Creep in Dispersion Strengthened Materials on Al Basis Prepared by Powder Metallurgy

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ABSTRACT

Some basic characteristics of creep and creep fracture for four fine grained aluminium alloys strengthened by aluminium carbide particles are described and briefly discussed. Steady state creep rate as well as time and creep strain to fracture were measured at temperatures ranging from 623 K to 723 K in a broad interval of applied stresses. The apparent activation energy of creep up to five times higher than the activation enthalpy of lattice diffusion, and the apparent stress exponent reaching values as high as 20 were found. The well-known Monkman-Grant relation was found to hold if modified introducing the strain to creep fracture. The creep data are correlated with three different models of creep, namely: (i) Model of dislocation creep as controlled by lattice diffusion (ii) Model assuming thermally activated detachment of dislocations from particles and (iii) Model of diffusional creep controlled by emission and absorption of vacancies by grain boundaries.

The creep fracture of the alloys is intergranular at low stresses with corresponding long times to fracture. The fracture is believed to be of the cavitational and constrained type.

1. INTRODUCTION

Since strengthening of aluminium alloys by (non-coherent) Al_4C_3 particles can be expected to have great potential for increasing their high temperature mechanical properties, creep and creep fracture in such alloys have been investigated extensively /1,2/. Aluminium alloys strengthened by Al_4C_3 particles are produced by mechanical alloying and/or reaction milling followed by consolidation and hot extrusion. The alloys are typically fine grained (the mean grain diameter is always less than 1 μm) and always contain a volume fraction of Al_2O_3 particles besides that of Al_4C_3 /3/. The particle size is typically 20 nm.

In the present paper, some basic characteristics of creep and creep fracture for four aluminium alloys strengthened by Al_4C_3 particles, measured in a broad enough region of temperatures and applied stresses, are presented. Also, the structure of the alloys as studied by means of the TEM and SED techniques is described. The creep data are correlated with some contemporary models of high temperature creep in dispersion-strengthened alloys.

2. EXPERIMENTAL MATERIALS AND PROCEDURES

The composition of the alloys investigated is given in Table 1. The alloys IN9021 and IN9052 were commercially produced by NOVAMET Co., USA, and supplied in the form of extruded bars 45 mm in diameter. The DISPAL alloys were laboratory produced at the Technical University Vienna, and supplied as extruded bars 6 mm in diameter.

Table 1

Dispersion strengthened aluminium alloys investigated: Nominal composition, grain size d, apparent activation energies of creep Q_c and creep fracture Q_f, apparent stress exponent of steady state creep rate m_c, and of time to creep fracture m_f.

Alloy^{+})	d [μm]	Q_c [kJ.mol^{-1}]	Q_f [kJ.mol^{-1}]	m_c	m_f
IN9021 4.0 Cu - 1.5 Mg - 4.5 Al_4C_3 + 1.8 Al_2O_3	0.86^{++})	712 ± 40	574 ± 36	16.2 ± 1.2	15.2 ± 1.2
IN9021 4.0 Cu – 0.5 Si - 4.5 Al_4C_3 + 1.8 Al_2O_3	0.55^{+++})	398 ± 11	269 ± 13	12.2 ± 0.4	10.9 ± 0.4
DISPAL (2.5) 2.5 Al_4C_3 + 2.1 Al_2O_3	0.82^{+++})	268 ± 19	247 ± 19	21.5 ± 0.9	18.4 ± 1.0
DISPAL (10.0) 10.0 Al_4C_3 + 3.8 Al_2O_3	0.72^{+++})	275 ± 19	264 ± 18	20.7 ± 0.8	18.1 ± 0.8

$^{+}$) Cu, Mg and Si in mass.%, Al_4C_3 and Al_2O_3 in vol.%

$^{++}$) after annealing at 773 K for 14.4 ks

$^{+++}$) in as received condition

From the bars of the alloys IN9021 and IN9022, creep specimens 50.0 mm in gauge length and 3.2 × 7.0 mm^2 in cross section were machined; the specimens of the former alloy were annealed at 773 K for 14.4 ks, while those of the latter one were creep tested in as received condition. From the bars of the alloys DISPAL(2.5) and DISPAL(10.0) creep specimens 50.0 mm in gauge length and 4.0 mm in diameter were machined. Both these alloys were tested in as received condition.

The tensile creep tests were performed at temperatures ranging from 623 K to 723 K in purified argon in creep machines allowing to keep applied stress constant. The creep strain ε was measured by means of the linear differential transducer and was continuously recorded during the creep test. The testing temperature was stabilized within 1 K.

The structure of the alloys, both prior to and after creep, was investigated using the TEM and SED techniques.

3. RESULTS

Relations between steady state creep rate $\dot{\varepsilon}_s$ and applied stress σ are illustrated in Fig. 1 relating to the IN9021 alloy. In the figure, the creep rate $\dot{\varepsilon}_s$ is normalized to the coefficient of lattice diffusion, D_L, and the stress σ is normalized to the shear modulus, G (for D_L and G, the values corresponding to pure aluminium were used). The fact that the data points for all temperatures cannot be fitted by a single curve demonstrates that the apparent activation energy of creep Q_c differs from the activation enthalpy of lattice diffusion ΔH_L. In fact, the value of 712 kJ.mol^{-1} was obtained for Q_c, while ΔH_L = 142 kJ.mol^{-1}. Also for the other alloys investigated, values of the apparent activation energy Q_c significantly higher than that of ΔH_L were found (Table 1).

For IN9021 alloy, the apparent stress exponent of steady state creep rate, m_c, is close to 16 as compared to the value of 4.5 for pure aluminium. The values of m_c for all the alloys are given in Table 1. It can be seen that m_c is as high as 21 for DISPAL alloys and as low as 12 for IN9052 alloy.

Note that the relations between $\dot{\varepsilon}_s/D_L$ and σ/G (Fig. 1) do not point at an existence of the true threshold stress; in fact, the apparent applied stress exponent is stress independent in the region of temperatures and applied stresses under consideration.

Relations between time to creep fracture, t_f, and applied stress are illustrated in Fig. 2 for both the

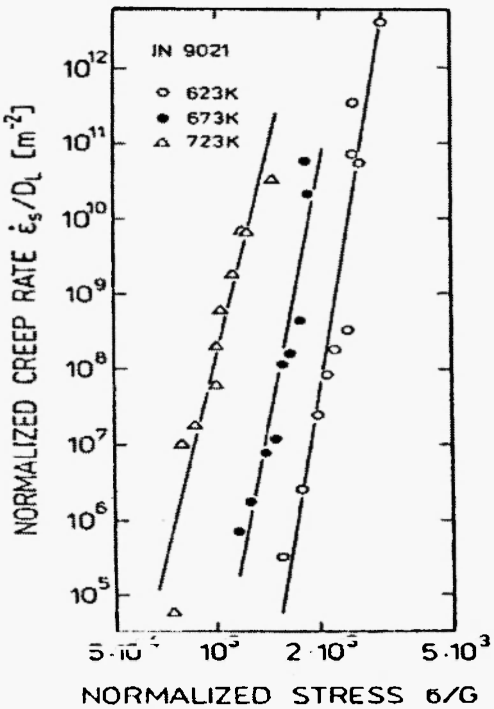

Fig. 1: IN9021 alloy. Normalized steady state creep rates plotted against normalized applied stresses.

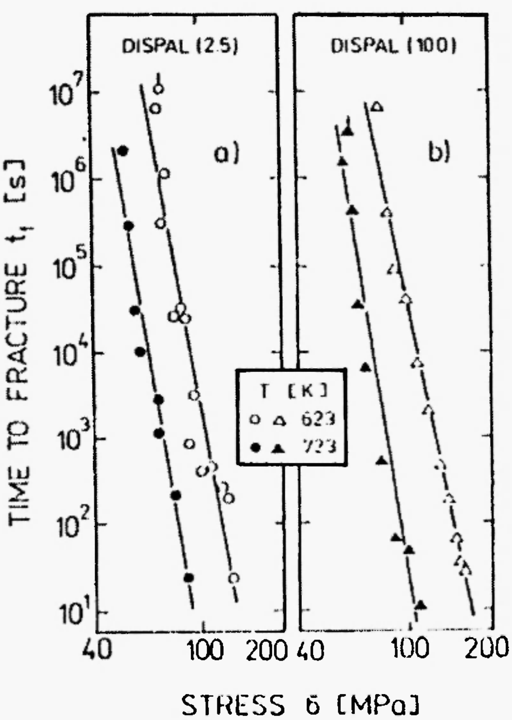

Fig. 2: DISPAL alloys. Relations between time to fracture and applied stress.

DISPAL alloys investigated. The relations are linear in double logarithmic coordinates; hence, the apparent stress exponent of time to creep fracture, m_f, is stress independent, its value being slightly lower than that of m_c. This applies to all the alloys investigated, Table I. At the same time, the value of the apparent activation energy of fracture, Q_f, is lower than that of Q_c. As it can be seen from Table I, $Q_f < Q_c$ for all alloys; at the same time, Q_f is significantly higher than ΔH_L.

In Figure 3, relations between "mean creep rate" ε_c/t_f and steady state creep rate $\dot{\varepsilon}_s$ is shown for the DISPAL alloys at temperatures 623 K and 723 K; ε_c is the creep strain to fracture. It can be seen that the well known Monkman-Grant relation as modified introducing ε_c /4/ holds:

$$\frac{\varepsilon_c}{f} = C\,\dot{\varepsilon}_s^p\,. \tag{1}$$

In eqn. (1), C and p are constants; $p = 0.99 \pm 0.10$ for both alloys. This result is typical for IN9021 and 9052 alloys, too (see ref. 5). The fact that $Q_f < Q_c$ and $m_f < m_c$ is related to the temperature and stress dependence of strain to fracture ε_c.

At low stresses, to which long times to fracture correspond, the creep fracture is intergranular. As shown elsewhere /6/, the validity of eqn. (1) with $p \simeq 1$ strongly suggests the constrained intergranular cavity growth as one of the basic processes in creep fracture.

The structure of the dispersion strengthened alloys investigated can be illustrated by that of IN9021 alloy /7/. In as received condition, the grains are uniform in size, the mean grain diameter being 0.56 μm. The dispersoid particles are of various shapes and their mean size ranges from 10 to 20 nm. Frequently, the particles form clusters in and around which dense dislocation tangles had been created during the alloy processing. Annealing the alloy at 773 K for 14.4 ks leads to slight grain coarsening (to 0.86 μm) and to a decrease of dislocation density, especially in the above mentioned tangles. The size of Al_4C_3 and Al_2O_3 particles remains without detectable changes.

After creep at the highest test temperature of 723 K and a stress of 15 MPa for 1.2×10^4 ks (up to fracture) the mean grain size of 1.03 μm was observed. However, the dispersoid as well as dislocation structure have

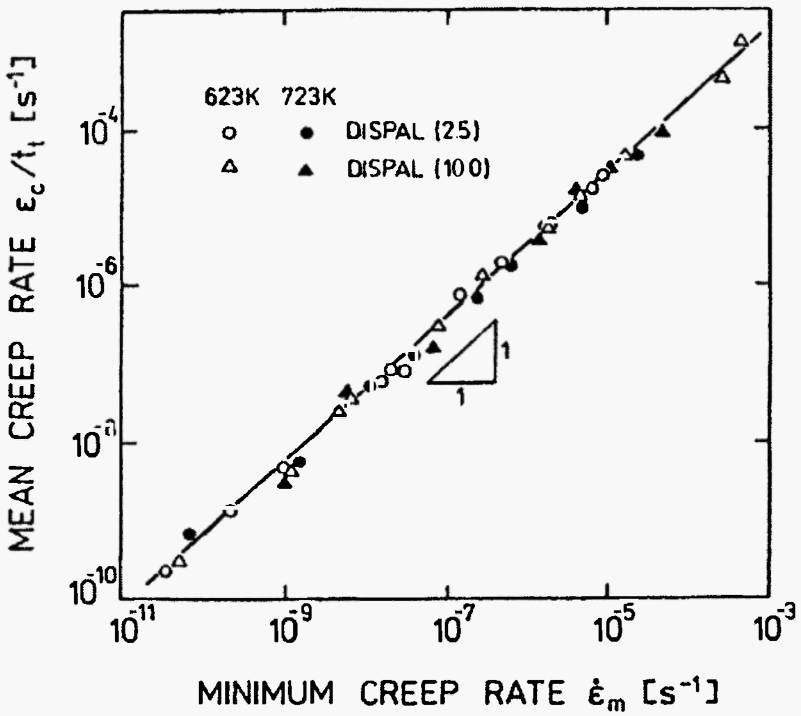

Fig. 3: DISPAL alloys. Relation between "mean" creep rate and steady state creep rate.

remained without detectable changes. Evidence of dislocation/particle interaction taking place during creep was obtained.

The structure and/or structure changes in creep in the alloy IN9052 /5/ are very similar to those in the alloy IN9021. The IN9052 alloy was creep tested in as received condition (as recommended by the producer) and the same applies to the DISPAL alloys. The structure of those laboratory produced alloys was in some respects different /2/ from that of commercially produced IN9021 and IN9052 alloys. First, the particles of dispersoid were less uniform in size; their size ranged from 20 to 80 nm. Second, the distribution of particles between grain interiors and grain boundaries was different: larger fraction of particles was situated at grain boundaries as compared to IN9021 and IN9052 alloys. However, the dispersoid structure was found to be equally stable.

4. A COMPARISON OF IN ALLOYS WITH DISPAL ALLOYS

Figures 4 and 5, in which stress dependences of steady state creep rate and time to creep fracture, respectively, of IN9021 alloy are compared with those of DISPAL alloys, demonstrate that the latter is superior to the former in the region of temperatures and applied stresses considered. One is quite naturally tempted to associate this different behaviour of two "classes" of alloys with different details of dispersoid structure following from differences in alloy processing. Such details may include not only different distribution of particles between grain interiors and grain boundaries but possibly also different structure of particle/matrix interface. Also, relatively higher volume fractions of Al_2O_3 particles in the DISPAL alloys may play a role though probably not dominant.

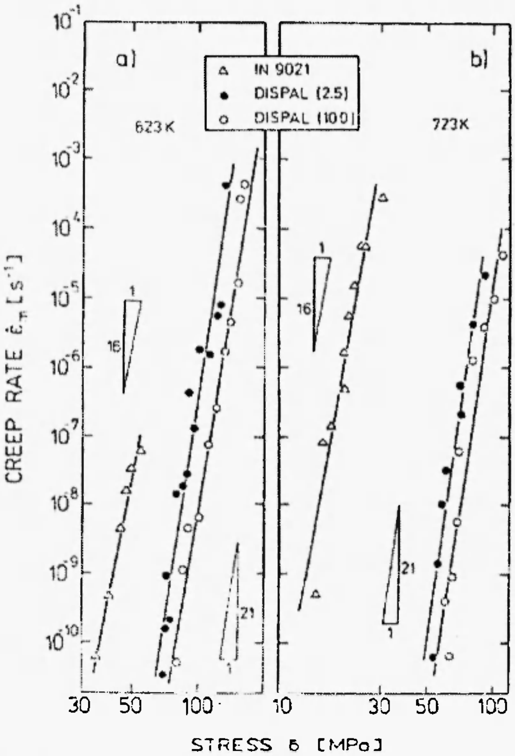

Fig. 4: A comparison of relations between steady state creep rate and applied stress for IN9021 alloy and DISPAL alloys.

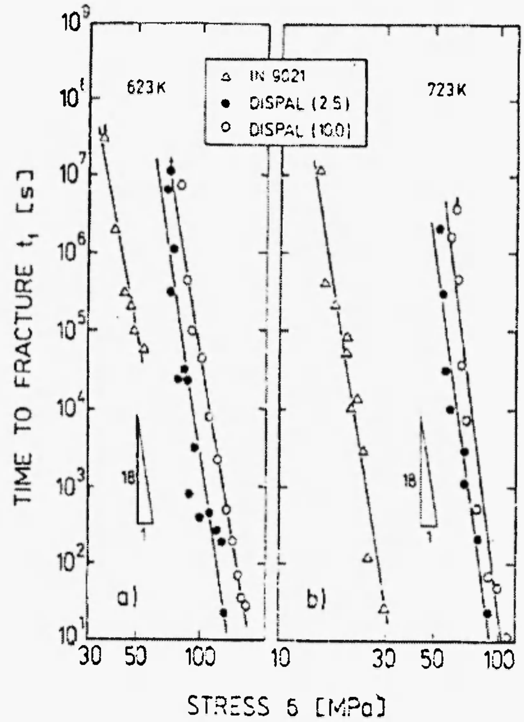

Fig. 5: A comparison of relations between time to creep fracture and applied stress for IN9021 alloy and DISPAL alloys.

The strains to creep fracture of the alloys IN9021 and IN9052 are as low as 0.02 at 623 K and low stresses to which long times to fracture correspond. However, the strains to creep fracture of the DISPAL alloys are still lower, ranging from 0.005 to 0.01 at the comparable times to fracture at 623 K. Again, this may be caused by the above mentioned differences in dispersoid structure. One promising way to improve creep plasticity of the alloys under consideration may consist in modifying the nature of particle/matrix interface by introducing a proper segregant. This segregant can be expected also to decrease grain boundary free energy and thus to affect beneficially not only the creep strain but also the time to creep fracture (creep life).

5. CORRELATION OF CREEP DATA WITH CONTEMPORARY CREEP MODELS AND DISCUSSION

1. The lattice diffusion controlled steady state creep rate of dispersion strengthened alloy is frequently described by the phenomenological relation (see e.g. ref. 8)

$$\frac{\dot{\varepsilon}_s\, k\, T}{D_L G\, b} = A\left(\frac{\sigma}{G} - \frac{\sigma_B}{G}\right)^n, \tag{2}$$

where A is a dimensionless constant, σ_B the back stress, n the true stress exponent (generally differing from m_c), b the length of Burger's vector and k the Boltzmann constant. Since the creep in the alloys investigated is not associated with a true threshold stress, σ_B is necessarily applied stress dependent. To account for the difference between Q_c and ΔH_L, this back stress must depend on temperature, too. The interpretation /9/ of creep in terms of eqn. (2) is purely phenomenological and does not contribute much to the identification of creep rate controlling mechanism.

2. Recently, Rösler and Arzt /10/ have developed a model of creep in dispersion (non-coherent particle) strengthened alloys in which attractive dislocation/particle interaction occurs. The model assumes that the

dislocation's climb past particles is easy and that the climbing dislocation acquires its critical configuration in the instant of its detachment from the particle. The athermal detachment stress σ_d is expressed as

$$\sigma_d = \sigma_{0B} \sqrt{1 - k_R^2}\ , \qquad (3)$$

where σ_{0B} is the Orowan bowing stress and k_R is the relaxation factor characterizing the strength of the attractive dislocation/particle interaction.

The correlation of the present creep data with the model has been described in detail by Orlová and Čadek /11/. For IN9021 alloy, the Orowan bowing stress σ_{0B} = 144 MPa at 623 K. The factor $k_R = 0.907$, thus $\sigma_d = 59.5$ MPa for this temperature. The correlation seems good, however, it requires the structure factor to increase from 1.05×10^{11} m^{-2} at 623 K to 3.66×10^{18} m^{-2} at 723 K, which cannot be accounted for easily.

3. The grain size of the alloys considered is very small, less than 1 μm. Consequently, one possibility which cannot be excluded is that their creep under the external conditions given occurs by the stress directed diffusional transport of matter. The particles situated at grain boundaries make the motion of grain boundary dislocations difficult so that grain boundaries do not act as perfect sources and sinks for vacancies. Under these conditions, despite the fact that creep results from diffusional matter transport, it is not diffusion controlled.

Similar to the correlations of creep data with the model based on eqn. (2), the correlation with the model of interface controlled diffusional transport of matter requires back stress depending strongly on temperature and on applied stress. Both these interpretations, discussed in detail by Kuchařová *et al.* /9/, are essentially phenomenological, this especially applies to that based on eqn. (2), which does not assume any specific dislocation model. On the other hand, the interpretation of creep data in terms of thermally activated detachment of dislocations from dispersed particles is well founded physically, and thus, is preferred to the interpretations mentioned above /11/.

6. CONCLUSIONS

Steady state creep rate as well as time and creep strain to fracture were measured at temperatures ranging from 623 K to 723 K in a broad interval of applied stresses. The apparent activation energy of creep up to five times higher than the activation enthalpy of lattice diffusion, and the apparent stress exponent reaching values as high as 20 were found. The well-known Monkman-Grant relation was found to hold if modified introducing the strain to creep fracture. The creep data are correlated with three different models of creep, namely: (i) Model of dislocation creep as controlled by lattice diffusion (ii) Model assuming thermally activated detachment of dislocations from particles and (iii) Model of diffusional creep controlled by emission and absorption of vacancies by grain boundaries. The creep fracture of the alloys is intergranular at low stresses with corresponding long times to fracture. The fracture is believed to be of the cavitational and constrained type.

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