MICROSTRUCTURE OF V-4Cr-4Ti ALLOY AFTER LOW-TEMPERATURE IRRADIATION BY IONS AND NEUTRONS*

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SUMMARY

Mechanical properties of V-4Cr-4Ti alloy were investigated after low-temperature (<420°C) irradiation. The effects of fast neutrons at 390°C were investigated by irradiation to ≈4 dpa in the X530 experiment in the EBR-II reactor; these tests were complemented by irradiation with single (4.5-MeV Ni^{++}) and dual ion beams (350-keV He^{+} simultaneously with 4.5-MeV Ni^{++}). TEM observations showed the formation of a high density of point-defect clusters and dislocation loops (<30 nm diameter) distributed uniformly in the specimens. Mechanical-property testing showed embrittlement of the alloy. TEM investigations of deformed microstructures were used to determine the causes of embrittlement and yielded observation of dislocation channels propagating through the undeformed matrix. Channels are the sole slip paths and cause early onset of necking and loss of work-hardening in this alloy. Based on a review of the available literature, suggestions are made for further research of slip localization in V-base alloys .

INTRODUCTION

Interest in application of V-4 wt.% Cr - 4 wt.% Ti (V-4Cr-4Ti) 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 in the EBR-II reactor in the X530 experiment, which attained a fluence of ≈4 dpa. Due to limited space and the imminent shutdown of the EBR-II reactor, a complementary study was undertaken using single (4.5-MeV Ni^{++}) and dual ion beams (350-keV He^{+} simultaneously with 4.5-MeV Ni^{++}); details of this experiment can be found in Ref. 1.

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 capability in tensile specimens and an increase in ductile-to-brittle-transition temperature (DBTT) to above 300 K, as measured from instrumented Charpy tests on miniature ($^1/_3$-size) CVN specimens [2, 3]. 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 to 275°C [4].

In this report, we include preliminary results of TEM work aimed at exploring the deformation mechanisms in irradiated V-4Cr-4Ti. 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 postirradiation ductility.

EXPERIMENTAL PROCEDURES

Procedures used to prepare the V-4Cr-4Ti alloy (Heat 832665, ANL ID: BL71) are described in detail in Ref. 5. Specimen preparation steps for ion and neutron irradiation were provided in Ref. 1. Annealed (1 hr at 1050°C in 10^{-5} Pa UHV ion pumped vacuum) 3-mm-diameter disks were irradiated at the Argonne Tandem Accelerator facility operated by the Materials Science Division with single (4.5 MeV Ni^{++}) or dual ion beams (350 keV He^{+} simultaneously with 4.5 MeV Ni^{++}). The disks were later sectioned (surface layer of 800~1000 nm removed) and back-jet-thinned for TEM observations. The neutron irradiation was conducted during the final run of the EBR-II reactor in August/September 1994. Specimens were irradiated in Li-filled capsules, located in the core

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position, to ≈4 dpa (2.4×10^{15} $n \cdot cm^{-2} \cdot s^{-1}$, E>0.11 MeV) at 390°C. Details of this irradiation experiment were reported in Ref. 6.

To evaluate deformation processes in the irradiated materials, three types of specimens were examined with TEM. First, 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. Second, 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 postirradiation mechanical properties (BL71-50 irradiated in EBR-II, X530 experiment, subcapsule S9; tensile tested to fracture at 390°C with strain rate of $1.1 \cdot 10^{-3}$ sec^{-1}: YS = 805 MPa, UTS = 808 MPa, Uniform Elong. = 0.45%, Total Elong. = 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.

RESULTS

The microstructure of the unirradiated V-4Cr-4Ti alloy consists of ≈20 μm grains with loosely dispersed ≈200-nm-diameter Ti(CNO) precipitates. These particles form in the ingot and are commonly found in all U.S. V-Ti-Cr alloys, as seen in Fig. 1(a). The irradiated microstructure of V-4Cr-4Ti alloy changes with fluence and temperature of irradiation; details on evolution of the irradiated microstructure are reported in Ref. 1. Ion irradiation caused formation of a high density (≈$2 \cdot 10^{22}$ m^{-3}) of small dislocation loops and "black dot" point-defect clusters after irradiation to 5 dpa at 350°C with 4.5 MeV Ni^{++} ions, as shown in Fig. 1(b). Similarly, neutron irradiation caused formation of uniformly distributed point-defect clusters and dislocation loops (≈5-10 nm in diameter, number density on the order of ≈10^{22} m^{-3}). Figure 1(c) provides an example of the X530 EBR-II irradiated V-4Cr-4Ti microstructure.

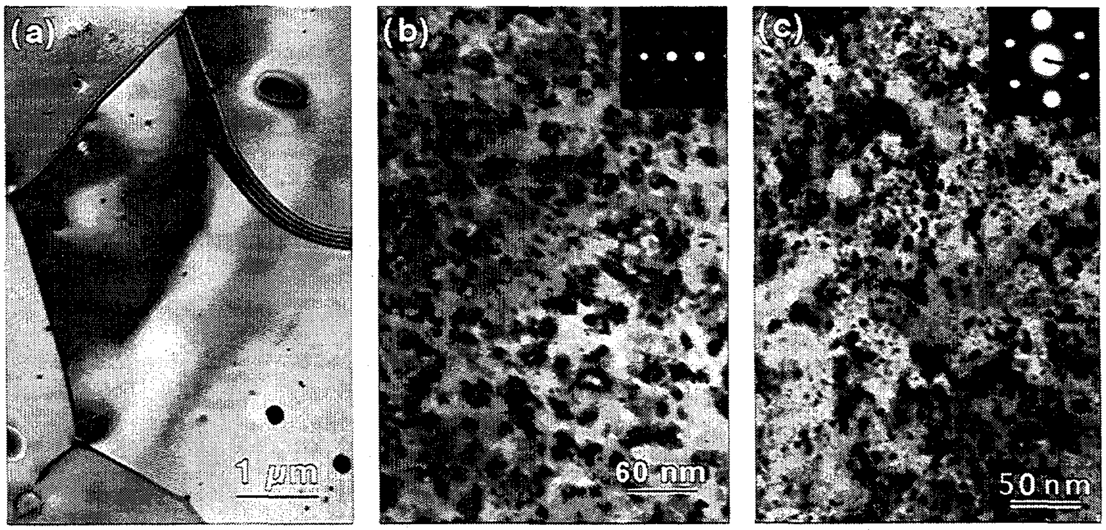

Figure 1. Typical microstructure of V-4Cr-4Ti alloy: (a) annealed for 1 hr at 1050°C in UHV furnace, (b) ion irradiated to 5 dpa at 350°C with 4.5 MeV Ni^{++}, and (c) irradiated to 4 dpa at 390°C in the X530 experiment.

Studies of the microstructure after plastic deformation were undertaken to determine the mechanisms for the radiation embrittlement of V-4Cr-4Ti irradiated below 420°C. 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. Figure 2 shows examples of the dislocation channels in ion-irradiated and indented V-4Cr-4Ti.

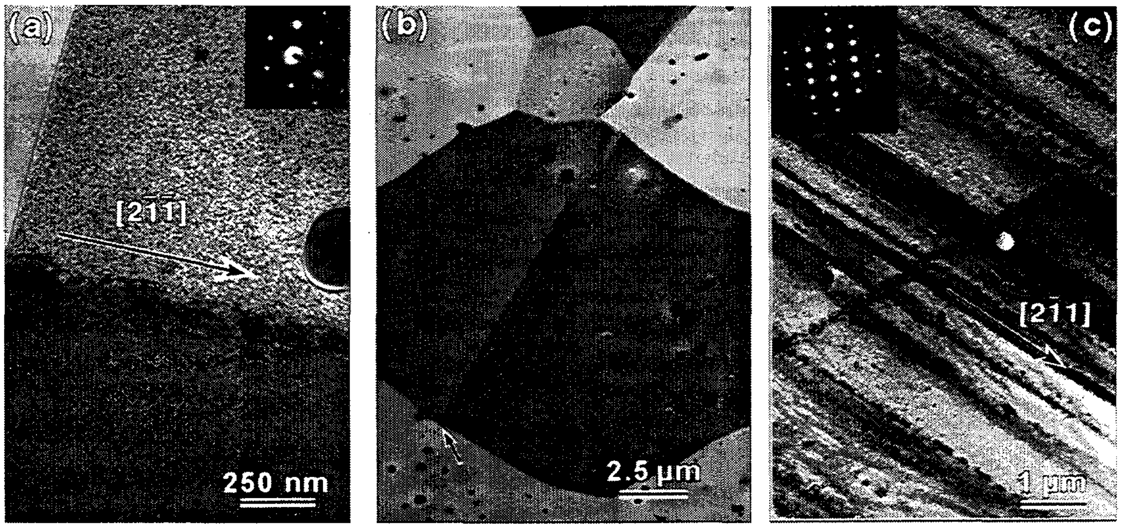

Figure 2. Dislocation channels observed in ion irradiated and indented specimens: (a) tangled dislocations forming a channel (0.5 dpa at 200°C), (b) channel arrested by grain boundary (10 dpa at 350°C), (c) channels in heavily deformed specimen (5 dpa at 420°C), two channel directions visible.

The miniature size of the limited number of the tensile specimens (gauge length dimensions: 0.6 × 1 × 5.6 mm) employed in the X530 experiment and the early onset of necking made the task of TEM specimen preparation extremely difficult. Therefore, sections of X530 neutron-irradiated Charpy specimens were first indented and TEM foils were then prepared from the region near indentation in the same way as in the ion-irradiated specimens. Again, the slip activity was found to be confined exclusively to 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 that massive cross-slips occurred during dislocation motion.

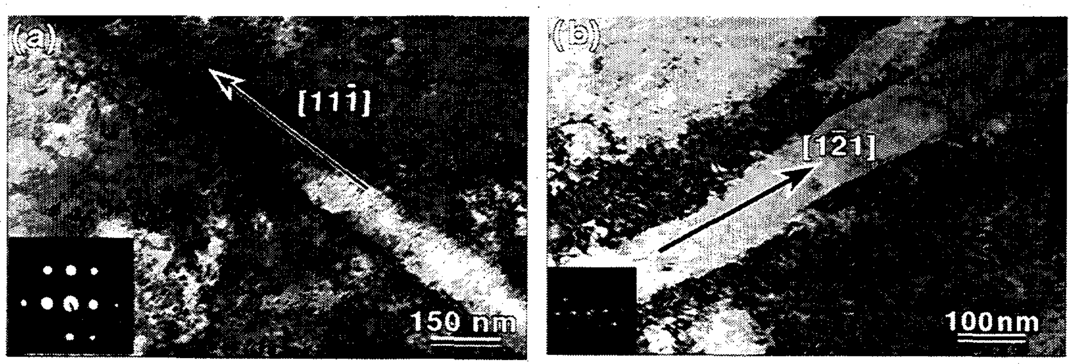

Figure 3. Dislocation channels in neutron-irradiated V-4Cr-4Ti alloy: (a) after indentation at room temperature, (b) after tensile deformation.

A limited number of TEM observations were carried out in the foils prepared from the necked regions of the tensile specimen (BL71-50). Although the fracture surface showed the ductile mode of fracture, TEM foils prepared from the necked region directly under the fracture surface exhibited dislocation channels as shown in Fig. 3(b). The channel direction was determined as <211> for the foil zone axis <111>, again consistent with the trace of {110} planes. The deviation of the channels from a straight line indicated the occurrence of cross-slip during deformation.

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 in neutron-irradiated material by TEM observation of the microstructure near a hardness indentation in Charpy specimens and parts of a tensile specimens within the necked region. In most cases, the channels contained dislocations, except for a few examples of channels nearly free of dislocations; these were found in exceptionally thin foils made from the neutron-irradiated and deformed specimen. This difference is attributed to dislocations slipping out of the foils in the vicinity of the surface, rather than a change in the character of the channels. 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.

Although our observations of dislocation channels are 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 [7] presented a detailed overview 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. 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.

Formation of dislocation channels was also observed by TEM in neutron-irradiated single crystals of vanadium alloys by Huang and Arsenault [8]. Dislocation channels studied in the present work bear a close resemblance to those observed by them.

Our observation of slip localization by dislocation channel formation explains the macroscopic loss of 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:

1. Predeformation of fully annealed material by cold working without significant rearrangement of the grain structure introduces dislocations which could be mobilized during the postirradiation deformation. In molybdenum, a critical density (6×10^8 cm^{-2} at 493 K) of obstacles is necessary for channel formation [9]. Below this density, dislocations distributed in the matrix act only as forest dislocations that provide the desired work-hardening effects. At higher dislocation densities, slip localization occurred upon redeformation. Dislocations uniformly distributed throughout the material could lead to more uniformly distributed slip in postirradiation deformation.
2. Introduction of incoherent, uncuttable particles can reduce slip localization. Dislocation channels were observed to form in Ti-base precipitation-strengthened alloys during deformation [10]. Aging studies showed that these small uniformly distributed coherent particles promote slip localization because dislocations were able to 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 homogenization of 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 uncuttable particles (V-17.7 wt.% Ti , ANL ID: BL15) and a cold-worked specimen of V-4Cr-4Ti alloy (experimental heat, ANL ID: BL47), which were used in earlier neutron irradiation studies and were replaced by the purer alloy in the current work, exhibited uniform elongation ≥2% [11]. The vanadium alloy development should be extended along this line to seek slip homogenization during deformation after low-temperature-irradiation.

CONCLUSIONS

1. The usefulness of ion irradiation as a tool in assessing the properties of fusion candidate materials should be noted. In the present case, ion irradiation was designed to provide information about both the swelling and embrittlement potential of a V-4Cr-4Ti alloy irradiated at low temperatures. In ion-irradiated specimens, voids/cavities were not observed by TEM and slip localization by dislocation channels was found near an indentation.

2. Dislocation channels were observed in V-4Cr-4Ti alloy deformed after irradiation. Slip localization resulted from the formation of defect-free, easy glide paths for slip dislocations. This is suggested to be the cause of premature necking and loss of work-hardening of the tensile specimen (BL71-50) 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 X530 experiment.

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 refinement is suggested as an alloy development task to minimize post-irradiation embrittlement at low temperatures (<420°C).

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