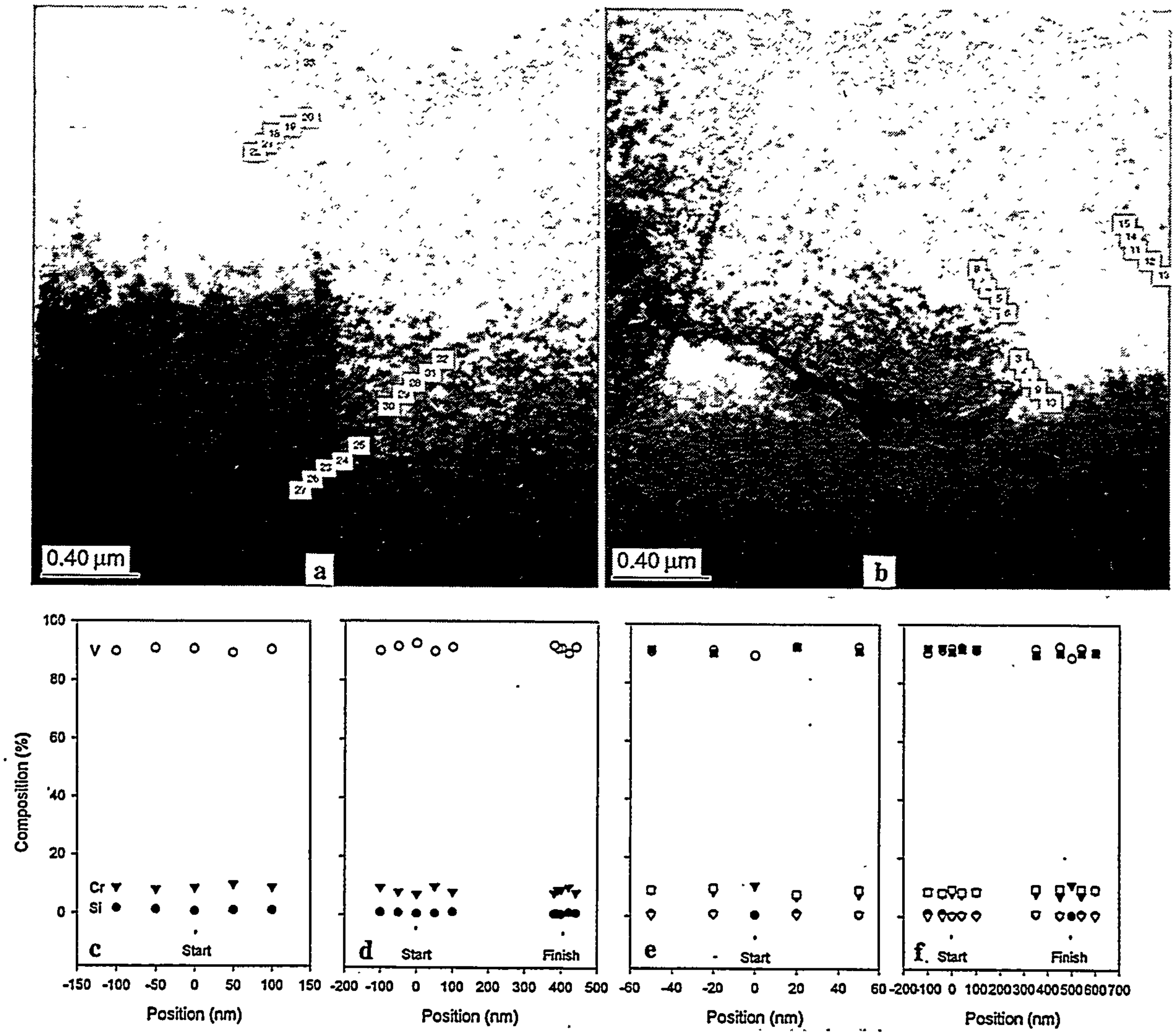

Figure 2 Two grain boundaries in specimen U006 of pure vanadium following irradiation at 500°C to 10 dpa, a), and b), and compositional analysis for locations indicated c), d), e), and f).

REFERENCES

1 D. S. Gelles, S. Ohnuki, B. A. Loomis, H. Takahashi, and F. A. Garner, in DOE/ER-0313/7 (1989) 193.

2 S. Ohnuki, D. S. Gelles, B. A. Loomis, H. Takahashi, and F. A. Garner, in DOE/ER-0313/7 (1989) 177.

3 S. Ohnuki, H. Kinoshita, F. A. Garner, and H. Takahashi, in DOE/ER-0313/19 (1996) 92.

4 S. Ohnuki, H. Takahashi, F. A. Garner, J. E. Pawel, K. Shiba and A. Hishinuma in DOE/ER-0313/19 (1996) 93.

5 A. M. Ermi and D. S. Gelles, in DOE/ER-0313/17 (1994) 35.

6 L. R. Greenwood and R. T. Ratner, to be published in Fusion Materials Semiannual Progress Report for the Period Ending June 30, 1996, DOE/ER-0313/20.

7 I. C. Gomes and D. L. Smith, in DOE/ER-0313/16 (1994) 33.

8 B. A. Loomis, H. M. Chang, L. J. Nowicki and D. L. Smith, J. Nucl. Mater., 212-215 (1994) 799.