ELSEVIER

Journal of Nuclear Materials 258–263 (1998) 1437–1441

journal of nuclear materials

Microstructure of V–4Cr–4Ti alloy after low-temperature irradiation by ions and neutrons [1]

J. Gazda [a,*], M. Meshii [a], H.M. Chung [b]

[a] Materials Science and Engineering Department Northwestern University, 2225 N. Campus Dr., Evanston IL 60208, USA
[b] Argonne National Laboratory, 9700 S. Cass Ave. Argonne IL 60439, USA

Abstract

Recent interest in the application of a V–4Cr–4Ti alloy in the ITER prompted an investigation of the effects of low-to-moderate temperature irradiation (<420°C) on the alloy's mechanical properties. Two sets of experiments were conducted. The effects of fast neutron irradiation to ≈4 dpa at 390°C were investigated in the X530 experiment in the EBR-II reactor. Irradiation with single (4.5-MeV Ni^{2+}) and dual ion beams (350-keV He^{+} simultaneously with 4.5-MeV Ni^{2+}) complemented this study. TEM observations showed the formation of a high density of point-defect clusters and dislocation loops (<30 nm diameter) distributed uniformly in both types of specimens. Mechanical property testing of neutron irradiated material showed embrittlement of the alloy. The deformed microstructures were examined by TEM to determine the causes of embrittlement and revealed dislocation channels propagating through the undeformed matrix. The channels are the sole slip paths and they cause early onset of necking and loss of work-hardening. Based on a review of the available literature, suggestions are made for further research of slip localization in V–4Cr–4Ti alloys. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

Extensive investigations of the post-irradiation properties of vanadium-base alloys suggest the V–4 wt% Cr–4 wt% Ti (V–4Cr–4Ti) alloy as combining the optimal physical and mechanical properties for fusion reactor applications. The main advantage of this alloy is its intrinsically low neutron activation and resistance to swelling and embrittlement after fast neutron irradiation at 420–700°C [1]. Interest in application of this alloy in the ITER prompted recent investigations of the effects of low-to-moderate temperature (200–420°C) irradiation on the alloy's mechanical properties. This irradiation temperature range is also relevant to transient situations in other fusion reactors. To address these issues, two sets of experiments were conducted within this temperature range. Effects of fast neutrons ($E > 0.1$ MeV) at 390°C were investigated with irradiation to of ≈4 dpa in the EBR-II reactor. Due to limited space and the imminent shutdown of the EBR-II reactor a complementary study was undertaken using single (4.5-MeV Ni^{2+}) and dual ion beams (350-keV He^{+} simultaneously with 4.5-MeV Ni^{2+}). Ion irradiation allowed flexibility in control of experimental parameters. Conditions chosen were 0.5, 2, and 5 dpa at 200°C, 350°C, and 420°C.

The irradiation-induced defect evolution was studied in ion-irradiated specimens and findings were reported in Ref. [2]. Both types of post-irradiation microstructure of V–4Cr–4Ti alloy, i.e. ion and neutron irradiation, consisted of dislocation loops and point-defect clusters (up to 30 nm diameter, $\sim 10^{22}$ m^{-3} number density); voids were not observed. Results of immersion density measurements confirmed insignificant swelling in the neutron-irradiated specimens.

The ion-irradiated specimens did not allow direct measurement of fracture properties of the material but they provided a warning about the possibility of extensive hardening due to radiation-induced defects. The neutron irradiation resulted in degradation of the V–4Cr–4Ti alloy, manifested by loss of work-hardening

---

[*] Corresponding author. Tel.: +1 630 252 4995; fax: +1 630 252 3604; e-mail: jerzy_gazda@qmgate.anl.gov.

[1] Work supported by the US Department of Energy, Office of Fusion Energy, under Contract W-31-109-Eng-38.

PII: S0022-3115(98)00159-7

capability in tensile specimens and an increase of the ductile-to-brittle-transition temperature (DBTT) to above 300 K, as measured in instrumented Charpy tests on miniature (one-third-size) CVN specimens [3,4]. A concurrent irradiation study in the HFBR reactor also reported dramatically increased DBTT and loss of work-hardening capability after irradiation to 0.5 dpa at temperatures of 100–275°C [5].

In this paper, we present preliminary results of TEM work aimed at exploring the deformation mechanisms in irradiated V–4Cr–4Ti. The common feature observed after post-irradiation deformation was the propagation of primary slip dislocations confined to “channels” surrounded by an undeformed matrix containing a high density of undisturbed radiation defects. The formation of such channels indicates plastic strain localization accompanied by local work softening, prompting the early onset of necking and limited work-hardening of the material. The dislocation channeling is not uncommon in materials hardened by structurally unstable barriers to dislocation glide and is generally recognized as one of the reasons for radiation embrittlement [6]. Based on the current work and studies available in the literature, suggestions are made for further research of dislocation channeling in irradiated vanadium alloys, directed to improve post-irradiation ductility.

2. Experimental procedures

The preparation of V–4Cr–4Ti alloy (Heat 832665, ANL ID: BL71) is described in detail in Ref. [7]. The composition of this alloy is shown in Table 1. The specimen preparation steps for ion and neutron irradiation were reported in Ref. [2]. Ion irradiation of annealed (1 h at 1050°C in 10^{-5} Pa UHV ion pumped vacuum) 3-mm-diameter disks were performed at the Argonne Tandem Accelerator facility with single (4.5 MeV Ni^{2+}) or dual ion beams (350 keV He^{+} simultaneous with 4.5 MeV Ni^{2+}). The disks were later sectioned and back-jet-thinned for TEM observations. TEM foils were made at the depth of ≈800–1000 nm from the irradiated surface. The neutron irradiation was conducted during the final run of the in EBR-II reactor in August/September 1994. Specimens were irradiated at 390°C in Li-filled capsules, located in the core position, to ≈4 dpa (flux of 2.4×10^{15} n cm^{-2}, $E > 0.11$ MeV). Details of this irradiation experiment were reported in Ref. [8].

To evaluate deformation processes in the irradiated materials three types of specimens were examined with TEM. Specimens with ion damage were indented at room temperature (RT) with a Berkovitch nano-indentor to induce plastic deformation. TEM foils were then prepared from regions near the indentation. Two types of neutron-irradiated specimens were also employed. First, disks were made from sections of irradiated Charpy specimens to evaluate the microstructure without and with plastic deformation. The disks were cut with a diamond saw and electropolished to remove damage due to cutting. Subsequently, some of the disks were polished for TEM examination, while others were indented at RT with a Vickers indentor to produce plastic deformation (typical indentations yielded hardness measurement of 320 VHN, as opposed to 178 VHN in annealed material). TEM foils were then prepared from regions under the indentations. For the third type, TEM foils were obtained from cross-sectional pieces of a fractured SS-3 miniature tensile sheet specimen used for determination of post-irradiation mechanical properties (BL71-50 irradiated in EBR-II, X530 experiment, subcapsule S9; tensile tested to fracture at 390°C with strain rate of 1.1×10^{-3} s^{-1}: YS = 805 MPa, UTS = 808 MPa, UE = 0.45%, TE = 4.7%). Cross-sections were obtained from the shoulder, gauge length, and necked regions of the tensile specimen.

All TEM specimens were electropolished with a South Bay Technology Single Jet Electropolisher 550B and electrolyte consisting of (by volume) 70% H_2SO_4, 15% CH_3OH, and 15% $C_6H_{14}O_2$ Butyl Cellosolve at −10°C. Microstructure observations were conducted in Phillips CM30 transmission electron microscope.

3. Results

The microstructure of the un-irradiated V–4Cr–4Ti alloy consists of ≈20 μm grains with loosely dispersed, ≈200-nm-diameter Ti(CNO) precipitates. These particles form in ingot and are commonly found in all U.S. V–Ti–Cr alloys. An example of this microstructure is shown in Fig. 1(a). The irradiated microstructure of V–4Cr–4Ti alloy changes with fluence and temperature of irradiation; for details see Ref. [2]. Ion irradiation caused formation of a high density ($\approx 2 \cdot 10^{22}$ m^{-3}) of small dislocation loops and “black dot” point-defect clusters, exemplified in Fig. 1(b) by the typical features after irradiation to 5 dpa at 350°C with 4.5 MeV Ni^{2+}

Table 1
Composition (impurities in wppm) of V–4Cr–4Ti alloy (Heat 832665, BL71)

ANL ID	Cr (wt%)	Ti (wt%)	Cu	Si	O	N	C	S	P	Ca	Cl	B
BL-71	3.8	3.9	<50	783	310	85	80	<10	<30	<10	<2	<5

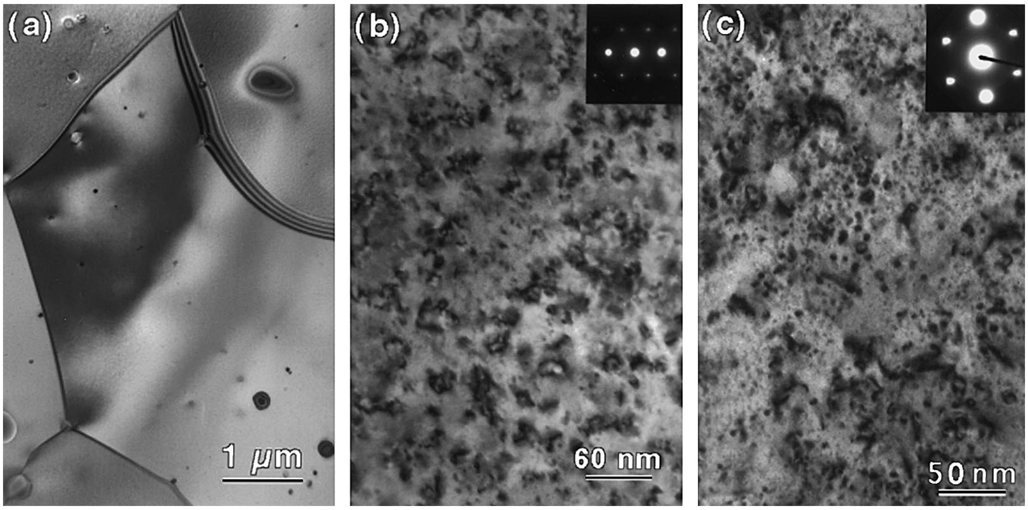

Fig. 1. Typical microstructure of V–4Cr–4Ti alloy: (a) annealed for 1 h at 1050°C in UHV furnace, (b) ion irradiated to 5 dpa at 350°C with 4.5 MeV Ni^{2+}, and (c) irradiated to 4 dpa at 390°C in the X530 experiment.

ions. Similarly, neutron irradiation caused formation of uniformly distributed point-defect clusters and dislocation loops ($\approx$5–10 nm in diameter, number density on the order of $\approx 10^{22}$ m^{-3}). Fig. 1(c) provides examples of X530 EBR-II irradiated V–4Cr–4Ti microstructure.

To determine the mechanisms for the radiation embrittlement of V–4Cr–4Ti irradiated at low-to-moderate-temperatures, studies of the microstructure after plastic deformation were undertaken. Ion-irradiated and nano-indented specimens were used in early experiments. TEM foils prepared from areas below the indentation showed bands filled with dislocations, also known as "channels". All plastic deformation appeared to be confined to these channels, because no slip dislocations were observed elsewhere. Fig. 2 shows examples of the dislocation channels in ion-irradiated and indented V–4Cr–4Ti.

The miniature size of the limited number of the tensile specimens (gauge length dimensions: 0.6 × 1.5 × 10 mm^3) employed in the X530 experiment and the early onset of necking made the task of TEM specimen preparation extremely difficult. Therefore first, sections of X530 neutron-irradiated Charpy specimens were indented and TEM foils were prepared from the region

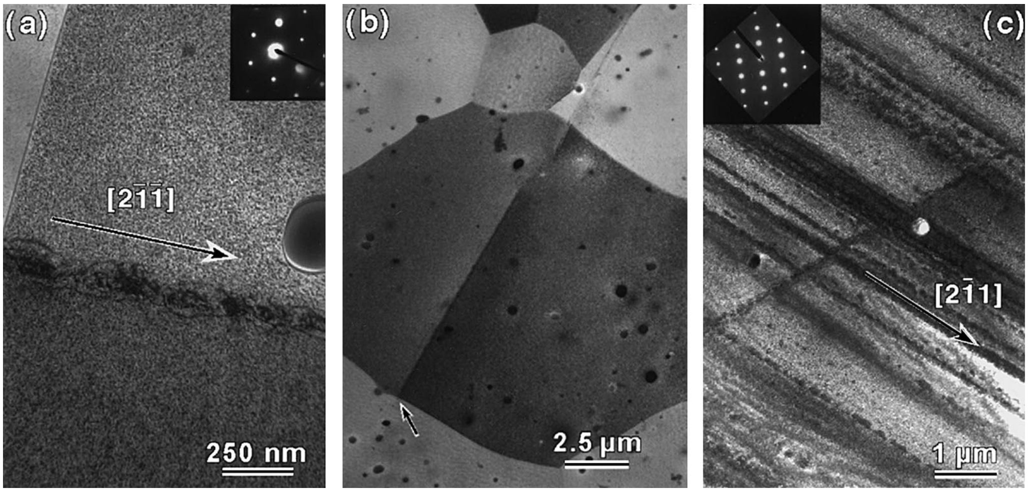

Fig. 2. Dislocation channels observed in ion-irradiated and indented specimens: (a) tangled dislocations forming a channel (0.5 dpa at 200°C), (b) channel arrested by grain boundary (10 dpa at 350°C), (c) channels in heavily deformed specimen (5 dpa at 420°C), two channel directions visible.

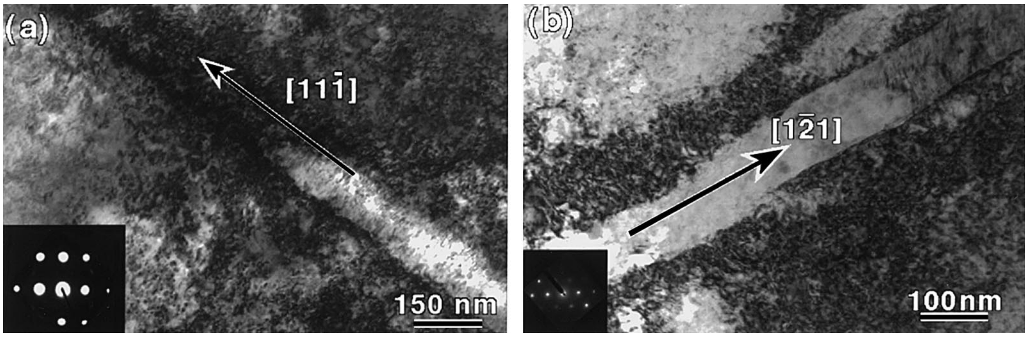

Fig. 3. Dislocation channels in neutron-irradiated V–4Cr–4Ti alloy: (a) after indentation at room temperature, (b) after tensile deformation.

near indentation in the same way as in the ion-irradiated specimens. Again, the slip activity was found to be confined exclusively in the dislocation channels. An example is given in Fig. 3(a). The crystallographic directions of the channels were determined and were consistent with the slip traces within the accuracy of the experiment, as the channels often deviated from a straight line, indicating massive cross-slips took place during dislocation motion.

A limited number of TEM observations were carried out in the foils prepared from the necked regions of the tensile specimen (BL71-50). Although fracture surface showed ductile mode of fracture, TEM foils prepared from the necked region contained dislocation channels as shown in Fig. 3(b). The channel direction was determined as $\langle 2\ 1\ 1\rangle$ for the foil zone axis $\langle 1\ 1\ 1\rangle$, again consistent with the trace of $\{1\ 1\ 0\}$ planes. The deviation of the channels from a straight line indicated the occurrence of cross-slip during deformation.

4. Discussion

Dislocation channels were observed in all irradiated and deformed materials investigated in this study. Microstructural evaluation of ion-irradiated and indented specimens provided an early warning of dislocation channel formation. Channel formation was confirmed by TEM observation in neutron-irradiated material near a hardness indentation in Charpy specimens and in the necked region of tensile specimens. In most cases, the channels contained dislocations. The few exceptions channels nearly free of dislocations were found, these were located in exceptionally thin foils. This difference is attributed to dislocations slipping out of the foils in the vicinity of the surface. In nearly all cases, the termination of a dislocation channel at a grain boundary triggered another channel in the adjacent grain. The crystallographic nature of the channels was the same in all three types of specimens.

Formation of channels was previously observed by TEM in neutron-irradiated single crystals of vanadium alloys by Huang and Arsenault [9] during an investigation of the effects of oxygen content on plastic deformation. In those vanadium alloys, as in the present experiment, test temperature ranging from 150°C to 300°C had little effect on the character of dislocation channeling, i.e., the channels formed were indistinguishable. However, oxygen content had a pronounced consequence on the appearance of the channels; in vanadium (40 wt.ppm O), channels were denuded of internal dislocations except for a few cases of channels containing dislocation arrays, but when the oxygen content was increased to 300 wt. ppm, only a few cleared channels were observed and none were found when the oxygen content was increased to 1000 wt. ppm. Indicating that dislocations in the channels readily disappear without the pinning effects of oxygen. The dislocation channels observed in the present study resemble these observed in the vanadium–oxygen alloys of Ref. [9].

Although our observations of the dislocation channels is the first in the V–4Cr–4Ti alloy, dislocation channels are common in a wide variety of materials where barriers to dislocation movement are uniformly distributed throughout the specimen. Luft [6] presented a review of dislocation channeling not limited to irradiated metals; he stated that in all investigated pure BCC metals that contained visible radiation damage (Fe, Nb, V, and Mo), defect-free slip channels were observed upon plastic deformation. The localization of strain was attributed to various strain-softening mechanisms. In irradiated and deformed metals, strain softening was explained by the loss of the defects from a channel by either annihilation or "plowing" the defects to the sides of the channel. Agglomeration of defect clusters at the edges of channels characteristic of the plowing mechanism were not observed in this study, therefore defect annihilation is the likely mechanism responsible for channel formation.

Our observation of slip localization by dislocation channel formation explains the loss of macroscopic ductility found in the irradiated vanadium base alloys. The following two types of observations are also well known in materials where channel formation takes place:

Pre-deformation of fully annealed material by cold working without significant rearrangement of the grain structure introduces dislocations which could be mobilized during the post-irradiation deformation. In cold rolled molybdenum a critical density (6×10^8 cm^{-2} at 493 K) of dislocations is necessary for channel formation [10]. Below this density, dislocations distributed in the matrix act only as forest dislocations that provide a work-hardening effect. When higher dislocation density is produced during pre-deformation, slip localization occurs upon re-deformation. Therfore, if this mechanism is adopted in V–4Cr–4Ti, a low density of dislocations uniformly distributed in the material could lead to slip homogenization during post-irradiation deformation.

Introduction of incoherent, uncuttable particles can reduce slip localization. Dislocation channels were observed to form in Ti-base precipitation-strengthened alloys during deformation [11]. These small uniformly distributed coherent particles promote slip localization because dislocations cut through them and form particle-free channels. However, when further aged, precipitates were replaced by larger semicoherent or incoherent particles that cannot be easily cut. In such cases, dislocations are forced to cross-slip around the precipitates during deformation, essentially work-hardening the alloy and leading to more homogenous macroscopic slip.

Therefore, providing uniformly distributed incoherent particles in the matrix, such as Ti(CNO), combined with a small degree of prestrain, could result in a higher macroscopic ductility in post-irradiated V–4Cr–4Ti. Limited supporting experimental evidence also exists from the recently compiled evaluations of tensile testing of vanadium-base alloys irradiated at low temperatures (<400°C). The alloy containing a high density of large particles (V-17.7 wt% Ti alloy, ANL ID: BL15) and a cold-worked specimen of V–4Cr–4Ti alloy (experimental heat, ANL ID: BL47) exhibited uniform elongation $\geqslant 2\%$ [12]. The vanadium alloy development should be extended along this line to promote slip homogenization during deformation.

5. Conclusions

1. The ion irradiation is useful as a tool in assessing the properties of fusion candidate materials. It has been shown that ion irradiation provides information about both the swelling and embrittlement potential of a V–4Cr–4Ti alloy irradiated at low temperatures. No voids/cavities were observed by TEM and slip localization by dislocation channels was found near indentations.

2. Dislocation channels were observed in V–4Cr–4Ti alloy deformed after irradiation. It is suggested that they promote premature necking and loss of work-hardening in tensile tests of the V–4Cr–4Ti alloy (Heat No.: 832665, BL71) after fast neutron ($E > 0.1$ MeV) irradiation to ≈4 dpa at 390°C in the EBR-II.

3. Channel formation is a common feature of the low-temperature deformation of BCC metals containing a dispersion of unstable barriers to dislocation glide. Microstructural modification is suggested as an alloy development task to minimize post-irradiation embrittlement at low temperatures (<420°C).

References

[1] D.L. Smith, H.M. Chung, B.A. Loomis, H.-C. Tsai, J. Nucl. Mater. 233–237 (1996) 356.

[2] J. Gazda, M. Meshii, H.M. Chung, in: I.M. Robertson, G.S. Was, L.W. Hobbs, T. Diaz de la Rubia (Eds.), MRS Fall' 96 Meeting Proceedings, Boston, vol. 439, Materials Research Society, Pittsburgh, p. 349.

[3] H.M. Chung, H.-C. Tsai, L.J. Nowicki, D.L. Smith, Fusion Reactor Materials – Semiannual Prog. Report, 1997, DOE/ER-0313/22, Oak Ridge Nat. Lab., Oak Ridge, TN, pp. 18–21.

[4] S.J. Zinkle, D.J. Alexander, J.P. Robertson, L.L. Snead, A.F. Rowcliffe, L.T. Gibson, W.S. Eatherly, H. Tsai, Fusion Reactor Materials – Semiannual Prog. Report, 1996, DOE/ER-0313/21, Oak Ridge National Laboratory, Oak Ridge, TN, pp. 73–78.

[5] D.J. Alexander, L.L. Snead, S.J. Zinkle, A.N. Gubbi, A.F. Rowcliffe, E.E. Bloom, Fusion Reactor Materials – Semiannual Prog. Report, 1996, DOE/ER-0313/20 Oak Ridge National Laboratory, Oak Ridge, TN, pp. 87–95.

[6] A. Luft, Prog. Mater. Sci. 35 (2) (1991) 97.

[7] H.M. Chung, H.-C. Tsai, D.L. Smith, R. Peterson, C. Curtis, C. Wojcik, R. Kinney, Fusion Reactor Materials – Semiannual Prog. Report, 1995, DOE/ER-0313/17, Oak Ridge National Laboratory, Oak Ridge, TN, pp. 178–182.

[8] H.-C. Tsai, R.V. Strain, A.G. Hins, H.M. Chung, L.J. Nowicki, D.L. Smith, Fusion Reactor Materials – Semiannual Prog. Report, 1995, DOE/ER-0313/17, Oak Ridge National Laboratory, Oak Ridge, TN, pp. 8–14.

[9] Y. Huang, R.J. Arsenault, Rad. Eff. 17 (1973) 3–11.

[10] C. Ritschel, Zur Stabilitat von Versetzungsstructuren in hochreinen Molybdaneikristallen bei plastischer Verformung, Dissertation, AdW der DDR, ZFW Dresden, Germany, 1982.

[11] A. Gysler, J. Lindigkeit, G. Lutjering, Acta Met. 22 (1974) 901.

[12] H.M. Chung, D.L. Smith, Fusion Reactor Materials – Semiannual Prog. Report, 1997, Oak Ridge National Laboratory, Oak Ridge, TN, pp. 33–38.