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Uniaxial creep behavior of V–4Cr–4Ti alloy

K. Natesan *, W.K. Soppet, A. Purohit

Energy Technology Division, Argonne National Laboratory, ET-212, 9700 South Cass Avenue, Argonne, IL 60439, USA

Abstract

We are undertaking a systematic study at Argonne National Laboratory to evaluate the uniaxial creep behavior of V–Cr–Ti alloys in a vacuum environment as a function of temperature in the range of 650–800 °C and at applied stress levels of 75–380 MPa. Creep strain in the specimens is measured by a linear-variable-differential transducer, which is attached between the fixed and movable pull rods of the creep assembly. Strain is measured at sufficiently frequent intervals during testing to define the creep strain/time curve. A linear least-squares analysis function is used to ensure consistent extraction of minimum creep rate, onset of tertiary creep and creep strain at the onset of tertiary creep. Creep test data, obtained at 650, 700, 725 and 800 °C, showed power-law creep behavior. Extensive analysis of the tested specimens is conducted to establish hardness profiles, oxygen content and microstructural characteristics. The data are also quantified by the Larson–Miller approach, and correlations are developed to relate time to rupture, onset of tertiary creep, times for 1% and 2% strain, exposure temperature and applied stress.

1. Introduction

Refractory alloys based on V–Cr–Ti are being considered for use in first-wall structures in advanced blanket concepts that employ liquid Li as a coolant and breeding material. Furthermore, advanced concepts that use He as a coolant also require structural alloys, such as the V–Cr–Ti alloys, that can withstand thermal loading at high temperature. For the advanced fusion system design concepts, it is important to base the establishment of the upper temperature limits for structural components on various design criteria. At temperatures above 600 °C, the time-dependent creep properties of V alloys must be considered when evaluating performance limits. Time-dependent allowable primary stress intensity is defined as

- Two-thirds of the minimum stress that corresponds to the average creep rupture time t at temperature T.
- 80% of the minimum stress that corresponds to time t and temperature T for onset of tertiary creep.
- Minimum stress necessary to cause a creep rate or a creep strain of some value (e.g., 1% in 10 000 h or 5% creep strain) in time t and at temperature T.

Limited data on the creep and stress-rupture properties of unirradiated V and V-base alloys have been reported by Chung et al. [1], Schirra [2,3], Böhm and Schirra [4], Bajaj and Gold [5], Kainuma et al. [6], Van Thyne [7], Carlander [8], Böhm [9] and Tesk and Burke [10]. Results from these studies showed that alloying of V with up to 3 wt%Ti produces a significant increase in creep strength, but that additional Ti concentrations cause a substantial decrease in creep resistance. Addition of up to 15 wt% Cr to V–(3–5)Ti alloy also produces significant strengthening in creep, whereas addition of 1 wt% Si to V–3wt%Ti causes a significant decrease in the creep strength of this alloy. The data reported in these studies indicate that the substitutional and interstitial element content of V-base alloys significantly affects the creep properties. Insufficient data are available on the reference composition of V–4wt%Cr–4 wt%Ti, especially at temperatures above 650 °C.

Long-term creep properties of the V-base alloys will be influenced by the time-dependent nucleation and growth of precipitates that involve non-metallic elements such as O, N and C. Several of the microstructural

* Corresponding author. Tel.: +1-630 252 5103; fax: +1-630 252 3604.
E-mail address: natesan@anl.gov (K. Natesan).

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studies of V-base alloys have identified precipitates such as face-centered-cubic Ti(O, N, C) with variable O, N and C ratios. It is essential to establish the time-dependent evolution of type, number and location of precipitates in V-base alloys to establish the creep mechanism(s) and to correlate the microstructural development with creep properties.

2. Experimental procedure

The program discussed here involves experimental evaluation of the uniaxial creep properties of V–4Cr–4Ti alloy in a high-vacuum environment at temperatures of 650–800 °C, with emphasis on baseline creep behavior of the alloy and on development of correlations among various creep properties. The test program is aimed at obtaining the steady-state creep rate, onset of tertiary creep, rupture strain, rupture life and times for accumulation of 1% and 2% strain. Flat creep specimens, 1 mm thick, were fabricated according to ASTM Standard E8-96a. Tests were conducted at 650, 700, 725 and 800 °C on specimens annealed at 1000 °C for 1 h in vacuum. The specimens were wrapped in Ti foil to minimize contamination of the sample, especially by O. The creep-test procedure was in accordance with ASTM E139-96. Creep strain in the specimen was measured by a linear-variable-differential transducer (LVDT), which was attached between the fixed and movable pull rods of the creep assembly. Displacements of 5×10^{-3} mm could be accurately determined with the LVDT. Before each test, the LVDT was calibrated by measuring its output for displacements that were set manually on a micrometer. The linear portion of the calibration curve was used to measure strain in a specimen during creep testing. The strain was measured at sufficiently frequent intervals during a test to define the creep strain/time curve.

A three-zone resistance-heated furnace was used in each testing machine to conduct creep tests at elevated temperatures. Chromel–alumel thermocouples with small beads were used to measure specimen temperature; ceramic insulators were used on the thermocouples in the hot zone. In general, three thermocouples were fed through the specimen chamber, one spot-welded onto each end of the grips on the specimen near the shoulder region, the third, held in the vacuum environment adjacent to the gauge-length portion of the specimen. Temperature was maintained within 2 °C of the desired value for each test. The specimens were loaded at a constant rate to full load at the test temperature.

3. Results and discussion

Fig. 1 shows the creep strain/time plot for V–4Cr–4Ti alloy specimens that were tested in vacuum at 650 and 725 °C. The data indicate that the primary creep period is negligible for all tests and the secondary (or linear) creep portion of the curve is small. The curves showed an accelerating creep behavior over the range of the present tests, especially at 725 and 800 °C. The creep strain/time curves were analyzed with a linear least-squares analysis function to provide a consistent method to extract minimum creep rate, onset of tertiary creep and creep strain at the onset of tertiary creep. Data are listed in Table 1 for tests that were conducted in the program. Fig. 2 shows variation in rupture time and minimum creep rate as a function of applied stress for the V–4Cr–4Ti alloy creep tested in vacuum at 650–800 °C.

To determine the extent of O contamination, if any, in the creep specimen, cross sections of the tested specimen were mounted and polished, after which Vickers hardness measurements were made along the thickness direction. In general, hardness values ranged from 145 to 195, and variation was negligible within a given specimen, indicating that the contamination was minimal and was confined to the near-surface region of the 1 mm thick specimen used in the current study. Examination of the fracture surfaces showed a ductile mode of fracture in all tested specimens. The specimens tested at

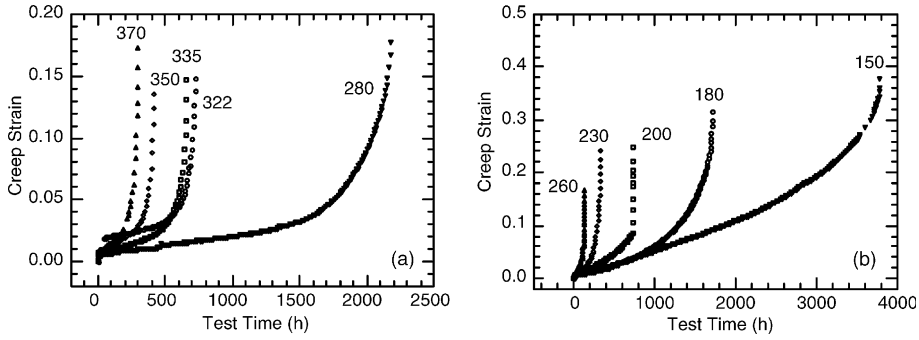

Fig. 1. Creep strain versus time plots for V–4Cr–4Ti alloys tested at (a) 650 and (b) 725 °C in vacuum environment.

Table 1
Creep test data obtained for V–4Cr–4Ti alloy at 650–800 °C

Temperature (°C)	Applied stress (MPa)	Time to rupture (h)	Rupture strain	Minimum creep rate (s^{-1})	Time-to-onset of tertiary (h)	Strain-to-onset of tertiary	Time for 1% strain (h)	Time for 2% strain (h)	Oxygen in 1 mm thick sample (wppm)	Nitrogen in 1 mm thick sample (wppm)
650	370	300	0.18	2.2×10^{-8}	160	0.021	41	156	470	100
	350	415	0.14	2.0×10^{-8}	252	0.023	76	212	430	80
	335	661	0.15	9.3×10^{-9}	325	0.017	118	400	390	100
	322	719	0.16	7.0×10^{-9}	440	0.029	–	–	–	–
	280	2176	0.18	4.2×10^{-9}	1250	0.024	360	1025	430	110
700	250	530	0.27	1.3×10^{-8}	250	0.016	120	282	430	100
	200	1715	0.28	6.1×10^{-9}	325	0.013	230	510	470	100
	180	1959	0.30	5.6×10^{-9}	525	0.014	300	650	–	–
	160[a]	>4000	>0.04	$<4.0 \times 10^{-9}$	–	–	–	–	–	–
725	260	139	0.17	7.2×10^{-8}	75	0.024	20	63	–	–
	230	280	0.25	3.3×10^{-8}	106	0.024	58	130	480	90
	200	737	0.27	2.2×10^{-8}	265	0.023	78	220	–	–
	180	1701	0.32	1.0×10^{-8}	530	0.029	50	315	470	70
	150	3783	0.38	6.6×10^{-9}	270	0.012	211	458	710	110
800	174	112	0.30	1.1×10^{-7}	45	0.023	14	38	–	–
	150	215	0.46	2.3×10^{-8}	85	0.011	77	115	450	100
	130	275	0.61	5.3×10^{-8}	45	0.013	37	59	580	100
	110	559	0.46	2.6×10^{-8}	67	0.012	61	101	1100	110
	90[b]	1147	0.55	1.2×10^{-8}	150	0.075	–	–	800	110
	75	2691	0.45	1.2×10^{-8}	200	0.014	120	270	–	–

[a] Terminated due to suspect O contamination; terminated before failure.
[b] Subjected to loading problem.

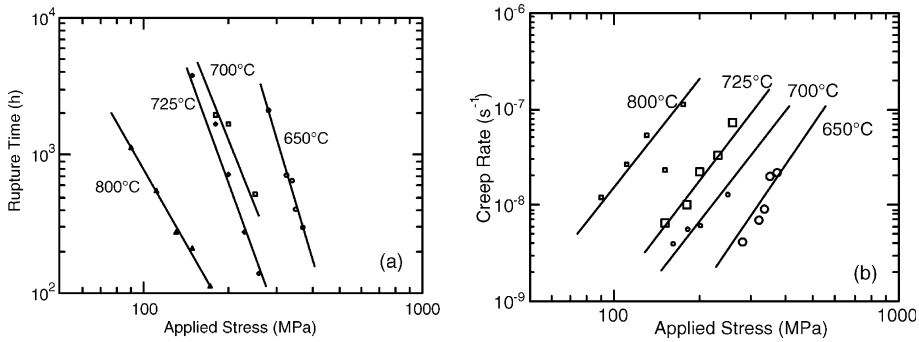

Fig. 2. Variation of (a) rupture time and (b) minimum creep rate as a function of applied stress for V–4Cr–4Ti alloy creep tested at 650–800 °C.

800 °C showed rupture strains of 30–61% and thinning of the cross section in the fracture zone was significant. Several creep-tested specimens were mechanically polished to remove ≈50 μm layers; subsequently, they were analyzed for O and N by the vacuum fusion method. Results of the analysis (see Table 1) indicate negligible O and N enrichment of the specimens.

Because the temperature range used in the present creep tests encompasses the narrow range of 650–800 °C and the stress levels employed at each temperature were such that the rupture time for the specimen will be <4000 h, only limited data are available to evaluate the possible mechanism for creep deformation. Furthermore, the data also indicate (in contrast with the face-centered-cubic materials) that the second stage or steady-state creep range for the V–4Cr–4Ti alloys is confined to creep strains of 2–3% at all the tested temperatures. However, the influence of applied stress and temperature on the creep rate can be determined because the microstructural changes will be minimal over this narrow strain range of the second stage. In as much as creep involves a thermally activated process, the minimum creep rate can be expressed by

$$\dot{\varepsilon} = A\sigma^n \exp(-Q/RT),$$

where σ is applied stress, n is the stress exponent, Q is activation energy for the creep mechanism, R is the universal gas constant and T is absolute temperature. Because the temperature window for the data developed in this program is small and the anticipated microstructural changes in various test specimens will be minimal due to a small second-stage strain range, the apparent activation energy calculated from the data will depict the activation energy for the underlying creep mechanism. On this basis, data were used to calculate the stress dependence of the minimum creep rate and the stress exponent ranged between 3.8 and 4.0. Using the computed stress exponent, we calculated the temperature dependence of the minimum creep rate for the temperature range 650–800 °C. The temperature dependence of the minimum creep rate, calculated at an applied stress level of 200 MPa indicated an activation energy of 262 kJ/mol. This value is in agreement with the activation energy of 270 kJ/mol for self-diffusion in pure V at 700–800 °C [11]. At present, the data are not sufficient to develop the stress dependence of the activation energy.

The Larson–Miller-parameter approach was used to correlate time to rupture, exposure temperature and applied stress. Similar correlations were developed for time to onset of tertiary creep and times for 1% or 2% strain accumulation. The Larson–Miller parameter is given by

$$P = (T \text{ in } ^\circ\text{C} + 273)[20 + \log(t \text{ in h})]0.001,$$

where t is time for rupture or to onset of tertiary creep or time for 1% or 2% strain accumulation.

The best-fit equations that relate the applied stress with Larson–Miller parameters for various creep parameters are

$$\text{Log}\sigma(\text{MPa}) = -0.81177 + 0.41488P(t_r) - 0.01219P(t_r)^2,$$

$$\text{Log}\sigma(\text{MPa}) = 0.79355 + 0.30003P(t_3) - 0.01043P(t_3)^2,$$

$$\text{Log}\sigma(\text{MPa}) = 8.9903 - 0.46601P(t_1\%) + 0.00729P(t_1\%)^2,$$

$$\text{Log}\sigma(\text{MPa}) = 6.4342 - 0.2228P(t_2\%) + 0.00168P(t_2\%)^2,$$

where $P(t_r)$, $P(t_3)$, $P(t_1\%)$ and $P(t_2\%)$ are the Larson–Miller parameters based on time (in h) to rupture and to onset of tertiary creep, for 1% and 2% strain, respectively. Fig. 3(a)–(d) show the best-fit curves for the Larson–Miller correlation, along with the data from individual tests for time to rupture and time to onset of

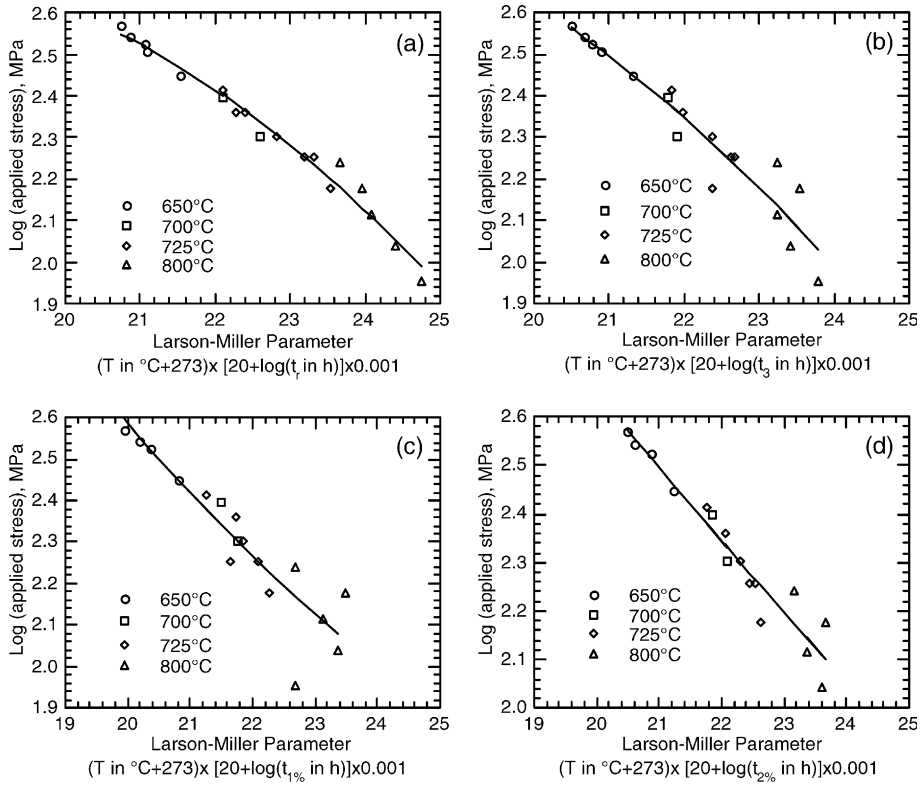

Fig. 3. Larson–Miller plots for (a) time to rupture, (b) time to onset of tertiary creep, (c) time for 1% strain accumulation and (d) time for 2% strain accumulation for V–4Cr–4Ti alloy creep tested in vacuum at 650–800 °C.

tertiary creep, and for 1% and 2% strain accumulation, respectively.

4. Summary

We have conducted a systematic study at Argonne National Laboratory to evaluate the uniaxial creep behavior of V–Cr–Ti alloys as a function of temperature in the range of 650–800 °C and applied stress levels of 75–370 MPa in a vacuum environment. Creep strains were calculated from continuous measurements of displacement in specimens. A linear least-squares analysis function was used to provide a consistent method to extract minimum creep rate, onset of tertiary creep and creep strain at the onset of tertiary creep. In the temperature range of 650–800 °C, the creep deformation followed a power-law creep with a stress exponent of ≈4, and the activation energy calculated from the data was 262 kJ/mol, which is similar to the value for self-diffusion in pure V. Microhardness profiles and postexposure O analysis of tested specimens showed minimal contamination by O during creep testing even over long times of ≈3800 h at 725 °C. The Larson–Miller approach was used to correlate several creep parameters with applied stress and test temperature.

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