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High temperature creep behaviour of lead-base particulate composites

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Lead-base particulate composites, namely Pb–5 vol% Zn, Pb–5 vol% Al_2O_3 and Pb–2.5 wt% PbO have been prepared using powders of lead (atomized), zinc (atomized) and Al_2O_3 by a powder metallurgy technique followed by deformation processing. Isothermal creep tests have been performed at 100, 150 and 180° C on the recrystallized composites. Internal stresses in the steady-state region have been measured in a Pb–PbO composite to evaluate rate controlling mechanisms. Threshold stress for steady-state creep is observed in P/M lead and Pb–Al_2O_3 composites at 100 and 150° C, whereas it is absent in Pb–Zn composites. Two distinct stress exponent regions with different activation energies associated with them, are observed over a shear strain rate range of 10^{-7} to 10^{-4} sec^{-1}. In the low stress exponent region, grain boundary sliding appears to be the rate controlling mechanism, whereas climb controlled dislocation creep is the operative mechanism in the other region.

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1. Introduction

Several investigators [1–5] have reported reduction in the steady-state creep rate of lead by the dispersal of particulate oxides in the matrix. These particulate composites were prepared by a powder metallurgy technique followed by deformation processing. The particulate size used in the composites was below 0.5 μm. Creep tests were conducted between room temperature and 100° C. In the dispersoid size range of 0.5 to 2 μm only a few studies have been reported. The systems that have been studied are zinc-base [6, 7] composites containing Al_2O_3, tungsten, carbon and ZnO as dispersoids and cadmium-base [8] composites containing boron and tungsten as dispersoids. No creep data have been reported for these types of composites. Some data are reported as strain rate against compressive stress for zinc composites [7]. An important feature of these data is the indication of a threshold stress below which creep rates are negligibly small.

The present investigation was undertaken to examine creep behaviour, at temperatures of 100° C and above, of recrystallized lead-base particulate composites containing zinc and Al_2O_3 dispersoids in the size range 0.5 to 1.2 μm and 5 vol%. The two dispersoids, Al_2O_3 (hard) and zinc (soft) were chosen with a view to examining and comparing their effects on the creep behaviour of lead.

2. Experimental procedure

Composites were prepared using powders of lead, zinc and Al_2O_3. Atomized lead and zinc powders were supplied by Messrs Khosla Metal Powders Ltd, India, with a mean particle size of 38 and 30 μm, respectively (as determined by a Fischer Sub-Sieve analyser). The zinc powder was further reduced to a mean size of 1.2 μm in a fluid mill using compressed air. Alumina powder with a mean particle size of 1 μm was obtained from the Union Carbide Corporation, USA. In the as-received condition, the lead powder contained its oxide (PbO) at a level of 1 wt% which is equivalent to 2.5 vol%.

Processing of the composites consisted of the following stages: (a) mechanical mixing of powders,

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(b) compaction, (c) vacuum sintering, (d) extrusion of the sintered compact and (e) annealing. Mixing of powders was done for a period of 1 h. The premixed powders were then compacted at room temperature under a pressure of 384 MPa. All the compacts were sintered at 280° C (about $0.92 T_m$ where T_m is the melting point) under vacuum (10^{-3} torr) for 100 h. The sintered compacts were extruded at room temperature at a speed of 1 cm min^{-1} and an extrusion ratio of 16:1. All the extruded composites were annealed at 280° C for 2 h.

Besides making Pb–Zn and Pb–Al_2O_3 composites, the as-received lead powder was compacted and pressed in the same manner as the composites. It has been designated as P/M lead and treated as a composite containing 2.5 vol% PbO dispersoids.

Constant stress creep tests were performed using double shear specimens [9] (Fig. 1). The advantage of a double shear specimen is that constant stress is maintained on a sample at constant load since the area of the specimen remains constant with shear strain. The tests were performed over a shear stress range of 1 to 11 MPa and at temperatures 100, 150 and 180° C. The tests were started after a period of 15 h of stabilization at the test temperature and continued well into the steady-state region.

Internal stress measurements were carried out in the steady-state creep region in the case of P/M lead. The procedure involved small stress reduction ($\Delta\tau$) of 0.1 τ_A, where τ_A is the applied shear stress, and recording creep strain for some time at the reduced stress. It was followed by a further reduction in stress of 0.1 τ_A and recording of the creep strain. This procedure was repeated till a negative creep rate was observed. The shear stress corresponding to the zero creep rate in such a test was taken as the internal stress (τ_i) corresponding to the applied shear stress and temperature.

3. Results

Comparative study of the microstructures of the annealed composites revealed [10] that the dispersoids are distributed both within grains and on grain boundaries in the case of P/M lead and Pb–Al_2O_3 systems. However, in the case of Pb–Zn most of the dispersoids were located on the grain boundaries. The data on grain size and porosity are presented in Table I.

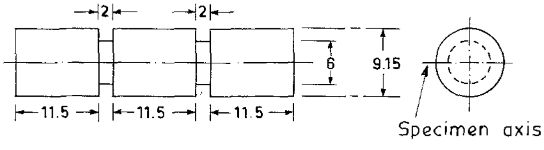

Figure 1 Creep testing specimen. All dimensions in mm.

The stress dependence of steady-state creep rate, $\dot{\gamma}_s$, observed in the lead-base composites at 100, 150 and 180° C is shown in Fig. 2. From Fig. 2 it can be noted that these composites have different stress exponents (n_a) based on applied stress at high and low stress levels. For a Pb–Zn composite, the value of n_a is 2.8 in the low stress region and 7.5 at high stress levels, at all the test temperatures. However, three distinct regions are obtained in the case of P/M lead and Pb–Al_2O_3, at 100 and 150° C. In these systems a threshold stress, below which no creep occurred, is observed at 100 and 150° C. The stress exponents of the other regions are shown in Fig. 2.

The relationship between applied stress (τ_a) and internal stress (τ_i) for P/M lead at 150° C is shown in Fig. 3. On the basis of internal stress measurement, the effective stress τ_e ($= \tau_a - \tau_i$) has been calculated and the plot of $\dot{\gamma}_s$ against τ_e is also shown in Fig. 2 for P/M lead. The plot indicates two regions of different stress exponents (n_e) based on the effective stress, namely 1 and 5.2 at low and high stress levels, respectively.

An Arrhenius plot of $\log \tau_a$ against $1/T$ is shown in Fig. 4 for the determination of activation energy for steady-state creep in low and high stress regions. The values of apparent activation energy (corresponding to the stress exponent, n_a) for these composites are summarized in Table II. In comparison with the self-diffusion activation energy (Q_{SD}) value of 95.9 kJ mol^{-1} [2] for lead, the Q_a values over the low stress exponent region are much lower. On the other hand, Q_a values in the high stress exponent region are closer to Q_{SD} for lead. Further, the Q_a value for Pb–Zn composite is lower than those of P/M lead and Pb–Al_2O_3 composites at both low and high stresses.

4. Discussion

Several possible dislocation creep mechanisms have

TABLE I Porosity and grain size of Pb-base composites

Composite	Porosity (%)	Mean intercept length (μm)
P/M lead	0.1	9
Pb–5 vol% Al_2O_3	1.0	9
Pb–5 vol% Zn	0.4	12.5

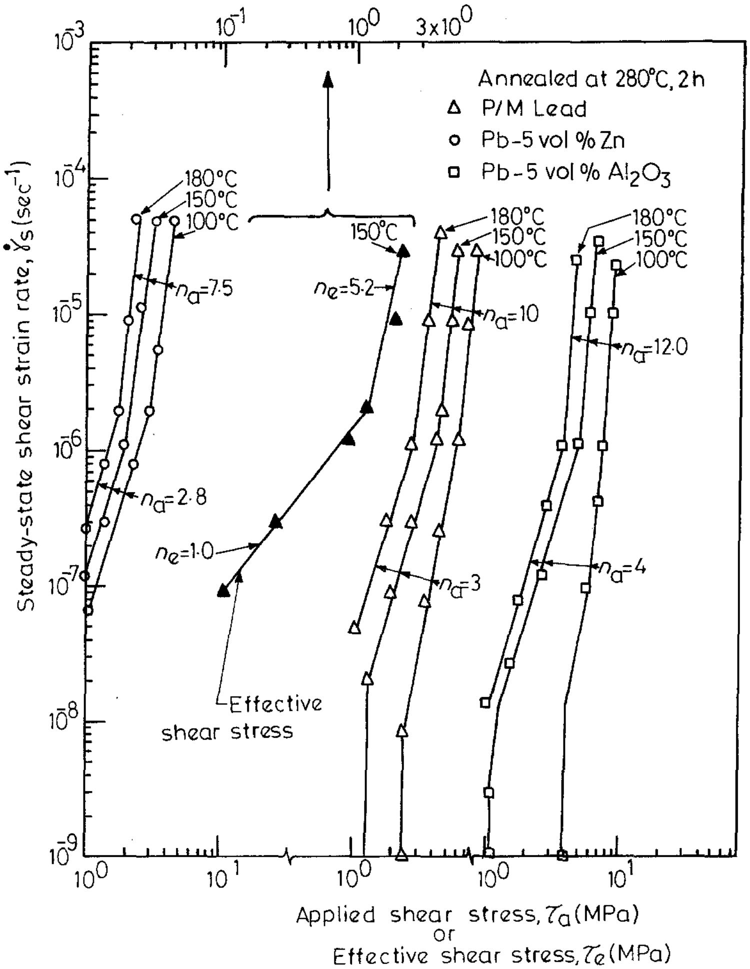

Figure 2 The stress dependence of the steady-state shear strain rate of lead-base composites.

been proposed to account for the creep behaviour of dispersion hardened alloys. The basis of these models is the original proposal by Ansell and Weertman [11]. In this proposal, the stress exponent for the steady-state creep rate is given as 4.5 which obviously does not explain the higher values obtained in several cases and those obtained in the lead-base composites. Also, the activation energy values proposed for the creep process are close to self-diffusion activation energy values. The model proposed by Ansell and Weertman has been modified by others [12–14] by invoking the concept of effective stress. The effective stress operating during creep deformation has been visualized as equal to $(\tau_a - \tau_i)$. It is observed that the stress exponents are modified to $n_e = 1$ and $n_e = 5.2$ in the steady-state creep range for P/M lead when the internal stresses are taken into account. Similar qualitative behaviour for Pb–Zn and Pb–Al_2O_3 composites is expected although no internal stress measurements were done.

It is to be noted that from the Arrhenius plots given in Fig. 3 for P/M lead, the observed apparent activation energies are 40.8 and 125.5 kJ mol^{-1} for regions of stress exponents $n_a = 3$ and 10, respectively. If the effective stress is used to estimate the activation energy, values would be lower than those obtained on the basis of applied stress. The activation energy value in the $n_e = 5.2$ would approach more closely to Q_{SD} for lead.

The stress exponent $n_e = 1$ is indicative of four possible operative creep mechanisms; namely, (a) Nabarro–Herring (N–H) [15], (b) Coble [16] (c) Harper–Dorn [17] and (d) grain-boundary sliding (GBS) [18].

In case of the Harper–Dorn mechanism, creep rate is proportional to the applied stress and the activation energy is equal to that for self-diffusion of the metal. On the basis of the measured activation energy values (below 45.6 kJ mol^{-1}) in all the composites studied in low stress exponent regions, the possibility of the Harper–Dorn mechanism can be ruled out.

If Coble or N–H creep controls the creep behav-

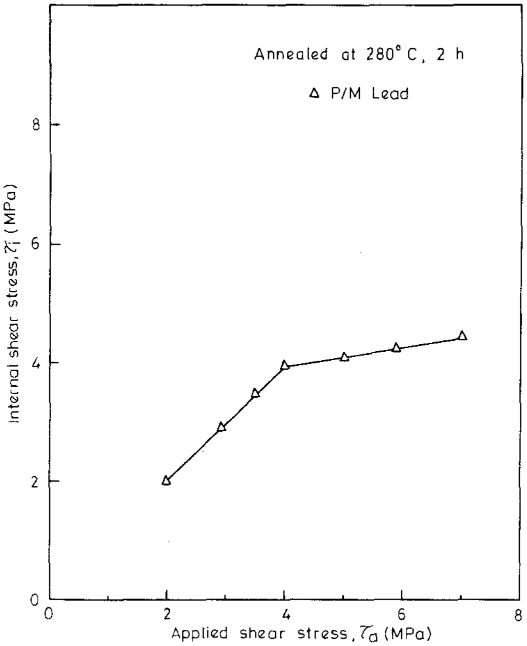

Figure 3 Arrhenius plots for lead-base composites at constant $\dot{\gamma}_s$.

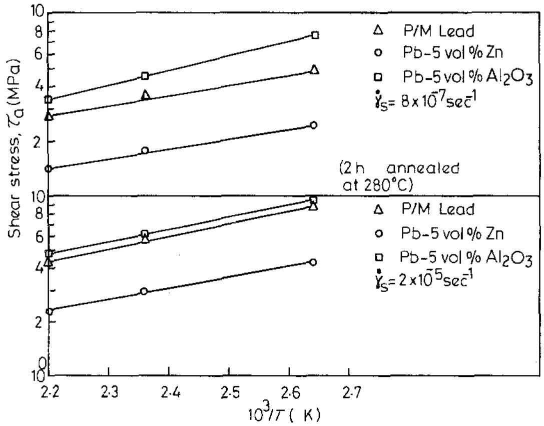

Figure 4 Internal shear stress as a function of applied stress in P/M lead measured in a creep test at 150° C.

iour, primary creep would be expected to be absent. However, there is a considerable amount of primary creep exhibited in all the lead-base composites. Moreover, estimated shear creep rates, according to these mechanisms, are below 10^{-9} sec^{-1} for the applied shear stress of the present investigation, whereas the observed creep rates are above 10^{-7} sec^{-1}. Hence both the N–H and the Coble mechanisms may also be excluded as operative mechanisms in this region.

The GBS mechanism predicts the stress exponent to be unity and an activation energy value corresponding to grain boundary diffusion activation energy which is about half or less than half of Q_{SD}. Since grain sizes of these composites are relatively small, the GBS can be the possible controlling mechanism in the lead-base composites above $0.5 T_m$ at low stress levels.

The observed threshold stress in P/M lead and Pb–Al_2O_3 may also be characteristic of the GBS mode of deformation. Because of the presence of dispersed particles at or near grain boundaries, there is a greater resistance to sliding in comparison to a particle free boundary [19] and the threshold stress may represent the minimum stress level below which sliding cannot occur. The threshold stress is observed to decrease with increasing temperature similar to the other reported cases [20, 21]. The threshold stress, if any, in the case of Pb–Zn composite, is below the lowest stress studied in the present investigation. This may be due to the softer nature of zinc dispersoid which is expected to offer much less resistance to sliding than other hard dispersoids.

In the higher stress exponent region, the modified stress exponent is 5.2 in case of P/M lead and the activation energy is 125.5 kJ mol^{-1}. The activation energy would be reduced below the apparent value after taking into account the internal stress, bringing it closer to Q_{SD} of lead. The modified values of the stress exponent and the activation energy would be in reasonable agreement with the dislocation climb model proposed by Ansell and Weertman [11].

5. Conclusions

The following conclusions are drawn from this investigation.

1. Threshold stress for steady-state creep is

TABLE II Q_a and n_a for lead-base composites

Composite	Low stress region		High stress region	
	n_a	Q_a (kJ mol^{-1})	n_a	Q_a (kJ mol^{-1})
P/M lead	3.00	38.7	10.00	125.5
Pb–5 vol % Al_2O_3	2.80	30.1	7.50	86.6
Pb–5 vol % Zn	3.50	43.5	12.00	138.1

observed in P/M lead and Pb–5 vol% Al_2O_3 composites below 150° C, whereas it could not be detected in Pb–5 vol% Zn composites in the same temperature range. The threshold stress, thus, depends on the nature of the dispersoid and the temperature.

2. In the creep of lead-base composites, high and low stress exponent regions with corresponding activation energies have been observed. In the low stress exponent region, grain boundary sliding appears to be the rate controlling mechanism, whereas climb controlled dislocation creep is the operative mechanism in the high stress range.

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