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Biaxial thermal creep of two heats of V4Cr4Ti at 700 and 800 °C in a liquid lithium environment

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Abstract

This paper presents biaxial thermal creep results for the vanadium alloy V4Cr4Ti in liquid lithium using US Heat 832665 and two batches of tubing made from NIFS-Heat-2. The tests were performed at 700 and 800 °C over a stress range of 30–120 MPa using pressurized tube specimens. Lithium environments changed C, N and O concentrations in V4Cr4Ti. Oxygen removal from the alloy to lithium was small when there was a significant pickup of nitrogen during exposures. The creep response of V4Cr4Ti was characterized by an inverted primary creep followed by a secondary creep or an accelerating creep up to creep rupture. A normal primary creep was also observed in US Heat 832665 when tested at 700 °C and 120 MPa. Improved creep rupture properties were observed in the JP-NIFS-Heat-2 specimens compared to the US-NIFS-Heat-2 specimens. Creep response of V4Cr4Ti is apparently dependent on the heat, tubing production, and stress and temperature conditions.

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1. Introduction

Vanadium alloys are candidates for lithium cooling and breeding blanket structural applications in advanced fusion systems. V4Cr4Ti is a reference alloy that has been studied extensively by the fusion community. The upper operating temperature limit of vanadium alloys is expected to be in the range of 650–750 °C, i.e. 0.43–0.47 T_m [1], where high temperature tensile properties and creep performance are critical for fusion structural applications. Therefore, it is of high importance to evaluate creep performance of V4Cr4Ti alloys in liquid lithium environments.

Vanadium alloys are highly susceptible to contamination by interstitial impurity elements, such as oxygen, nitrogen and carbon. Previous investigations have shown that interstitial impurities can

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significantly affect tensile properties and load-carrying capacity at high temperatures [2]. The strength is increased and the ductility is decreased by pickup of interstitial solutes. The susceptibility to pickup of interstitial elements is more serious if long exposure times are involved. Interstitial impurities can be introduced during fabrication, welding and joining processes and in service. Liquid lithium creates a unique environment that can substantially change the interstitial concentration (C, N, O) in vanadium alloys. Thermodynamic calculations indicated that oxygen preferentially transfers from vanadium to lithium, and nitrogen and carbon move in the opposite direction [3]. The additions of substitutional solutes such as Cr and Ti do not change the impurity transfer direction, but may alter the kinetics of impurity reactions. There are insufficient data characterizing the interstitial reactions of V4Cr4Ti in liquid lithium and their effects on mechanical behavior, particularly on creep performance.

Preliminary work has been carried out to measure the creep properties of V4Cr4Ti in lithium environments at 665 and 765 °C on the US Heat 832665 by Grossbeck [4,5]. Grossbeck's results indicated that the creep strain rate of V4Cr4Ti in liquid lithium was higher than in vacuum due to loss of oxygen from exposure to lithium. Additional data are needed to characterize lithium thermal creep of different heats and tubing batches of V4Cr4Ti. This paper summarizes the experimental results on the creep deformation of pressurized creep tubes made from US Heat 832665 and from two batches of tubing of NIFS-Heat-2 in the stress range of 30–120 MPa while exposed to liquid lithium at 700 (0.45 T_m) and 800 °C (0.50 T_m) for up to 2500 h. The creep behavior of V4Cr4Ti is characterized by the curves of creep strain vs. exposure time and creep rate vs. creep strain. The creep response of three batches of V4Cr4Ti tubing in liquid lithium was compared.

2. Experimental procedure

Pressurized creep tube specimens were fabricated from V4Cr4Ti alloy tubing from the US Heat 832665 and from two batches of tubing of the NIFS-Heat-2. The Japan tubing of NIFS-Heat-2 was fabricated using tube vendors in Japan (designated JP-NIFS-Heat-2) and the US tubing of NIFS-Heat-2 was fabricated using vendors in US (designated US-NIFS-Heat-2). The detailed manufacturing processes of JP-NIFS-Heat-2 creep specimens were reported by Fukumoto et al. [6], and the fabrication of US Heat 832665 and US-NIFS-Heat-2 creep tube specimens can be found in the references of [7,8]. The US Heat 832665 pressurized tubes contained 161 wppm N, 509 wppm C and 700 wppm O for a total impurity content of 1370 wppm. The US-NIFS-Heat-2 creep tube specimens contained 277 wppm N, 586 wppm C and 519 wppm O for a total impurity content of 1382 wppm. The JP-NIFS-Heat-2 creep tube specimens had 120 wppm N, 120 wppm C and 330 wppm O for a total impurity content of 570 wppm. Note that the JP-NIFS-Heat-2 tubing has a higher purity than the other two batches of V4Cr4Ti tubing. All creep specimens were annealed at 1000 °C for 1–2 h which produced an average grain size of 20–30 μm. The creep tube specimens have a nominal outer diameter of 4.57 mm and a wall thickness of 0.254 mm with a total length of 25.4 mm.

The US-NIFS-Heat-2 and JP-NIFS-Heat-2 creep tube specimens were tested at 800 °C over a stress range of 30–90 MPa. The US Heat 832665 and JP-NIFS-Heat-2 creep tube specimens were tested at 700 °C at stresses of 90–120 MPa. All the thermal exposure tests of pressurized creep tubes were performed in static liquid lithium. The details of the experimental setup were described previously [4,9]. Pure lithium (>99.9%) was supplied by Sigma–Aldrich (161 ppm N) or by Chemetall Foote Corp. (65 ppm N). To minimize contamination of lithium metal by nitrogen and carbon, the commercial lithium was either purified by hot-trapping with Zr getters prior to exposure tests, or included Zr or Ti getters during lithium exposure tests. A batch of lithium was used for no more than two exposure tests and then replaced with a new batch of lithium. After exposure, all specimens were removed from lithium and cleaned using liquid anhydrous ammonia. This cleaning method generates very little hydrogen so that hydrogen embrittlement can be minimized. The creep specimens were then measured for creep deformation and reloaded into liquid lithium for further exposure. This process was repeated at exposure intervals of 255, 499, 1019 and 2019 h at 700 °C and at intervals of 428, 688, 943, 1443, 1963 and 2580 h for JP-NIFS-Heat-2 creep tube specimens at 800 °C and at intervals of 260, 515, 1015, and 1535 h for US-NIFS-Heat-2 creep tube specimens at 800 °C.

The outer diameters (OD) of pressurized creep tubes were measured at room temperature before and after each exposure using a laser profilometer with a resolution of 0.2 μm. A specimen was loaded vertically into the laser profilometer with the same orientation each time, and the tube profile was obtained by measuring the central 12.7 mm of the tube at 500 unique locations using a standard helical pattern program. The mean OD of a tube was computed by averaging the central 300 measurements to preclude any end effects. A standard gauge pin was periodically measured to compensate for any errors from the laser profilometer and the environment. Each specimen was measured three times in a non-consecutive manner, and the average from these three measurements was used to determine the creep strain. The mid-wall effective creep strain of a pressurized creep tube was determined by assuming that the material is incompressible and the deformation is uniform in plastic flow. The mid-wall von Mises effective stress of a pressurized creep tube was calculated using the analytical solution of stress state for a thick-walled pressured cylinder [10]. The dimensions of the creep specimen at the test temperature were compensated by the linear thermal expansion coefficient of vanadium alloys [11].

Coupon specimens of V4Cr4Ti (nominal dimensions of 25 × 6 × 3 mm) were exposed to liquid lithium along with pressurized creep tubes for chemical analysis of O, N and C after each exposure to monitor contamination from lithium environments. Coupon specimens were machined from a 40% cold-rolled R-plate of the US Heat 832665. The initial contents of interstitial impurities in coupon specimens were 322 wppm O, 99 wppm N and 122 wppm C.

3. Results and discussion

3.1. Chemical changes

The changes in interstitial impurity (C, N, O) concentration in coupons specimens are shown in Fig. 1 as a function of lithium exposure time at 700 and 800 °C. Significant contamination by nitrogen was observed at both temperatures. The source of nitrogen contamination is uncertain. The decrease in nitrogen content in the coupon specimen at 2000 h and 700 °C is likely due to stronger chemical affinity of nitrogen with Ti getters that were included during exposure. There was a slight pickup of carbon as exposure time increased. The decrease in oxygen contents in V4Cr4Ti coupon specimens was much smaller than expected. Oxygen concentration dropped only about 30 ppm after 1500 h at 800 °C or after 2000 h at 700 °C. Grossbeck [5] reported a loss of 600 ppm oxygen and an increase of ~50 ppm nitrogen after ~2500 h at 800 °C. It is possible that the significant pickup of nitrogen in the present tests altered the kinetics of interstitial solute transfer in the V4Cr4Ti–lithium system, leading to reduced oxygen egress (e.g., due to an enhanced formation of Ti (C, O, N) precipitates). It should be mentioned that changes in interstitial impurity contents in creep tube specimens may not be the same as in coupon specimens, due to different volume and area ratios between the two specimen geometries. Note also that creep tubes were under straining during exposure, while coupon specimens were in the stress-free condition. However, the data shown in Fig. 1 demonstrate the general trend of chemical changes in creep tube specimens that were exposed to liquid lithium.

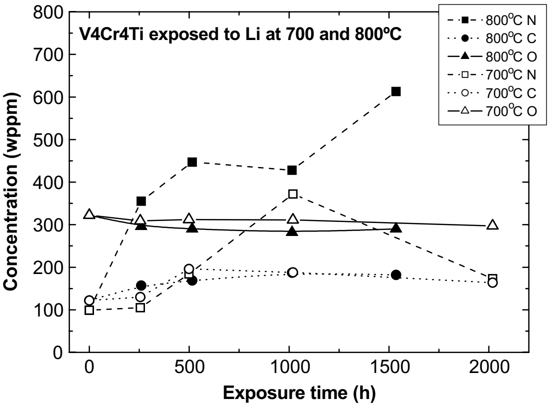

Fig. 1. Interstitial impurity contents (O, N, C) in V4Cr4Ti after lithium exposure at 700 and 800 °C.

3.2. Creep curves

The US-NIFS-Heat-2 and JP-NIFS-Heat-2 pressurized creep tubes were tested in liquid lithium at 800 °C at stress levels 30–90 MPa. Their creep strain–exposure time curves are shown in Fig. 2(a). To illustrate the change in creep rate during the test, the creep strain–time curves were differentiated to obtain the creep strain rate using finite difference formulae:

$$\dot{\varepsilon}(\bar{\varepsilon}_n) = \frac{\varepsilon_{n+1} - \varepsilon_n}{t_{n+1} - t_n}, \quad \bar{\varepsilon}_n = \frac{\varepsilon_{n+1} + \varepsilon_n}{2}, \tag{1}$$

where ε_{n+1} and ε_n represent the effective strain at the two consecutive exposure time t_{n+1} and t_n, respectively, and $\bar{\varepsilon}_n$ is the average strain between the two

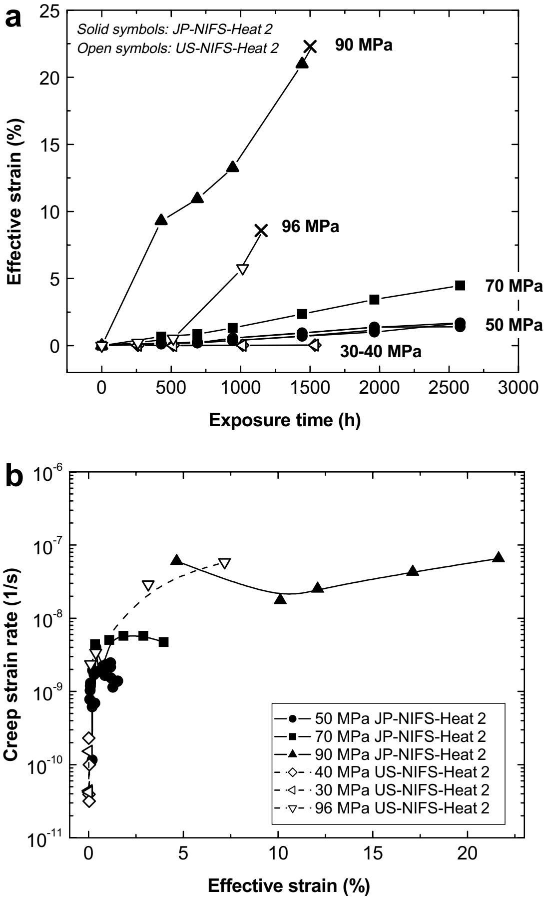

Fig. 2. Creep curves of V4Cr4Ti exposed to liquid lithium at 800 °C (a) effective strain vs. exposure time, and (b) creep strain rate vs. effective strain.

time intervals. The creep strain rate is plotted as a function of the average strain on a semi-log scale and shown in Fig. 2(b) for the 800 °C lithium exposure tests. Creep deformation of V4Cr4Ti was found to be sensitive to the magnitude of the applied stress. The creep of V4Cr4Ti did not follow the classical three-stage creep pattern (primary, secondary and tertiary) under the test conditions shown in Fig. 2. The US-NIFS-Heat-2 specimen tested at 96 MPa exhibited a continuously increasing creep rate with increasing strain up to creep failure. The 30–40 MPa US-NIFS-Heat-2 creep specimens showed an increasing creep rate with creep strain in the primary stage as well. This inverted primary transient behavior is often observed in solid solution strengthened alloys [12]. The creep response of the JP-NIFS-Heat-2 specimens is similar to that of the US-NIFS-Heat-2 specimens, i.e. an inverted primary transient followed by either a steady-state or an accelerating creep up to creep rupture. If a steady-state is observed, it often occurred at about 1–2% creep strain. However, there was a short decelerating creep (creep rate decreasing with increasing strain) in the primary stage that was observed only in the JP-NIFS-Heat-2 specimens but not in the US-NIFS-Heat-2 specimens, as shown in Fig. 2.

Lithium thermal exposure tests were performed at 700 °C on the US Heat 832665 and JP-NIFS-Heat-2 pressurized creep tubes in the stress range of 90–120 MPa. The creep strain–time curves and creep rate–strain curves of the 700 °C tests are shown in Fig. 3(a) and (b), respectively. The JP-NIFS-Heat-2 creep tube specimens exhibited an increasing creep rate followed by a decreasing creep rate and later another increasing creep rate with increasing creep strain, similar to their creep

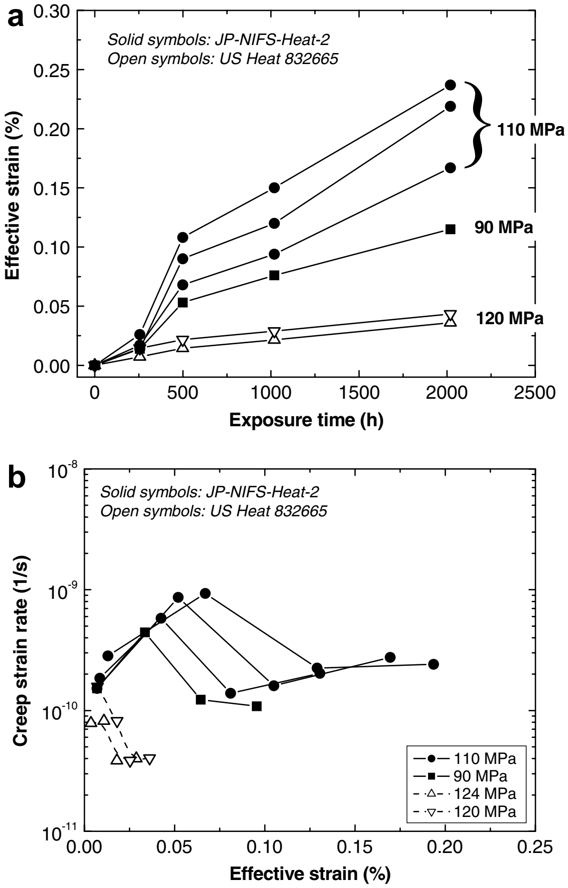

Fig. 3. Creep curves of V4Cr4Ti exposed to liquid lithium at 700 °C (a) effective strain vs. exposure time, and (b) creep strain rate vs. effective strain.

behavior at 800 °C. The US Heat 832665 creep tube specimens, however, showed a normal primary transient behavior when tested at ~120 MPa. There were not sufficient data to characterize the secondary and tertiary creep behavior at 700 °C.

The method of production of tubing apparently played an important role in the creep response of V4Cr4Ti when exposed to liquid lithium. The JP-NIFS-Heat-2 tubing and the US-NIFS-Heat-2 tubing were made from the same heat (NIFS-Heat-2) but by different vendors. The JP-NIFS-Heat-2 tubing has less interstitial impurity contamination than the US-NIFS-Heat-2 tubing (the total interstitial impurity level in the JP-NIFS-Heat-2 was about 40% of that in the US-NIFS-Heat-2). Both the JP-NIFS-Heat-2 and the US-NIFS-Heat-2 exhibited an inverted primary transient behavior followed by either a steady-state or an acceleration of creep until failure for the stress levels and temperatures examined. The difference in creep character between these two batches of tubing was a deceleration of creep in the early stage of creep that was observed only in the JP-NIFS-Heat-2 specimens. Similar decelerating creep phenomena were reported in the literature for nickel alloys [13,14]. This behavior was explained by strengthening of oxide and nitride formed on specimen surfaces of nickel alloys when tested in air. Visual inspection of creep tube specimens of V4Cr4Ti indicated discoloration of the JP-NIFS-Heat-2 specimens, but no discoloration was observed on the US-NIFS-Heat-2 creep tube specimens. Auger Electron Spectroscopy detected nitrogen in the discolored area of specimen surfaces. The formation of a nitride film on the JP-NIFS-Heat-2 specimen surface was likely associated with the observed deceleration of creep. It is not understood why the discoloration and decelerating creep was observed in JP-NIFS-Heat-2 specimens but not in US-NIFS-Heat-2 creep tube specimens.

The tubing processing affected rupture properties as well. The 96 MPa US-NIFS-Heat-2 creep tube specimen failed at 1148 h at a creep strain of 8.6%, while the 90 MPa JP-NIFS-Heat-2 pressurized tube failed at 1499 h at a creep strain of 22.3%. The more ‘pure’ JP-NIFS-Heat-2 specimen exhibited a higher rupture strain and longer rupture life than the less ‘pure’ US-NIFS-Heat-2. This implies that alloy purity may be important to rupture properties. Small changes in grain size, texture, tubing quality, etc. could also be important factors in controlling creep strength.

4. Conclusions

Thermal creep behavior of three batches of V4Cr4Ti tubing made from NIFS-Heat-2 and US Heat 832665 was investigated in a liquid lithium environment at 700 and 800 °C. Significant pickup of nitrogen was observed in both 700 and 800 °C lithium exposure tests. The loss of oxygen from V4Cr4Ti to lithium was much smaller than that reported in previous studies. The kinetics of oxygen transfer in a V alloy–Li system apparently was affected by nitrogen contamination of the V4Cr4Ti. The creep properties of V4Cr4Ti are dependent on alloy heat, tubing production method, stress and test temperature. The creep behavior of V4Cr4Ti in liquid lithium is characterized by an inverted primary transient period followed by a steady-state or an accelerating creep to rupture. Normal primary creep was observed in US Heat 832665 at 700 °C. The JP-NIFS-Heat-2 with less initial interstitial impurities showed a higher rupture strain and a longer rupture life than the US-NIFS-Heat-2 at 800 °C, a difference due to impurity content and/or material variables introduced in the production process. In addition, a short deceleration in the primary creep stage was observed only in the JP-NIFS-Heat-2 creep specimens, not in the US-NIFS-Heat-2 creep specimens.

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