

The Stress-Dependence of High Temperature Creep of Tungsten and a Tungsten-2 Wt Pct ThO₂ Alloy

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Constant-load creep tests were conducted with pure tungsten and a W-2 wt pct ThO₂ alloy at temperatures between 1600° and 2200°C and at strain rates of about 1×10^{-8} to 4×10^{-5} sec⁻¹. The results were evaluated by the empirical correlations of Robinson and Sherby and also Mukherjee *et al.* which describe the stress dependence of the creep of metals and alloys. The agreement of the present experimental data with these correlations was found to be poor. However, when the following empirical relationship was used:

$$\dot{\epsilon}_c = A'(\sigma_c/\sigma_f)^n$$

the present creep data for tungsten and the tungsten alloy at various temperatures were much better correlated. Here, $\dot{\epsilon}_c$ is the experimental creep rate, σ_c is the applied stress for creep, σ_f is the flow stress of the material at the same temperature in a constant strain rate tensile test, and A' is function of temperature, structure, and strain rate.

THE stress dependence of creep at high temperatures is often expressed by the following equation:

$$\dot{\epsilon} = B(T, S)\sigma_c^n \quad [1]$$

where $\dot{\epsilon}_c$ is the steady state creep rate and σ_c is the applied creep stress. The term $B(T, S)$ is a function of structure (S) and temperature (T). Eq. [1] is obeyed by a number of metals when tested at constant temperature and at strain rates of about 10^{-7} to 10^{-4} sec⁻¹. In these cases, the exponent n has usually been found to vary from 3 to 7.^{1,2} In order to correlate creep data obtained at different temperatures, the term $B(T, S)$ must be explicitly defined. Robinson and Sherby³ and also Mukherjee, Bird, and Dorn⁴ have proposed explicit relationships which compensate for the effect of temperature and stress on the steady-state creep rate of materials at high temperatures.

In the present investigation, creep data on pure powder metallurgy (PM) tungsten and a W-2 wt pct ThO₂ alloy are reported. The creep results were found to be in poor agreement with the proposed correlations.^{3,4} A new relationship based on a power law of creep yielded a significantly improved fit for the data.

EXPERIMENTAL PROCEDURE AND RESULTS

Pure tungsten and a W-2 wt pct ThO₂ alloy with a finely dispersed second phase were produced by a powder metallurgy technique. The mean ThO₂ particle size in the alloy was about 750 Å with an interparticle spacing of about 0.3 μm. The alloy was tested in both the recrystallized (annealed 6 hr at 2850°C), and the recovered condition (annealed ½ hr at 2400°C), whereas the pure tungsten was tested only in the recrystallized condition (annealed ½ hr at 2400°C). The grains in the alloy were highly elongated parallel to the rod axis, with a width of about 20 μm in the recovered condition and about 130 μm in the recrystallized condition. The recovered alloy also had a stable substructure with

subgrains of ~2 μm diam. The grains of recrystallized pure tungsten were equiaxed and their mean size was about 63 μm. Details about the production and microstructures of these materials have been previously reported.^{6,7}

Constant-load creep tests were made on buttonhead type specimens (0.36 cm diam by 2.5 cm gage length) in an Instron Testing Machine equipped with a radiant vacuum furnace (~ 1×10^{-5} torr at 2400°C). The test loads were suspended outside the furnace through a bellows-type vacuum seal. The extension of the specimen during creep was measured by a dial gage with a 1 μm scale. The dial gage was actuated by a lever arm connected to the pull rod. The movement of the gage pointer was recorded with a cinematic camera operated in a time-lapse mode for slow creep rates.

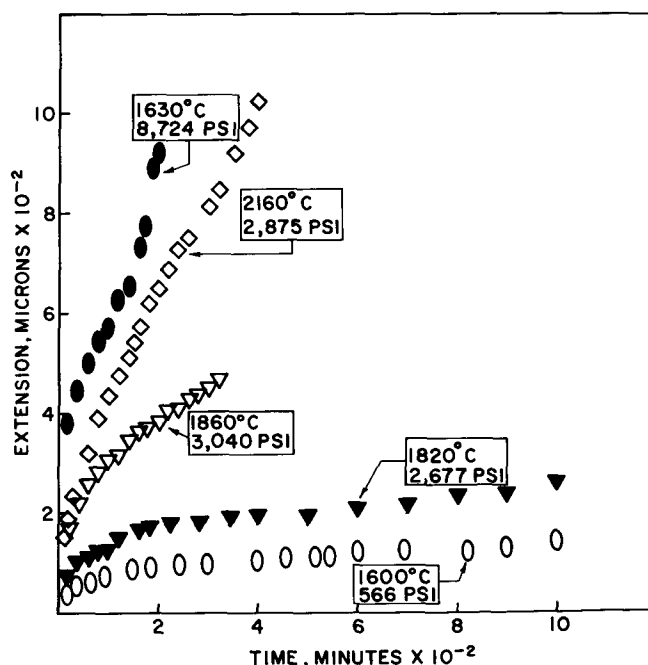


Fig. 1—Typical creep curves for recrystallized pure tungsten (open symbols) and W-2 wt pct ThO₂ (closed symbols) under various test conditions.

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Table I. Summary of Creep Data for Pure Tungsten and W-2 Wt Pct ThO₂

Test Temp, °C	Applied Stress, σ_c	Flow* Stress, σ_f	σ_c/σ_f	Steady-State Creep Rate, sec ⁻¹	Tot. Creep Strain, Pct
Pure Tungsten, Recrystallized ($\frac{1}{2}$ hr, 2400°C)					
1600	566	5,500	0.103	9.97×10^{-9}	0.22
1580	3,100	5,650	0.549	1.87×10^{-7}	0.10
1590	4,661	5,600	0.832	1.16×10^{-6}	1.58
1595	7,704	5,550	1.390	1.38×10^{-5}	2.28
1850	1,651	4,100	0.403	7.93×10^{-8}	0.34
1860	3,040	4,070	0.747	5.60×10^{-7}	0.88
1870	5,924	4,000	1.480	3.84×10^{-5}	4.33
2200	1,103	2,150	0.513	2.35×10^{-7}	1.34
2160	2,875	2,400	1.200	1.29×10^{-6}	2.70
2230	3,050	2,000	1.530	1.65×10^{-5}	2.06
W-2 Wt Pct ThO ₂ , Recrystallized (6 hr, 2850°C)					
1610	3,113	10,700	0.291	8.39×10^{-8}	0.46
1630	5,394	10,600	0.504	2.08×10^{-7}	0.32
1630	8,724	10,600	0.823	1.40×10^{-6}	0.92
1870	909	8,400	0.108	1.09×10^{-8}	0.24
1820	2,677	8,800	0.304	5.81×10^{-8}	0.35
1845	6,161	8,500	0.723	5.33×10^{-7}	0.25
1860	8,744	8,400	1.040	4.82×10^{-7}	0.88
2220	3,301	5,100	0.647	2.88×10^{-7}	1.42
2190	5,344	5,400	0.990	1.08×10^{-6}	0.42
W-2 Wt Pct ThO ₂ , Recovered ($\frac{1}{2}$ hr, 2400°C)					
2200	10,000	16,500	0.610	7.7×10^{-6}	
2200	13,000	16,500	0.790	4.4×10^{-5}	
2200	14,000	16,500	0.850	9.3×10^{-5}	

*Flow Stress \equiv Proportional Limit.

Table II. Summary of Tensile Data

Test Temp, °C	Stress, σ_c	$\dot{\epsilon}_1$, sec ⁻¹	Flow Stress, σ_f ($\dot{\epsilon} = 8.3 \times 10^{-4}$ sec ⁻¹)	σ_c/σ_f
Pure Tungsten, Recrystallized ($\frac{1}{2}$ hr, 2400°C)				
1650	13,500	3.33×10^{-2}	5,250	2.6
1850	9,600	3.33×10^{-2}	4,100	2.3
2250	6,300	3.33×10^{-2}	1,900	3.3
W-2 Wt Pct ThO ₂ , Recrystallized (6 hr, 2850°C)				
1650	14,030	3.33×10^{-3}	10,400	1.35
1875	12,460	3.33×10^{-2}	8,250	1.52
W-2 Wt Pct ThO ₂ Recovered ($\frac{1}{2}$ hr, 2400°C)				
2200	26,600	3.33×10^{-2}	16,500	1.6

Typical creep curves are depicted in Fig. 1, and the results of all creep tests are recorded in Table I. The change in cross-section during creep was negligible; and, therefore, the tests were essentially performed at constant stress. Tensile data from previous investigations,^{6,7} which will be used later in this paper, are recorded in Table II. The flow stress values, σ_f , reported in Tables I and II were the proportional limits in constant strain rate tensile tests at a strain rate of 8.33×10^{-4} sec⁻¹. The values of the steady-state creep rates, and also the flow stress data reported in Tables I and II, were determined from linear regression analyses.

DISCUSSION OF RESULTS

1) Evaluation by Established Models

The creep data in Table I were plotted according to the expression proposed by Robinson and Sherby—Eq.

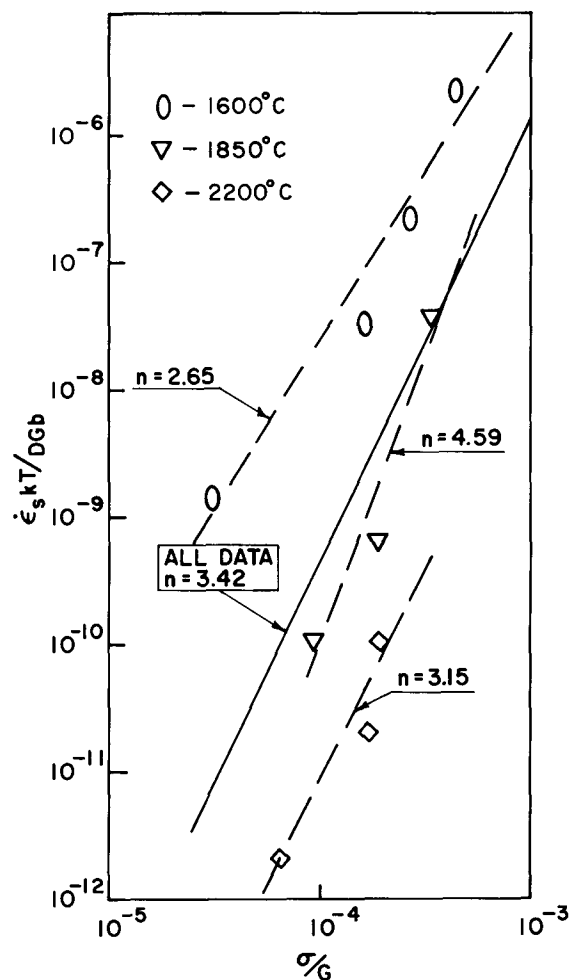


Fig. 2—Stress dependence of the creep rate of pure tungsten plotted according to Ref. 4.

[8b], Ref. 3—and also according to the expression proposed by Mukherjee *et al.*—Eq. [8], Ref. 4. Values of the elastic moduli, E , used in the expression by Robinson and Sherby were taken from the work of Armstrong and Brown⁸ after extrapolation of the linear portion of their data to higher temperatures. A value of $42.8 \times \exp(-153,000/RT)$ was employed in the expression by Mukherjee *et al.* for the self-diffusion coefficient of tungsten,⁹ and the shear modulus, G , was taken to be $\frac{3}{8}E$. Plots of the data according to both expressions were very similar and, therefore, only the results obtained by employing the expression of Mukherjee *et al.*, have been reproduced. These results are shown in Fig. 2 for pure tungsten and in Fig. 3 for W-2 wt pct ThO₂.

As is immediately apparent, the data plotted in Figs. 2 and 3 are poorly represented by a common curve—solid line in Figs. 2 and 3. However, the curves computed from constant temperature data (dashed lines) show a good fit with slopes, n , ranging from 2.65 to 4.59 for pure tungsten and from 1.89 to 2.28 for the W-2 wt pct ThO₂ alloy. In comparison, the slopes of the common curves for pure tungsten and for W-2 ThO₂ are 3.42 and 2.58, respectively, but, as previously stated, are a poor fit for the data.

The values of the temperature-compensated creep rates plotted in Fig. 2 are inversely proportional to temperature at a given value of σ/G . They are 2 to 3 orders of magnitude lower at the highest test tempera-

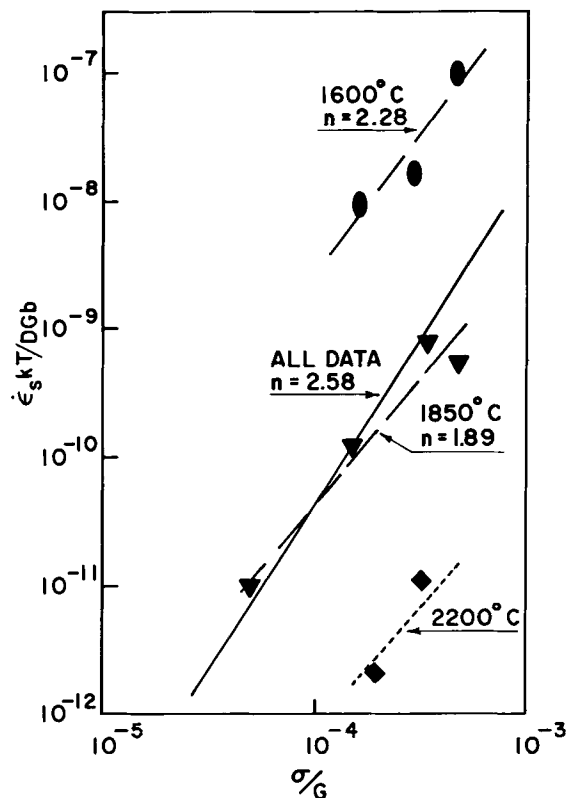


Fig. 3—Stress dependence of the creep rate of W-2 wt pct ThO₂ plotted according to Ref. 4.

ture (2200°C) than those at the lower test temperature (1600°C).

2) New Empirical Approach

In an earlier investigation,⁶ it was found that at high temperatures ($>0.5T_m$); and at nominal strain rates of $\sim 10^{-3} \text{sec}^{-1}$, the ultimate strength of tungsten was thermally activated. The activation energy at a strain rate of $1 \times 10^{-4} \text{sec}^{-1}$ was found to be about 135,000 Cal/mole, which is approximately the same as for self-diffusion of tungsten. In the same investigation,⁶ it was also found that the dependence of the ultimate strength on the strain rate at high temperatures could be expressed by a power law similar to Eq. [1], where the stress exponent, n , had a value of ~ 5 .

In a later investigation,¹⁰ it was found that the 1 hr creep rupture stress at 2200°C for the same two materials investigated here was about equal to their flow stress (proportional limit) in a constant strain rate tensile test ($8.33 \times 10^{-4} \text{sec}^{-1}$). Also, their steady-state creep rates were about the same under these conditions, although the flow stress of the two materials differed by a factor of about 3 at a given temperature and strain rate. These results suggested that the creep rates of pure tungsten and the W-2 wt pct ThO₂ alloy might be the same under test conditions in which both materials were loaded to the same fraction of the flow stress.

As the data in Table I show for stresses which are equal to the same fraction of the flow stress, the steady-state creep rates of the two materials are, indeed, as nearly the same as can reasonably be expected. Hence, one can conclude that the strain rate dependence of the flow stress in high temperature

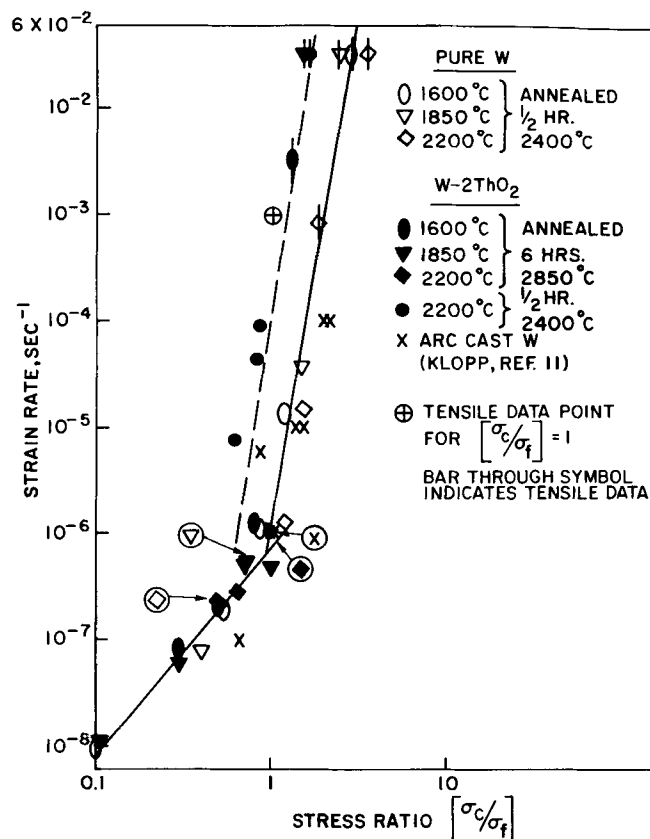


Fig. 4—Stress dependence of the creep rate of pure tungsten and W-2 wt pct ThO₂ plotted according to Eq. [3a].

tensile deformation can be expressed by an equation similar to that of Eq. [1] as follows:

$$\dot{\epsilon}_k = B'(T, S)\sigma_f^n \quad [2]$$

Here, $\dot{\epsilon}_k$ is the strain rate in a constant strain rate tensile test, and σ_f is the resulting flow stress. The term $B'(T, S)$ has a similar meaning as the term $B(T, S)$ already defined for Eq. [1]. By assuming that the constants in Eqs. [1] and [4] have the same dependence on temperature and structure, one can form the ratio of $\dot{\epsilon}_c/\dot{\epsilon}_k$ which gives:

$$\dot{\epsilon}_c = A'(\sigma_c/\sigma_f)^n \quad [3a]$$

where:

$$A' = \frac{B(T, S)}{B'(T, S)} \cdot \dot{\epsilon}_k \quad [3b]$$

A plot of the data according to Eq. [3a] is shown in Fig. 4. Included in this figure are data reported by Klopp *et al.*¹¹ for arc-cast tungsten, which further demonstrate the usefulness of Eq. [3a].

Fig. 4 shows the following pertinent features: 1) Independent of temperature, the data for each material falls on one curve which has a change in slope at about $\sigma_c/\sigma_f = 1$. 2) With decreasing stress and ratios of $\sigma_c/\sigma_f \leq 1$, the data for pure tungsten and the W-2 wt pct ThO₂ alloy are represented by a common curve with slope $n = 2.05$; however, for ratios of $\sigma_c/\sigma_f > 1$, the respective data fall on two separate curves which have about the same slope ($n = 9.63$ for pure tungsten and $n = 10.03$ for the alloy), but different intercepts. 3) The data for arc-cast tungsten (not included in determining the slopes) have a reasonably good fit within

the stress range ($\sigma_c/\sigma_f \geq 1$) in which they apply, and 4) the tensile data (indicated by a bar through the symbol) correlate well with the creep data. When all test data are presented by a single curve, the slope has a value of $n = 5.05$; however, the fit is much poorer than that represented by the curves drawn in Fig. 4.

A value of $n = 5$, derived by combining data from the literature,^{11,12} has been reported by Robinson and Sherby³ for arc-cast (AC) and electron beam melted (EB) tungsten. On the other hand, for powder metallurgy (PM) tungsten, these authors determined a stress exponent of $n = 7$ based on the other published data.^{13,14} An exception to the PM results was the data reported by King and Sell,⁷ which conformed better to a slope of $n = 5$. Robinson and Sherby attributed the difference in the stress dependence between AC- and EB-tungsten as compared to PM-tungsten, to the formation of a substructure in the former materials and its absence in the latter. On the other hand, Mukherjee *et al.*, in analyzing part of the same data cited before^{7,11,13} and using their Eq. [8], made no distinction between the values of n for the different grades of tungsten. In both grades of tungsten, n ranged from 5 to 7—see Fig. 6 of Ref. 3. More recently, Vandervoot and Barmore¹⁵ determined the stress dependence of PM-tungsten and obtained a value of $n = 5.8$. It should be noted that in the present work, as also in the work of Vandervoot and Barmore,¹⁵ a substructure was found to form during creep in the PM-tungsten. A typical example is shown in Fig. 5.

The good correlation shown in Fig. 4 for creep and tensile data taken over a wide range of temperature for materials with significantly different microstructures, (*i. e.* large vs small grain sizes or subgrain sizes, arc-cast vs pure tungsten vs W-2 ThO₂), differences in grain morphology (equiaxed vs elongated), presence of fine dispersed second phases vs precipitate-free materials (W-2 ThO₂ vs pure AC- and PM-tungsten) demonstrates the usefulness of Eq. [3a] in

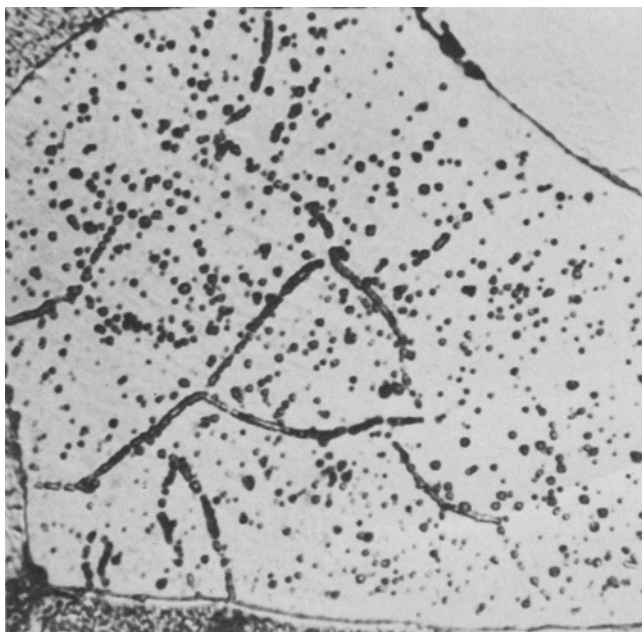


Fig. 5—Photomicrograph showing substructure formed during creep of pure tungsten (Temp. = 1870°C, $\dot{\epsilon} = 3.84 \times 10^{-5} \text{sec}^{-1}$). Magnification 1080 times.

compensating for both the temperature dependence and the structure dependence of the high temperature creep rate of tungsten.

The results obtained, Fig. 4, by the use of Eq. [3a] are consistent with previous investigations (*e. g.* Ref. 5) which have shown that the power law of creep breaks down at high stress. Also, the values of the stress exponent in certain ranges of stress are consistent with various theoretical models which have predicted values of n of 1, 3, and 4.5.¹⁶⁻²⁰ In the present work, the stress exponent is equal to about 2 over approximately one order of magnitude of the stress ratio σ_c/σ_f , Fig. 4. This result could well be caused by two competing mechanisms having a stress dependence of 1 and 3, respectively, operating concurrently or in sequence as discussed elsewhere.^{21,22} On the other hand, the value of n equal to 10 which applies in the region of high strain rates ($>10^{-5} \text{sec}^{-1}$) is without theoretical basis at present. It is obvious, however, that by restricting the range of strain rates to approximately 10^{-7} to 10^{-4}sec^{-1} (which is the range of strain rates usually employed in creep tests) a value of n equal to 5 is obtained which is usually assumed to show support for the theoretical model which predicts a value of n equal to 4.5. The main point is, however, that the results presented here indicate that the creep properties are not controlled by a single mechanism over a wide range of stress and/or temperature.

SUMMARY AND CONCLUSIONS

In summary, it is concluded that a single mechanism based on a power law expression cannot account for the stress dependency of creep in tungsten over a wide range of stress and temperature. Also, the expressions proposed by Mukherjee *et al.* and Robinson and Sherby do not adequately compensate for the effect of temperature on the creep rate for the materials presently investigated. The empirical relationship, Eq. [3a], which best correlates the data over a wide range of temperature for materials with greatly varying structures is based on the assumption that the flow stress measured in a constant strain rate tensile test ($\leq 10^{-3} \text{sec}^{-1}$) has about the same dependence on temperature and structure as does the applied stress under steady-state creep conditions.

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