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Effect of magnesium content on the stress exponent and effective stress in the steady state creep of Al–Mg alloys

D. O. NORTHWOOD*, L. MOERNER, I. O. SMITH
Department of Mining and Metallurgical Engineering, University of Queensland, St Lucia, Queensland 4067, Australia

The stress exponent of steady state creep, n, and the internal (σ_i) and effective stresses (σ_e) have been determined using the strain transient dip test for a series of polycrystalline Al–Mg alloys creep tested at 300° C and compared with previously published data. The internal or dislocation back stress, σ_i, varied with applied stress, σ, but was insensitive to magnesium content of the alloy, being represented by the empirical equation $\sigma_i = 1.084\sigma^{1.802}$. Such an applied stress dependence of σ_i can be explained by using an equation for σ_i of the form $\sigma_i \ \alpha (\text{dislocation density})^{1/2}$ and published values for the stress dependence of dislocation density. Values of the friction stress, σ_f, derived using the equation $\sigma_e/\sigma = (1-c)\ (1-\sigma_f/\sigma)$, indicate that σ_f is not dependent on the magnesium content. A constant value of σ_f can best be rationalized by postulating that the creep dislocation structure is relatively insensitive to the magnesium content of the alloy.

1. Introduction

Aluminium–magnesium alloys have been classified with Class I alloys [1] in which the applied-stress exponent, n, of the steady-state creep rate, $\dot{\epsilon}_s$, in the following equation is about 3.

$$\dot{\epsilon}_s = \alpha\sigma^n \exp(-Q_c/RT) \qquad (1)$$

where σ is the applied creep stress, Q_c the apparent activation energy for creep, α a constant, R the gas constant, and T the test temperature. A stress exponent of about 3 is considered characteristic of a viscous glide process [2, 3]. Pure metals, where the recovery creep process is rate controlling, have on the other hand been found to have higher values for the stress exponent with values of $n =$ 4.0 to 4.5 being reported for pure aluminium [2].

Several authors, however, have proposed that creep in alloys is determined not by the applied stress, σ, but by an effective stress, σ_e, which differs from the applied stress by a quantity σ_i which is termed either an internal, friction or back-stress [4–7]. Equation 1 can then be rewritten either as:

$$\dot{\epsilon}_s = \alpha\sigma_e^{n'} \exp(-Q_c/RT) \qquad (2)$$

$$\dot{\epsilon}_s = \alpha(\sigma-\sigma_i)^{n'} \exp(-Q_c/RT) \qquad (3)$$

where n' is the stress exponent based on the effective stress. Obviously in the extreme case where $\sigma_i \simeq 0$ the two stress exponents n and n' would be approximately equal. However, the bulk of the experimental data for Al–Mg alloys indicates that σ_i is of an appreciable level [7–11] and thus n and n' would not be expected to be equal.

The present authors have published some initial results for Al–Mg alloys containing 1.77 to 7.7 at % Mg alloys at 300° C. These are compared with previous published data, particularly that of Oikawa *et al.* [10] and an empirical equation has been developed relating effective stress to the applied stress.

2. Experimental procedure

2.1. Materials

Aluminium alloys containing 1.77, 2.73, 4.20 and 7.72 at % Mg were vacuum cast using high purity

*On leave from Engineering Materials Department, University of Windsor, Windsor, Ontario N9B 3P4, Canada.

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TABLE I Chemical composition and grain size

Concentration of magnesium		Average grain size
(at %)	(wt %)	(mm)
1.77	1.67	0.26 ± 0.08
2.73	2.47	0.19 ± 0.08
4.20	3.80	0.13 ± 0.03
7.72	7.01	0.13 ± 0.04

(99.999%) metals. The alloys were swaged and drawn to 2.95 mm diameter wire with appropriate intermediate annealing treatments. Creep specimens with a 76 mm gauge length were cut from the wire. These specimens were then annealed for 1/2 h at 400° C and quenched into water to obtain a complete α-phase solid solution prior to creep testing. Chemical compositions and grain sizes are listed in Table I.

2.2. Creep tests

All creep tests were performed in air at 300° C with a temperature gradient along the gauge length of less than 1° C. A uniaxial constant stress creep machine with high sensitivity was used. The strain is detected by a pair of LVDT transducers outside the hot zone. Invar rods are used to transmit the strain from the wire grips so that temperature fluctuations have a minimal effect on the strain signal. The temperature in the room was controlled to better than ± 0.5° C and the resulting stability of the strain signal was excellent. Steady state creep rates down to $10^{-8}\,\text{sec}^{-1}$ could be measured with confidence and reproducibility. The experimental set-up is described in more detail by Smith [13].

The effective stress, σ_e, was measured using the stress-dip transient method [14] where once the specimen is creeping at the steady state rate, part of the applied stress is removed and the nature of the strain transient is observed. The sign of the strain rate just after the reduction depends on the magnitude of the stress change with positive creep rates being obtained after small changes and negative transients being recorded for large reductions [4, 15, 16]. At one particular stress reduction, the creep rate after the instantaneous drop is zero, and there is an incubation period before creep resumes in a forward manner. It is this particular stress reduction to cause zero creep that is considered to be σ_e, the effective stress. It is estimated that σ_e could be determined to an accuracy of ± 0.125 MPa.

3. Results

3.1. Stress exponent, *n*

Values of the stress exponent (n and n') for steady state creep found in the present work are given in Table II. It will be noted that, in general, n increases with increasing magnesium content whereas n' remains approximately constant (with the exception of the 2.73 at % Mg alloy where n' is somewhat lower). Fig. 1 in which the stress exponent, n, is plotted against the magnesium content, summarizes previously determined values for creep at 300° C and compares them to the present results. In general the values for n obtained in the present work are higher than previously published data, particularly for magnesium contents above about 3 at %. It is interesting to note that the values obtained in this work for n', the stress exponent of steady state calculated using σ_e rather than σ, are more in line with previously published results for n for the higher magnesium content alloys. However, comparing the present results with those of Oikawa *et al.* [10], which was the only other fairly extensive study of effect of magnesium content on stress exponent, a number of similarities can be seen, namely:

1. when the magnesium content is increased above about 1 at % the stress exponent, n, decreases from a value, 4.2, which is typical of recovery creep in metals [3] to a value closer to 3 which is typical of control by a viscous glide process [3];
2. the stress exponent remains fairly constant

TABLE II Stress exponents of steady state creep at 300° C for Al–Mg Alloys

Composition	Stress Exponents			
	Equation I		Equation II	
at % Mg	n	(Correlation coefficient)	n'	(Correlation coefficient)
1.77	3.37	(0.9991)	3.04	(0.9920)
2.73	3.38	(0.9986)	2.76	(0.9959)
4.20	3.68	(0.9928)	3.06	(0.9738)
7.72	3.98	(0.9922)	3.05	(0.9920)

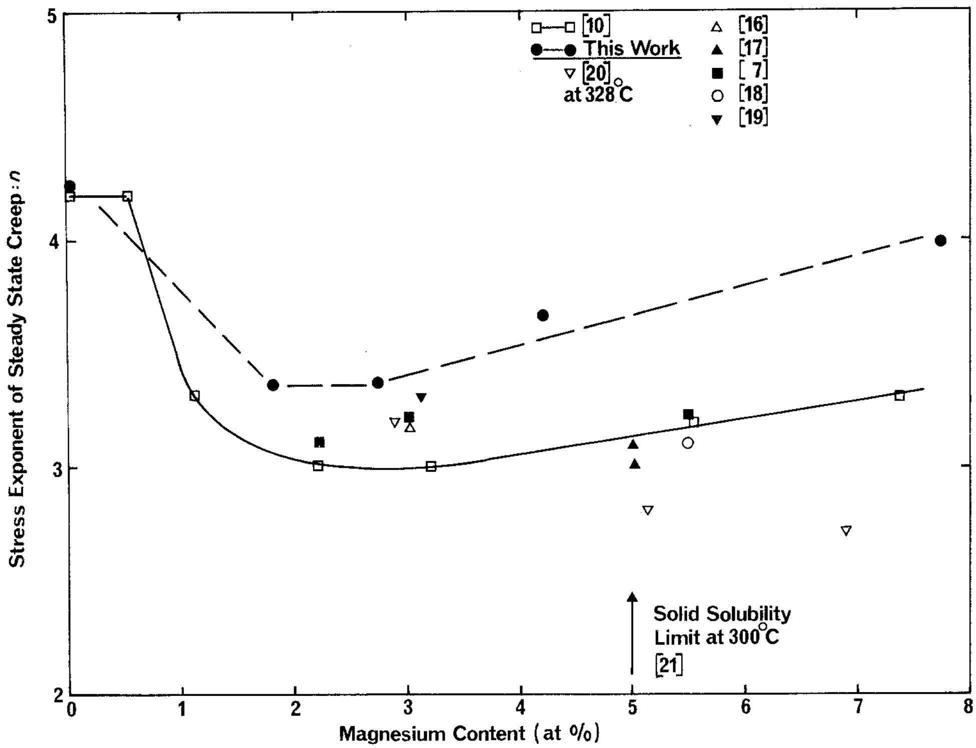

Figure 1 Variation of the stress exponent of steady-state creep, n, with the magnesium content of Al–Mg alloys creeping at 300° C.

for magnesium contents from about 1 to 4 at %;

3. for magnesium contents above about 4% there is an increase in n with increase in magnesium content. The data of Horiuchi and Otsuku [20] show the opposite trend but this may be due to the different testing temperature (328 cf. 300° C). Oikawa *et al.* [10] tentatively ascribed this increase in n to precipitation since the solid solubility limit of magnesium in aluminium at 300° C is just under 5 at % [21], and any deformation could assist precipitation.

3.2. Effective stress (σ_e) and internal stress (σ_i)

As was detailed in the experimental procedure section, it is σ_e that is actually measured and σ_i is then calculated from the relationship $\sigma_i = \sigma - \sigma_e$. The variation of the internal stress, σ_i, with the applied stress, σ, for the four alloys in the present investigation is shown in Fig. 2. In all alloys, σ_i increased, although the ratio σ_i/σ decreased with increasing σ. This is similar to the trend noted by Oikawa *et al.* [10]. Magnesium content appears to have little or no effect on σ_i as can be seen from Fig. 3 where σ_i is plotted against σ for all four alloys. The fact that σ_i is independent of concentration of magnesium is also demonstrated in Fig. 4 where a set value of applied stress has been chosen (14.7 MPa since data for this value of σ were published by Oikawa *et al.* [10] in their series of tests on Al–Mg alloys) and σ_i has been plotted against concentration of magnesium (at %). Also shown in Fig. 4 are the data of Oikawa *et al.* [10] and the agreement between those results and our own is nothing short of startling.

The complete set of data for effective stress, σ_e, from the present work are plotted in Fig. 5 against applied stress on a log-log scale. Also plotted are the data of Oikawa *et al.* [10] for Al–Mg alloys at 300° C. As was found by Oikawa *et al.* previously, log–log plots of σ_e against σ give a good straight line relationship between the two quantities. The two sets of data are complementary in the sense that our data are mainly for lower stresses, $\sigma < 20$ MPa, whereas the data of Oikawa *et al.* are mainly for higher stresses, $\sigma > 20$ MPa. Linear regression analysis was used to fit the data to an equation of the form:

$$\sigma_e = A\sigma^m \quad (4)$$

Table III lists values for A and m together with

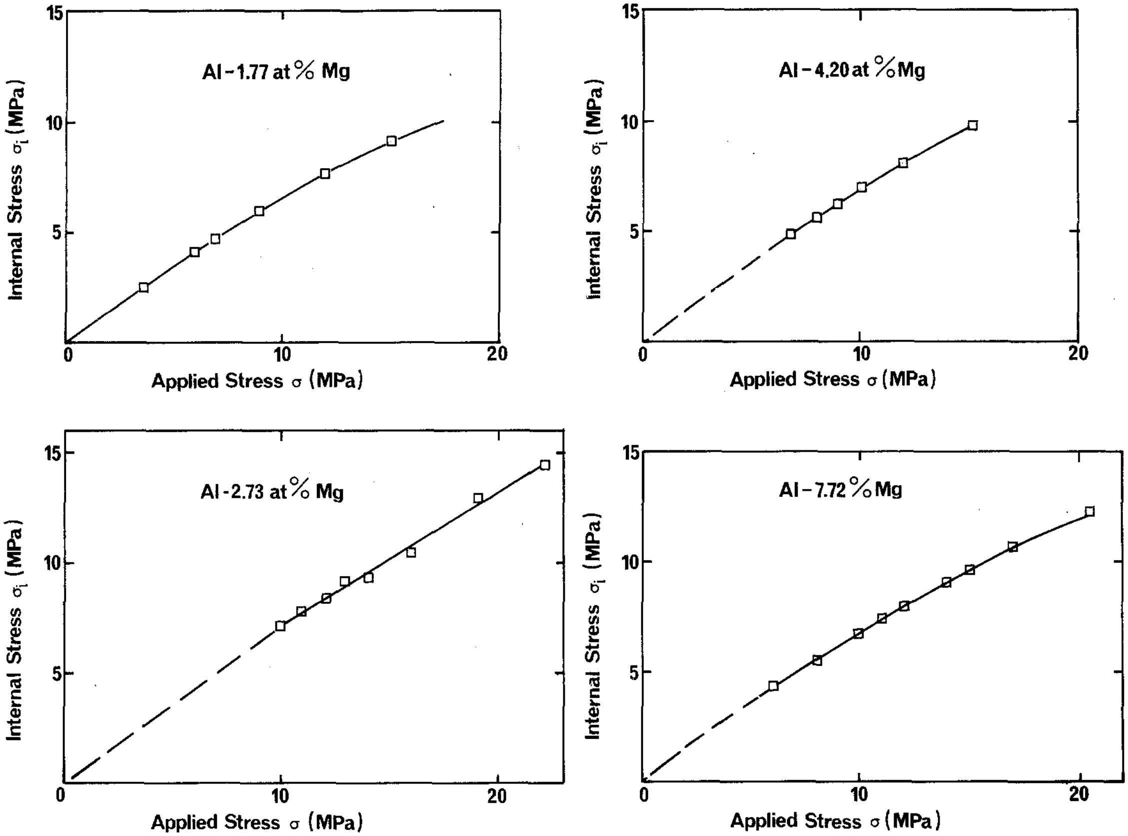

Figure 2 Variation of the internal stress, σ_i, with applied stress, σ, for a range of Al–Mg alloys creeping in steady-state at 300° C.

correlation coefficients for the linear regression analysis using our data, the data of Oikawa *et al.* [10], or both sets of data. It is interesting that the highest correlation coefficient is obtained when analysing both sets of data together.

4. Discussion

The fact that the stress exponent of steady state creep determined using applied stress, n, is not equal to the stress exponent of steady state creep determined using the effective stress, n', indicates that the internal stress, σ_i, is of an appreciable level. This is confirmed by the σ_e and σ_i measurements where σ_i/σ varied from 0.7 to 0.65, decreasing with increasing σ and reaching a value of 0.65 at $\sigma = 20$ MPa (Fig. 3).

Looking at the published values for n at 300° C it can be seen from Fig. 1 that magnesium additions greater than about 1 at % reduce n suddenly from a value of about 4.2 for pure aluminium to about 3 to 3.5 for the Al–Mg alloys. Such a change in n is consistent with a change in rate controlling creep mechanism from high temperature climb controlled recovery creep for pure aluminium, to viscous glide controlled creep in the Al–Mg alloys [5, 22]. The increase in n noted both by Oikawa *et al.* [10] and ourselves for magnesium contents greater than about 4 at % could, as suggested in the results section, be due to the presence of a small amount of precipitate. The precipitates would reduce creep rates slightly without affecting the rate-controlling process

TABLE III Values of A and m in the equation $\sigma_e = A\sigma^m$ obtained using linear regression analysis

Reference for Data	Number of data points	Values in equation A	m	Correlation coefficient for $\sigma_e = A\sigma^m$
Present work	30	0.217	1.16	0.986
Oikawa *et al.* [10]	24	0.135	1.33	0.994
Present work and Oikawa *et al.* [10]	54	0.165	1.28	0.995

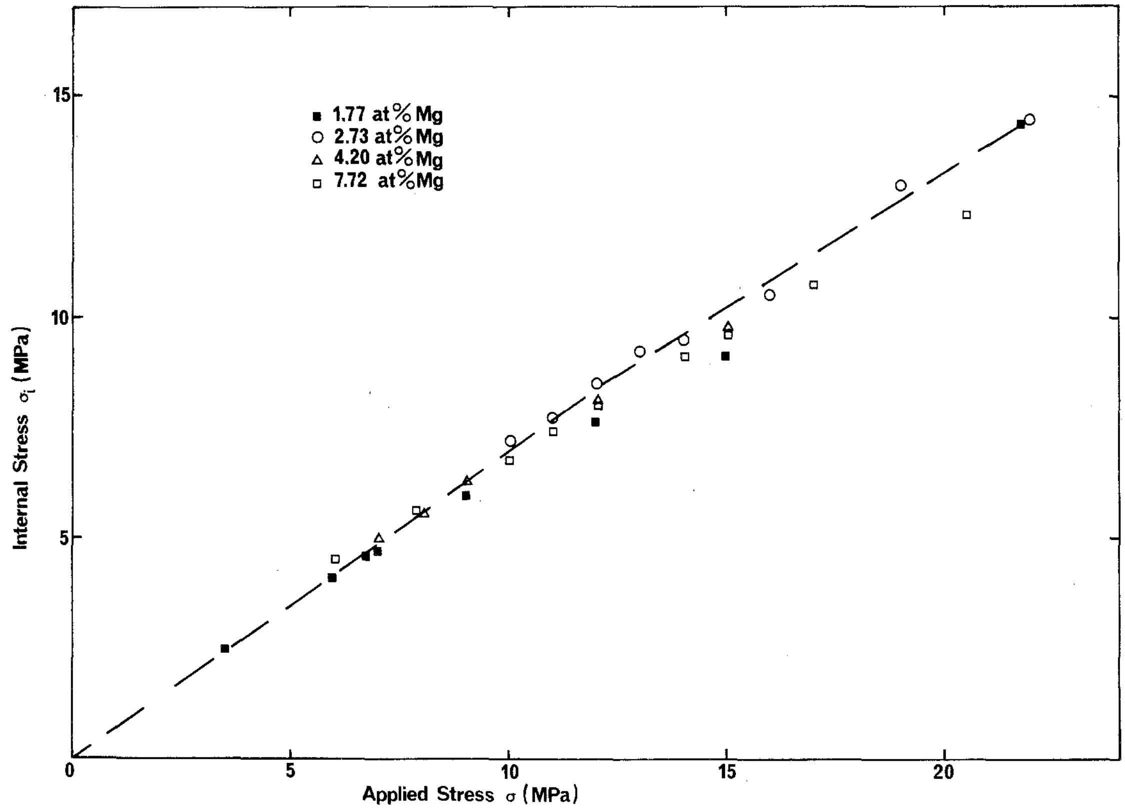

Figure 3 Applied stress dependence of internal stress for Al–1.77, 2.73, 4.20 and 7.72 at% Mg alloys creeping in steady-state at 300° C.

(i.e. viscous glide would remain rate controlling). It is expected that the reduction of creep rates would be more significant at lower values of applied stress, with the net effect that over the whole range of applied stresses n would become greater than the value that would be obtained in the absence of precipitates. The feature that is difficult to explain is the slightly higher values of n obtained in the present work compared to those found by Oikawa *et al.* [10], given the fact that the internal stresses or effective stresses are almost identical in the two sets of tests. One possible explanation could be the differences in grain size with grain sizes varying from 0.13 to 0.26 mm in

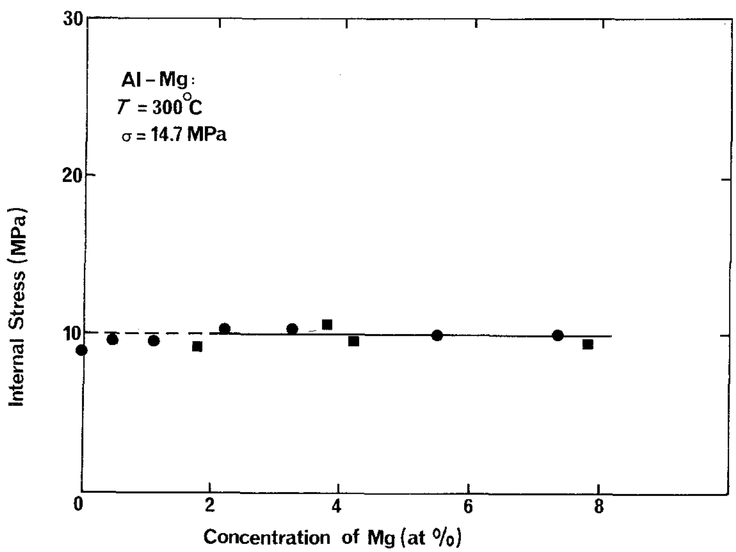

Figure 4 Concentration dependence of the internal stress, σ_i, in Al–Mg alloys creeping in steady-state at 300° C under an applied stress at 14.7 MPa. Both the present data (■) and previous data (•) of Oikawa *et al.* [10] are given.

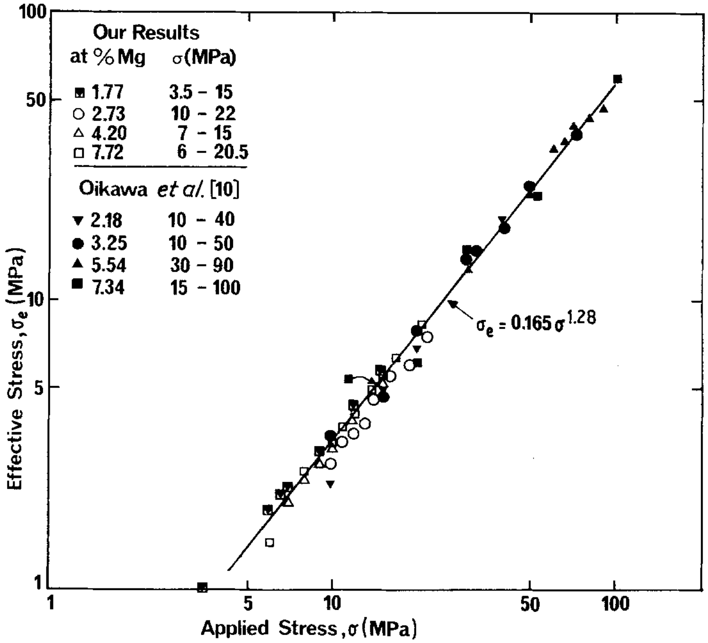

Figure 5 Applied stress dependence of the effective stress, σ_e, for Al–Mg alloys creeping in steady-state at 300° C.

the present work and 0.5 to 1.1 mm in the tests of Oikawa *et al.* [8, 10]. (Note that slightly different grain sizes are given in [9] and [10] for apparently the same materials.)

The values of σ_i and σ_e reported in this work are obviously in excellent agreement with the results of Oikawa *et al.* [10]. Thus the difference in the purity of the metals (99.99% pure for Oikawa *et al.* [10] compared to 99.999% in the present study) or the grain sizes (Oikawa *et al.*'s grain sizes being approximately four times larger than in the present work) made little or no difference to σ_i or σ_e. The agreement with the results of Ahlquist and Nix [7] for Al–3% Mg at 300° C is also fairly good with σ_i decreasing from 0.66 at $\sigma = 13.8$ MPa to 0.58 at $\sigma = 21.8$ MPa. (This compares to $\sigma_i/\sigma = 0.69$ at $\sigma = 13.8$ MPa and $\sigma_i/\sigma \simeq 0.66$ at $\sigma = 21.8$ MPa in the present series of tests.) The agreement with the results of Kucharova *et al.* [18] for Al–5.5 at% Mg at 300° C is not quite so good where σ_i/σ was found to decrease from 0.57 at $\sigma = 10$ MPa, to 0.46 at $\sigma = 20$ MPa, and finally down to 0.13 at $\sigma = 102.5$ MPa. However, the general trend of a decreasing σ_i/σ ratio with increasing σ is to be found in all the results.

In the results section the data for σ_e were fitted to an equation of the form $\sigma_e = A\sigma^m$ and the parameters A and m evaluated by least squares regression analysis. Since many theoretical models exist for estimating σ_i rather than σ_e it was considered useful to fit the data for σ_i to a similar type of equation namely:

$$\sigma_i = B\sigma^x \tag{5}$$

Table IV lists values for B and x together with correlation coefficients for the linear regression analysis using either our data, the data of Oikawa *et al.* [10], or both sets of data together. Again the agreement between the data of Oikawa *et al.* [10] and present results is fairly good and an average stress exponent (x) of about 0.8 is found when taking both sets of data.

Argon *et al.* [23] have developed an equation relating σ_i to σ as follows:

$$\sigma_i/\mu = C_i\frac{(1-\nu)}{\pi}(K\theta)^{1/3}\,[\sigma/\mu]^{2/3} \tag{6}$$

TABLE IV Values of B and x in the equation $\sigma_i = B\sigma^x$ obtained using linear regression analysis

Reference for data	Number of data points	Values in equation B	Values in equation x	Correlation coefficient for $\sigma_i = B\sigma^x$
Present work	30	0.815	0.922	0.995
Oikawa *et al.* [10]	24	1.307	0.749	0.992
Present work and Oikawa *et al.* [10]	54	1.084	0.802	0.994

where μ is the shear modulus, ν the Poissons ratio, C_i a constant $= 0.317$, K a constant $= 30$ (from the equation relating subgrain size, d, to stress: $d = K \mu b/\sigma$), and θ the sub-boundary misorientation. Thus if such an equation represents the situation for the Al–Mg alloys we would expect a stress exponent, x, of about 0.67. This is not that far removed from the measured empirical value of about 0.8 but the difference is probably large enough to suggest that either the equation does not apply, or that the terms apart from $(\sigma/\mu)^{2/3}$ on the right-hand side of Equation 6 are not constant. Of the terms on the right-hand side of Equation 6 the only term that could be expected to vary with σ is θ, the lattice misorientation across a sub-boundary. θ is known to vary with the square root of dislocation density as does the internal or flow stress. Since θ appears in the equation for σ_i/μ as a $\sigma^{1/3}$ term then an additional $\sigma^{1/3}$ term could be expected, thus giving an overall linear dependence of σ_i on σ. A stress exponent, σ, of 1 is not in good agreement with the measured value of about 0.8. However, if the lattice misorientation across a sub-boundary did not vary directly with σ but with a fractional power of σ then better agreement would be obtained. A more compelling reason why Equation 6 does not fit the data is that it was developed for materials that from sub-grains during high-temperature creep. Such materials are usually considered to be pure metals and Class II alloys [24, 25]. Class I alloys (e.g. Al–Mg) are, however, considered to form no subgrains and the dislocations generally show a minimum of clustering [3, 24, 26, 27]. Thus our materials would not be expected to obey Equation 6. However, limited transmission electron microscope examination of thin foils prepared from our creep specimens has indicated some degree of subgrain formation and dislocation clustering [28]. An equation such as Equation 6 would explain why σ_i (or σ_e) is virtually independent of the concentration of magnesium in the alloy. Of the terms on the right-hand side of the equation, C_i and K are constants. The elastic properties $(\mu + \nu)$ are considered fairly insensitive to small additions of magnesium [29] and values for aluminium are generally used [9]. The sub-boundary misorientation term, θ, could possibly vary with magnesium content but the variation, if any, is considered to be quit: small. Thus, overall, σ_i would be fairly insensitive to changing magnesium content.

Equations have been developed to relate the internal stress to the dislocation arrangement (configuration) in the material. One such equation is [30]:

$$\sigma_i = \alpha M G \boldsymbol{b}\, \rho^{1/2} \tag{7}$$

where α is a geometric constant, M an orientation factor, G the shear modulus, $\boldsymbol{b}$ the burgers vector, and ρ the dislocation density. The dislocation density is dependent on the applied stress and can be expressed as a power function

$$\rho = \rho_0\, \sigma^{n} \rho \tag{8}$$

Combining Equations 7 and 8 gives:

$$\sigma_i = \alpha M G \boldsymbol{b}\, \rho_0^{1/2}\, \sigma^{n} \rho = \alpha M G \boldsymbol{b}\, \rho_0^{1/2}\, \sigma^{\rho n/2} \tag{9}$$

Values of the stress exponent n_ρ have been determined as 1.62 for Al–5 Mg creeping at 300° C [9], 1.7 for Al–5 Mg at 360° C [20] and 0.93 for Al– 5.5 at % Mg at 300° C [31].

Relating Equations 9 and 5, $B = \alpha M G \boldsymbol{b}\, \rho_0^{1/2}$ and $\sigma = n_\rho/2$. Using the values for n_ρ from [9] and [20], since they are reasonably consistent, σ would be expected to be 0.81 to 0.85. This compares to the values of 0.80 determined by least squares analysis of the present σ_i against σ data, plus that of Oikawa *et al.* [10], Table IV. Such a relationship as Equation 1 also explains why σ_i is virtually independent of the concentration of magnesium, since Oikawa *et al.* [9] have shown for Al–Mg alloys creeping at 300° C that ρ is independent of magnesium concentration for magnesium contents greater than about 1 at %.

Weertman [32] has developed a relationship between σ, σ_e and σ_f (the friction stress) which gave a not unreasonable account of the effective stress that had been measured on Al–5.5 at % alloys by other researchers in high temperature creep experiments [7, 18]. The relationship is as follows:

$$\sigma_e/\sigma = (1 - c)(1 - \sigma_f/\sigma) \tag{10}$$

where c is a temperature independent constant and σ_f is defined as the stress that must be exceeded if the dislocation is to move in any microcreep mechanism and is one of the components of internal stress. We have previously attempted to fit this equation to some of our data for the 1.77, 4.20 and 7.72 at % Mg alloys and found the fit to be fairly good [11]. That analysis indicated that σ_f increased with increasing magnesium content from a value of 1.05 MPa for the

TABLE V Values of the constants c and σ_f in the equation $\sigma_e/\sigma = (1-c)(1-\sigma_f/\sigma)$ for the steady state creep of Al and Al–Mg alloys at 300° C

Alloy composition	Applied stress range (MPa)	Temperature Independent constant, C	Friction stress, σ_f (MPa)	Reference
Al–1.77 at % Mg	3.5 – 15.0	0.61 ± 0.02	1.05 ± 0.36	This work
Al–2.73 at % Mg	10.0 – 22.0	0.17 ± 0.03	1.22 ± 0.07	This work
Al–4.20 at % Mg	7.0 – 15.0	0.65 ± 0.03	1.17 ± 0.78	This work
Al–7.72 at % Mg	6.0 – 20.5	0.55 ± 0.02	2.73 ± 0.42	This work
Polycrystalline Al	4.0 – 16.0	0.08 ± 0.04	1.66 ± 0.20	[10]
Al–2.18 at % Mg	10.0 – 0.02	0.09 ± 0.02	1.47 ± 0.05	[10]
Al–3.25 at % Mg	10.0 – 50.0	0.20 ± 0.04	1.24 ± 0.06	[10]
Al–5.54 at % Mg	30.0 – 90.0	0.55 ± 0.02	1.00 ± 0.10	[10]
Al–7.74 at % Mg	15.0 – 100.0	0.15 ± 0.04	1.30 ± 0.06	[10]
99.995% pure Al	3.5 – 10.6	0.24 ± 0.08	3.56 ± 0.44	[7]
Al–3.0 at % Mg	13.8 – 21.8	0.45 ± 0.01	5.54 ± 1.64	[7]

Al–1.77 at % Mg alloy to 2.73 MPa for the Al–7.72 at % Mg alloy. All our data for σ_e (all four alloys) plus the data of Oikawa *et al.* [10] and Ahlquist and Nix [7] have now been used to compute values of C and σ_f using a statistical fit to a non-linear least squares estimation of the parameter, and the results are presented in Table V. The data for σ_f are plotted in Fig. 6 as a function of magnesium content. The data of Ahlquist and Nix [7] give much higher values for σ_f than in the present work or the previous work of Oikawa *et al.* [10]. The agreement

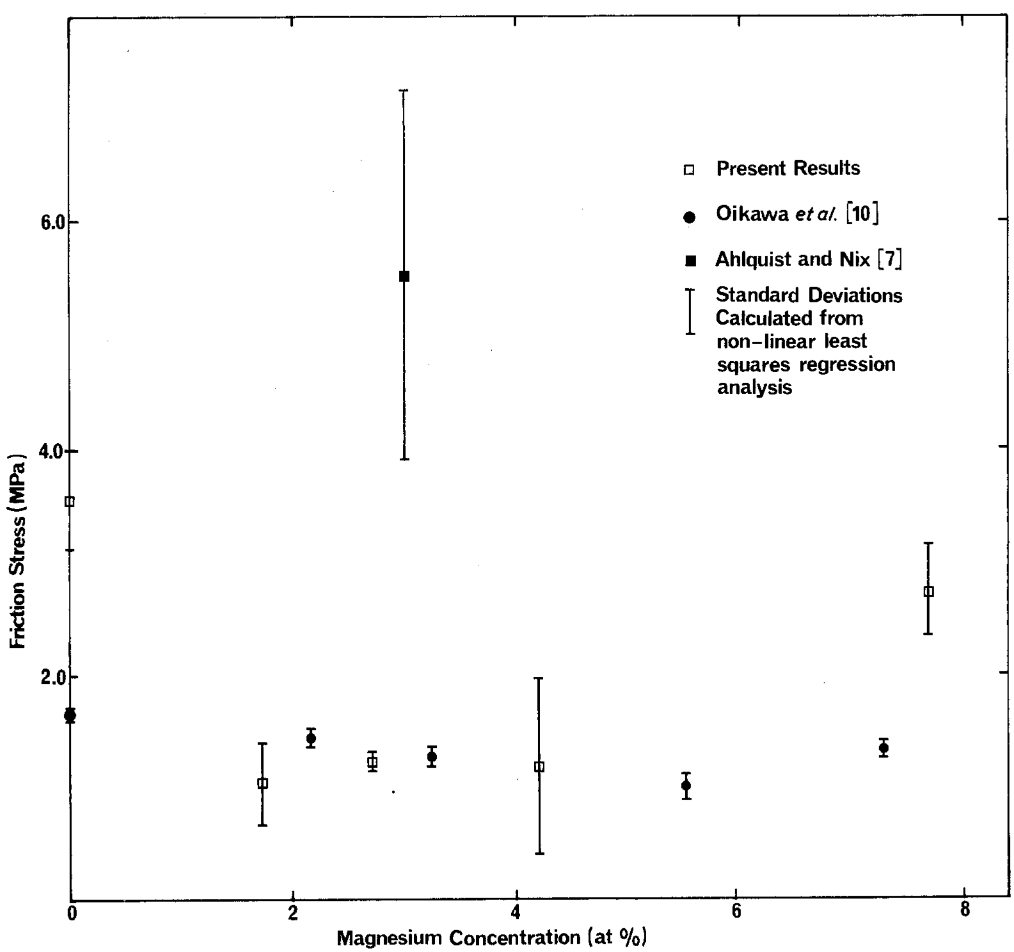

Figure 6 Concentration dependence of the friction stress, σ_f, for Al–Mg alloys creeping in steady state at 300° C.

between our work and that of Oikawa *et al.* is excellent. The apparent trend noted before of increasing σ_f with increasing magnesium content does not appear to be a real trend and was only indicated by the somewhat high value of σ_f for the 7.72 at% Mg alloy. In fact, if one can believe the limited data for pure aluminium, σ_f is of the same order of magnitude as for the Al–Mg alloys.

The various origins of the friction stress σ_f are considered to be [32]:

1. the Peierls hills. The Peierls (shear) stress, τ_ρ, is given by an equation of the form [33]:

$$\tau_\rho \simeq \frac{2G}{1-\nu}\, e^{-2\pi w/b} \qquad (11)$$

where w is the width of dislocation, and b the distance between atoms in slip direction. Given that the elastic constants (G and ν) vary little, if any, with small magnesium additions, τ_ρ (and hence σ_f) would remain approximately constant if w and b were relatively insensitive to magnesium content. This would seem to be reasonable assumption;

2. the stress fields of immobile dislocations and of precipitate particles can presumably be disregarded. Assuming that the arrangement and number of dislocations in the Al–Mg alloys is not overly sensitive to magnesium content when this source for a friction stress would given a fairly constant value for all magnesium contents. Whether this is so could only be confirmed by an extensive TEM study of all samples;

3. the presence of forest dislocations. Certainly forest dislocations have been seen in these materials [28] but again as in (2) more detailed TEM is required;

4. the presence of some stress induced order microcreep mechanism that is enhanced by an increased substitutional solute concentration near dislocations. Such a mechanism would presumably be dependent on solute concentration and σ_f would be expected to vary with magnesium content. Thus the more reasonable explanations for a constant σ_f with changing magnesium content would seem to be those which postulate a creep dislocation structure that is relatively independent of magnesium content of alloy.

5. Conclusions

1. The stress exponent, n, of steady state creep in Al–Mg alloys at 300° C decreases from a value of about 4.2 for pure aluminium, typical of recovery creep, to a value of about 3 for magnesium contents greater than about 1 at%. Such a value of n is typical of control by a viscous glide process.

2. The stress exponent remains fairly constant for magnesium contents from about 1 to 4 at% but increases with higher magnesium contents. A possible cause for the increase in stress exponent is considered to be precipitation.

3. The magnesium content has little or no effect on either σ_e, the effective stress, or σ_i, the internal stress but both quantities vary with the applied stress, σ.

4. Using data from the present tests plus previously published data empirical equations have been developed relating σ_e and σ_i to σ, namely

$$\sigma_e = 0.165\,\sigma^{1.28}$$

and

$$\sigma_i = 1.084\,\sigma^{0.802}.$$

5. The applied stress dependence of σ_i can be explained by using an equation for σ_i of the form $\sigma_i \propto(\text{dislocation density})^{1/2}$ and previously measured values for the stress dependence of dislocation density for Al–Mg alloys crept at 300° C.

6. Values of the friction stress, σ_f, derived from an equation $\sigma_e/\sigma = (1-c)\ (1-\sigma_f/\sigma)$ indicate that σ_f is virtually independent of magnesium content. A fairly constant σ_f can best be explained by postulating a creep dislocation structure that is relatively independent of the magnesium content of the alloy.

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