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Constant Stress Creep and Constant True Strain-Rate Tensile Tests of the Superplastic Alloy PbSn

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Strain-time curves and stress-strain curves have been obtained for the superplastically deformed PbSn eutectic tested in creep under constant stress and in tension under true strain-rate conditions at temperatures ranging between −44 and 30°C. It is shown that the flow stress does not depend on strain and time and is only a function of the true strain rate, of the temperature and of the initial grain size. The result is that this superplastic alloy does not strain-harden and that the grain size is constant. The apparent activation energy does not depend on stress and temperature and is equal to 11.5 ± 0.5 kcal/mole.

1. Introduction

Some alloys with very fine and stable equiaxed grains of about a micron in size exhibit a superplastic behaviour characterised by elongations up to 2000% at rupture [1]. As long as the deformation remains uniform, the stress σ depends on the true strain ϵ, the true strain-rate $\dot{\epsilon}$, the test temperature T and the average size of the alloy grains $\bar{L}$:

$$\sigma = f(\epsilon, \dot{\epsilon}, T, \bar{L}) \quad (1)$$

In the differential form this relation can be represented by [2]:

$$\mathrm{d}\ln\sigma = \gamma \mathrm{d}\epsilon + m\,\mathrm{d}\ln\dot{\epsilon} + m\frac{Q_a}{R}\,\mathrm{d}\left(\frac{1}{T}\right) + \alpha(\mathrm{d}\bar{L}_i + \mathrm{d}\bar{L}) \quad (2)$$

The following coefficients γ, m, Q_a and α generally depend on the value of ϵ, $\dot{\epsilon}$, T and $\bar{L}$. The coefficient γ is positive when the alloy strain-hardens or equal to zero when it does not. The strain-rate sensitivity coefficient m ranges from 0 to 1 for all materials [3]. Q_a is the apparent activation energy. The coefficient α is positive because in superplastic materials the stress increases with the grain size [4].

During an elongation, $\mathrm{d}\epsilon$ is positive, $\mathrm{d}\ln\dot{\epsilon}$ can be either positive or negative or equal to zero. $\mathrm{d}\bar{L}_i$ is the difference between the average grain sizes of the different specimens studied before elongation and $\mathrm{d}\bar{L}$ is the variation of the grain size during the deformation process; in the superplastic range $\mathrm{d}\bar{L}$ is either positive [5] or equal to zero.

It is important to know the values of the coefficients in equation 2 and to know if the size of the grains remains constant or if it varies during the elongation in order to study the model of superplastic deformation from a theoretical point of view.

Numerous tensile tests at constant cross-head velocity [6-10] have been carried out with the famous superplastic alloy, the eutectic alloy PbSn, but it is difficult to use these results because the velocity v does not directly appear in equations 1 and 2. It is possible to study the coefficient m using the velocity change tests [10-11] but the subsequent extrapolation remains open to discussion [12]. The first tensile tests at constant true strain-rate have been recently carried out with this alloy by Newbury and Joy [13]; their study is very interesting because $\dot{\epsilon}$ appears directly in the previously mentioned relations.

The aim of the present work is to study the behaviour of this alloy in constant stress creep tests because the parameter σ also appears directly in equations 1 and 2. The results will then be confirmed mainly by tensile tests at constant true strain-rate and finally compared to previously published papers.

TABLE I Creep tests at constant true stress

Tests	A						B				C
True stress kg/cm^2	71	86	142	186	284	310	425	425	425	425	306
Temperature °C	25	25	25	25	25	25	-44	-30	-16	0	-30
True strain-rate ($\times 10^2$) mn^{-1} (experimental)	0.72	1.1	3.3	6.2	19.4	27.5	0.33	1.33	3.56	16.1	0.31

Tests	C			D			E			F	
True stress kg/cm^2	306	306	306	200	200	200	129	129	129	82.5	82.5
Temperature °C	-16	0	19	-16	0	19	-16	0	19	0	19
True strain-rate ($\times 10^2$) mn^{-1} experimental)	1.35	4.54	12	0.52	1.7	6.2	0.2	0.8	2.78	0.3	1.2

2. Experimental

The eutectic alloy PbSn was prepared from 99.999% pure lead and tin and cast in a mould 20 mm diameter. The as-cast ingot is homogenised for two days at 130°C. It was extruded into rods of 3 mm diameter at 20°C. Grains of about the size of a micron develop during an anneal of about 15 min at 130°C. Specimens of 25 or 50 mm in effective length are cut from the rods. Then they are maintained at 25°C for one day to allow them to be glued to the grips.

A creep machine was built [14, 15] which permits us to apply constant stress, with an accuracy of ± 1%, up to values of the true strain equal to 120% (engineering strain $e = 230\%$) when the deformation is uniform. The specimens are maintained at constant temperatures within a variation of ± 0.5% in a thermostatically controlled liquid bath.

3. Results and Discussion

The tests marked with the same letter A, B, C . . . were made with specimens from the same rod and we assume that these specimens have the same initial grain size $\bar{L}_i$. Only the tests where the elongation is uniform are kept, in which case, the diameter all along the specimen remains constant within ± 2%. If this is not observed, it can lead to wrong results [9]. Table I summarises the values of the stress and the temperature T for each test.

The behaviour of the strain ϵ as a function of the time t during each creep test at constant stress are plotted in fig. 1. A steady state is observed after a strain of about 20% and during this state, the true strain ϵ is linearly related to time and the true strain-rate $\dot{\epsilon}$ remains constant (see table).

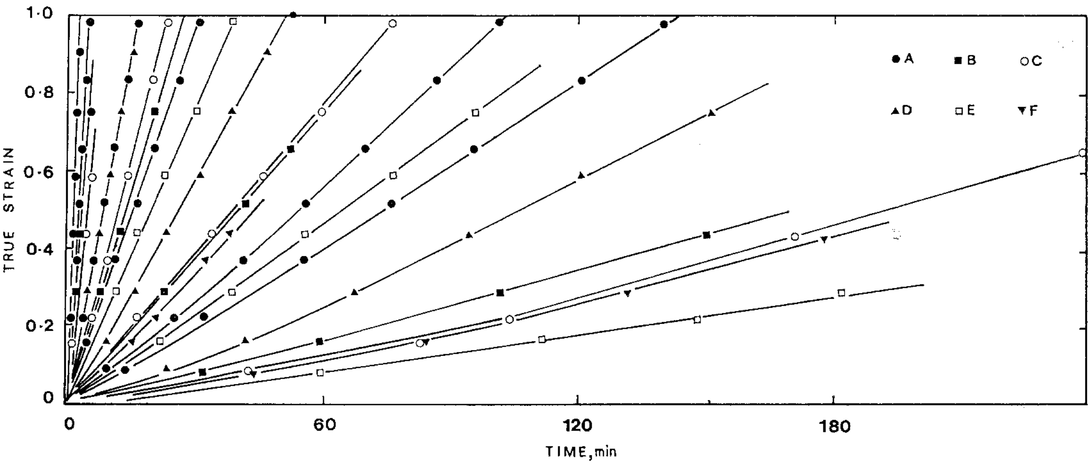


Figure 1 The true strain-time behaviour of the super-plastically deformed PbSn eutectic tested in creep under constant stress. The experimental conditions are shown on the table.

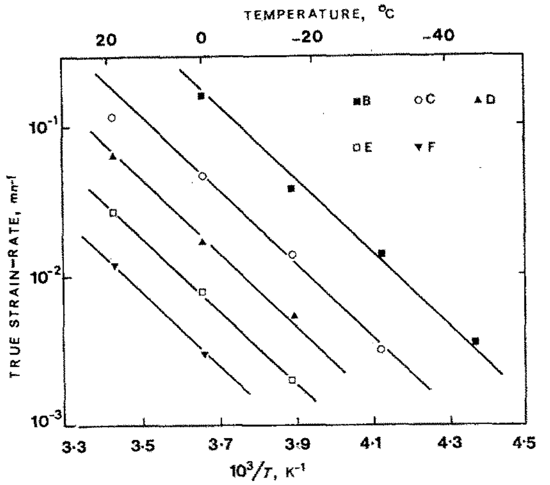


Figure 2 Variation curves of true strain-rate against temperature at identical values of stress.

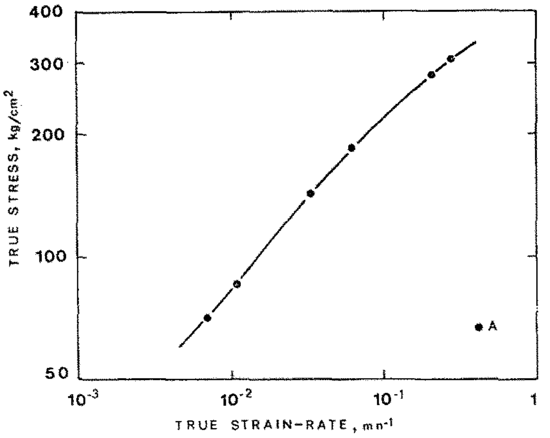


Figure 3 Variation curve of true stress against true strain-rate at 25° C.

For each creep test at constant stress, equation 2 is now represented by this equation:

$$0 = \gamma \mathrm{d}\epsilon + \sigma \mathrm{d}\bar{L}$$

The two terms are of the same sign, consequently the strain-hardening coefficient γ must be equal to zero and the grain size must remain constant during the creep test at temperatures lower than 25°C. The stress σ is only a function of the true strain rate of the temperature and of the initial grain size of this alloy. In this case equation 2 becomes:

$$\mathrm{d}\ln\sigma = m\mathrm{d}\ln\dot{\epsilon} + m\frac{Q_a}{R}\mathrm{d}\left(\frac{1}{T}\right) + a\mathrm{d}\bar{L}_1 \qquad (3)$$

For the same true stress and initial grain size (see table) equation 3 can be represented by:

$$\mathrm{d}\ln\dot{\epsilon} + \frac{Q_a}{R}\mathrm{d}\left(\frac{1}{T}\right) = 0 \qquad (4)$$

The curves for the true strain-rate $\dot{\epsilon}$ versus the temperature at identical values of the stress are plotted in fig. 2. The variation of the true strain-rate against the temperature is effectively an Arrhenius function as it is predicted by equation 4. The apparent activation energy in the superplastic range is independent of the stress and the temperature. The value of this energy is equal to 11.5 ± 0.5 kcal/mole (0.50 eV/atom). This value was confirmed by temperature changes during constant creep tests. It agrees perfectly with the results obtained by constant cross-head velocity tensile tests carried out in the superplastic range at higher temperatures between 0 and 80°C [7]. This value seems to be a feature of the deformation of this superplastic alloy. The self-diffusion energy of the vacancies along the tin grain-boundaries seems to control this deformation [10]: the same energy is obtained with the superplastic alloy Sn 5% Bi [16] where the bismuth precipitates do not play any part in the deformation process and this value is approximately equal to 9.6 kcal/mole. This last value was found for the self-diffusion energy of the vacancies along the tin grain-boundaries [17].

The curve of the stress versus the true strain rate at 25°C for the same series of specimens is plotted in fig. 3. The values of the coefficient m can be obtained from the slope of this curve, They agree with the published results. As in the case of the stress, this coefficient depends neither on the strain nor the time.

These results do not agree with those obtained by Newbury and Joy [13]. They observed that the stress increases by approximately 20% for an engineering strain of 100% and have explained this increase by the grain growth during the deformation process.

Our results were confirmed by tensile tests at constant cross-head velocity and especially by tensile tests at constant true strain-rate similar to those already made [13].

During tensile tests at constant cross-head velocity, the true strain rate $\dot{\epsilon}$ can be represented by the following equations:

$$\dot{\epsilon} = \frac{v}{l_0 + vt}$$

and

$$\dot{\epsilon} = \frac{v}{l_0} \exp(-\epsilon)$$

l_0 is the initial length of the specimen.

If the ratio v/l_0 varies, the same true strain-rate $\dot{\epsilon}$ can be obtained for different strains and for different times. These experiments show that the stress σ is independent of strain and time. These results confirm the creep tests and agree with those obtained by Cline and Alden [7] with the same alloy tested in the same conditions.

The tensile tests at constant true strain-rate were made on an Instron tensile testing machine and the motion of its cross-head was electronically controlled (see appendix 1). With this apparatus the true strain-rate remains constant with an accuracy of $\pm 0.5\%$ up to engineering strain values equal to 500%. The specimens were deformed at true strain-rates equal to 5.7×10^{-3} and 11.4×10^{-3} mn^{-1} at 30°C. The stress remains constant during each test (curves a and b on fig. 4); it is independent of the strain and the time and only a function of $\dot{\epsilon}$, T and $\bar{L}_i$. These results agree perfectly with the creep tests at constant stress but disagree with those found by Newbury and Joy [13] in the same conditions.

This disagreement may arise from a systematic error in their experiments. Thus, when a part of the specimen is deformed in the grips, the non-corrected values of σ and $\dot{\epsilon}$ are necessarily wrong. In appendix 2, it is shown that this systematic error leads to an apparent increase of the stress σ similar to that observed by Newbury and Joy [13]. In our experiments the deformation in the grips is negligible compared to the length of the specimen, and the final diameter of the specimen is effectively equal to the theoretical diameter.

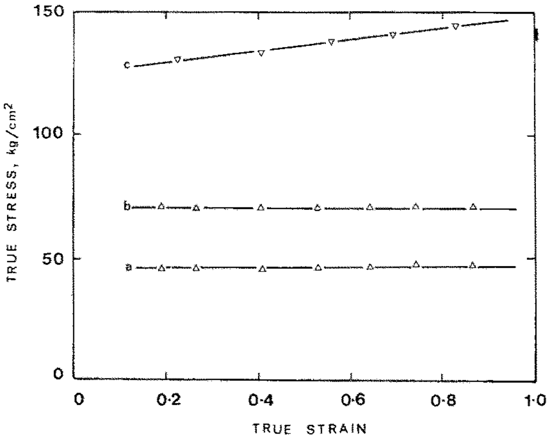


Figure 4 The true stress-time behaviour of the superplastically deformed PbSn eutectic tested at 30°C in tension at constant true strain-rate: (a) $\dot{\epsilon} = 5.7 \times 10^{-3}$ mn^{-1} $\theta = 30$°C, with a negligible elongation of the specimen in the grips. (b) $\dot{\epsilon} = 11.4 \times 10^{-3}$ mn^{-1} $\theta = 30$°C, with a negligible elongation of the specimen in the grips. (c) $\dot{\epsilon} = 12.1 \times 10^{-3}$ mn^{-1} $\theta = 0.5$°C, with an elongation ($\delta l/l_0 = 20\%$) of the specimen in the grips.

4. Conclusion

The superplastic deformation of the eutectic alloy PbSn at temperatures ranging from −44 to 30°C is as follows:

1. The stress and the strain-rate sensitivity coefficient are only functions of the true strain-rate, the temperature and the initial size of the alloy grains since strain-hardening does not appear and the grain size remains constant in this material.
2. The apparent activation energy is constant in the superplastic range and equal to 11.5 ± 0.5 kcal/mol (0.5 eV/atom).

The deformation in the superplastic range is said to be mainly due to grain-boundary sliding [18-19]. This sliding does not exhibit any strain-hardening as in the case of bicrystals [20] and does not lead to any grain growth. It seems to be controlled by the vacancy self-diffusion in tin grain-boundaries.

Appendix 1

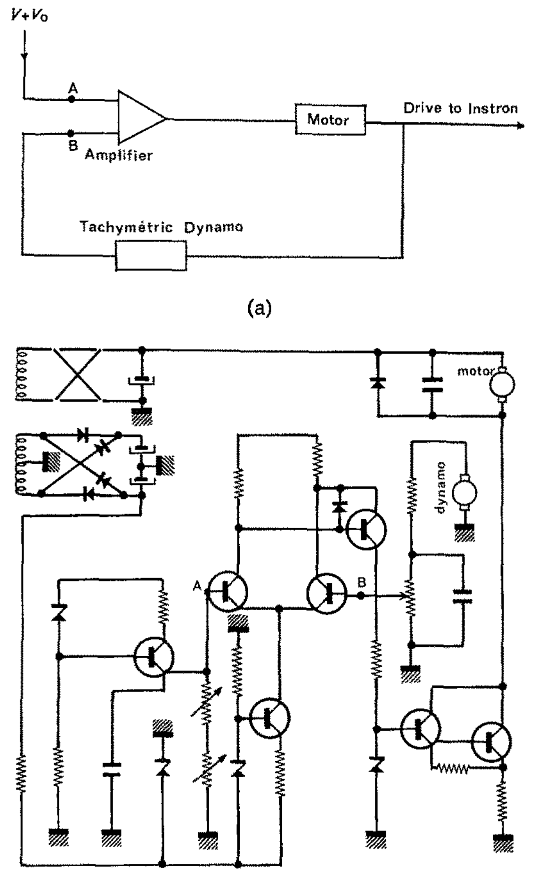
During a tensile test at constant true strain-rate, the length l of the specimen is proportional to the cross-head velocity and varies exponentially with the time. A voltage V proportional to the elongation Δl of the specimen is produced by a measuring device. A voltage V_0 corresponding to the initial length l_0 of the specimen is superimposed to this voltage. The total voltage, proportional to the length l is compared to the voltage produced by a tachymetric dynamo. The difference between these voltages is amplified and runs a direct current engine. This engine controls the speed of the cross-head by means of a reductor and a selsyn (fig. 5).

Appendix 2

If a part δl of the specimen is deformed in the grips, the corrected values σ' and $\dot{\epsilon}'$ can be represented by the following equations:

$$\sigma' \simeq \sigma \left[1 - \frac{\delta l}{l_0} \frac{e}{l + e}\right] \quad \dot{\epsilon}' \simeq \dot{\epsilon} \left[1 - \frac{\delta l}{l_0} \frac{1}{1 + e}\right]$$

σ and $\dot{\epsilon}$ are the values of stress and true strain



(a)

(b)

Figure 5 Device to adapt a normal tensile testing machine for tests at constant true strain-rate; (a) Theoretical diagram. (b) Electrical circuit diagram.

rate calculated without the correction resulting from the elongation of the specimen in the grips; e is the engineering strain, l_0 the initial length.

The flow stress during a tensile test where the true strain rate is effectively constant is obtained by means of a second correction. If we assume that δl remains constant during the deformation, the new equation can be represented by:

$$\sigma'' \simeq \sigma \left[1 - \frac{\delta l}{l_0} \frac{e(1+m)}{1+e}\right]$$

During a tensile test where the flow stress σ'' is constant, the non-corrected stress σ increases up to 15% of its initial value if the strain e is equal to 100%, $\delta l/l_0$ to 20% and m to 0.5. The apparent increase of the stress σ is observed each time that the specimen is deformed in the grips and the rate of this increase agrees with the previous equation (see curve c on fig. 4).

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