

EFFECT OF GRAIN SIZE ON THE STEADY STATE CREEP RATE OF INCONEL 718

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Introduction

The influence of grain size on steady state diffusional creep of pure metals and alloys has been extensively studied and recently reviewed by Lasalmonie and Strudel(1). However, most of the studies have been concerned with the creep of pure metals or single phase alloys (2-4) and few studies have been reported concerning the effect of grain size on the creep of two phase alloys. Also, it has been generally assumed that the steady state creep rate in the dislocation climb power law creep region is independent of grain size but recent studies (1) suggest some grain size dependence for the creep rate in this region. Therefore, a study was initiated to determine the effect of grain size on the steady state creep rate of a two phase, precipitation hardened alloy in both the diffusional creep and dislocation climb power law creep regimes.

In a previous investigation (5) the creep deformation mechanisms for the precipitation hardened alloy Inconel 718 were established for the stress range 620 - 840 MN/m² and temperature range 853 - 943K for a grain size of 80µm. The results of this study are summarized in Fig. 1.

The material used in the present investigation was 3.2 mm thick sheets of commercial alloy Inconel 718 provided by International Nickel Company of Canada. The creep specimens, with a cross-section of 1.3 x 5.5 mm² and a gauge length of 25.4 mm, were machined from 1.3 mm thick strips cold rolled from the as-received sheets. These specimens were solution treated for 2 hours at 1243K, 1273K and 1373K, respectively, to obtain various grain sizes, water quenched, aged at 998K for 15 hours to obtain the same precipitate particle sizes and water quenched. The grain sizes of the alloys measured by the line-intercept method using a Leitz Image plus analyser were found to be 10 ± 2, 40 ± 2, and 100 ± 10 µm and the particle sizes were found to be 16 nm for γ' and 27 nm for γ". The creep experiments were conducted in an argon atmosphere in two T48 Avery-Denison constant stress creep machines. The creep strain was monitored during the tests by measuring the displacement of an extensometer attached to the specimen grips using an LVDT connected to a strip chart recorder. The details of the experiments were included in a previous communication (5).

Thin foil electron microscopy was used for the examination of microstructures of different parts of crept specimens and for the observation of precipitate free zones in the gauge length part of specimens. Thin foils were prepared by electropolishing 0.15 - 0.20 mm thick and 3.0 mm diameter discs in a jet electropolishing bath of 15% perchloric acid and 85% methanol at 223 - 233K. The thin foils were examined in a Philips 300 electron microscope.

The creep results for different grain sizes tested at various temperatures and stresses are given in Table I which shows that at 873K and stresses of 670 and 700 MN/m² the creep decreases with increase in grain size whereas at 873K and a stress of 820 MN/m² the creep rate increases with increase in grain size. At 898K and 923K and stresses of 690 and 650 MN/m², respectively, the creep rate first decreases with increase in grain size then increases with further increase in grain size. This peculiar behavior will be discussed in detail later but it is desirable to first establish the dominant creep mechanism for the various conditions.

TABLE I. Effect of Grain Size on Steady State Creep Rate

Solution Treatment Temp. (K) (2 h.)	Grain Size (μm)	Testing Temp. (K)	Applied Stress (MN/m^2)	Creep Rate $\dot{\epsilon}_s (\text{s}^{-1})$	n_e (From Eq.[1])	n_e^*	σ_o^* (MN/m^2)
1243	10	873	670	1.48×10^{-9}	1.16		576
1273	40	873	670	1.10×10^{-9}	1.44	1.4 ± 0.1	576
1373	100	873	670	0.93×10^{-9}	1.37		576
1243	10	873	700	2.04×10^{-9}	1.16		576
1273	40	873	700	1.64×10^{-9}	1.44	1.4 ± 0.1	576
1373	100	873	700	1.36×10^{-9}	1.37		576
1243	10	873	820	3.40×10^{-9}			605
1273	40	873	820	7.39×10^{-9}		5.1 ± 1.5	605
1373	100	873	820	8.6×10^{-9}			605
1243	10	898	650	5.13×10^{-9}	1.34		540
1273	40	898	650	4.60×10^{-9}	1.31	1.6 ± 0.2	540
1373	100	898	650	5.89×10^{-9}	1.54		540
1243	10	898	690	7.74×10^{-9}	1.34	1.6 ± 0.2	540
1273	40	898	690	6.83×10^{-9}	1.31		540
1373	100	898	690	9.44×10^{-9}	1.54		540
1243	10	923	650	1.49×10^{-8}		2.3 ± 0.3	510
1273	40	923	650	1.36×10^{-8}			510
1373	100	923	650	2.00×10^{-8}			510

* From ref. (5). Grain size = 80 μm .

Lasalmonie and Strudel (1), among other investigators, suggest that, in general, the steady state creep rate, $\dot{\epsilon}_s$, can be expressed by:

$$\dot{\epsilon}_s = A \frac{D G b}{k T} \left(\frac{b}{d} \right)^m \left(\frac{\sigma - \sigma_o}{G} \right)^{n_e}, \quad (1)$$

where A is a material constant, D is the diffusivity, G is the shear modulus, b is the Burgers vector, k is Boltzmann's constant, T is temperature, d is grain size, m the grain size exponent, σ the applied stress, σ_o the threshold stress, and n_e the stress exponent. The values of D, m and n_e need to be determined to establish the dominant creep mechanism. Slopes of log-log plots of $\dot{\epsilon}_s$ vs. $\left(\frac{\sigma - \sigma_o}{G} \right)$ for constant temperatures and grain size give the values of the stress exponent, n_e .

These plots are shown in Fig. 2. The values of n_e are listed in Table I and compared with values obtained previously under similar conditions (5). The results show that in the stress region 650 to 700 MN/m^2 and temperature range 873K to 898K the values of the effective stress exponent, n_e , are close to unity, the average value being $n_e = 1.35$.

To determine the value of the grain size exponent, m in equation [1], $\log \dot{\epsilon}_s$ vs. $\log (b/d)$ have been plotted for a constant temperature of 873K and are shown in Fig. 3. The values of m calculated from the slopes of these plots are 0.18 and 0.20 for applied stresses of 700 MN/m^2 and 670 MN/m^2 , respectively, giving an average value of $m = 0.19$. This value is much smaller than the values of 2 and 3, predicted by the models of Nabarro-Herring and Coble (6-8) for diffusional creep in single phase alloys and pure metals. However, the mechanism of creep in two phase alloys may be quite different than that for single phase alloys and therefore a different grain size exponent might be expected.

The value of diffusivity, D, cannot be determined explicitly from the creep results but if the diffusivity is assumed to be of the normal form, $D = D_o e^{-Q/RT}$, then the value of the activation

energy, Q , can be determined and a decision can thus be made as to whether lattice diffusion or grain boundary diffusion is the mass transport mechanism for creep deformation. From equation [1], the slopes of plots of $\ln \left\{ \frac{\dot{\epsilon}_s T}{G} \left(\frac{G}{\sigma - \sigma_0} \right)^{n_e} \right\}$ vs. $1/T$ for constant grain size will yield values of Q/R from which Q can be calculated. These plots for $n_e \approx 1.35$ are shown in Fig. 4. As indicated earlier, there is an anomaly in the creep rate vs. grain size relationship for the tests at 650 MPa and 690 MPa in that the creep rate exhibited a minimum at a grain size between 10 μm and 100 μm . However, the results for the 10 μm and 40 μm grain sizes are consistent and give an activation energy of 264 kJ/mol. If the data for 100 μm grain size were included the activation energy would be only slightly higher. Therefore, it is clear that lattice diffusion is the mass transport mechanism since the value of 264 kJ/mol is close to the lattice self diffusion activation energy for Ni, 280 kJ/mol (9).

If it is assumed that the coefficient for diffusion in Inconel 718 is similar to that for diffusion in nickel, i.e.: $D = 1.98 \times 10^{-4} e^{-280,000/RT} \text{ m}^2/\text{s}$ (9), then the material constant, A , in equation [1] may be determined. The average value of the constant for all the results excluding only those for which $n_e = 5.1$ and for the 100 μm grain sizes at 898 and 923 K is $A = 1.6 \times 10^{-5}$.

Thin foil electron microscopy was used to observe the microstructure before and after creep. Figs. 5 and 6 show precipitate-free zones in specimens with a grain size of 10 μm deformed at 873K at a stress of 700 MN/m² for 1086 h and 690 MN/m² for 1500 hours, respectively. Precipitate-free zones were not observed in specimens solution treated and aged at the same temperature and time as the creep specimens but with zero applied stress. Also, no precipitate-free zones were observed in the grip portions of creep specimens where the applied stress is low nor were precipitate-free zones observed in specimens creep tested under conditions where creep occurred via the dislocation climb mechanism. The theoretical models of diffusional creep in two-phase alloys proposed by Harris (10) and Burton (11) predict that precipitate-free zones (PFZ) should be present around grain boundaries that are in tension when a creep test lasts for a sufficiently long time. Since the stress exponent for the creep results discussed thus far is close to unity and since precipitate-free zones were observed in the creep specimens, we conclude that diffusional creep is the rate controlling mechanism for these experimental conditions. The creep rate for this diffusional creep in Inconel 718 is therefore given by

$$\dot{\epsilon}_s = 1.6 \times 10^{-5} \frac{D_b G b}{kT} \left(\frac{b}{d} \right)^{0.19} \left(\frac{\sigma - \sigma_0}{G} \right)^{1.35} \quad (2)$$

It was previously established for specimens tested at 873K and 820 MN/m² that dislocation climb was the creep mechanism with $n_e = 5.1$ (5). In this region, the creep rate increases with increasing grain size. The $\log \dot{\epsilon}_s$ vs. $\log (b/d)$ plot for dislocation climb creep is shown in Fig. 7. By taking the value of threshold stress, σ_0 , to be 610 MN/m² (5), the calculated values of m and A for equation [1] are -0.42 and 75, respectively. Therefore, the creep rate equation for grain size dependence in the power law creep region of Inconel 718 is given by

$$\dot{\epsilon}_s = 75 \frac{D_b G b}{kT} \left(\frac{b}{d} \right)^{-0.42} \left(\frac{\sigma - \sigma_0}{G} \right)^{5.1} \quad (3)$$

The grain size exponents determined from the experimental data show that the creep rate for diffusional creep decreases with increase in grain size while the creep rate for power law creep increases with increase in grain size. These observations suggest the possibility of the existence of an optimum grain size exhibiting a minimum creep rate in the region between the diffusional creep and power law creep regions and, indeed, this is where the rate minima were observed as shown in Fig. 1. Similar results were observed by Mannan and Rodriguez (12) for creep in 316 stainless steel. For specimens creep tested in the region between the diffusional and power law creep regions it is reasonable to assume that both processes contribute to the creep rate which may be written as

$$\dot{\epsilon}_s (\text{total}) = 1.6 \times 10^{-5} f_D \frac{D_b G b}{kT} \left(\frac{b}{d} \right)^{0.19} \left(\frac{\sigma - \sigma_0}{G} \right)^{1.35} + 75 (1 - f_D) \frac{D_b G b}{kT} \left(\frac{b}{d} \right)^{-0.42} \left(\frac{\sigma - \sigma_0}{G} \right)^{5.1} \quad (4)$$

where f_D is the fraction of diffusional creep. By taking $\partial \dot{\epsilon}_s(\text{total})/\partial d = 0$ one can calculate the optimum grain size for minimum creep rate assuming that $f_D = 0.5$. For the creep conditions of 650 MN/m² at 898K, 690 MN/m² at 898K and 650 MN/m² at 923K, where the effective stresses are equal to 110, 150 and 140 MN/m², respectively, the values of optimum grain size were calculated to be 217 μm , 32 μm and 47 μm , respectively, in fair agreement with the observed values of approximately 40 μm indicated in Table I. The value of 217 μm is somewhat high but the calculation is extremely sensitive to the value of the threshold stress, σ_0 , which is difficult to determine accurately and therefore the agreement between the calculated and observed values of optimum grain size is considered reasonable.

It is concluded that the creep rate for power law creep in Inconel 718 varies as $d^{0.42}$ whereas the creep rate for diffusional creep varies as $d^{-0.19}$. In the region between the diffusional creep and power law creep regions it is suggested that both mechanisms contribute to the creep rate and this gives rise to an optimum grain size exhibiting a minimum creep rate.

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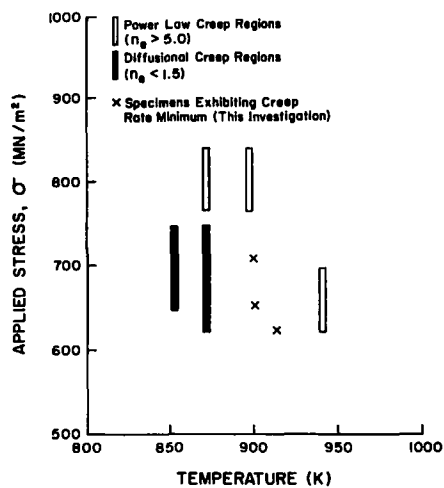


Fig. 1 Steady state creep region for Inconel 718.

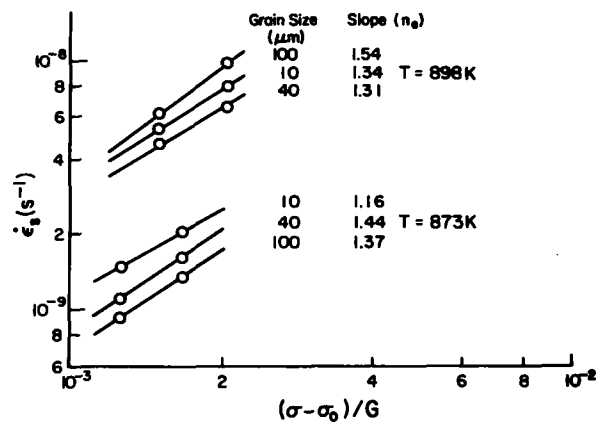


Fig. 2 Log-log plots of $\dot{\epsilon}_s$ vs $(\sigma - \sigma_0)/\sigma$ to determine stress exponent, n_e .

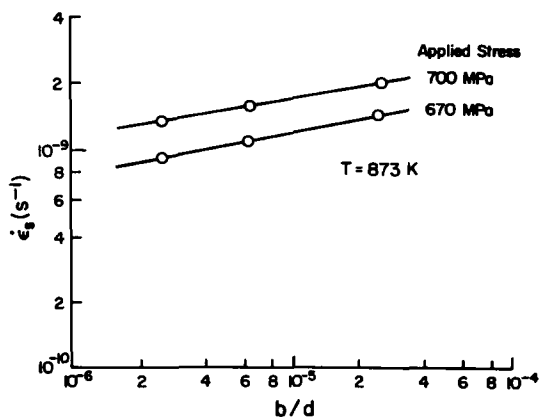


Fig. 3 Effect of grain size on diffusional creep rate.

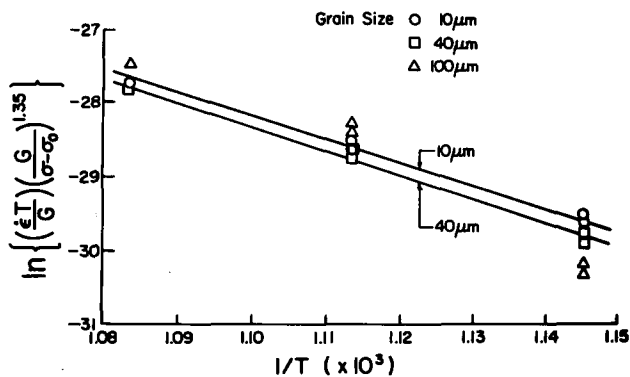


Fig. 4 Activation energy plots for $n_e = 1.35$.

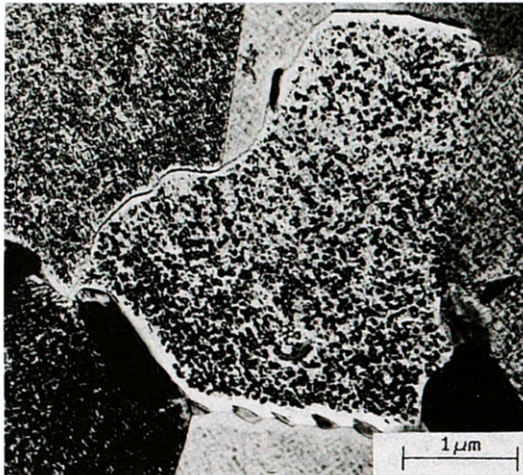


Fig. 5 Precipitate-Free Zone in a 10 μm grain size creep specimen tested under a stress of 700 MN/m^2 at 873 K for 1086 h.

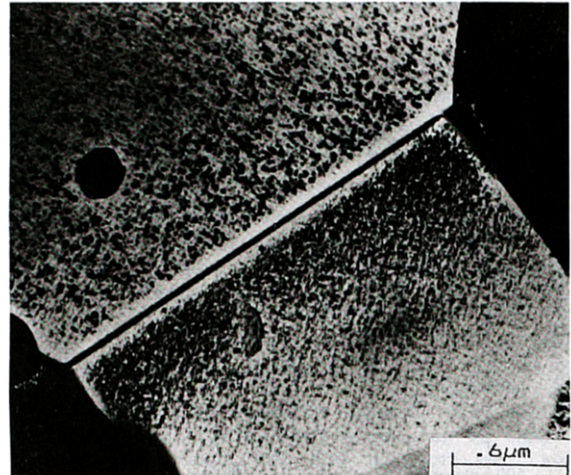


Fig. 6 Precipitate Free Zone in 10 μm grain size specimen tested under a stress of 690 MN/m^2 at 873 K for 1500 h.

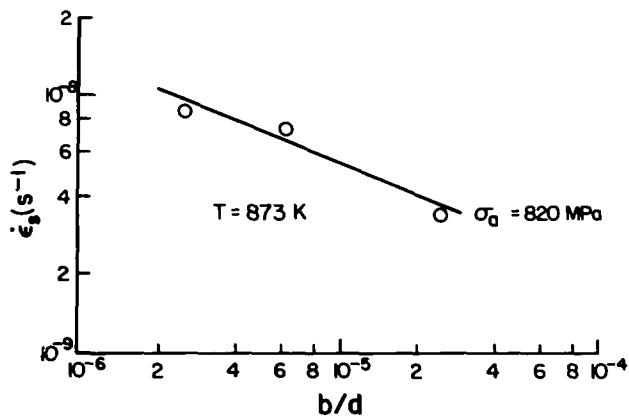


Fig. 7 Effect of grain size on power law creep rate.