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TENSILE AND LOW-CYCLE FATIGUE MEASUREMENTS ON CROSS-ROLLED TUNGSTEN[a]

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Low-cycle fatigue and tensile tests were performed on specimens fabricated from 14-mm (0.55-in.) cross-rolled tungsten plate which was prepared by a powder metallurgy process. Tests included measurements on both as-received and recrystallized specimens. Data have been obtained at 1088 K (1500°F) in vacuum, and at room temperature. Low-cycle fatigue data at both 1088 K and room temperature are in fair agreement with predictions based on the "universal slopes" equation for the as-received material condition. In contrast, fatigue data for recrystallized specimens at 1088 K fall considerably below prediction, except in the high cycles-to-fail (10^5 cycles) regime. Details of the test procedure as well as modification of the specimen configuration which was required for room temperature testing are reported.

1. INTRODUCTION

Tungsten has been utilized in a number of fusion experiments to date, as a plasma limiter and as a protector plate to shield sensitive components from the plasma. Its merits include high mechanical strength, high melting point, and good thermal shock properties. In spite of the fact that recent successes with the Princeton Large Torus were attributed partly to the removal of the tungsten limiter from that machine [1], areas still exist in which tungsten can contribute to the success of fusion.

In order to make the best use of tungsten or any other material in the difficult fusion environment, experimental data are needed to apply in component design. A review of the literature [2] indicates that the properties of tungsten are quite sensitive to the process history of the material. Often, the material history is poorly documented or not documented at all. Many items are made from powder metallurgy base material due to the high melting point (3683 K) of the element. Matters are further complicated by the occurrence of a ductile-brittle transition above room temperature, tungsten being brittle at room temperature but becoming ductile above the transition temperature of ~573 K [3].

In this effort low-cycle fatigue and tensile measurements were performed on powder metallurgy tungsten specimens which were provided by the Princeton Plasma Physics Laboratory. Measurements were obtained for two material conditions, as-received stress-relieved (wrought) and recrystallized, and two temperatures, 296 K (75°F) and 1088 K (1500°F). Data for the recrystallized form are considered important because accident scenarios suggest that thermal shock conditions could be realized in which some transformation to the recrystallized form would take place. Data for both material conditions and the two temperatures are reported here. All tests at elevated temperature were made with the specimen contained in an evacuated chamber.

2. EXPERIMENTAL PROCEDURES

Specimens tested in this work were prepared from powder metallurgy tungsten plate, 14 mm thick, which was obtained from GTE Sylvania. The plate material was prepared from 99.9% purity starting material and was cross-rolled in the reduction process to provide maximum uniformity of properties in the plane of the plate. The microstructure of the material was examined and found to show no preferential grain orientation in the plane of the plate. Before an order was placed for the specimens, a single hourglass specimen was obtained and used for strain measurements to determine if any anisotropy was observable. These strain measurements were made using a diametral extensometer. The specimen was rotated 30° between measurements; a constant stress level of 135 MPa (19.6 ksi) was reapplied for each measurement. A second set of strain measurements was made with the stress level increased to 270 MPa (39.2 ksi). No anisotropy was observed in these measurements of the as-received material, and it was concluded that the hourglass configuration could be used for low-cycle fatigue tests.

Specimens were prepared from plate material by Thermo Electron Corporation using grinding techniques followed by a final electropolish to remove surface scratches. Due to the previously observed lack of directionality in the grain structure, specimen blanks were cut from the cross-rolled plate without regard to a

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longitudinal or transverse direction. For the high temperature tests, hourglass and uniform gage specimens were prepared for the low-cycle fatigue and tensile tests, respectively. Both specimen configurations incorporated button-head ends for gripping the specimens during the tests. The gage section of the hourglass specimen had an hourglass radius of 50.8 mm with a minimum diameter of 5.08 mm for the elevated temperature tests. The uniform gage specimens were 6.35 mm in diameter by 19.05 mm long in the test section. Due to the notch sensitivity of tungsten at room temperature, a modification of the end configuration was required in order to run tests at that temperature. Figure 1 shows the specimen configuration that was used for the room temperature low cycle fatigue tests. A corresponding change was made in the collett sets that were used to hold the specimen.

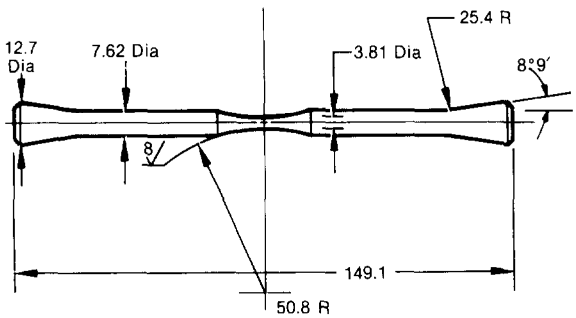

Figure 1 : Hourglass fatigue specimen showing the modified end configuration used in testing at room temperature.

The preparation of specimens for testing in the recrystallized condition was accomplished in a vacuum furnace. Based on metallographic examination and microhardness measurements, it was found that a temperature of 1755 K (2700°F) was needed to recrystallize the powder metallurgy material. Although changes in microstructure were observed at lower temperatures, the microhardness change which accompanies the transition was not observed until the temperature was raised to 1755 K. With recrystallization the hardness was observed to decrease from 453 to 382 VHN.

A vacuum chamber with an approximately four liter volume was installed inside the corner posts of the die set of the fatigue frame for the tests at 1088 K. Roughing and diffusion pumps were used to evacuate the chamber, and a liquid nitrogen cold trap was included in the system. A pressure level of 1.33×10^{-4} Pa (1×10^{-6} torr) was achieved with the gauge section of the specimen at temperature before starting each test. Induction heating was used to heat the specimens, and the specimen temperature was monitored and controlled by chromel-alumel thermocouples. Before fatigue and tensile testing, the axial temperature profile was measured along the gage section of a specimen with a number of thermocouples. Then, during testing, three thermocouples were attached to each specimen, all of them displaced from the center of the gauge section by $\pm$ 7.6 mm, two below and one above the center. One thermocouple each side of the center was used to check the positioning of the induction coil by the symmetry of the heating, and the third thermocouple was used to control the induction heater.

A diametral extensometer, positioned at the minimum diameter of the gage section of the specimen, was used on all tests at 296 K and 1088 K. Strain-controlled, fully-reversed uniaxial fatigue tests were conducted using a closed-loop servohydraulic fatigue frame. Fatigue tests were run in strain control, except for the room temperature tests on recrystallized specimens which were tested in load control because the loading was fully elastic and some anisotropy was noted at these very low strain ranges. A triangular wave form was utilized, and the frequency was adjusted to maintain a constant strain rate of 4×10^{-3} s^{-1}. A constant frequency of one hertz was used for all load-control fatigue tests. Tensile data were obtained on the same system by adjusting the crosshead velocity to obtain a constant strain rate of 8.3×10^{-5} s^{-1}.

3. RESULTS AND DISCUSSION

The low-cycle fatigue data for 1088 K (1500°F) are listed in Table 1 and are displayed in Figure 2. Also displayed in Figure 2 are the fatigue curves predicted by the universal slopes equation [4] for both as-received (solid curve) and recrystallized (dashed curve) material conditions. These curves were used as a guide in running the initial fatigue tests. Clearly, the fatigue data for the as-received condition agree with the equation better than the recrystallized data, particularly at low values of N_f. It is not clear whether the high strain ranges encountered here would ever occur in a reactor situation or not.

One anomaly was observed in the 1088 K tests that should be reported. For total strain ranges greater than approximately 0.75%, a slight bulging occurred at the center of the gage section during the tests. However, the bulging did not appear to interfere with the operation of the diametral extensometer. Attempts were made to observe the bulge formation during a test but without success. It is thought that the bulge formation relates to a reduced effective cross section in the gage area once a crack has initiated. Table 2 lists the low-cycle fatigue data for 296 K

TABLE 1. TUNGSTEN LOW-CYCLE FATIGUE DATA, 1088 K

Specimen Number	Mat'l Condition[a]	$\Delta\varepsilon_t$ (%)	$\Delta\varepsilon_p$[b] (%)	$\Delta\sigma$[b] (MPa)	$\Delta\sigma_{Ten}$[b] (MPa)	N_o[c]	N_f	t (h)
5-28	A-R	8.02	3.92	800	401	103	114	1.20
5-26	A-R	5.00	4.73	773	384	23	26	0.18
5-1	A-R	3.03	2.88	673	--	530	713	2.97
5-29	A-R	2.96	2.71	711	363	384	440	1.84
5-25	A-R	0.66	0.53	655	335	3,643	4,692	4.17
5-27	A-R	0.65	0.43	596	310	--	4,609	3.84
5-15	R	2.01	0.95	501	251	127	154	0.40
5-13	R	1.02	0.89	438	223	490	502	0.69
5-30	R	0.70	0.64	325	169	1,021	1,021	0.97
5-22	R	0.50	0.37	332	167	8,713	10,537	7.15
5-23	R	0.31	0.26	302	151	54,744	58,094	24.25
5-11	R	0.25	0.18	284	146	32,671	33,139[d]	11.40

a. A-R = As-received, stress relieved; R = Recrystallized.
b. Properties at $N_f/2$, $\Delta\varepsilon_p$ = plastic strain, $\Delta\sigma$ = stress range, $\Delta\sigma_{Ten}$ = tensile stress.
c. N_o = Number of cycles where load starts to drop due to crack initiation.
d. Failed at thermocouple weld connection

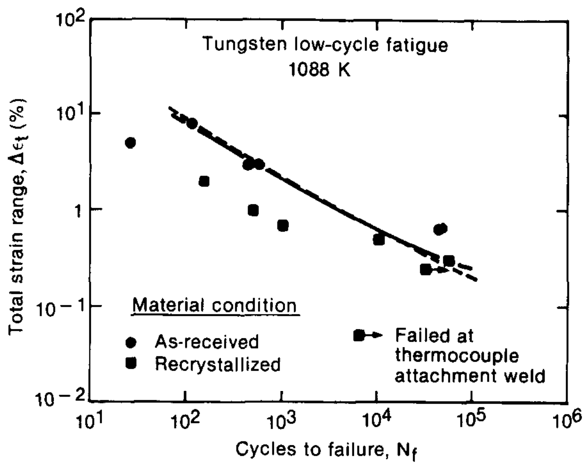

Figure 2 : Tungsten low-cycle fatigue data at 1088 K. The solid (dashed) curves give the predictions of the universal slopes equation for the as-received (recrystallized) material condition which was used as a guide in starting the tests.

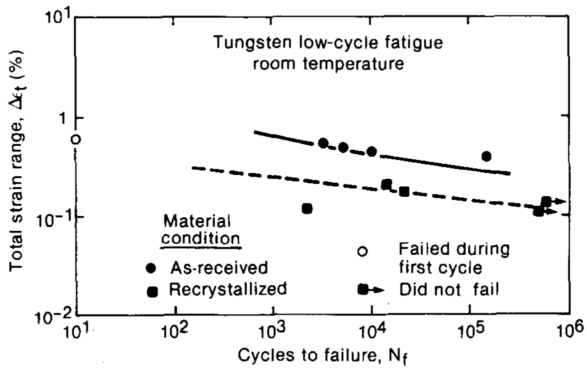

Figure 3 : Tungsten low-cycle fatigue data at room temperature. The solid (dashed) curves give the predictions of the universal slopes equation for the as-received (recrystallized) material condition which was used as a guide in starting the tests.

and these data are plotted in Figure 3. Since the room temperature tests for the recrystallized condition were run in load control, the strain range values were calculated from the stress range values and Young's modulus [5]. The data for both material conditions agree reasonably well with predictions from the universal slopes equation. At higher cycles-to-fail the data appear to be near the endurance limit. For the recrystallized condition, both tests that ran beyond 500,000 cycles were terminated without failure. Except for the greater degree of caution that was needed in loading the specimens for the room temperature tests, the modified specimen configuration presented no problems and functioned well.

Oxidation of tungsten is a potential problem in the current experiment which is addressed in the following paragraphs. In the temperature range 773-1173 K, initial oxidation results in the formation of a compact protective scale which follows a parabolic rate [6]. Subsequently the rate becomes linear corresponding to the formation of a more porous layer of tungsten trioxide. The composition

TABLE 2. TUNGSTEN LOW-CYCLE FATIGUE DATA, 296 K

Specimen Number	Mat'l[a] Condition	$\Delta\varepsilon_t$ (%)	$\Delta\varepsilon_p$[b] (%)	$\Delta\sigma$[b] (MPa)	$\Delta\sigma_{Ten}$[b] (MPa)	N_f	t (h)
	A-R	0.50	.021	1774	889	5,220	3.57
3	A-R	0.40	.004	1362	624	150,502	82.74
5	A-R	0.60	NA	NA	NA	1	--
7	A-R	0.45	.008	1649	716	10,337	6.37
8	A-R	0.55	.066	1870	976	3,343	2.50
4	R	0.118[c]	--	$\pm$236.4		2,196	0.62
2	R	0.108[c]	--	$\pm$206.8		512,727[d]	142.4
6	R	0.137[c]	--	$\pm$275.7		611,572[d]	169.9
10	R	0.172[c]	--	$\pm$344.7		21,535	5.99
9	R	0.206[c]	--	$\pm$413.6		14,317	4.0

a. Material Condition: A-R = As-received, stress relieved; R = Recrystallized
b. Properties at $N_f/2$, $\Delta\varepsilon_p$ = Plastic strain, $\Delta\sigma$ = Stress range, $\Delta\sigma_{Ten}$ = Tensile stress
c. Tests on recrystallized material run in load control, $\Delta\varepsilon_t$ calculated from $\Delta\sigma$ and Young's Modulus.
d. No failure, test terminated.

TABLE 3. TUNGSTEN TENSILE DATA, 1088 K[a]

Specimen Number	Material[b] Condition	UTS (MPa)	Y.S. (MPa)	Total Elongation (%)	Reduction in Area (%)
1	A-R	428	411	22	62
4	A-R	425	419	27	80
X	R	248	96	51	68
Y	R	232	134	51	84

a. $\dot{\varepsilon} = 5 \times 10^{-3}$ in./in./min

b. A-R = As-received, stress relieved; R = Recrystallized

and structure of the initial film has been the subject of some debate. Between 1173 and 1523 K the formation of oxide scale continues with the evaporation of some trioxide. Above 1523 K oxidation occurs without the formation of an appreciable oxide film or scale. Evaporation of tungsten trioxide occurs as rapidly as it is formed.

In the present experiment, some oxidation was possible in tests at 1088 K. However, the use of the liquid nitrogen cold trap was intended to minimize the presence of oxygen in the system as much as possible. Post-test examination of specimen surfaces showed that they remained bright and shining with no evidence of scale formation. Recrystallization of specimens before testing was accomplished in a separate vacuum furnace at 1755 K and a vacuum of 1×10^{-6} torr. Hence, any oxide that formed during recrystallization should have evolved from the specimens. Differences in fatigue test results for as-received and recrystallized material do not appear to be attributable to oxidation due to the fact that the differences observed were at low values of N_f (short test duration) for which oxidation should have the least effect.

Fracture surfaces of failed fatigue specimens were examined by scanning electron microscopy for both material conditions and both temperatures. Specimens selected for examination were taken from the extremes of the total strain range. At 296 K, for both as-received and recrystallized material, all specimens examined exhibited intergranular fracture. At 1088 K the as-received material showed transgranular failure, whereas the recrystallized material showed transgranular failure for low total strain range (high N_f) and intergranular failure for high total strain range (low

N_f). Hence, the deviation from the universal slopes equation for the recrystallized material at 1088 K appears to be related to a change in the mode of failure as a function of total strain range or number of cycles-to-fail.

Tensile data were obtained only at 1088 K. Attempts to obtain data at room temperature were abandoned due to repeated failures outside the gage section. The data for 1088 K are listed in Table 3. They fall within the range of data given in a compilation some years ago [2]. Yield strength values were obtained from the first quarter cycle of each low cycle fatigue test at 1088 K and are listed separately in Table 4. These data are in fair agreement with yield strength values from the tensile tests. However, it should be noted that the data in Table 4 were obtained at a strain rate that was a factor of forty-eight times that for the data in Table 3. These data are considered of value because of the higher strain rates which may be encountered in fusion reactors.

4. CONCLUSIONS

- The tensile data and yield strength data extracted from the low-cycle fatigue data at 1088 K are in agreement with published data.
- The universal slopes equation gives good agreement with the low-cycle fatigue data for the as-received condition.
- The rather sluggish transformation to the recrystallized form which was observed here may not apply in a reactor system due to the presence of stress fields which were not present during the recrystallization process of this experiment.

TABLE 4. YIELD STRENGTH DATA FROM LOW-CYCLE FATIGUE TEST, 1088 K[a]

Specimen Number	Material[b] Condition	Yield Strength (MPa)
5-28	A-R	437
5-26	A-R	445
5-1	A-R	425
5-29	A-R	447
5-25	A-R	452
5-27	A-R	403
	Average =	435
5-13	R	123
5-15	R	89.6
5-11	R	87.6
5-22	R	100
5-30	R	88.2
	Average =	115

a. $\dot{\varepsilon} = 4 \times 10^{-3}$ in./in./s for fatigue tests.

b. A-R = As-received, stress relieved
R = Recrystallized

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