

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 in the range 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.

INTRODUCTION

Refractory alloys based on V-Cr-Ti are being considered for use in first-wall structures in advanced blanket concepts that involve 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 involve nonmetallic elements such as O, N, and C. Several of the 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 microstructural 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, and He of various purities.

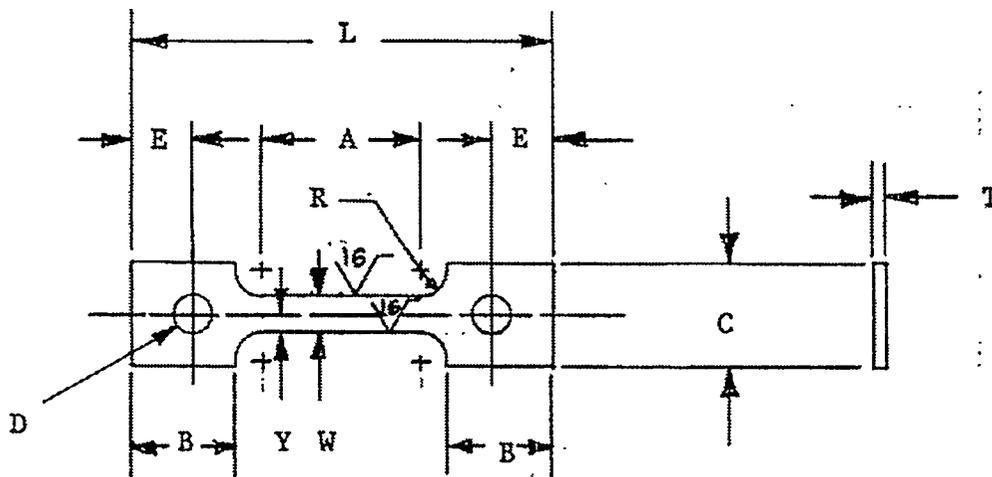
*This work has been supported by the U.S. Department of Energy, Office of Fusion Energy Research, under Contract W-31-109-Eng-38.

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. 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 Four uniaxial creep-test machines have been allocated for this program. All of the machines are equipped with high-vacuum systems and furnaces capable of 900°C. Flat creep specimens, 1 mm in thickness (see Fig. 1 for details), were fabricated according to ASTM Standard E8-69 and used in the initial phase of the program. A few specimens with cylindrical cross sections will be tested to validate the effects, if any, of specimen geometry on creep properties. 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, 725, and 800°C. The specimens were wrapped in Ti foil to minimize contamination of the sample, especially by O.



Dimensions in mm

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

Fig. 1. Schematic diagram of creep specimen designed according to ASTM Standard E8-69.

Creep strain in the specimen is measured by a linear-variable-differential transducer (LVDT), which is 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 is used to measure strain in a specimen during creep testing. The strain measurements are made at sufficiently frequent intervals during a test to define the creep strain/time curve.

A three-zone resistance-heated furnace 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 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 is maintained within 2°C of the desired value for each test. The specimens are loaded at a constant rate to full load at the test temperature.

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 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, 725, and 800°C. Furthermore, a test was initiated at 700°C to complement and compare the data generated in the biaxial creep test program at 700°C conducted at Pacific Northwest National Laboratory (PNNL).² Figure 2 shows the creep strain/time plot for a V-4Cr-4Ti alloy specimens that were tested in vacuum at 650, 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. The creep strain/time curves have been analyzed in detail to extract data on the onset of tertiary creep, creep strain at the onset of tertiary creep, minimum creep rate (linear portion of the curve), and time and strain at rupture. Data are listed in Table 1 for tests that have been completed.

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 3 shows the hardness profile for several specimens after testing at 650, 725, and 800°C. Hardness values ranged from 145 to 195, with negligible variation in hardness 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%, with significant thinning of the cross section in the fracture zone. Detailed examination of the tested specimens by scanning and transmission electron microscopy is planned for the future.

A comparative analysis was attempted in order to correlate the uniaxial creep data developed at ANL and the biaxial creep data developed at PNNL. At the common test temperature of 800°C, ANL tests were conducted at applied stress levels of 174, 150, and 130 MPa. PNNL tests were conducted at effective stress levels of 136.8, 117.7, 92.6, and 70.6 MPa. Figure 4 shows a comparative correlation of the data between time-to-rupture and applied or effective stress developed at 800°C in ANL and PNNL programs. The data developed at 800°C can be correlated as follows:

$$\text{Time to rupture (h)} = 1.5 \times 10^{12} [\text{Applied or effective stress (MPa)}]^{-4.58}$$

$$\text{Applied or effective stress (MPa)} = 565.85 \times [\text{Time to rupture (h)}]^{-0.2548}$$

Additional tests are in progress at 650, 700, 725, and 800°C; results will be reported as they become available. Furthermore, the creep curves will be analyzed in detail to evaluate the time for accumulation of 1 and 2% strain and to develop correlations that relate stress with 1 and 2% strain in 10,000 h.

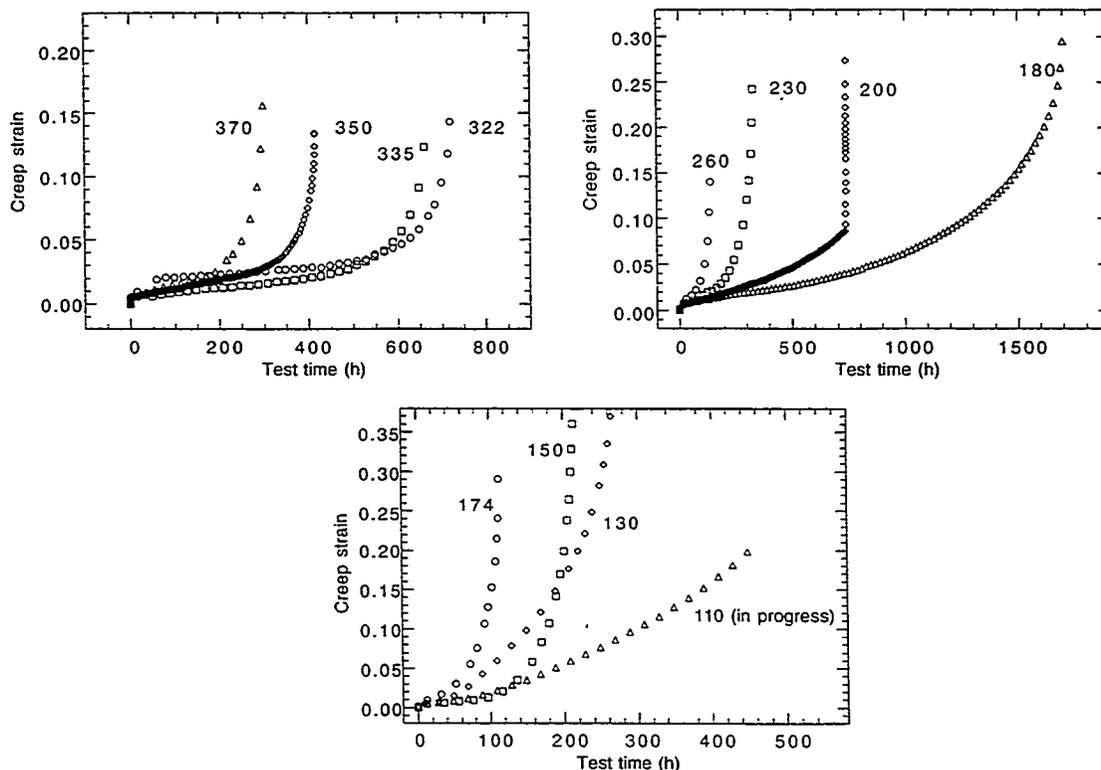


Fig. 2. Uniaxial creep strain-vs.-time plot for V-4Cr-4Ti alloy tested at 650 (top left), 725 (top right), and 800°C (bottom) in vacuum environment. Numbers next to the curves indicate applied stress in MPa.

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
650	370	300	0.18	2.2×10^{-8}	210	0.029
	350	414	0.14	2.0×10^{-8}	280	0.029
	335	661	0.15	9.3×10^{-9}	450	0.028
	322	718.5	0.16	7.0×10^{-9}	515	0.029
	280 ^a	-	-	-	-	-
700	250 ^a	-	-	-	-	-
725	260	139.2	0.17	7.2×10^{-8}	80	0.024
	230	329	0.25	3.3×10^{-8}	160	0.024
	200	737.4	0.27	2.2×10^{-8}	380	0.033
	180	1701.4	0.32	1.0×10^{-8}	530	0.029
800	174	112.2	0.30	1.1×10^{-7}	45	0.036
	150	214.9	0.46	2.3×10^{-8}	85	0.015
	130	275	0.61	$\approx 10^{-8}$	40	0.012
	110 ^a	-	-	-	-	-
	90 ^a	-	-	-	-	-

^aIn progress.

REFERENCES

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

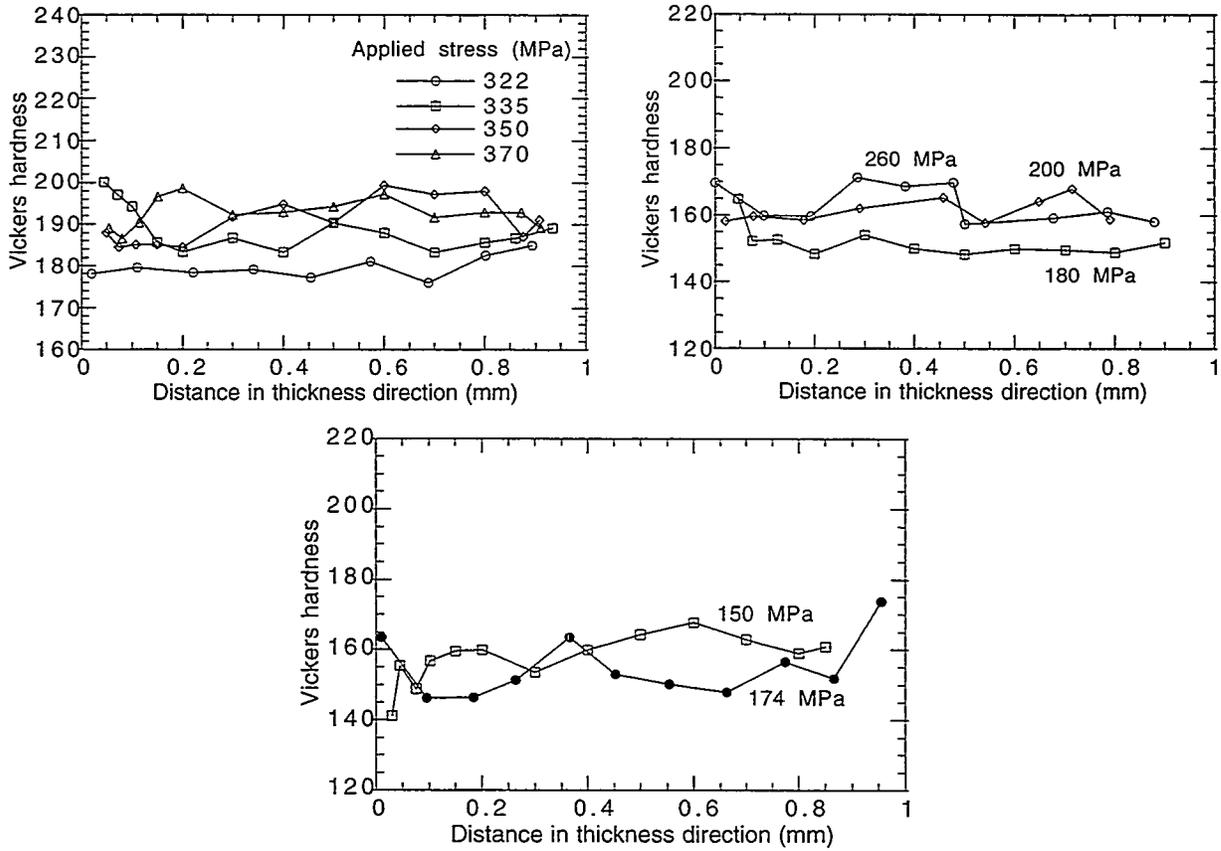


Fig. 3. Vickers hardness profiles in thickness direction for V-4Cr-4Ti specimens, after creep testing at various stress levels at 650 (top left), 725 (top right), and 800°C (bottom) in vacuum environment. Numbers next to curves indicate applied stress in MPa.

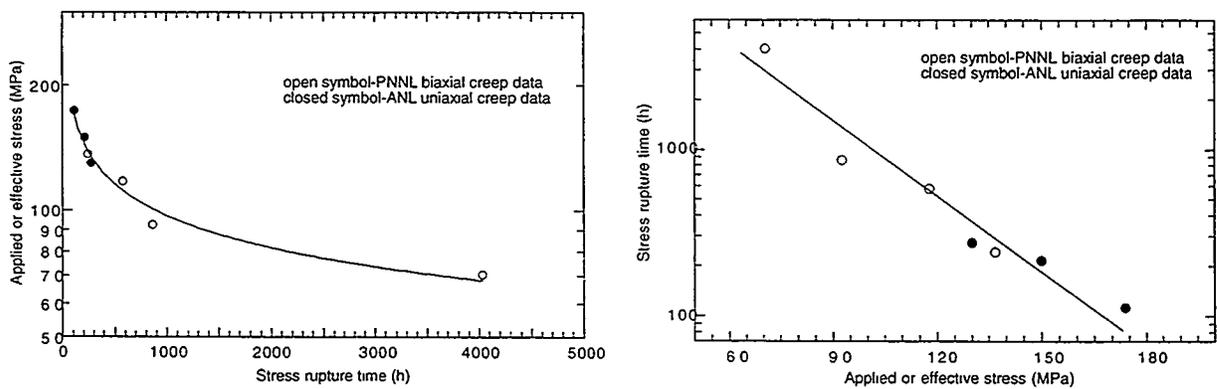


Fig. 4. Two correlations between time-to-rupture and applied stress, where independent variable is time-to-rupture (left) and applied stress (right). Curves represent best fit of data at 800°C obtained in both ANL and PNNL programs.