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Method to determine the strain-rate sensitivity of a superplastic material from the initial slopes of its stress–strain curves

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New method to estimate quantitatively the strain-rate sensitivity of a superplastic material is suggested. It is based upon measurements of the initial slopes of the stress–strain curves. In contrast with known techniques the method suggested is appropriate to apply to small deformations, so that the result obtained are to be assigned to the initial structural state of the material under consideration. The experimental verification of the method suggested is fulfilled in practice for the example of Wood's alloy. It is shown that the strain-rate sensitivity of this alloy decreases monotonically with increasing strain, ε; the value of m determined by means of the method suggested are in good agreement with that determined independently using standard techniques. An important feature of the method suggested is that in contrast with standard techniques its accuracy improves as the value of strain, ε, becomes smaller, since the tilts of σ–ε curves have a maximum when $\varepsilon \to 0$.

1. Introduction

The main feature of the rheological behaviour of a superplastic material is believed to be the anomalous high sensitivity of the flow stress, σ, to the strain rate, ξ [1–5]. In order to characterize this property numerically, one can introduce the so-called *strain-rate sensitivity index*, m:

$$\sigma = K\xi^m \tag{1}$$

where K is a constant of the material. The value of m does not usually exceed 0.1 for most known materials. However, for the materials in a superplastic state the value of m commonly lies in the range 0.3–0.9 and may achieve approximately 1 [3], which corresponds to Newtonian viscous flow. It should be noted that the boundary between superplastic and non-superplastic states for a given material at a given temperature is usually determined experimentally on the basis of an empirical condition $m \geqslant 0.3$. Thus the high strain-rate sensitivity is the most essential criterion of a superplasticity; therefore the development of special methods intended to evaluate this feature numerically is of interest.

The simplest method to determine the value of m is to fulfil a number of constant cross-head velocity tests during which the time dependence of the axial force, P, is recorded for specimens having the same initial structure. Then the standard σ–ε (stress–strain) curves are plotted using experimental P–t diagrams. Subsequently the values of σ corresponding to the same strain, $\varepsilon = \varepsilon_0$, and different strain rates, ξ, are plotted as the logarithmic coordinates of stress, σ, against the strain rate, ξ. Finally the value of m is determined as the slope of the corresponding $\log\sigma$–$\log\xi$ straight line. The main disadvantage of this method is the necessity to use a large number of specimens (at least five specimens are required for the same temperature). It is a highly time-consuming procedure, especially for high-temperature tests. On the other hand, in spite of the large number of specimens, there is considerable scattering in the values of m measured by this method.

There are some other methods known in the literature that are used to determine the value of m [1]. In practice, the value of m is usually determined during a step strain-rate test. In this case the strain rate is increased in successive steps and an attempt is made to measure the corresponding steady (or saturated) flow stress. Different variants of this method are distinguished by the way the results obtained are treated. Common assumptions for all of them are that the testing machine is absolutely rigid and the change in the cross-head velocity takes place almost instantly. It should be emphasized that these factors may, in principle, considerably distort the experimental results [6]; however, in the literature on superplasticity their influence is not usually considered. On the other hand, even if the above-mentioned assumptions are sufficiently warranted, the deformation-induced changes in the material's structure may considerably distort the experimental results as well. In particular, it is well known [7–9] that superplastic deformation is often accompanied by grain growth, the rate of which depends upon both strain and strain rate, and is usually well in excess of that found in the absence of deformation.

It is evident that, from the mechanical point of view, one can consider the influence of the deformation-induced structure changes (at least, as a first

approximation) as the influence of the strain, ε, on the flow stress, σ, and, as a consequence, on the value of m. A load relaxation test is one effective way to diminish the influence of strain. The method to determine the value of m during a load relaxation test has been suggested recently in [10]. However, as shown in [11], Bingham-type constitutive relations are not always simultaneously applicable for both the active and the passive loading conditions. Since Equation 1 is a Bingham-type constitutive relation, the value of m determined from the relaxation curve will not always coincide with that determined using the results of experiments with the active loading, e.g., constant cross-head velocity tests. Also, it is evident that the structural state of the material at the very beginning of the load relaxation test, $t = t_0$ (immediately after cross-head arrest), will depend upon the deformation history, i.e., on the time dependences of strain ($\varepsilon(\tau)$, $0 \leqslant \tau \leqslant t_0$) during the preliminary active deformation. Therefore the average grain size of every specific specimen will depend upon its particular deformation history, $\varepsilon(\tau)$, $0 \leqslant \tau \leqslant t_0$, since all specimens have the same structure with average grain size d_0 at the initial moment of time, $t = t_0$.

In order to reduce the influence of the deformation-induced structure changes to a minimum, it is reasonable to determine the value of m for an initial structural state. This may be done at the early stages of plastic deformation, i.e., at small ε, when the influence of the structure changes is minimal. In this connection it is interesting to determine the value of m from the initial parts of the σ–ε curves. In the present paper the method to evaluate numerically the strain-rate sensitivity of a superplastic material at the initial state based upon measurements of the initial slopes of the σ–ε curves is suggested.

2. Fundamentals

According to the standard method, one can calculate the value of m in the following way:

$$m|_{\varepsilon=\varepsilon_0} = \frac{\ln(\sigma_1/\sigma_2)}{\ln(\xi_1/\xi_2)} \tag{2}$$

where the values of stress, σ_1 and σ_2, correspond to the same strain, $\varepsilon = \varepsilon_0$, and the strain rates, ξ_1 and ξ_2, respectively (Fig. 1). Thus, m_{40}, for example, denotes the value of m calculated for $\varepsilon = 40\%$. If there are $N > 2$ experimental pairs, σ_i, ξ_i ($i = 1, 2, \ldots, N$), available at $\varepsilon = \varepsilon_0$, one can determine the value of m as the slope of the straight line in logarithmic coordinates, $\log\sigma$–$\log\xi$. Unfortunately, as already mentioned above, the deformation-induced structure changes take place in the material so that the values of $\sigma_1(\xi_1)$ and $\sigma_2(\xi_2)$ will correspond, in general, to different values of average grain sizes, d_1 and d_2, respectively. Step strain-rate tests allow us to bring together the material structure states that are correlated in calculating the value of m. However, in this case there

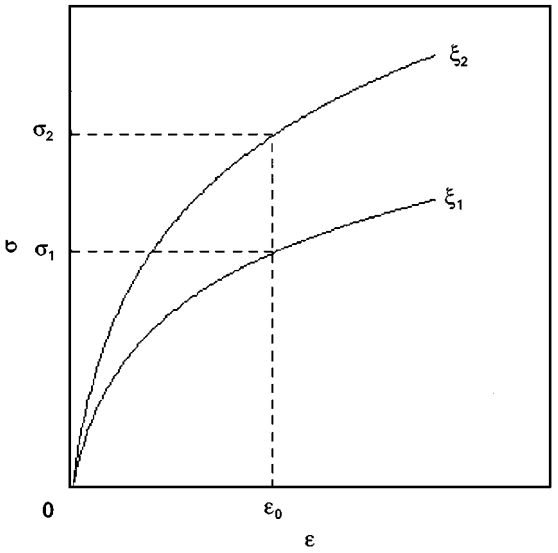

Figure 1 Standard procedure to determine the value of strain-rate sensitivity index, m.

is not the unique point of view in the literature concerning the way to treat the experimental time dependences of the axial force, P [1].

In order to exclude the influence of the deformation-induced structure changes it is attractive to use the initial parts of the σ–ε curves. Unfortunately, the standard methods to calculate the value of m, e.g., according to Equation 2, are not applicable in this case, since in any event $P \to 0$ when $t \to 0$ (i.e., $\sigma \to 0$ when $\varepsilon \to 0$). Thus, Equation 2 leads to the 0/0 type uncertainties when $\varepsilon \to 0$. Also, as a rule, an initial part of any actual experimental P–t diagram is usually distorted owing to the influence of various imperfections of the testing machine, such as free plays, clearances and limited rigidity. However, it turns out that it is possible to determine appropriately the initial tilt of an experimental P–t diagram, since one can easily exclude the effects of the above-mentioned factors. Let us now consider the possibility of determining the value of m using the information concerning the tilts of the initial parts of the experimental P–t diagrams.

It is known that an elastic deformation of metals and alloys rarely exceeds 0.1%. Let $L = 10$ mm be the characteristic length of the specimen, using experimentally to determine the value of m. In this case, 0.1% of its length will be equal to 0.01 mm, i.e., beyond the measurement's accuracy. Therefore we shall further assume that elastic deformation is negligible in comparison with plastic strains. Consequently, the initial tilt of the experimental P–t diagram (and, correspondingly, the σ–ε curve) will not be determined by the elastic properties of the specimen; in particular, the initial slope of the σ–ε curve will not be determined by Young's modulus for the tested material. Also, we shall assume further that the testing machine is absolutely rigid*. Thus, the initial tilt of the experimental

* One can take into consideration the influence of the limited rigidity of the testing machine in using the following standard procedure; it is necessary to load "absolutely rigid" (in comparison with standard specimen) solid body and to record the resulting $(P$–$t)_s$ curve. This resulting $(P$–$t)_s$ diagram should be subtracted from all experimental P–t diagrams before their further treatment.

P–t diagram is fully determined by the plastic properties of the material under consideration.

The following constitutive relation is often introduced in the literature in order to take into consideration the strain dependence of the flow stress, σ [4, 12]:

$$\sigma = A\varepsilon^n \xi^m \tag{3}$$

where A is a temperature-dependent constant of the material. Equation 3 is widely used in engineering practice of metal working in approximating σ–ε curves for commercial metals and alloys. It is known [13] that Equation 3 provides reasonable accuracy in the field of well-developed plastic flow. For the initial stage of plastic deformation, Equation 3 seems to be not applicable. Therefore let us consider the following generalization of the Equation 3:

$$\sigma = f_1(\varepsilon) f_2(\xi) \tag{4}$$

where f_1, and f_2 are arbitrary functions.

Elementary analysis of Equation 4 shows that a set of curves σ–ε, plotted according to Equation 4 at different strain rates $\xi = \xi_i = \text{constant}$ ($i = 1, 2, \ldots, N$) will represent a number of similar curves, the coefficients of similarity being equal to the corresponding meanings of the function f_2: $K_i = f_2(\xi_i)$ ($i = 1, 2, \ldots, N$). Thus, for a material having a constitutive relation corresponding to Equation 4, one can find the functional form f_1 by approximating the σ–ε curve, plotted at a constant strain rate $\xi = \text{constant}$. At the same time the functional form f_2 may be found by approximating the dependence of the above-mentioned coefficients of similarity, K, on the strain rate, ξ.

Substituting $f_2(\xi) = \xi^m$ into Equation 4, one can obtain after differentiation with respect to ε the following relationship:

$$\sigma_\varepsilon = \frac{df_1}{d\varepsilon} \xi^m \tag{5}$$

Here σ_ε denotes the full derivative of σ with respect to ε: $\sigma_\varepsilon = d\sigma/d\varepsilon$. Hence it follows that

$$m = \frac{\ln(\sigma_\varepsilon^1/\sigma_\varepsilon^2)}{\ln(\xi_1/\xi_2)} \tag{6}$$

According to Equation 6, one can calculate the value of m at arbitrary ε, including $\varepsilon \to 0$. It is appropriate to apply Equation 6 for small ε, because in this case the tilt of σ–ε curves has a maximum value.

It should be noted that standard Equation 2 remains valid (at $\varepsilon \neq 0$) for materials, the constitutive relation of which has the form of Equation 4. At the same time, in contrast with Equation 6, the standard Equation 2 is best suited to sufficiently large ε, when the values $\sigma_1(\xi_1)$ and $\sigma_2(\xi_2)$ entering into Equation 2 are sufficiently large in magnitude.

3. Experimental procedure

The method suggested was realized in practice for Wood's alloy (B_i–25 wt % Pb–12.5 wt % Sn–12.5 wt % Cd). Extrusion of rods of diameter 8 mm from the casting alloy of size 25 mm × 35 mm allowed us to prepare fine-grained microstructure needed to transfer the material under consideration into a superplastic state. Specimens of diameter 8 mm and length 12 mm had concentric grooves on their contact surfaces intended to keep the lubricant (plumbago with oil). Compression tests were carried out at room temperature and constant cross-head velocities in the range 0.01–1.0 mm min^{-1}. Load relaxation curves have been recorded for every specimen after the cross-head arrest.

Fig. 2 shows σ–ε dependences for the material under consideration calculated by means of the standard procedure. The numbers near the curves indicate the initial value of the strain rate (the ratio of the cross-head velocity to the initial height of the specimen). The initial parts of these curves were treated as follows. The linear portion of the P–t curve is detected on the experimental P–t diagram; then one should measure the slope of this straight line and finally it is necessary to recalculate it into the slope of the corresponding part of σ–ε curve. The results obtained are represented in Table I. One can find the value of m, e.g., using the first two couples of the experimental data represented in Table I:

$$m = \frac{\ln(408/282)}{\ln(2.27 \times 10^{-4}/1.28 \times 10^{-4})} = 0.64$$

In order to calculate the value of m using all the experimental data contained in the Table I, let us

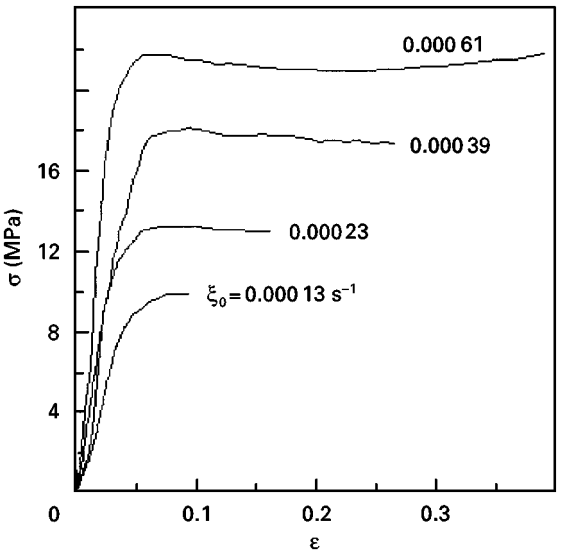

Figure 2 Stress–strain curves for Wood's alloy at room temperature and different strain rates (122 days after extrusion).

TABLE I Calculated values of the initial tilt of σ–ε curves at different strain rates, ξ, for Wood's alloy at room temperature

Strain rate, ξ (s^{-1})	Initial tilt of the σ–ε curve, $\sigma_\varepsilon = d\sigma/dt$ (MPa)
1.28×10^{-4}	282
2.27×10^{-4}	408
2.61×10^{-4}	368
4.86×10^{-4}	582
6.11×10^{-4}	773

TABLE II The values of m, calculated according to Equation 9 using the data in Table I (k denotes the number of the pair σ_ε^k, ξ_k ($k = 1, 2, \ldots, 5$) used as the reference point σ_ε^0, ξ_0

k	m
1	0.58
2	0.58
3	0.60
4	0.59
5	0.62

rewrite Equation 5 as follows:

$$\frac{\sigma_\varepsilon}{\sigma_\varepsilon^0} = \left(\frac{\xi}{\xi_0}\right)^m \tag{7}$$

where σ_ε^0 and ξ_0 are some characteristic values of corresponding parameters (we shall further designate this couple as the *reference point*).

In accordance with standard method of least squares, one can consider the following goal function:

$$\Phi(m) = \sum_{i=1}^{N}\left[\ln\left(\frac{\sigma_\varepsilon^{(i)}}{\sigma_\varepsilon^0}\right) - m\ln\left(\frac{\xi^{(i)}}{\xi_0}\right)\right]^2 \to \min \tag{8}$$

where $\sigma_\varepsilon^{(i)}$, $\xi^{(i)}$ ($i = 1, 2, \ldots, N$) are the experimental data (see Table I). Then one can find from the standard condition $d\Phi/dm = 0$ the following expression:

$$m = \left[\sum_{i=1}^{N}\ln\left(\frac{\sigma_\varepsilon^{(i)}}{\sigma_\varepsilon^0}\right)\ln\left(\frac{\xi^{(i)}}{\xi_0}\right)\right] \Big/ \sum_{i=1}^{N}\left[\ln\left(\frac{\xi^{(i)}}{\xi_0}\right)\right]^2 \tag{9}$$

It should be noted that Equation 9 remains valid if one substitutes in it the values of σ_i instead of the corresponding strain derivatives, σ_ε. Indeed, it is easy to see that one can rewrite Equation 1 as follows:

$$\frac{\sigma}{\sigma_0} = \left(\frac{\xi}{\xi_0}\right)^m \tag{10}$$

where the new reference point includes σ_0, ξ_0 which are the characteristic values of the stress and strain rate, respectively. Then the following expression for m may be found in a similar way:

$$m = \left[\sum_{i=1}^{N}\ln\left(\frac{\sigma_i}{\sigma_0}\right)\ln\left(\frac{\xi_i}{\xi_0}\right)\right] \Big/ \sum_{i=1}^{N}\left[\ln\left(\frac{\xi_i}{\xi_0}\right)\right]^2 \tag{11}$$

The results obtained in treating the data contained in Table I are incorporated into Table II.

4. Discussion

As Table II suggests, the value of m for Wood's alloy at room temperature turned out to be equal to 0.60 ± 0.02 for the initial structural state. In order to verify the applicability of the method suggested, it is necessary to compare the results obtained with those found by known methods.

Earlier it was shown [10], that the values of m, calculated for the Wood's alloy using load relaxation curves, are in a good agreement with that determined by standard procedure. That is why the values of m were determined in the present work by means of relaxation tests as well. With that in view let us write the theoretical time dependence of the stress during load relaxation test as follows [10]:

$$\sigma(t) = \frac{\sigma_0}{[1 + (t - t_0)/\tau]^p} \qquad t \geqslant t_0 \tag{12}$$

where σ_0 is the value of stress at the very beginning of the relaxation test ($t = t_0$); $p = m/(1 - m)$ and τ are parameters of the material. The values of p and τ were found by non-linear regression methods. To do this all relaxation curves were represented as σ_i, t_i ($i = 1, 2, \ldots, N_{\text{exp}}$), where N_{exp} is the number of points measured on the experimental plot; the following goal function was chosen in order to determine the values of parameters p and τ:

$$\Phi(p, \tau) = \sum_{i=1}^{N}\left[1\frac{+ t_i}{\tau} - \left(\frac{\sigma_0}{\sigma_i}\right)^p\right]^2 \to \min \tag{13}$$

The results obtained are given in Table III. The scattering turned out to be sufficiently small that the theoretical curves almost coincide with corresponding experimental data for all tested specimens.

As one can see in Table III, the value of m turned out to be approximately equal to 0.40 ± 0.02 (if one omits the last point). It is smaller than that determined from the initial slopes of σ–ε curves by a factor of 1.5 (according to Table II, $m = 0.60 \pm 0.02$). Since the values of m for Wood's alloy in analogous experimental circumstances are approximately the same at the active and passive loading conditions [10], this discrepancy may account for the deformation-induced structure changes occurring in the material during active deformation at $0 < t < t_0$. In order to verify this hypothesis, estimations of the value of m using standard procedure were carried out. For this purpose the couples σ_i, ξ_i ($i = 1, 2, \ldots, K$) were found on the curves σ–ε for a given value of $\varepsilon = \varepsilon_0$. The values of m were calculated according to Equation 11. The results obtained are given in Table IV. The couples σ_i, ξ_i ($i = 1, 2, \ldots, K$) available for a given $\varepsilon = \varepsilon_0$ have been chosen one after another as the reference point σ_0, ξ_0. The value of m was accepted as equal to the arithmetic mean while the scattering, Δm, was evaluated according to conventional procedure as the square root of the dispersion, D: $\Delta m = D^{1/2}$.

One can see that the value of m listed in Table II (about 0.6) is different from that reported for small strains in Table IV (about 0.51). In general, this discrepancy may account for the low accuracy of measurements of σ_i, ξ_i at small ε. In particular, it is pertinent to note that the initial part of the σ–ε diagram may be distorted owing to the influence of the specimen's concentric grooves and/or that of the imperfections of the testing machine. On the other hand, some disagreement between the values of m measured by means of different procedures is not surprising (see, e.g., [10]); it would be strange if this were not the case.

The data presented in Table IV show certainly that the m value of the alloy under consideration decreases with increasing strain. Therefore the above-mentioned discrepancy between m values, listed in Table II (about 0.6) and those listed in Table III (about 0.4) in

TABLE III The results of treatment of the relaxation curves for Wood's alloy ($\sigma_0 = \sigma(t_0)$ and $H_k = H(t_0)$ are the values of the stress and height of the specimen, respectively, at the very beginning of the load relaxation test (cross-head is arrested at $t = t_0$); H_0 is the initial height of the specimen (at $t = 0$); v = constant is the cross-head velocity at $0 < t < t_0$; dispersion was calculated by means of the standard procedure)

σ_0 (MPa)	N_{exp}	$\varepsilon = 1 - H_k/H_0$	m	τ (s)	$\xi_0 = v/H_k$ (s^{-1})	Dispersion
14.8	32	0.064	0.38	5.50	2.79×10^{-4}	7.4×10^{-4}
17.1	27	0.289	0.39	3.66	5.48×10^{-4}	1.3×10^{-4}
10.2	22	0.085	0.39	9.98	1.40×10^{-4}	5.2×10^{-4}
20.0	27	0.316	0.41	4.03	7.10×10^{-4}	3.1×10^{-4}
21.0	29	0.409	0.41	3.66	10.70×10^{-4}	5.4×10^{-4}
13.2	19	0.160	0.40	5.50	2.70×10^{-4}	0.82×10^{-4}
21.1	24	0.401	0.45	5.50	10.2×10^{-4}	10.2×10^{-4}

TABLE IV Strain dependence of m for Wood's alloy at room temperature. (K is the number of couples σ_i, ξ_i ($i = 1, 2, \ldots, K$) used to calculate m according to Equation 11)

ε	K	m	Δm
0.05	7	0.51	0.01
0.10	6	0.51	0.01
0.15	5	0.48	0.01
0.20	4	0.37	0.02
0.25	4	0.36	0.02

fact may account for the deformation-induced changes in the material's structure. At the same time it is often argued in the literature on superplasticity that the main micromechanism of superplastic deformation (grain-boundary sliding) is developing gradually in the early stages of plastic deformation. Within the framework of this hypothesis the m value should increase at small strains. The tendency for a decrease in m observed for Wood's alloy (see Table IV) contradicts this concept. It is of interest to find out the reasons for this disagreement; however, such discussion are not within the scope of the present paper. It is evident that further investigations should be made in order to elucidate this problem.

5. Summary

The method to estimate the value of the strain rate sensitivity index, m, of a superplastic material based upon measurements of the initial tilts of σ–ε diagrams, is proposed in this paper. As distinct from the known techniques, the method suggested allows us to exclude the influence of the deformation-induced structure changes and thus to assign the measured values of m directly to the initial structural state of the material under consideration.

The applicability of the method suggested is demonstrated for Wood's alloy (Bi–25 wt %, Pb–12.5 wt %, Sn–12.5 wt % Cd). It is shown that the strain rate sensitivity of this alloy decreases monotonically with increasing strain, ε; the values of m determined by means of the method suggested are in good agreement with that determined independently using standard techniques.

An important feature of the method suggested is that in contrast with standard techniques its accuracy improves as the value of strain, ε, becomes smaller, since the tilts of σ–ε curves have a maximum when $\varepsilon \to 0$.

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