UNIAXIAL CREEP BEHAVIOR OF V-4Cr-4Ti Alloy*

K. Natesan, W. K. Soppet, and D. L. Rink (Argonne National Laboratory)

OBJECTIVE

The objectives of the creep test program are to (a) to establish time/temperature relationships for creep properties, such as creep rupture strength, 1% creep in 10,000 hr, onset of third-stage creep, etc., all of which are key parameters in designing structural components for service at elevated temperatures; (b) provide a basis to establish the upper-use temperature associated with creep limits for application of V-base alloys; and (c) evaluate the influence of variations in the concentrations of substitutional and interstitial elements on the creep properties of fusion-reactor-relevant V-base alloys.

SUMMARY

A systematic study is currently being conducted at Argonne National Laboratory (ANL) to evaluate the uniaxial creep behavior of V-Cr-Ti alloys as a function of temperature in the range of 650-800°C and at applied stress levels of 75-380 MPa. At present, the principal effort has focused on the V-4Cr-4Ti alloy of Heat 832665; however, another heat of a similar alloy from General Atomics (GA) will also be used in the study. The Larson-Miller approach is used to correlate several creep parameters such as time to rupture, time-to-onset of tertiary creep, and times for 1 and 2% strain accumulation with applied stress and temperature. Best-fit equations are presented for several creep parameters.

INTRODUCTION

Refractory alloys based on V-Cr-Ti are being considered for use in first-wall structures in advanced blanket concepts that use liquid Li as a coolant and breeding material. Furthermore, advanced concepts that involve He as a coolant also require structural alloys such as V-Cr-Ti, which can withstand thermal loading at high temperature. It is important that for advanced fusion systems, design concepts establish the upper temperature limits for structural components based on various design criteria. At temperatures above 600°C, the time-dependent creep properties of V alloys must be considered when evaluating performance limits.

The long-term creep properties of the V-base alloys will be influenced by the time-dependent nucleation and growth of precipitates that contain nonmetallic elements such as O, N, and C. Several microstructural studies of V-base alloys have identified precipitates such as face-centered-cubic Ti(O, N, C) with variable O, N, and C ratios. To correlate microstructual development with creep properties, It is essential to establish the time-dependent evolution of type, number, and location of precipitates in V-base alloys. Furthermore, development of several of these precipitates can be influenced by the exposure environment during creep testing. Over the long term, creep data are needed for environments with a wide range of chemistry and that encompass high vacuum to low partial pressures of O and H, as well as He of various purities.

---

*This work has been supported by the U.S. Department of Energy, Office of Fusion Energy Research, under Contract W-31-109-Eng-38.

SCOPE OF WORK

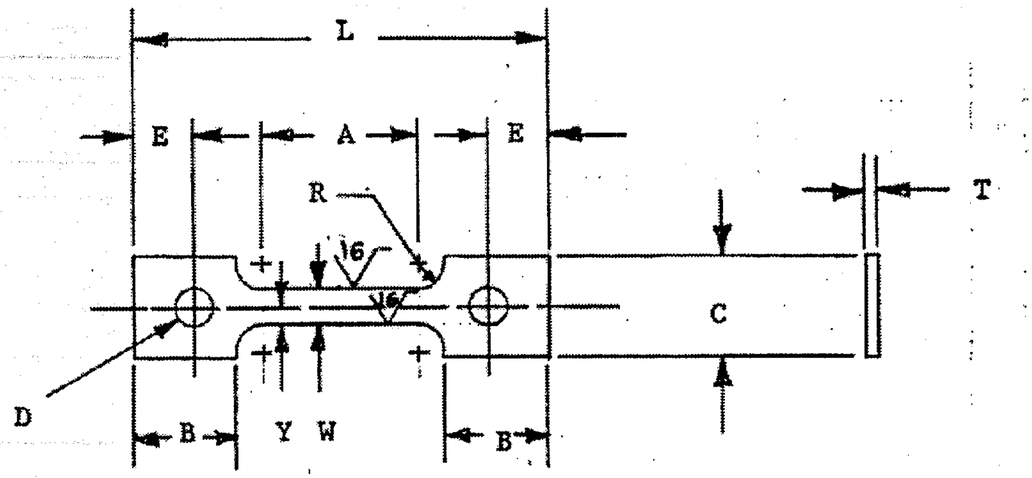
In the near term, the program will experimentally evaluate uniaxial creep properties of V-Cr-Ti materials in high-vacuum environments at temperatures of 650-800°C, with emphasis on baseline creep behavior of the alloys and correlations between microstructures and properties. Furthermore, the test program will examine the effects of specimen geometry and heat-to-heat variation on the creep properties. Another aspect of the program will be creep tests on heats of V-base alloys that represent a range of variations in the concentrations of both substitutional and interstitial elements to provide an understanding of the effects of these variables on creep behavior.

EXPERIMENTAL PROGRAM

The effort is focused on the ANL-procured large heat of nominal composition V-4Cr-4Ti and on the GA heat of a similar composition.[1, 2] Flat creep specimens, 1 mm in thickness (see Fig. 1 for details), were used in the initial phase of the program. A few specimens with cylindrical cross sections (2.5 mm diameter; see Fig. 2 for details) are being tested to validate the effects, if any, of specimen geometry on creep properties. All specimens were fabricated according to ASTM Standard E8-96a with the gauge length oriented parallel to the rolling direction. Initial tests were conducted on specimens annealed at 1000°C for 1 h in vacuum. During this reporting period, several tests were conducted at 650, 700, 725, and 800°C. The specimens were wrapped in Ti foil to minimize contamination of the sample, especially by O.

The creep-test procedure is in accordance with ASTM E139-96. Four ATS model 2140 uniaxial direct constant-load creep-test machines are used for this program. All of the machines are equipped with high-vacuum systems and furnaces capable of 900°C. Creep strain in the specimen is measured by a linear-variable-differential transducer (LVDT) 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 over an operating range of 25 mm. Before each test, the LVDT was calibrated by measuring its voltage output for displacements that were set manually on a standard micrometer. The LVDT is operated over the linear portion of the calibration curve to measure specimen strain during creep testing. Each creep-test system is monitored with a dedicated 66 MHz Intel-486-based PC data acquisition system (DAS) that records LVDT displacement versus time and fracture event. The strain measurements are made at sufficiently frequent intervals during a test to define the creep strain/time curve. The DAS software provides a continuously variable data-sampling rate that adapts for transients and different creep-rate stages.

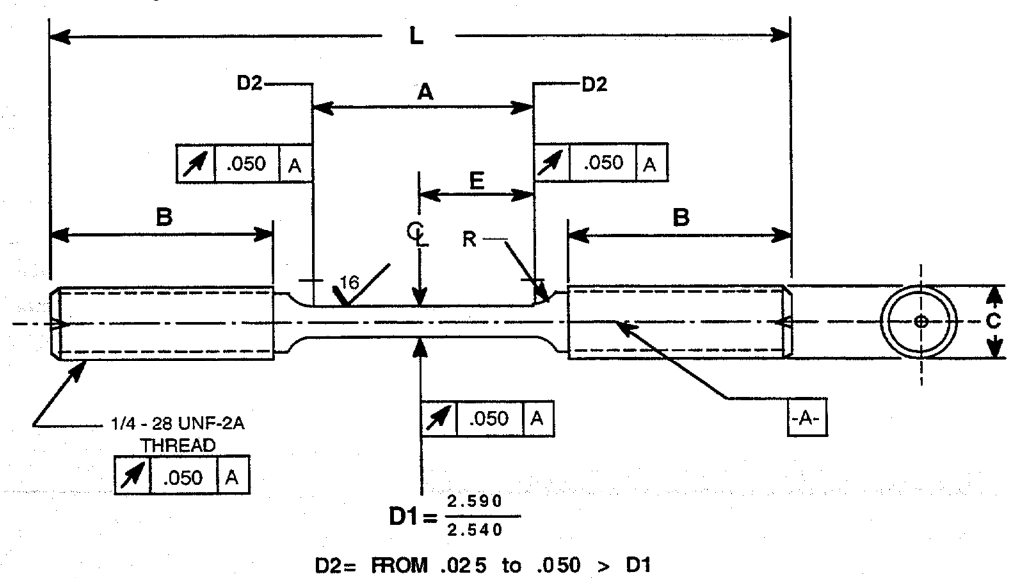
A three-zone resistance-heated furnace with three-mode proportional-integral-derivative temperature controller is used in each testing machine to conduct creep tests at elevated temperatures. Chromel-Alumel thermocouples with small beads are used to measure specimen temperature. Ceramic insulators are used on the thermocouples in the hot zone. In general, three thermocouples are fed through the specimen chamber, one spot-welded onto each end of the specimen grips near the



Dimensions in mm

A = 19 ± 0.50	Y = 2.2
B = 12.7	W = 4.5
C = 12.7	T = 1.0
D = 4.0	R = 3.175
E = 7.6	L = 50.8

Fig. 1. Schematic diagram of flat creep specimen designed according to ASTM Standard E8-96a.



Dimensions in mm

A = 19.05	E = 9.52
B = 19.05	R = 4.75
C = 6.35	L = 63.50
D1 = 2.54 +0.05	

Fig. 2. Schematic diagram of round creep specimen designed according to ASTM Standard E8-96a.

shoulder region and the third held in the vacuum environment adjacent to the gauge section of the specimen. Temperature is maintained within ±2°C of the desired value for each test. The specimens are loaded at a constant rate to full load after test-temperature equilibrium is achieved.

A detailed microstructural evaluation of the tested specimens is planned to characterize the morphologies as a function of exposure temperature and time and to establish the mechanisms of creep deformation and failure. The test program is aimed at obtaining the steady-state creep rate, onset of tertiary creep, rupture strain, and rupture life. At least four stress levels are planned at each temperature to obtain sufficient data to develop Larson-Miller correlations between time, temperature, and applied stress. The information will be used to assess the upper-use temperature for the material, based on appropriate design criteria and as a basis for alloy improvement.

RESULTS

During this period, several creep tests were conducted at 650, 700, 725, and 800°C. Two additional creep tests were completed at 700°C to complement and allow comparison with the data generated in the biaxial creep test program at 700°C conducted at Pacific Northwest National Laboratory.[3] Figure 3 shows creep strain/time plots for V-4Cr-4Ti alloy specimens that we tested in vacuum at 650, 700, 725, and 800°C at ANL. 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 show an accelerating creep behavior over the range of the present tests, especially at 725 and 800°C. Since the last reporting period, the creep strain/time curves have been reanalyzed with KaleidaGraph graphical analysis software (Version 3.5 from Synergy Software). A linear least-squares analysis (LLSA) function is used to provide a consistent method to extract minimum creep rate, onset of tertiary creep, and creep strain at the onset of tertiary creep. Figure 4 shows an example of a typical LLSA applied to a creep strain/time curve. Data are given in Table 1 for completed tests. Figure 5 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 examine 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. Figure 6 shows hardness profiles for several specimens after testing at 725 and 800°C. Hardness values ranged from 145 to 195, with negligible variation within a given specimen, indicating that the contamination is minimal over the range of the current study. Examination of the fracture surfaces of tested specimens showed a ductile mode of fracture in all of the specimens. The specimens tested at 800°C showed rupture strains of 30-61%, and significant thinning of the cross section is seen in the fracture zone. Detailed examination of the tested specimens by scanning and transmission electron microscopy is planned.

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

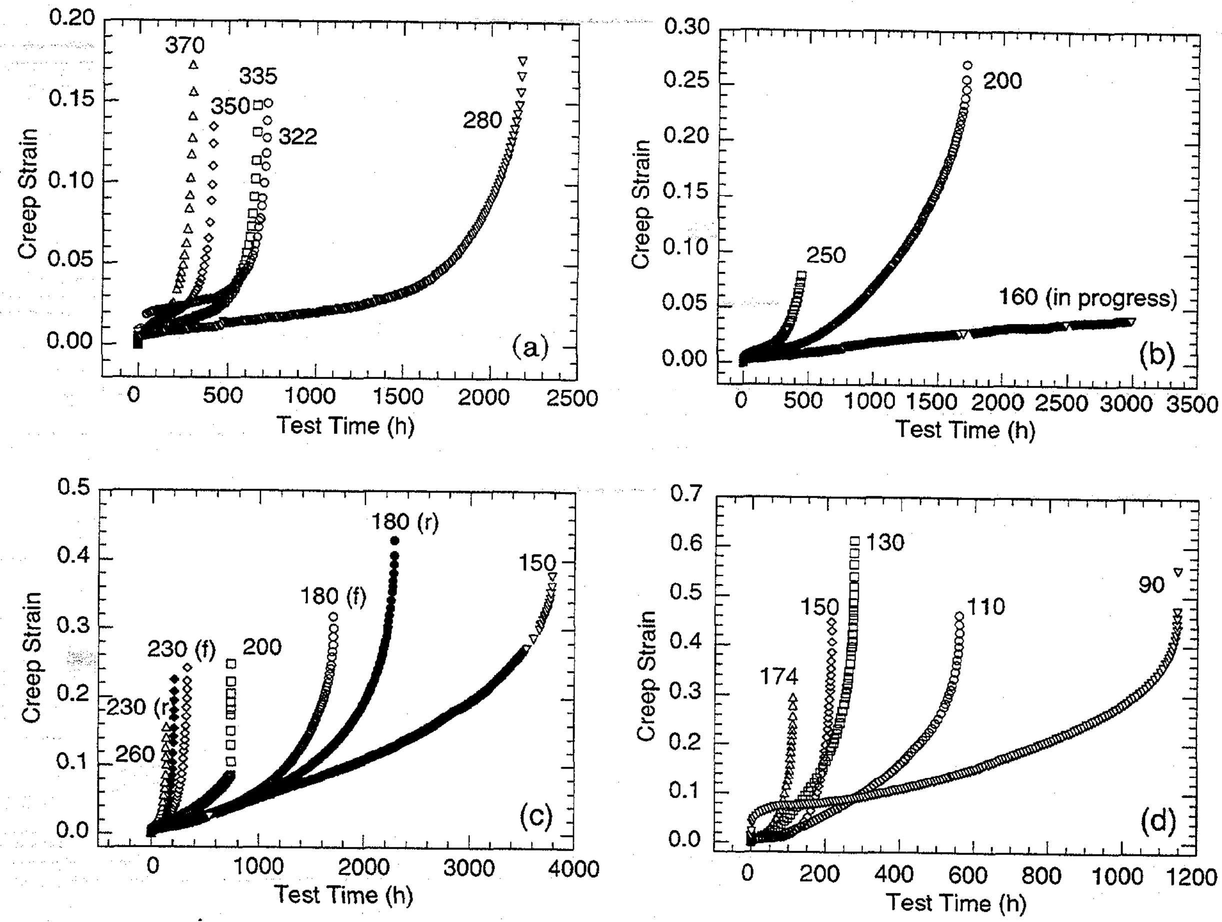


Fig. 3. Creep strain versus time plots for V-4Cr-4Ti alloys tested at (a) 650, (b) 700, (c) 725, and (d) 800°C in vacuum environment.

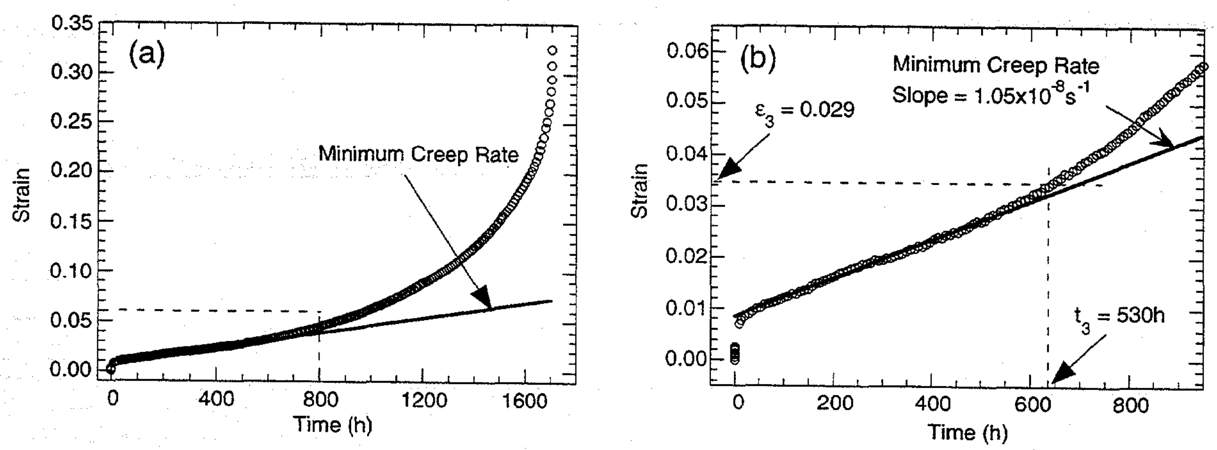


Fig. 4. (a) Typical creep strain versus time curve obtained for V-4Cr-4Ti alloy at 725°C and 180 MPa and (b) magnified plot of portion of curve indicated by rectangular box shown in (a), along with application of linear least-squares analysis to extract minimum creep rate, onset of tertiary creep, and creep strain at onset of tertiary creep.

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

T (°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)
650	370	300	0.18	2.2×10^{-8}	160	0.021	41	156
	350	415	0.14	2.0×10^{-8}	252	0.023	76	212
	335	661	0.15	9.3×10^{-9}	325	0.017	118	400
	322	719	0.16	7.0×10^{-9}	440	0.029	-	-
	280	2176	0.18	4.2×10^{-9}	1250	0.024	360	1025
700	250	530	0.27	1.3×10^{-8}	250	0.016	120	282
	200	1715	0.28	6.1×10^{-9}	325	0.013	230	510
	160 [a]	-	-	-	-	-	-	-
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
	230 [b]	215	0.23	2.4×10^{-8}	105	0.014	61	127
	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
	180 [b]	2281	0.43	1.2×10^{-8}	475	0.024	140	379
	150	3783	0.38	6.6×10^{-9}	270	0.012	211	458
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
	130	275	0.61	5.3×10^{-8}	45	0.013	37	59
	110	559	0.46	2.6×10^{-8}	67	0.012	61	101
	90	1147	0.55	1.2×10^{-8}	150	0.075	c	c

[a]In progress.

[b]Round cross-section specimen.

[c]Times for 1 and 2% strain could not be deduced because of a large scatter in strain/time data in the early part of the test.

$$P = (t \text{ in } ^\circ C + 273)\,[20 + \log (t \text{ in h})] \times 0.001 \qquad (1)$$

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

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

$$\text{Log}\,\sigma\,(\text{MPa}) = 0.81177 + 0.41488 \times P1 - 0.01219 \times P1^2 \qquad (2)$$

$$\text{Log}\,\sigma\,(\text{MPa}) = 0.79354 + 0.30003 \times P2 - 0.01219 \times P2^2 \qquad (3)$$

$$\text{Log}\,\sigma\,(\text{MPa}) = 8.9903 - 0.46601 \times P3 + 0.00729 \times P3^2 \qquad (4)$$

$$\text{Log } \sigma \text{ (MPa)} = 6.4342 - 0.2228 \times P4 - 0.00168 \times P4^2, \quad (5)$$

where P1, P2, P3, and P4 are the Larson-Miller parameters based on time (in hours) to rupture and to-onset of tertiary creep, for 1% strain and 2% strain, respectively. The values for the Larson-Miller parameters calculated from data from various tests are given in Table 2. Figure 7 shows the best-fit curves for Larson-Miller correlations, along with data from individual tests for time to rupture and time-to onset of tertiary creep, for 1 and 2% strain accumulation, respectively.

Table 2. Larson-Miller parameters calculated from creep test data developed in vacuum for V-4Cr-4Ti alloy at 650-800°C in the ANL program

Test temperature (°C)	Applied stress (MPa)	P1 (Time to rupture)	P2 (Time to tertiary)	P3 (Time for 1% strain)	P4 (Time for 2% strain)
650	370	20.746	20.494	19.949	20.484
	350	20.876	20.676	20.196	20.607
	335	21.063	20.778	20.372	20.862
	322	21.097	20.900	-	-
	280	21.541	21.318	20.819	21.239
700	250	22.111	21.793	21.483	21.844
	200	22.607	21.904	21.758	22.094
725	260	22.099	21.831	21.258	21.756
	230	22.402	21.981	21.720	22.070
	230 [b]	22.288	21.977	21.742	22.060
	200	22.822	22.378	21.848	22.298
	180	23.184	22.679	21.656	22.453
	180 [b]	23.311	22.631	22.102	22.533
	150	23.531	22.387	22.280	22.616
800	174	23.659	23.234	22.690	23.155
	150	23.963	23.530	23.484	23.671
	130	24.077	23.234	23.143	23.360
	110	24.408	23.419	23.376	23.611
	90	24.743	23.795	22.690	-

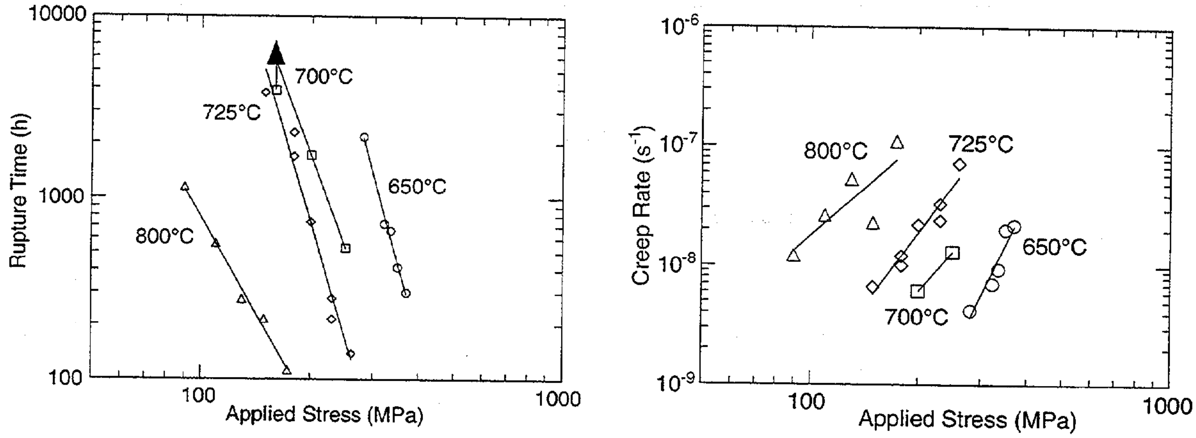


Fig. 5. Variation in (a) rupture life and (b) minimum creep rate as a function of applied stress for V-4Cr-4Ti alloy tested in vacuum at 650-800°C.

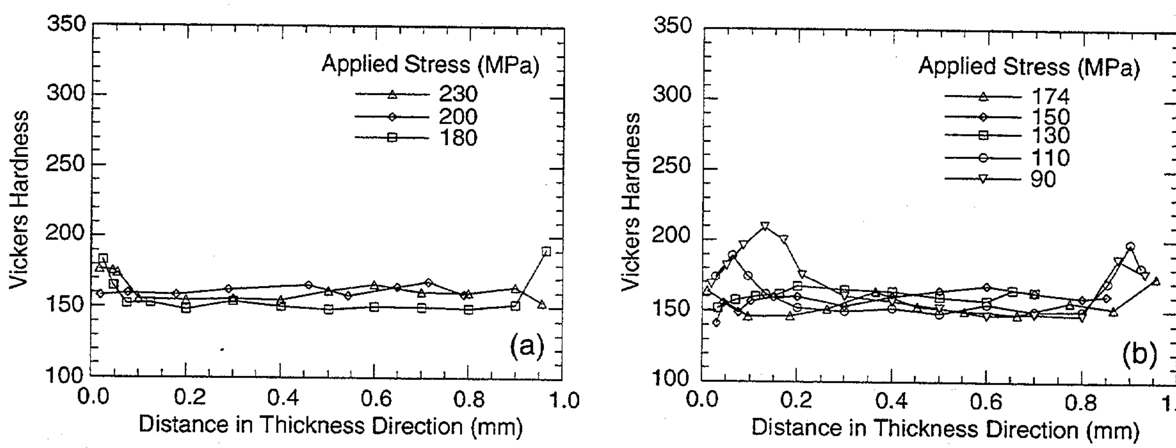


Fig. 6. Vickers hardness profiles across specimen gauge section obtained for several V-4Cr-4Ti specimens after creep testing at (a) 725 and (b) 800°C in vacuum environment.

REFERENCES

1. K. Natesan, W. K. Soppet, and D. L. Rink, Uniaxial Creep Behavior of V-Cr-Ti Alloys, Semiannual Progress Report for Period Ending June 30, 1999, DOE/ER-0313/26, Sept. 1999, p. 20.
2. K. Natesan, W. K. Soppet, and D. L. Rink, Uniaxial Creep Behavior of V-Cr-Ti Alloys, Semiannual Progress Report for Period Ending December 31, 1999, DOE/ER-0313/27, March 2000, p. 17.
3. R. J. Kurtz and M. L. Hamilton, Biaxial Thermal Creep of V-4Cr-4Ti at 700 and 800°C, ibid, p. 3.

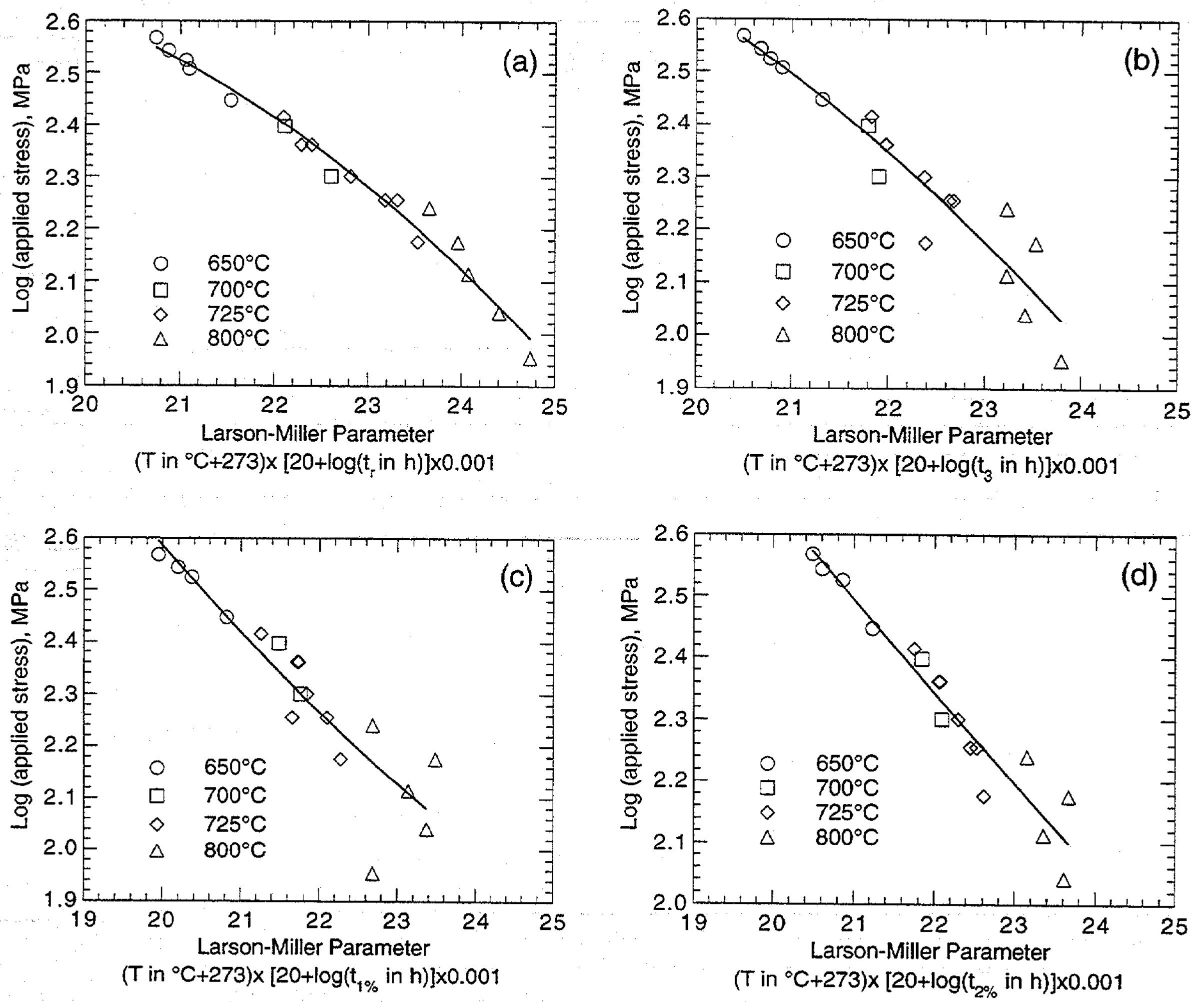


Fig. 7. Larson-Miller plots for (a) time to rupture, (b) time-to-onset of tertiary creep, (c) time for 1% strain accumulation, and (d) time for 2% strain accumulation for V-4Cr-4Ti alloy creep tested in vacuum at 650-800°C.