EFFECT OF PREINJECTED HELIUM ON THE RESPONSE OF V-20Ti
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Abstract

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Vanadium-20% titanium tubes, pressurized to stresses of 34 and 39 MPa, were irradiated in the Experimental Breeder Reactor (EBR-II) at 700°C to a displacement damage level of 22 dpa. Sections of the tubes were injected with 15 appm He prior to irradiation to determine the effect of helium on the microstructural and creep response of this alloy to irradiation. It was found that helium promoted cavity formation, primarily within existing precipitates, but total swelling remained low. Helium also significantly enhanced creep deformation. The results indicate that the increase in creep deformation in the presence of helium may be very sensitive to stress.

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Introduction

Vanadium alloys are of interest as candidate alloys for possible structural applications in fusion reactor devices. One of the advantages of this class of alloy is its potential for use at a higher service temperature than other candidate alloys such as ferritic or austenitic stainless steels. More recently, interest in vanadium alloys has increased because of their inherent low activation following neutron irradiation [1]. Vanadium alloys maintain promising properties following irradiation [2]. As candidate materials for fusion applications, though, one concern that requires investigation, in addition to displacement damage effects, is how the postirradiation properties are affected by the helium production and stress in a fusion environment. Unfortunately, a direct evaluation of simultaneous helium production and displacement damage during neutron irradiation is not possible at present. Therefore, one must resort to techniques which simulate the fusion environment as closely as possible. One such technique, used previously and also in the present study, is to implant helium into the specimens prior to neutron irradiation. In order to make as direct a comparison as possible to evaluate helium effects on the irradiation response of the vanadium alloys, helium was implanted into only a section of the vanadium tubes which were subsequently pressurized and neutron irradiated. In this way, comparisons could be made between stressed regions with and without helium in the same specimen and possible variations among specimens in temperature, neutron spectrum, or preparation would be avoided.

The use of helium implantation prior to irradiation has often been used in the past. Examination of tensile properties of specimens

preinjected with helium has shown that helium decreases the ductility for a number of vanadium alloys [2,3]. In addition, the preinjected helium promoted cavity formation although negligible total swelling was found [3,4]. In the present study, the helium was injected into pressurized tube samples so that the additional factor of irradiation under stress could be examined. In a similar study on helium injected pressurized tubes of type 316 stainless steel [5], it was found that the helium suppressed swelling.

Experimental Procedure

The alloy used in the present investigation was vanadium with 20 wt % Ti (V-20Ti), one of the several vanadium alloys being evaluated for potential fusion applications. A list of minor element concentrations in the alloy is given in Table 1. The tubing was manufactured by Superior Tube Company and swaged down to its final size at the Oak Ridge National Laboratory (ORNL). After swaging, the tube was given a final anneal at 900°C for 30 min at $\sim 5 \times 10^{-5}$ Pa.

The specimens employed in this study were thin-walled tubes pressurized with helium. The tubing had an outer diameter of 4.57 mm and a wall thickness of 0.25 mm. They were approximately 51 mm in length and had welded end caps. Tubes V23 and V24 were pressurized to produce a hoop stress of 34 MPa (5000 psi) at the irradiation temperature, whereas tube V22 had a hoop stress of 39 MPa (5700 psi). The central section (~ 13 mm in length) of each specimen was preinjected with helium at 20°C at the Oak Ridge Isochronous Cyclotron (ORIC). The end sections of the tubes contained no preinjected helium. An accelerator energy of 51.6 MeV was degraded with a beryllium wedge-shaped wheel in order

to uniformly distribute the helium throughout the thickness of the tube walls. Helium preinjection levels of 15 appm were attained. The tube specimens were then irradiated in the EBR-II at a nominal temperature of 700°C to a dose of 3.9×10^{26} neutrons/m² ($E > 0.1$ MeV), corresponding to a total damage of 22 dpa and a direct helium production of 1 appm [6].

The diameters of the tube specimens were measured after filling with helium (prior to irradiation) and after neutron irradiation. These measurements were made at the Hanford Engineering Development Laboratory (HEDL). After the diameter measurements, the tubes were cut into ring sections corresponding to the regions with and without preinjected helium. Immersion density measurements were made on these ring sections prior to the preparation of TEM specimens. Specimens for TEM examination were cut from the ring sections and ground flat on 600-grit emery paper. The wafers were subsequently jet polished in a 12.5 vol % solution of conc. H₂SO₄ in methanol at -25°C and examined in a 100 kV analytical electron microscope.

Experimental Results

Dimensional Measurements

Tube diameter measurements are available for tubes V22 and V24 and the change in tube diameter is plotted in Fig. 1 for both of these specimens. The increase in tube diameter found for both specimens is a combination of plastic deformation and swelling. Except for the last data point in Fig. 1(a), which seems to be an outlier, the tube expansion was fairly uniform across its length. A slight increase in the tube

expansion may exist in the helium preinjected area. For the tube with the greater hoop stress [V22, Fig. 1(b)], a large increase in tube expansion is found in the center section of the tube, corresponding to the region that contains 15 appm preinjected helium. Thus, for sample V22, it is clear that the presence of helium leads to a larger tube expansion.

Density Measurements

After irradiation, the three tubes were cut into rings and density measurements were made on these rings from all three tubes. The results are plotted in Fig. 2. Also shown as a dashed line is the density for identical, but unirradiated, ring sections. The data indicate that a slight (1.2%) decrease in density occurs upon irradiation. No significant difference in density is seen between the regions with and without helium.

Microstructure

The microstructure of the unirradiated vanadium tube is typical of that for a cold-worked material that has undergone some recovery as shown in Fig. 3(a). The microstructure contains an extensive network of dislocation segments and subgrain boundaries. Second phase particles, ranging in size from less than 0.1 μm to several microns in diameter, were scattered throughout the material [Fig. 3(b)]. Quantitative x-ray dispersive spectroscopy (EDS) analyses performed on an analytical electron microscope showed that the detectable element composition ($Z > 11$) of the second phase was 96 wt % Ti and 4 wt % V. It is likely

that the phase is a Ti-V oxide, nitride, or carbide. Unpublished electron diffraction data by Tanaka [7] indicates that it is probably an oxide.

Irradiation to 22 dpa produced cavities in the second phase of the sections of V-20Ti tubes without implanted helium, as shown in Fig. 4. Stereo-microscopy confirmed that the cavities were within the second phase particles and not along the particle/matrix interface. Cavities were not observed in the matrix. Under the same irradiation conditions, much larger and often elongated cavities in the second phase particles in the helium-implanted section of the same tube were found, as illustrated in Fig. 5. Stereo-microscopy showed that the unusual cavity distribution was within the particles and appeared to be aligned along a specific crystallographic plane. Such alignment of cavities was observed frequently in the second phase particles. Although no quantitative measurements were made, it was obvious that cavitation or swelling in the second phase was significantly greater in the section of the tube containing helium. Limited matrix swelling of the helium-injected sections was also found, but the actual volume change was small as evident from the micrograph in Fig. 6. In this typical matrix area, only a few small cavities are present. Under all conditions, in sections with and without preinjected helium, the total swelling was minimal, well below 1%.

Discussion

The dimensional changes measured on the pressurized tubes can be due to two factors: creep deformation and cavitation swelling. Based on the TEM observations, swelling is minimal and therefore contributes

only marginally to the change in tube diameters. This is supported by the density measurements which showed only a small decrease in density, even in the tube specimen that changed in diameter by more than 5%. Therefore, the diameter changes can be attributed primarily to creep deformation. The fact that the diameter change was significantly greater in the tube with a higher stress (V22) agrees with this conclusion. The creep deformation is a combination of thermal and irradiation creep, but these two mechanisms will not be distinguished in the subsequent discussion.

The aim of the present study was to evaluate the effect of helium on the irradiation behavior of the V-20%Ti alloy. Two effects can be identified, one on the microstructural behavior and the other on the creep response. Considering the former effect first, it is apparent that helium promoted swelling within the titanium-rich second phase particles (compare Figs. 4 and 5). The region with implanted helium also experienced some limited cavity formation within the matrix. Thus, although V-20 Ti is quite resistant to swelling when irradiated in the absence of helium [8], some limited swelling is found when helium is present. The promotion of swelling by helium is characteristic of systems in which cavity formation is restricted. The helium bubbles act as nuclei for voids. Such an effect has been observed in other vanadium alloys [4,9] as well as in other systems, such as ferritic steels [10]. The opposite effect was found in helium-injected pressurized tubes of type 316 stainless steel [5], where helium inhibited swelling. The difference in behavior can be attributed to the fact that voids form in the stainless steel without helium and the presence of helium changes the cavity growth rates by influencing the relative sink strengths [5].

It is interesting to note that nearly all of the voids observed were located within second phase particles. Such behavior is unusual, but not unique. The presence of cavities within precipitates has recently been observed in other EBR-II-irradiated vanadium alloys [9] as well as in large precipitates in nickel-doped ferritic alloys [11]. In the present case, such void formation within precipitates may be due to helium production during irradiation. Although the average amount of helium produced in the matrix during irradiation is small (1 appm), within the titanium-rich precipitates over 5 appm He is produced due to the titanium concentration alone [6]. In the region with preinjected helium, bubbles already exist and the irradiation-produced helium may preferentially segregate to these bubbles. With this additional helium, the critical radius for cavity growth may be surpassed, leading to void growth during irradiation.

Since the major portion of the diameter changes measured was due to creep deformation, it is clear from Fig. 1(b) that helium promotes creep. This result is in agreement with an earlier study on stainless steels [12] which showed that helium promoted creep when grain boundaries were free to slide and were not inhibited by precipitates, as in the case of V-20Ti. This enhanced creep effect is not as large when the stress and resultant creep deformation are smaller [Fig. 1(a)]. The increase in creep deformation due to helium at the higher stress is quite dramatic and the effect is unexpectedly strong. Whereas the stress in tube V22 was only 15% higher, a 250% increase in tube expansion was measured. This effect should be confirmed by additional experimentation, for if it is real, it will have major consequences on the evaluation of postirradiation creep properties. It should be noted

that among the vanadium alloys under consideration for fusion applications, other alloys, such as V-15Cr-5Ti, have better creep resistance than the V-20Ti alloy considered here [13]. The V-15Cr-5Ti alloy also has precipitates along its grain boundaries and therefore it may not show the same helium-enhanced creep found here. Such considerations should be important in any future vanadium alloy development work.

Summary

Examination of pressurized tubes of V-20%Ti irradiated in EBR-II at 700°C indicated that substantial creep deformation occurs at a hoop stress of 39 MPa. The presence of implanted helium significantly enhanced the creep deformation. Microstructural examination indicated that helium also promoted cavity formation, primarily within titanium-rich second phase particles. However, after irradiation to a damage level of 22 dpa, only very modest total swelling was found in all samples. Because of the potential sensitivity of creep deformation to helium, creep may be an important property to monitor in future studies on the irradiation response of vanadium alloys.

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Table 1. Impurity element concentrations
in V-20%Ti tubing (wt ppm)

Mg	10
Al	10
Si	80
S	20
Ca	10
Cr	90
Fe	200
Ni	20
Cu	30
Sn	40
W	20

Figures

Fig. 1. Percent change in tube diameter vs. position for tubes (a) V24 and (b) V22. Shaded region corresponds approximately to helium-containing section.

Fig. 2. Immersion density measurements vs. tube position for ring sections from tubes (a) V22, (b) V23, and (c) V24. Dashed lines indicate unirradiated density.

Fig. 3. Microstructure of unirradiated V-20Ti tube showing (a) the cold-work and recovered structure with characteristic dislocation networks and numerous subgrain boundaries, and (b) a second phase particle in the grain matrix.

Fig. 4. Cavities within second phase particle in section of tube V23 without helium.

Fig. 5. Large elongated cavities aligned on crystallographic plane in second phase particle in section of irradiated tube V23 containing ~15 appm He.

Fig. 6. Typical matrix region in section of irradiated V-20Ti tube containing ~15 appm He that contains a few small cavities.

FIGURE 1

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FIGURE 2

a

b

FIGURE 3

FIGURE 4

FIGURE 5

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FIGURE 6