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Some Creep and Fatigue Properties of Dispersed-Oxide-Strengthened Lead

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Creep and fatigue tests have been made on dispersed-oxide-strengthened lead. The presence of dispersed oxide in the lead resulted in a marked improvement in creep and fatigue resistance. Observations showed that the presence of oxide particles affected the incidence of slip and grain-boundary migration. It is suggested that the role of the dispersed oxide, in improving the creep and fatigue properties of lead, arises from the effect of oxide particles on the amount of slip, the amount of grain-boundary sliding and migration, and, in fatigue failure, the number of vacancies available for cavity nucleation.

1. Introduction

There has been considerable interest in the recent developments in dispersion-strengthened leads, since Lenel [1], Roberts *et al* [2, 3], and Lund *et al* [4] reported significant improvements in the thermal stability and in the tensile and creep properties of lead due to the presence of dispersed oxide.

The present work was undertaken to examine the effects of dispersed oxide on the creep and fatigue properties of lead and, in particular, to determine how the dispersed oxide affected the deformation and fracture processes.

2. Materials and Methods

Experiments were made on two types of dispersions of oxide in lead. The first material, designated DS lead A, was prepared from commercial-purity lead powder extruded at a temperature between 160 and 250° C, at a pressure of 40 ton/in.^{2} (1 ton/in.^{2} = 1.6 kg/mm^2). This material had an oxide content of 2.0 to 2.5 wt % PbO and an ultimate tensile strength of 3×10^3 lb/in.^{2} (1 lb/in.^{2} = 7×10^{-4} kg/mm^2).† The average grain size was 2.5×10^{-3} in. (1 in. = 25.4 mm).

The second material, designated DS lead B, was prepared by melting commercial-purity lead, 99.998 % lead, in air, at various temperatures in the range 400 to 900° C. The lead was saturated with oxygen by keeping the melt at a particular temperature for 30 min, and was cast into a water-cooled mould to reduce segregation of the oxide during solidification. The $1 \times 1 \times 5$ in. ingots were reduced to strip 0.160 in. thick by rolling. Metallographic examination showed that the oxide was distributed in oxide-rich layers parallel to the rolling plane. This material was tested in fatigue only, because of its less attractive properties compared with those of DS lead A, as discussed below.

The grain structures of DS lead A and DS lead B are shown in figs. 1a and 1b.

Creep specimens of DS lead A were prepared by clamping the sheet in a hardened steel template and cutting around the edges of the template with a sharp knife. Specimens were taken from the sheet so that the extrusion direction was parallel to the tensile axis. The gauge section of the creep specimen was either $1 \times 2 \times 0.036$ or $1 \times 2 \times 0.064$ in. Specimens were chemically polished in Worner and Worner's solution [25] and were tested under conditions of constant load at $20 \pm 2°$ C ($0.5\ T_m$, where T_m is the melting point of lead).

Flat cantilever specimens with tapered sides [5] were tested at 500 c/min in a reverse-plane-

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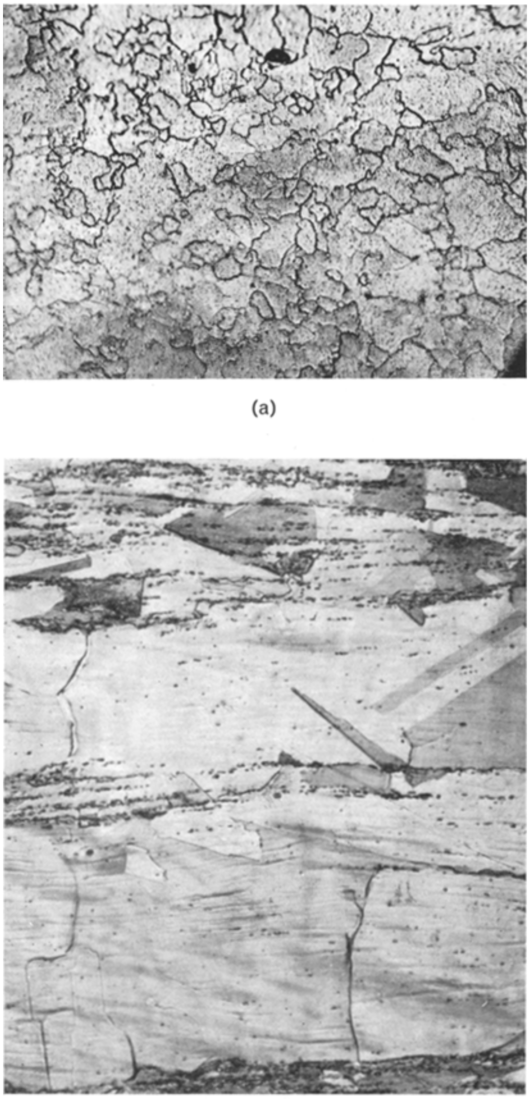
†Samples of DS lead A and production details were kindly supplied by Mr R. D. Semmens of Broken Hill Associated Smelters Pty Ltd.



(a)

(b)

Figure 1(a) DS lead A – etched surface of sheet which was formed by extruding lead powder (×210). (b) DS lead B – rolled ingot cast at 800° C; the section plane was orthogonal to the rolling plane and contained the rolling direction (×187).

bending fatigue machine which has been described elsewhere [6].

3. Results

3.1. Creep

3.1.1. Creep Tests on DS Lead A

Typical creep curves obtained for the DS lead A specimens are shown in fig. 2a except for the curve at 2×10^3 lb/in.^{2}, in which the elongation was too rapid to be plotted on this scale. The specimen at 2×10^3 lb/in.^{2} failed after 4.5 h at an extension of 16.8%. It was noted that the curve at 1.45×10^3 lb/in.^{2} showed a restricted tertiary stage, and that small changes in the secondary creep rates occurred during the tests at 1.2×10^3 and 1.45×10^3 lb/in^2. This latter feature has also been reported by Ansell and Weertman [7] for a dispersion-strengthened aluminium alloy which showed the effect to a more marked degree than the present material.

The instantaneous extension of the creep specimens was approximately proportional to the square of the creep stress up to a stress of 1.75×10^3 lb/in.^{2} (fig. 2b). This behaviour is similar to that for pure lead reported by Feltham [8] and is similar to the tensile stress/strain behaviour of other dispersion-strengthened materials [9]. The data shown in fig. 2b can be approximated by the relation:

$$\sigma^2 = 1.06 \times 10^9 \, \epsilon$$

where σ is the applied stress (in pounds per square inch) and ϵ is the initial elongation (in inch per inch).

The secondary creep rate ($\dot{\epsilon}$) of DS lead A was found to show a high stress sensitivity (fig. 3a). In this figure, the stress sensitivity of $\dot{\epsilon}$ for annealed commercial-purity lead (grain size $\sim$0.2 cm) and that for a 0.05% Ca/Pb alloy [10] (grain size $\sim$0.2 cm) are given for comparison. The Ca/Pb alloy data are given because the alloy has a low secondary creep rate compared with that of pure lead and other common binary lead alloys [10]. It is seen that DS lead A has a lower creep rate than that of either the Ca/Pb alloy or the pure lead. Each of the three curves (fig. 3a) shows that the stress sensitivity of creep decreases as the stress level decreases. Fig. 3b shows that stress for a particular creep rate for DS lead A is 2 to 7 times the stress for the same creep rate of pure lead.

The relation between σ and $\dot{\epsilon}$ can be represented in the form of the Bailey equation [11]:

$$\dot{\epsilon} = A' \sigma^n \tag{1}$$

where n and A' are constants. For DS lead A, the stress exponent n decreased smoothly from $n \simeq 27$ at the higher stresses to $n \simeq 6$ at the lower stresses. For pure lead, the exponent decreased from $n \simeq 7$ at 1000 lb/in.^{2} to $n = 1$ at below 300 lb/in^2. The significance of this for pure lead is discussed elsewhere [12, 13]. The exponent for the 0.05% Ca/Pb alloy also

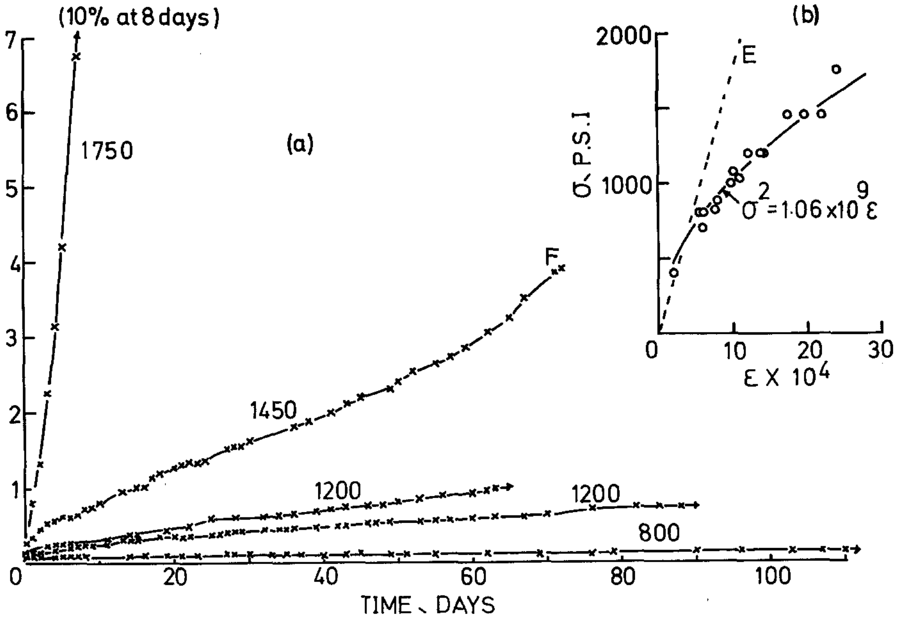


Figure 2 (a) Creep curves for DS lead A stressed at 800, 1200, 1450, 1750 lb/in^2. *F* indicates failure of specimen. (b) Instantaneous elongation of DS lead A. Dashed line marked *E* indicates elastic line. (*Note* psi units are used in the illustrations; 1 psi = 1 lb/in.^{2} = 7 × 10^{-4} kg/mm^2.)

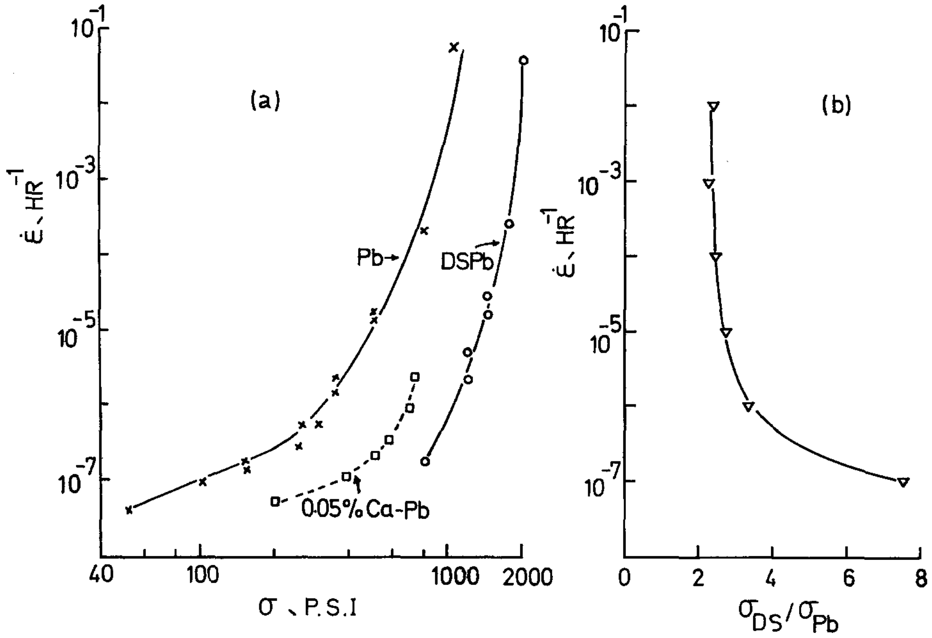


Figure 3 (a) Stress dependence of the creep rate for lead (from reference 13), a 0.05 wt % Ca/Pb alloy (from reference 10), and DS lead A. (b) The ratio of the stress on DS lead A to that on pure lead for various creep rates.

approached $n = 1$ at low stress. The high values of n for DS lead A are consistent with the high values for n for other dispersion-strengthened materials (e.g. Wilcox and Clauer [14] obtained

$n = 40$ for thoriated Ni at temperatures above $0.5\ T_m$, and Takahashi *et al* [15] found $n = 12$ for Ni + Al_2O_3).

The creep rupture behaviour of DS lead A is compared with that of pure lead in fig. 4. The comparison shows that, for a given time to fracture, the rupture strength of DS lead A was about twice that of pure lead. However, the ductility at fracture of DS lead A was substantially less than that of pure lead, as is usual for DS-type materials. (The percentage ductility is given in parenthesis at each point.)

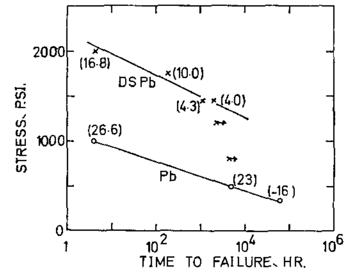


Figure 4 The stress rupture behaviour of DS lead A and pure lead.

3.1.2. *Observations on Creep Deformation and Fracture of DS Lead A*

Metallographic examination of the specimens after creep showed that: (i) slip was very dispersed and difficult to detect, except for the unusual clustering of slip into bands in some grains near the main fracture (e.g. the slip bands marked A in fig. 5); (ii) little or no grain-boundary migration had occurred; (iii) some boundaries showed surface steps which were apparently due to sliding (e.g. at B in fig. 5); and (iv) cracks were evident (e.g. at C in fig. 5). No metallographic evidence for recrystallisation during creep was detected, such as slip crossing a number of grains, marked changes in grain size, or rapid grain-boundary migration [22]. Crack formation occurred uniformly over the gauge section, and cracks extended by propagating at right-angles to the tensile-stress direction. The slip density, as detected with the light microscope at magnifications of up to ×1000, decreased with decreasing stress level and was less than that in pure lead at similar creep strains [22]. Specimens repolished after failure showed that the cracks were located at grain boundaries (fig. 6) and that no marked change of grain size had occurred, except near the main fracture where grains were elongated in the stress direction (fig. 6).

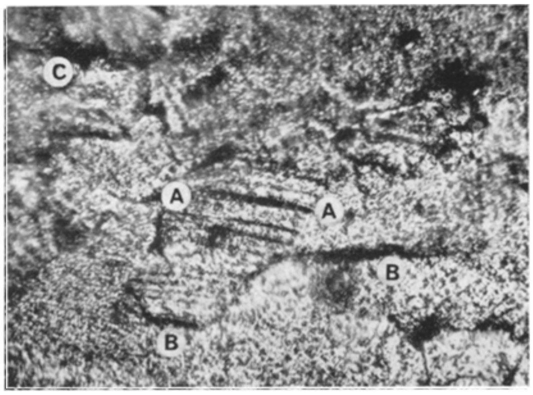


Figure 5 DS lead A stressed at 2×10^3 lb/in.^{2}; strain is 17% (×590). *A* indicates an unusual example of clustering of slip into bands; in general, the slip was dispersed and hard to detect. *B* indicates grain-boundary steps. *C* indicates crack.

Figure 6 DS lead A stressed at 2×10^3 lb/in^2. Chemically polished after failure at 16.8%. Area showing intercrystalline cavities and grain elongation (×280).

3.2. Fatigue

3.2.1. *The Fatigue Behaviour of Dispersed-Oxide-Strengthened Lead*

Fatigue tests were made on DS lead A and DS lead B to determine the influence of the presence and distribution of oxide. Specimens were fatigued at strain amplitudes from ±0.034 to ±0.090%; the results are given in table I.

The table shows that DS lead A had a greater endurance than that of DS lead B, which in turn was greater than that of pure lead. Specimens

TABLE I Endurance of DS lead and pure lead specimens.

Strain	Pure lead*	DS lead A	DS lead B (cast 900° C)
±0.090%	0.27 × 10^6	5 × 10^6 (average 3 specimens)	1 × 10^6 (average 2 specimens)
±0.034%	1.5 × 10^6	$\gg$6 × 10^6 (3 specimens)	6.5 × 10^6 (average 2 specimens)

*data from reference 16

of DS lead B prepared from ingots cast at intermediate temperatures between 400 and 900° C had endurances which were between that of pure lead and the values given in the table for 900° C.

3.2.2. Observations on the Fatigue Behaviour of Dispersed-Oxide-Strengthened Lead

(*a*) *DS lead A* Metallographic examination of specimens after various amounts of fatigue showed that deformation was first evident as regions of very fine slip in isolated grains at 1% of the fatigue life (fig. 7a). The scale of this fine slip may be appreciated by comparison with slip shown by pure lead fatigued for a comparable number of cycles at almost the same strain amplitude (fig. 7b). These two figures (figs. 7a and 7b) illustrate the marked suppression of slip due to the presence of dispersed oxide. With continued fatigue-straining, the regions of slip in the dispersion-strengthened lead tended to form into transverse bands (fig. 8). Specimens showed no evidence of grain-boundary migration and did not develop the rectangular grain structure observed in pure lead after fatigue [5, 16].

Small, serrated, fatigue cracks (fig. 9a) were found to develop in association with the transverse bands of fine slip such as those shown in fig. 8. Repolished specimens showed, however, that the cracks were intercrystalline (fig. 9b) and that these tended to be less rounded than the cracks formed during creep at high stress.

(*b*) *DS lead B* These specimens were sectioned after fracture to determine the influence of the presence of oxide-rich layers parallel to the rolling plane. Sections perpendicular to the rolling plane and parallel to the rolling direction are shown in figs. 10a and 10b. Fig. 10a indicates that the intercrystalline crack growing from the surface (near the top of the figure) was deviated at the stringers of oxide shown by arrows. Fig. 10b shows that a fatigue crack (growing from left to right in the figure) was propagating along the oxide-rich layers. This behaviour suggests that the metal/oxide interfaces were weak in comparison with grain-boundary interfaces.

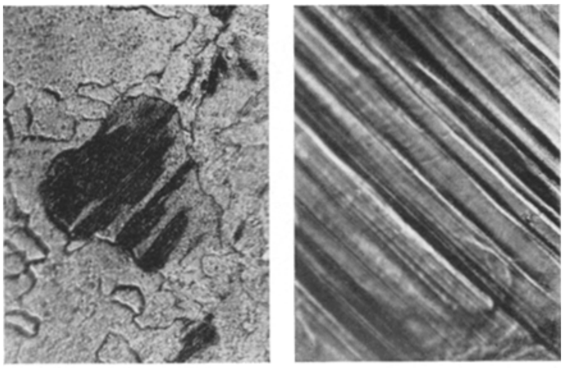

(a) (b)

Figure 7 (a) DS lead A subjected to 6 × 10^4 cycles at a strain amplitude of ±0.090% – grain showing fine slip – (×400). (b) Pure lead (99.998% Pb) after 5 × 10^4 cycles at a strain amplitude of ±0.092% (×500).

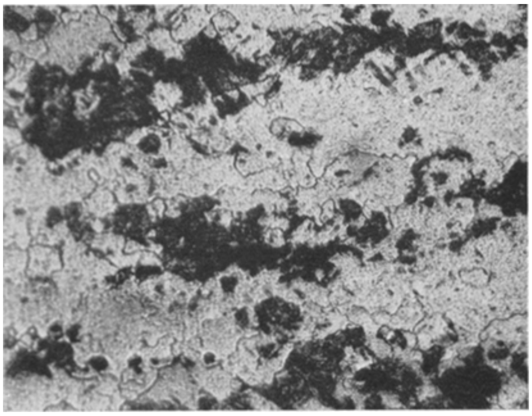
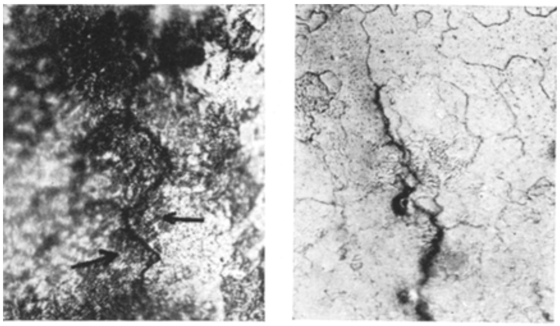


Figure 8 DS lead A. Area showing transverse bands of slip at 6 × 10^5 cycles (×280). Specimen axis parallel to short edges of the figure.

4. Discussion

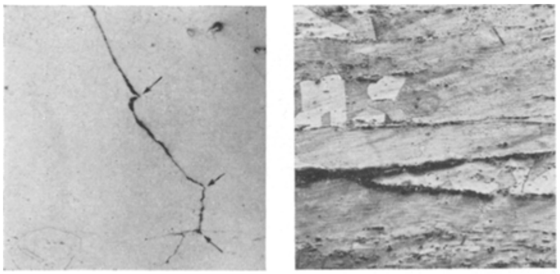
4.1. Creep

The present work shows that the dispersed-oxide-strengthened lead has a significantly greater creep resistance than that of either pure lead or a creep-resistant 0.05% Ca/Pb alloy. The high stress sensitivity of the creep rate of DS lead A is consistent with the reported



(a) (b)

Figure 9 DS lead A: (a) fatigue crack at 2.8×10^6 cycles at a strain of $\pm 0.090\%$ – note dark patches of fine slip associated with the crack which are marked by arrows (×350); (b) repolished after failure at 2.8×10^6 cycles (×200).



(a) (b)

Figure 10 DS lead B: (a) specimen prepared from rolled ingot which had been cast at 600° C, strain amplitude $\pm 0.034\%$ (×47); (b) specimen prepared from rolled ingot cast at 900° C, fatigued at $\pm 0.034\%$ (×94). Plane of section was orthogonal to the rolling plane and contained the rolling direction.

behaviour of other dispersion-strengthened materials [14, 15] and is much greater than that predicted by Ansell and Weertman's theory for dispersion-strengthened materials [7]. Other investigations [9, 15, 17] have suggested that, at high stress levels, the behaviour of dispersion-strengthened materials appears to be related to high rates of dislocation interaction during creep, which are due to retention of strain-hardening and suppression of the normal recovery and recrystallisation processes. For example, the possibility of such dislocation interactions is suggested by the observations of Takahashi *et al* [15], who found that the stress sensitivity of the creep rate of Ni + 1 wt % Al_2O_3 alloy could be increased by cold-work. Also by the observations of Ebeling and Ashby [9], and Greetham and Honeycombe [17], who found that single crystals, which contained dispersions, were more prone to exhibit multiple slip and show multiple slip strain-hardening behaviour than pure single crystals under similar test conditions. In the dispersion-strengthened single crystals, multiple slip occurred in the vicinity of particles [17].

At low stresses, the strengthening effects of the dispersed oxide may be considered in relation to the nature of the creep deformation of the basis lead. It has been pointed out that for this material, creeping at rates less than $\dot{\epsilon} \simeq 5 \times 10^{-7}$ h^{-1}, grain-boundary sliding makes a contribution to the overall creep extension which is at least comparable to that due to slip within grains [12, 13]. Fig. 3b shows that the strengthening influence of the oxide dispersion increased sharply in this range of creep rates. This suggests that oxide particles not only interfere with the movement of dislocations but may also inhibit grain-boundary sliding.

Similarly, the creep failure of DS lead A by intercrystalline cracking is probably associated with oxide particles suppressing slip and grain-boundary migration. In the absence of boundary sliding, the condition for the growth of a stable cavity at an oxide particle, which is assumed to be "non-wetting" with respect to the metal, is [18-20]:

$$r > \frac{2\gamma}{\sigma} \qquad (2)$$

where r is the radius of the particle which must be exceeded to form a stable cavity and γ is the surface energy, taken as ~350 dyn/cm [21]. For applied stresses in the range 0.8×10^3 to 2×10^3 lb/in.^{2}, the critical radii are 5×10^{-6} to 1.5×10^{-5} cm. Particles below this size may nucleate cavities, but then would require the assistance of grain-boundary sliding above the critical rate of sliding which overcomes the effects of sintering [19]. However, there are probably sufficient oxide particles present in the dispersion-strengthened lead, with radii above the critical values predicted by (2), to form stable crack nuclei without the aid of sliding, provided there is little or no cohesion at the metal/oxide interface as suggested by the observations on DS lead B (fig. 10b).

In contrast to the behaviour of dispersion-strengthened lead, pure lead exhibits recrystallisation, substantial amounts of slip, grain-boundary sliding and migration, and normally fails in creep by necking down to a knife-edge [22]. In this case, the slip and grain-boundary migration are probably sufficient to relax any stress concentrations capable of nucleating intercrystalline cavities.

4.2. Fatigue

The fatigue resistance of DS lead A and DS lead B was greater than that of pure lead. This is in agreement with the results for a dispersion-strengthened lead reported by Roberts *et al* [3], and with the results for other dispersion-strengthened materials reported by Bradshaw and Wheeler [23] and by Ham [24]. Further, the present results showed that the lead which contained the more uniform dispersion of oxide (DS lead A) had the better fatigue properties.

The observations revealed that the dispersion-strengthened leads failed by intercrystalline cracking, which is the same mode of failure as that for pure lead [26]. (The intercrystalline failure of pure lead in fatigue is in contrast to the ductile knife-edge-type failure of pure lead under creep conditions noted above.) The presence of the dispersed oxide in the lead was observed to reduce the amount of slip at a given cyclic strain amplitude and to prevent grain-boundary migration. Comparison of the observations on dispersion-strengthened lead with those previously obtained on pure lead suggests that the importance of the dispersed oxide in the lead is threefold.

(*a*) It is known that, under the action of fatigue straining, grain boundaries in pure lead migrate and line-up in directions of maximum shear stress. In adopting these orientations, boundaries form a rectangular pattern which is conducive to crack nucleation by processes involving grain-boundary sliding [21]. In the dispersion-strengthened lead, however, the rectangular grain structure did not develop, because of the suppression of grain-boundary migration; so that there would be less tendency for grain-boundary sliding to occur with the associated nucleation of cracks.

(*b*) The tendency for crack nucleation by processes involving grain-boundary sliding would also be reduced by oxide particles "keying" grain-boundary sliding. In the short time available during a half-cycle (<0.05 sec), there would be little chance for diffusion to relax stresses at oxide particles preventing grain-boundary sliding. Harris [19] has pointed out that such boundaries may slide if the metal surrounding the particles can deform plastically under the applied stress. This may account for the localised slip near grain-boundary fatigue cracks (fig. 9a).

(*c*) The presence of dispersed oxide in the lead reduced the amount of slip produced by fatigue-straining. This suggests that, at a given strain amplitude (=plastic + elastic strain), the plastic component of strain would be less in dispersion-strengthened lead than in pure lead under the same total strain. This suggests further that, since the plastic strain is associated with the generation of dislocations and vacancies, there would be fewer dislocations and mechanically produced vacancies in the dispersion-strengthened lead for crack formation.

These features of the fatigue deformation suggest that the improved fatigue resistance of the dispersion-strengthened lead, compared with that of pure lead, is due to the reduced amount of grain-boundary sliding and to fewer dislocations and mechanically produced vacancies available for crack formation in the presence of particles of dispersed oxide.

5. Conclusions

The present experiments on dispersed-oxide-strengthened lead have indicated the following conclusions.

(*a*) The creep resistance of DS lead A was greater than that of either pure lead or a 0.05 wt % Ca/Pb alloy.

(*b*) The instantaneous extension of DS lead A was proportional to the square of the applied stress up to 1.75×10^3 lb/in^2.

(*c*) The stress dependence of the secondary creep rate, $\dot{\epsilon}$, of DS lead A increased with increasing creep stress, σ (e.g. for DS lead A at low stress $\dot{\epsilon} \propto \sigma^6$ and at high stress $\dot{\epsilon} \propto \sigma^{27}$).

(*d*) The creep rupture strength of DS lead A was about twice that of pure lead.

(*e*) The creep and fatigue failure of DS lead A occurred by intercrystalline cracking.

(*f*) The fatigue resistance of DS lead A, which contained a uniform dispersion of oxide, was greater than that of DS lead B, which contained oxide dispersed in oxide-rich layers parallel to the plane of rolling.

(*g*) The improved creep and fatigue properties of DS lead A are suggested to arise from the effects of the dispersed oxide particles on the ease of slip, and on the amount of grain-boundary migration and sliding.

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