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The Measurement of Strain-Rate Sensitivity in Superplastic Alloys

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The currently used methods of determining strain-rate sensitivities, m, of superplastic alloys from strain-rate change tests are examined and found to be unsatisfactory. The stress-relaxation method is also investigated and is shown to be applicable to the superplastic Al-Cu eutectic. However, the m-values derived by this method, and by a variant of the strain-rate change technique, are significantly lower than those obtained using the conventional strain-rate change tests and this is explained in terms of microstructural factors.

1. Introduction

Superplastic alloys are characterised by large neck-free elongation in a tensile test, low flow stress and high strain-rate sensitivity. This mode of plastic deformation normally occurs at low strain-rates ($< 1 \text{sec}^{-1}$), at high homologous temperatures ($T > 0.5\, T_m$), and is associated with fine microstructures. Backofen, Turner and Avery [1] first recognised that superplasticity is characterised by highly strain-rate sensitive deformation and showed that the index of strain-rate sensitivity, m, was related to the maximum elongation ϵ(max) attainable in a tensile test. Subsequently, other workers have reaffirmed that ϵ(max) increases with an increase in m [2-8]. Strain-rate sensitivity is, therefore, a parameter which is of practical significance as well as of fundamental interest.

It is the purpose of this paper to discuss the experimental determination of "m" and to illustrate the procedures (and their limitations) with reference to the superplastic Al-Cu eutectic alloy.

2. Experimental Procedures

The Al-Cu eutectic (33 wt % Cu) alloy used in this work was prepared from high purity aluminium supplied by The British Aluminium Company Research Laboratories (BARL) and high purity copper supplied by Johnson Matthey and Company. The alloy was melted in an argon atmosphere and chill-cast into graphite crucibles. The casts were then hot extruded at $\sim 770°K$ into a 9.5 mm diameter rod at BARL (reduction ratio 50:1) in order to refine the grain size and phase dispersion (Stowell *et al* [9]).

Tensile specimens having 12.7 mm gauge length and 6.35 mm diameter were machined from the extruded rod. Testing was carried out on an Instron testing machine (model TT-C-L) at constant cross-head velocity. A triple-wound furnace was used in which a constant temperature zone ($\pm 2°K$) ~ 130 mm long was maintained. The load cell was effectively thermally protected from the furnace by a thick aluminium plate (through which the shackle passed) and by passing air continuously between this plate and the load cell. This ensured that the initial calibration of the load cell was maintained throughout the duration of the tests.

The results which are discussed in this paper are representative of detailed experiments carried out on about twenty test pieces. The errors noted in the text refer to the particular set of results being described; they make allowance for uncertainties in the measured parameters and, where m-values are averaged over a range of strain-rates, for statistical variations in the individual results. However, the confidence limits do not allow for m-variations with strain or with variations among nominally identical specimens.

3. Theory

Hart [10] has proposed a definition of a strain-hardening parameter, γ, and strain-rate sensi-

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tivity, m, which does not rely on any specific dependence of applied stress on strain or on strain-rate. His phenomenological approach demands that small imposed changes in strain and strain-rate are linearly related to the corresponding (small) change in applied stress; his arguments lead to the relation

$$\mathrm{d}(\ln\sigma) = \gamma\,\mathrm{d}\epsilon + m\mathrm{d}(\ln\dot{\epsilon}) \qquad (1a)$$

where σ is the applied stress, ϵ is natural plastic strain and $\dot{\epsilon}$ is strain-rate. For viscous materials, $\gamma = 0$ so that

$$m = \mathrm{d}(\ln\sigma)/\mathrm{d}(\ln\dot{\epsilon}) \qquad (1b)$$

Hart [10] emphasised that γ and m need not be constant and may be functions of specimen history and of microstructure.

Most workers in the field of superplastic deformation have followed Backofen, Turner and Avery [1] by assuming a stress strain-rate relationship of the form

$$\sigma = \kappa\dot{\epsilon}^{m'} \qquad (2a)$$

where κ is taken to be dependent on temperature and microstructure. Equation 2a does not adequately describe superplastic behaviour and some authors [3, 11, 12] have suggested that a relationship of the form

$$\sigma^* = \sigma - \sigma_i = \kappa^*\dot{\epsilon}^{m*'} \qquad (2b)$$

is more appropriate. Here, σ_i is some "internal" stress below which the plastic strain-rate is zero; σ^* is referred to here as an "effective" stress in analogy with usage of similar expressions in the field of dislocation dynamics. In principle, σ_i can be a function of strain-rate, temperature, grain size, etc. although we shall consider it to be constant in much of the following analysis. Hart's [10] analysis does not consider effective stresses but we may readily modify equation 1a to allow for this parameter, viz.,

$$\mathrm{d}(\ln\sigma^*) = \gamma^*\,\mathrm{d}\epsilon + m^*\mathrm{d}(\ln\dot{\epsilon}) \qquad (1c)$$

Note that m^* of equation 1c is not necessarily equal to $m^{*\prime}$ in equation 2b, just as the m′ of equation 2a need not be the same as the m of equation 1a. Also, all of the equations quoted so far are phenomenological in character.

The most commonly used method of obtaining strain-rate sensitivities in superplastic alloys is the strain-rate change test. This entails, in principle, deforming a specimen at one strain-rate $\dot{\epsilon}_1$ until a steady state flow stress σ_1 is established then changing the strain-rate to $\dot{\epsilon}_2$ and measuring the second steady state flow stress σ_2. Superplastic alloys are assumed to be viscous so, provided κ is constant, $m = \log(\sigma_2/\sigma_1)/\log(\dot{\epsilon}_2/\dot{\epsilon}_1)$. Clearly, this relation attains greater validity the smaller the difference is between $(\dot{\epsilon}_1, \dot{\epsilon}_2)$ and (σ_1, σ_2). Often, it is convenient to carry out several tests on one specimen and to plot $\log\sigma$ against $\log\dot{\epsilon}$; the slope of the experimental curve gives m. This technique is only useful if equation 1a is applicable since only σ and not σ^* is measured.

Another method of obtaining strain-rate sensitivities is the stress-relaxation test [10, 13]. Here the cross-head of a hard tensile machine is arrested and the relaxation of the applied load is measured. The elastic strain in the specimen and machine is compensated by plastic flow in the specimen, so

$$\dot{\sigma}^* = -E\dot{\epsilon} \qquad (3)$$

where E is the effective Young's modulus of the specimen and machine. For materials that are viscous or have a very small value of γ, equation 1c gives

$$\mathrm{d}(\ln\sigma^*) = m^*\mathrm{d}[\ln(-\dot{\sigma}^*)]$$

Therefore, if σ_i and m^* are constant,

$$\frac{\mathrm{d}}{\mathrm{d}\sigma}\left\{\ln(-\dot{\sigma})\right\} = \frac{1}{m^*(\sigma - \sigma_i)} \qquad (4)$$

so that m^* is obtained from a plot of $[\mathrm{d}\{\ln(-\dot{\sigma})\}/\mathrm{d}\sigma]^{-1}$ against σ; σ_i is the intercept on the σ-axis. An equation similar to (4) has been derived on the basis of dislocation dynamics by Kelly and Round [14]. If $\sigma_i = 0$ and m is constant

$$\frac{1}{m} = \frac{\mathrm{d}[\ln(-\dot{\sigma})]}{\mathrm{d}(\ln\sigma)} \qquad (5)$$

If m is not constant, it may be derived from the slope of a plot of $\ln(-\dot{\sigma})$ against $\ln\sigma$.

4. Experimental Results

4.1. Strain-rate Change Tests

Figure 1a represents schematically the form of a typical load-time curve for the superplastic Al-Cu eutectic used in this work. The specimen has been subjected to two changes in cross-head velocity, from $V_1 \rightarrow V_2$ and then from $V_2 \rightarrow V_1 (V_1 < V_2)$. The two most commonly used methods for obtaining m-values from such curves are discussed in the following paragraphs.

4.1.1. Method 1

Backofen, Turner and Avery [1] extrapolated the load-time curve at V_1 to a point B such that the

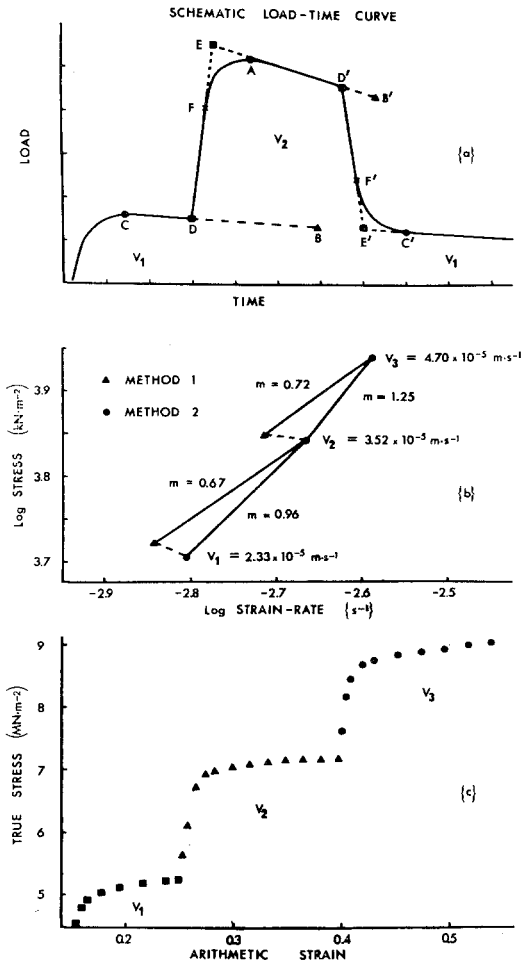

Figure 1 (a) Schematic load-time curve for strain-rate cycling. (b) m-value determination from log stress-log strain-rate data. (c) Typical stress-strain curves encountered in constant velocity change tests.

accumulated plastic strains at B and at A (the position of maximum load at V_2) were equivalent. The strain-rate sensitivity was calculated according to $m = \log(P_A/P_B)/\log(V_2/V_1)$, where P_A, P_B correspond to the loads at A, B, respectively.

4.1.2. Method 2

This entails calculating the stresses corresponding to maximum loads (A and C) at the two velocities, calculating the corresponding true strain-rates, $\dot{\epsilon}_A$ and $\dot{\epsilon}_C$, and evaluating m as $\ln(\sigma_A/\sigma_C)/\ln(\dot{\epsilon}_A/\dot{\epsilon}_C)$, e.g. Naziri *et al* [15] and Morrison [6, 7]. A variant of this method is to measure the maximum loads during a series of incremental velocity changes, to plot log σ against log $\dot{\epsilon}$ and to deduce m from the slope of a smooth curve drawn through the data points.

In fig. 1b, it is shown that these two methods can lead to quite different m-values. Here, a typical set of experimental results for the Al-Cu eutectic tested at $\sim 750°K$ and subjected to two velocity increments yield $m = 0.67 \pm 0.01$ and $m = 0.72 \pm 0.01$ when analysed by method 1, and $m = 0.96 \pm 0.02$ and $m = 1.25 \pm 0.02$ when analysed by method 2. The reasons for these discrepancies become apparent when the load-time data from which these results were obtained is plotted as a stress-strain curve, fig. 1c, and when it is realised that the true strain-rate decreases continuously with time. Even though the *load* attains an approximately constant value, the corresponding flow stress is not constant. Indeed, if the Al-Cu eutectic is tested under constant strain-rate conditions the flow stress increases continuously with time [16] and this is true also for the Zn-Al eutectoid [17].

A further disadvantage of the two methods discussed above is that they both lead to inconsistent results when incremental and decremental velocity changes are made. We have considered only incremental velocity changes so far, but there is no reason to exclude decremental velocity change experiments. When the cross-head velocity is decreased a *minimum* load is not obtained but an approximately constant load, or a region over which the load changes very slowly with time, is achieved, as in fig. 1a. To utilise this information, we choose point C′ as our reference at V_1. The strain-rate sensitivity calculated according to method 1 was derived by using the loads at C′ and B′ in fig. 1a B′ is the point on the extrapolated load-time curve at V_2 corresponding to the accumulated strain at C′) whereas that calculated by method 2 was obtained from the stresses and instantaneous strain-rates at points A and C′. The results of using these two methods in an experiment in which V was first increased and then decreased in six steps are shown in fig. 2a.

It is clear from this graphical representation that inconsistent m-values are obtained. We have already seen in fig. 1b that method 1 yields lower m-values than does method 2 when incremental velocity changes are made; fig. 2a demonstrates that the opposite result obtains when decremental velocity changes are used. The data analysed by method 1 give average values over the strain-rate range used of $m = 0.68 \pm 0.02$ for incremental changes and $m = 0.64 \pm 0.02$ for

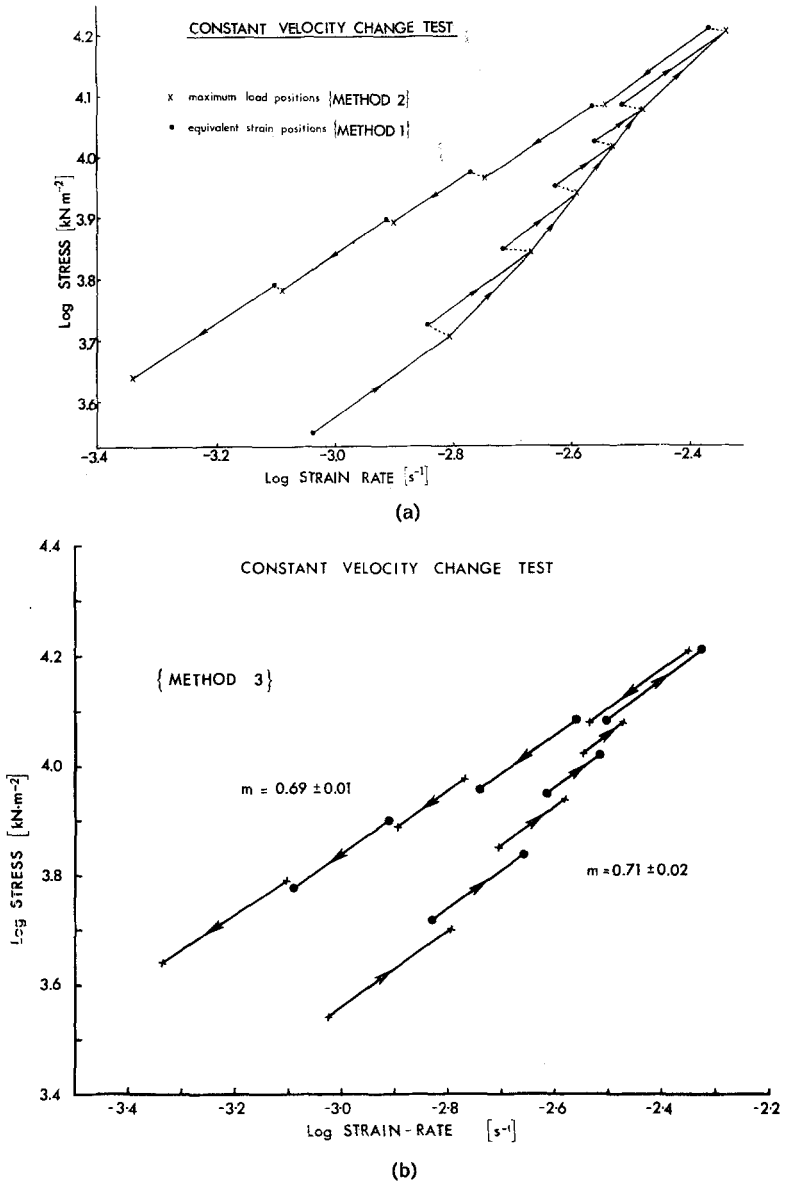

Figure 2 (a) Log stress—log strain-rate plot from velocity change test. Data derived employing methods 1 and 2. (b) Log stress—log strain-rate data derived employing method 3.

decremental changes, which is not too large a discrepancy; however, method 2 clearly yields much greater differences which vary significantly with strain-rate over the range illustrated. This is a very unsatisfactory situation and we now describe a method whereby these inconsistencies may be resolved.

4.1.3. Method 3

The method suggested here entails extrapolating the slowly changing (or approximately constant) part of the load-time curve back to the instant the velocity change was made (E or E′ in fig. 1a). The strain-rate sensitivity is obtained from $m = \log(\sigma_E/\sigma_D)/\log(\dot{\epsilon}_2/\dot{\epsilon}_1)$ or $\log(\sigma_D'/\sigma_E')/\log(\dot{\epsilon}_2/\dot{\epsilon}_1)$. The result of using this method to analyse the same data used to obtain fig. 2a is seen in fig. 2b, where it is illustrated that not only are consistent m-values obtained for both incremental and decremental velocity changes but the apparent variations in m with strain-rate seen in fig. 2a are not real. This method is in effect a variant of

method 1, the difference being that the data are evaluated at the common strain corresponding to the time of the velocity change. There is no *sound* physical reason for it being preferable to method 1, but possible explanations for the slight discrepancies between these two methods will be discussed in section 5. However, we emphasise that method 3 has the advantage of being much simpler to employ in practice.

It is pointed out here that all of the methods discussed so far in this section have doubtful physical validity. It is often admitted that the term κ in equation 2a is somehow dependent on the microstructural state of the specimen, but normally only its dependence on grain size or on phase dispersion is recognised or explored. Workers in the fields of creep and dislocation dynamics are usually careful to be less restrictive in their assumptions and we now show that such care is needed in the analysis of data from superplastic materials.

4.1.4. Method 4

Let us admit the possibility that the flow stress of a superplastic alloy depends upon factors other than grain size, for example on matrix and/or interface dislocations, and that the densities of such "primitive" defects are both strain- and strain-rate-dependent. A change in cross-head velocity will produce a change in these defect densities and, hence in the factor κ in equation 2a or, with greater generality, a contribution from the term $\gamma d\epsilon$ in equation 1a.

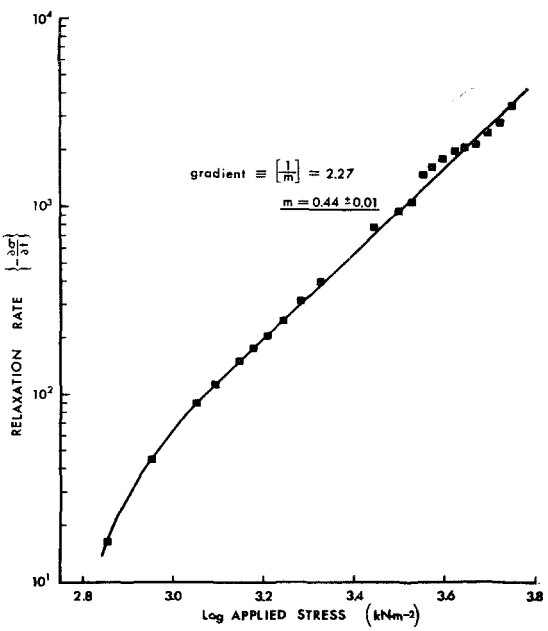

Figure 3 Logarithmic plot of relaxation rate ($-\delta\sigma/\delta t$) against applied stress for an aluminium-copper eutectic alloy.

During a velocity change experiment, the load initially increases or decreases very rapidly as soon as the cross-head velocity is changed, and, thereafter, increases slowly as in fig. 1a. Gibbs [27] has pointed out that the initial stress increment (i.e. DF in fig. 1a) or decrement (D'F') represents the change in load required for the structure at D (or D') to accommodate the changed rate of plastic flow. In this case $m = \log(P_F/P_D)/\log(V_2/V_1)$ or $\log(P_D'/P_F')/\log(V_2/V_1)$ and so is smaller than the m-value derived by methods 1, 2 and 3. The average value of m calculated by this method from the data used to construct figs. 2a and b is 0.42 ± 0.04. This result is markedly different from those determined by the other methods of this section. Discussion of possible reasons for this discrepancy will be delayed until after the results of stress relaxation tests have been detailed.

4.2. Stress-relaxation Tests

It was noted in section 3 that m may be obtained from the slope of a plot of $\log(-d\sigma/dt)$ against $\log\sigma$. Such a plot is shown in fig. 3; for $\sigma > 1.1$ MN. m^{-2} a straight line is obtained, from the slope of which $m = 0.44 \pm 0.01$. The same data are shown in fig. 4 plotted according to equation 4; again a straight line is obtained from which we deduce $m^* = 0.51 \pm 0.01$, $\sigma_i = 300$ kNm^{-2}. In all cases analysed in this manner, σ_i was always small (< 350 kNm^{-2}) although we cannot discount the possibility that, at lower temperatures and higher strain-rates than those employed here, σ_i may be very much greater. Note here that the value of m derived from fig. 3 is significantly lower than the values obtained from this material by the conventional strain-rate change tests, although it is similar to the results deduced in section 4.1.4 from method 4. In order to emphasise the agreement between m-values deduced from stress-relaxation tests and method 4, the results are shown in the following table of two experiments in which a stress-relaxation test was carried out following a velocity change (thus minimising any influence of strain on the m-values). It is seen that the results in the second and third columns exhibit excellent agreement, indicating that they represent the same physical parameter, the significance of which will be discussed in section 5.

Strain-rate cycling (Method 3)	*Stress relaxation*	*Strain-rate cycling Method 4*
0.71	0.48 ± 0.01	0.48 ± 0.05
0.70	0.48 ± 0.01	0.47 ± 0.05

In their analysis of stress-relaxation in a superplastic Ni-Fe-Cr alloy, Hayden and Brophy [13] assumed that equation 2a was valid during the relaxation event. Using this equation in equation 3 and integrating with respect to time, one obtains with $m \equiv m^*$

$$\sigma_0^{(m-1)/m} - \sigma^{(m-1)/m} = \frac{m-1}{m} \cdot \frac{Et}{\kappa} \quad (6a)$$

(provided $m \neq 1$). They assumed $m = 0.5$, plotted $\log [\sigma^{-1} - \sigma_0^{-1}]$ against $\log t$ and obtained a straight line with unit slope over a limited period of time (< 2 sec) from which they concluded that they had proved that $m = 0.5$ for their alloy. We wish to point out that this is not only a very poor way of proving that $m = 0.5$, but that the obtained result is to be expected, independent of the value of m. This may readily be seen analytically by expanding the left-hand side of equation 6a to first order about σ_0 to obtain

$$\ln \sigma + (1 - 1/m) \ln \sigma_0 + \ln(\sigma^{-1} - \sigma_0^{-1}) = \ln[(E\kappa^{-1/m})t] \quad (6b)$$

It is evident that, until σ deviates significantly from σ_0 a plot of $\ln(\sigma^{-1} - \sigma_0^{-1})$ against $\ln t$ will *always* yield a straight line of unit slope *independent of the value of m*. The extent of this region of unit slope is greater the smaller the value of m. The fact that an extensive linear region of unit slope was not obtained by Hayden and Brophy [13] casts considerable doubt on their results. Indeed, we obtained similar behaviour in both our Al-Cu eutectic alloy, for which m derived via strain-rate change tests was ~ 0.7 and in a non-superplastic Al-Si eutectic, for which $m \simeq 0.2$.

5. Discussion

The methods commonly used by workers in the superplasticity field for obtaining m by strain-rate cycling have been compared and found to be unsatisfactory when tests are made on a constant cross-head velocity testing machine. A major difficulty in accurately determining m-values from a constant velocity test arises because the flow stress is a function both of strain and strain-rate (equation 1a or c). In addition, a true steady-state flow condition cannot be obtained. Even though a region of a stress-strain curve can be found for which the true stress is approximately constant, this is not indicative of steady-state conditions because the strain-rate is continuously decreasing (assuming the gauge-length remains uniform). Incremental and decremental velocity change tests yield inconsistent "m"-values when analysed by methods 1 and 2, but the discrepancies may be removed if method 3 is employed. This last method, which is much simpler to employ because it eliminates some tedious arithmetical calculation, does compensate partly for strain hardening at the second velocity, but the physical justification for using it in conjunction with a constant velocity testing machine is somewhat dubious.

The stress-relaxation experiments reported in section 4.2 can be accurately analysed on the basis of assumed plastic flow laws given by equations 2a and b; for $\sigma \leq 1$ MN. m^{-2}, deviations from these laws occurred and possible reasons for this will be discussed later. Under our testing conditions σ_i was small (< 350 kNm^{-2}) and so was the difference between m and m^*. However, the values of m derived from these tests agreed with those obtained from velocity change tests using method 4 (section 1), but were significantly different from the results derived from the other methods of analysing velocity change tests (i.e. ~ 0.45 compared to ~ 0.70). In many cases, both types of test were carried out on the same specimen at approximately the same strain, so that a fundamental difference appears to exist between these two testing techniques.

One significant difference between methods 1, 2 and 3 of analysing velocity change tests and method 4 and the stress-relaxation test is the amount of plastic strain involved during each test. In the former case, engineering strains of several per cent are necessary before a "pseudo steady-state" flow condition is obtained, whereas in the latter tests total plastic strains less than about 10^{-3} are involved. Now, the stress-relaxation test is frequently employed to study dislocation dynamics [14, 18-26] and it is invariably assumed that the dislocation substructure (cell size) and the density of mobile dislocations remain constant during the greater part of a test. However, during creep experiments in which the deformation conditions are changed (e.g. by changing the load or stress), it is recognised that constant structure conditions do not prevail. We propose that similar considerations apply to the tests reported in this paper.

However, by "constant structure" we do not mean just constant grain size; this expression is taken to apply to primitive crystalline defects which are responsible for the deformation process, e.g. matrix and grain boundary dislocations, point defects etc.

As Gibbs [27] has pointed out, the physical significance of method 4 is that the instantaneous stress increment (or decrement) upon changing the cross-head velocity from V_1 to V_2 represents the change in stress necessary to cause the structure formed at V_1 to deform at the new rate V_2. Thus the value of strain-rate sensitivity so derived corresponds closely to that for constant structure conditions. Similarly, since the stress-relaxation test is restricted to very small total plastic strains, we expect constant structure conditions to be more closely approximated than in the conventional strain-rate change test. However, we do not suggest that constant structure conditions are accurately maintained during stress relaxation. The variation of flow stress under constant velocity conditions involves microstructural variations due both to strain and strain-rate changes which, when discussed in terms of the physical processes usually considered in high temperature creep theories, includes both strain-hardening and recovery phenomena. Therefore, we must not exclude the possibility that recovery takes place during stress relaxation. Indeed would be surprising if no recovery occurred and we tentatively attribute the departure from linearity of the data shown in figs. 3 and 4 in part to such a process. There exists another possibility for this behaviour. Many authors [3, 4, 17, 28, 29] have reported a decrease at low strain-rates (or low stresses) in m-values obtained from velocity-change tests and this has been attributed [11, 30-32] to a change in deformation mechanism. We cannot discount this possibility, but, in view of the doubts that must now be expressed concerning the validity of many of the published variations of m with strain-rate (on the basis of our evidence in section 4.1) we can only suggest that some very carefully executed experiments be performed to check on this point. Until this point is resolved, it is recommended that the low stress portion of relaxation curves be disregarded when m-values are determined by this technique.

The term "constant structure" has so far been used without reference to specific deformation mechanisms relevant to superplasticity. Until the many different proposals for defect models of superplastic behaviour have been whittled down to a small number of realistic possibilities, it is pointless to expand at length on the physical significance of this term.

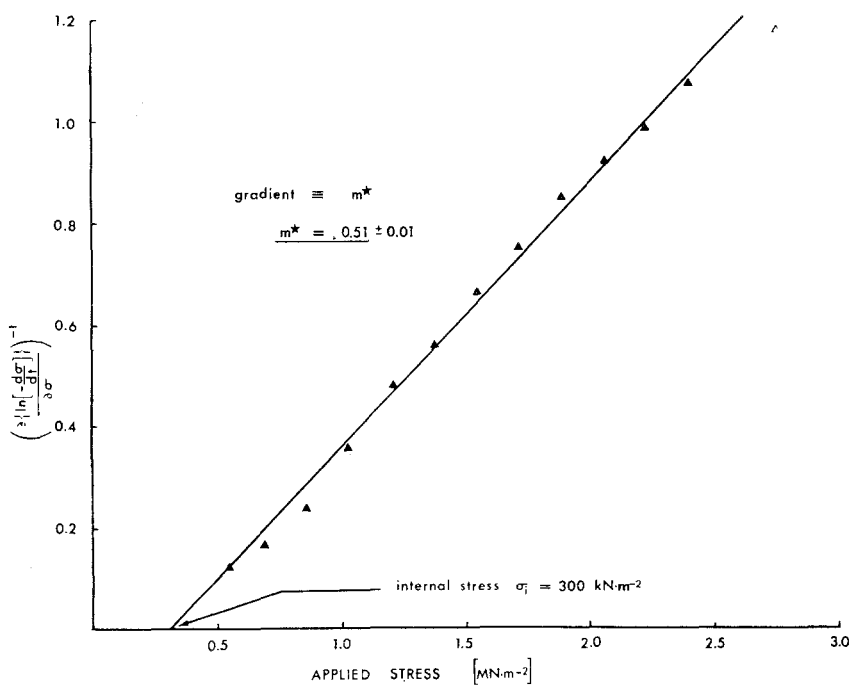

Figure 4 Plot of $[\delta\{\ln - \dot{\sigma}\}/\delta\sigma]^{-1}$ against applied stress.

We must consider now which methods of determining m-values are the most reliable and have the greatest physical significance. The strain-rate change tests, as employed in section 4.1 (methods 1, 2 and 3) and by many other workers in this field, are susceptible to the influence of structural changes which arise from both coarsening of the grain structure and from changes in primitive defect structure (which could be reflected in work hardening). The m-values derived from such tests are therefore not true strain-rate sensitivities as defined by equations 1a and c. However, since this is a well-established form of test, the results of which have been shown to be related to ductility of superplastic alloys [2-8], we recommend that method 3 of section 4.1 be adopted for future use because of its ease of application and since it is equally applicable to both incremental and decremental strain-rate change tests. *We emphasise that the* m-*values so obtained have dubious physical significance.* The stress-relaxation test and the variant of the strain-rate change test (designated method 4 in section 4.1) yield m-values which approximate more closely to those defined via equations 1a and c and should always be used in fundamental studies which seek to correlate mechanical properties with models of the physical mechanism of superplasticity. Also, in order to investigate the possibility of a (σ, ϵ) relationship of the form given by equation 2b, the stress-relaxation test is clearly the most direct and useful of the two recommended methods. We note that by combining all of these techniques one may obtain considerably more insight into the mechanical behaviour of superplastics and derive both m- and γ-values.

We remark that care must be taken to ensure that machine transients are not influencing the data used for obtaining strain-rate sensitivities from stress-relaxation tests. Some discussion on this point has already been made (Guiu and Pratt [33], Guiu [34]). In the work reported here, the data recorded during the first few (<2) sec of a stress-relaxation curve have not been given much weight for the following reasons. Firstly, they are in error because of the relatively slow response of the recording system and also because of the finite time taken to arrest the cross-head (~ 1.2 sec at 0.04 mm sec^{-1}). Secondly, during calibration of the instrument, it was noted that, at the highest cross-head speeds available (which, fortunately were not used in the detailed experimental study reported in this paper) a large reverse motion of the cross-head occurred, i.e. a negative strain was imposed. This negative displacement was measurable on all but the lowest velocities and undoubtedly influenced the stress-relaxation process during the initial stages. This adds further weight to our comment that, even if $m = 0.5$, the method of analysis used by Hayden and Brophy [13] is inferior to the method illustrated in section 4.2, since it depends strongly upon the value of σ_0. If a small negative elastic strain is imposed on the system at $t = 0$, the true σ_0 is *not* the last recorded during the straining period prior to relaxing the stress. However, the method of analysis illustrated by fig. 3 is independent of σ_0 and of any negative imposed elastic strain, provided this is small compared to the total elastic strain in the specimen at $t = 0$.

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