JOURNAL OF MATERIALS SCIENCE 6 (1971) 1178–1181

The Fatigue Behaviour of Dispersed-Oxide-Strengthened Lead in Air and in Vacuum

K. U. SNOWDEN*
The Baillieu Laboratory, Metallurgy Department, Melbourne University, Parkville, Victoria, Australia

Specimens of dispersed-oxide-strengthened lead containing 4.5 wt % oxide were fatigued at room temperature ($\sim$ 0.5 T_m where T_m is the melting point in degrees K) in air and in vacuum ($< 2 \times 10^{-4}$ torr). Metallography of the damage during fatigue and after fatigue fracture showed that the improved fatigue resistance of dispersed-oxide-strengthened lead (DS lead) over that of pure lead was due mainly to the mechanical strengthening effects of the dispersed oxide rather than an increase in the resistance to atmospheric corrosion fatigue. The ratio of the fatigue life of DS lead in vacuum to that in air was $\sim$ 8.5 at a strain of $\pm$ 0.145%. In specimens fatigued in air, failure occurred at grain boundaries and in those fatigued in vacuum it occurred by a mixture of intercrystalline and transcrystalline modes.

1. Introduction

Dispersion-strengthened metals have been found to have attractive fatigue properties at ambient and elevated temperatures. However, little information appears to be available on the structural stability of dispersion-strengthened metals subject to fatigue stressing and on the mode of fatigue deformation and fracture of these materials under conditions of low strain fatigue at temperatures $\sim$ 0.5 T_m, where T_m is the melting point in degrees Kelvin. In addition, the fatigue properties of many materials are affected by small amounts of oxygen or water vapour in the test atmosphere. It is therefore of interest to distinguish between the mechanical strengthening effects of the dispersion and the influence of the dispersion on the material resistance to atmospheric corrosion fatigue.

The present work was undertaken to examine (a) the stability of the structure of dispersed-oxide-strengthened lead (DS lead) under fatigue stressing and (b) to determine the effects of the ambient air atmosphere on the fatigue behaviour of a DS lead since it was known that pure lead is strongly affected by the presence of oxygen in the test atmosphere [1].

Previous work [2] on the fatigue properties of DS lead in air had shown that these materials did not exhibit the grain-boundary migration which resulted in the marked changes in grain shape typical of the fatigue deformation of pure lead. In spite of the different grain-boundary migration behaviour, fatigue crack propagation in both DS lead and pure lead was found to follow an intercrystalline path. The lives of the DS leads were greater than those of pure lead at the same strain amplitudes. However, it was not clear from this previous work whether the greater fatigue resistance of the DS lead was due to an increase in mechanical strength or an improvement in the resistance to atmospheric corrosion fatigue.

2. Experimental Procedures

Samples of DS lead were obtained from the St Joseph Lead Company in the form of plates 5.08 × 76.2 × 152.4 mm (0.2 × 3 × 6 in.). The average grain size of the samples was 8×10^{-4} cm in the extrusion or principal specimen axis direction and 2×10^{-4} cm at right angles to this direction. The hardness of the samples was 14.5 VPN when measured with a 10 kg load. This

*Now with Materials Division, Australian Atomic Energy Commission Research Establishment, Private Mail Bag, Sutherland, N.S.W., Australia.

© *1971 Chapman and Hall Ltd.*

hardness number indicated an oxide content of about 4.5 wt % [3]. Analysis by the B.H.A.S. Pty Ltd Assay Department at Port Pirie, South Australia showed the presence of the following major metallic impurities; Cu 0.042, Sb 0.020, Sn 0.032, Bi 0.039, Zn $<$ 0.0004, As $<$ 0.002 and Ag 0.0004 wt %.

The reverse-plane-bending fatigue machine and the specimen geometry have been described elsewhere [4, 5]. The fatigue tests in vacuum were made at pressures of about 2×10^{-4} torr or better, i.e. in the range below 5×10^{-3} torr where the fatigue life becomes independent of air pressure [1]. Specimens were annealed at 100°C for 1 h and chemically polished in Worner and Worner's solution [6] before fatigue testing. Back reflection X-ray diffraction patterns showed rings of sharp spots with little evidence of preferred orientation.

3. Results

3.1. Fatigue Life in Air and in Vacuum

The results of the fatigue tests on the DS lead specimens in air and in vacuum are given in fig. 1. The two dashed curves in fig. 1 are taken from previous work [1] and represent the endurance of pure lead in air and in vacuum. These pure lead specimens had a grain size of $\sim 2 \times 10^{-1}$ cm whereas the present DS lead had a grain size of 2 to 8×10^{-4} cm. Fig. 1 shows that the DS lead specimens had lives which were 10 to 20 times those for pure lead at the same strain amplitudes. Further, the DS lead specimens which were fatigued in vacuum had lives significantly greater than those of the DS lead specimens fatigued in air at the same strain amplitude. The magnitude of the increase in life was similar to that shown by pure lead, i.e. at a strain amplitude of $\pm$ 0.145% the ratio of the life in vacuum to that in air was $\sim$ 8.5 for the DS lead and $\sim$ 9.5 for pure lead.

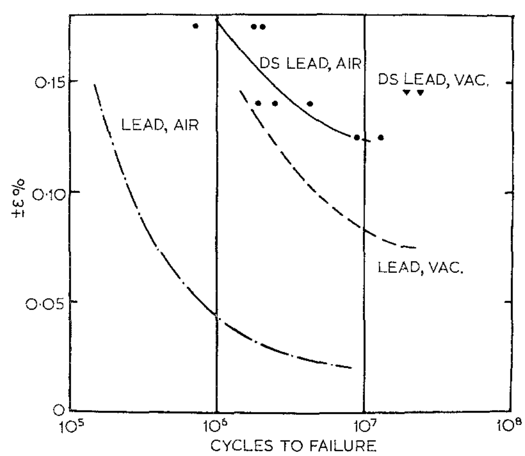

Figure 1 Strain amplitude ($\pm$ ϵ) versus fatigue life for DS lead and for pure lead (dashed curves). The latter curves are from reference [1].

3.2. Metallographic Examination

3.2.1. *Air Tests*

Observations made on the DS lead specimens which were fatigued in air to various fractions of

Figure 2 Grain growth associated with a small crack in DS lead fatigued to failure at 2.5×10^6 cycles in air at a strain of $\pm$ 0.145% (chemically polished after failure) ($\times$ 233).

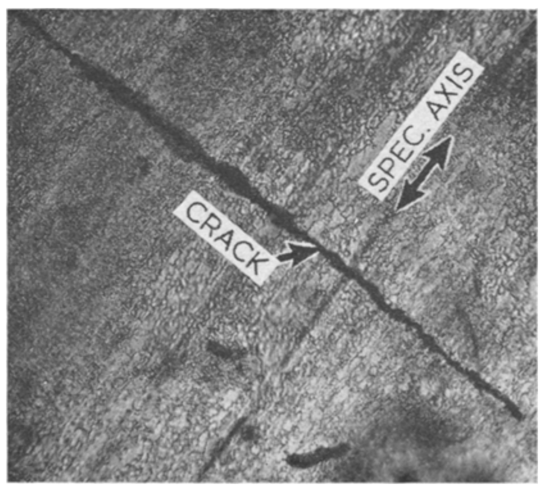

Figure 3 Crack propagation at 90° to specimen axis. Same specimen as shown in fig. 2 ($\times$ 233).

the fatigue life showed that slip traces were finely dispersed and it was difficult to resolve individual slip markings at a magnification of × 1000. It was noted however that, as the fatigue-straining increased, isolated clusters of grains appeared to blacken owing to the intensification of fine slip in these grains. These blackened grains were often associated with the formation of small irregular cracks. Later repolishing revealed that these cracks occurred in areas of local grain growth and distortion (fig. 2).

The general direction of crack propagation was 90 degrees to the specimen axis, fig. 3, and secondary cracking confirmed that the path was intercrystalline as reported previously [2]. The absence of significant grain growth in the DS lead studied in the previous work was probably due to the thinness of the samples, 1.63 mm (0.064 in.), which permitted the fatigue machine to apply only moderate or small strains to specimens (i.e. to strain amplitudes $< |\pm 0.09\%|$).

3.2.2. *Vacuum Tests*

Examination of the present DS lead specimens fatigued in vacuum showed that at about 25% of the life, small areas not much bigger than the grain size were present in which sheet-like extrusions had developed. By about 50% of the life, these areas had grown to a size which was several orders of magnitude greater than the original grain size (fig. 4). One particular area was noted which was 0.5 mm wide, and 11 mm

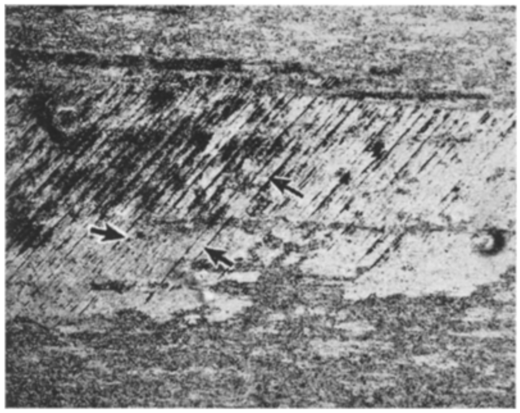

Figure 5 An area showing deformation markings (arrows) in a large grain revealed by repolishing DS lead specimen after $1.2 \times .0^7$ cycles of a strain of $\pm$ 0.140% in vacuum (× 103).

in the direction of the principal specimen (or extrusion) axis. Repolishing one of the vacuum-fatigue specimens at about half the life revealed that the clusters of extrusions coincided with areas in which extensive grain growth had taken place (fig. 5).* The largest of these grains could be seen with the unaided eye. Comparison of micrographs of the same area taken before and after repolishing showed that extrusions grew at deformation markings such as those shown in fig. 5. These deformation markings and extrusions were similar in many ways to the slip bands and extrusions formed during the fatigue of pure metals and alloys.

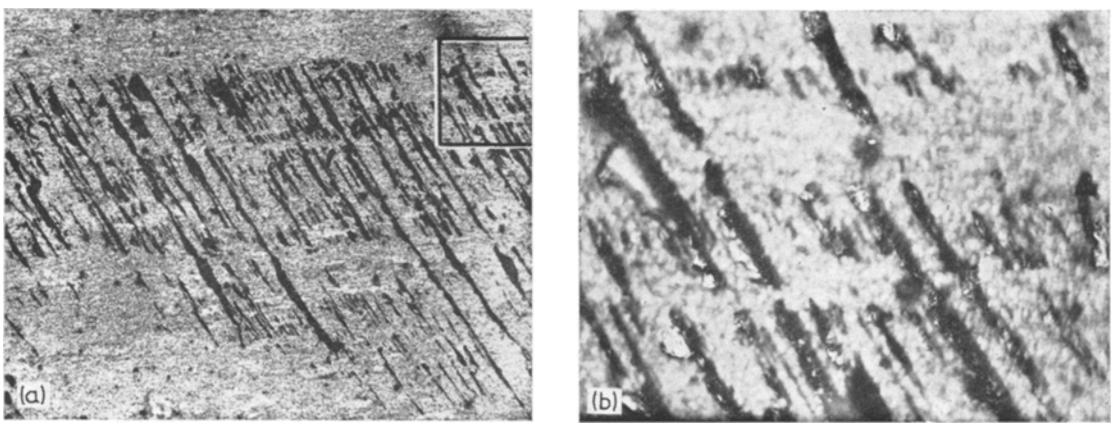

Figure 4 (a) Extrusions from surface of DS lead specimen fatigued for 10^7 cycles in vacuum at a strain of $\pm$ 0.140% (× 140). (b) Area marked in (a) shown at a higher magnification (× 630).

*These large grains were not present in the original specimens: this is known because specimens were chemically polished and examined before testing.

The overall direction of crack-propagation in the vacuum-tested DS lead specimens was, like that in the air tests, at 90° to the specimen axis, but on a micro-scale the cracks were serrated, with sections parallel to the deformation markings which were usually at an angle of about 45°. Other sections of the crack appeared to have propagated by a plastic tearing process.

4. Discussion

The DS lead specimens had fatigued lives in vacuum which were about 9 times those in air, at a strain-amplitude of $\pm 0.145\%$. This increase in life is similar in magnitude to that found for pure lead under the same test conditions [1] and suggests that similar corrosive effects due to oxygen occur in both cases. It is suggested that the superior fatigue resistance of the DS lead over that of pure lead is due to (i) the difference in grain size (2×10^{-1} cm for lead and 2 to 8×10^{-4} cm for DS lead), and (ii) the stability of the fine grain structure and oxide dispersion in DS lead under cyclic stressing. Forsyth [7] has pointed out that if the tensile strength changes with grain size then the fatigue strength changes in the same direction. Lund *et al* [8] reported that the tensile strength of DS lead increased with decreasing grain size according to a Hall-Petch type relationship. The greater fatigue-resistance of the fine-grained DS lead compared with that of pure lead is therefore in accord with the trend expected from the grain-size dependence of the tensile strength.

In both air and vacuum-tested DS lead specimens, observations showed that crack initiation was associated with areas of local grain growth. These areas were presumably subjected to a higher than average local stress or were softer than the surrounding matrix and therefore more favourable for crack initiation. After crack initiation had occurred in the air-tested specimens, cracks propagated along an intercrystalline path without the need for further grain growth (fig. 3).

The most marked grain growth was found in the vacuum-tested specimens where the rate of growth was highest along the principal specimen (or extrusion) axis. There appeared to be no general redistribution of oxide particles during the growth process. This suggests, in the light of the work of Ashby and Palmer [9], that the driving force for boundary migration was large and sufficient to pluck a boundary free from oxide particles whenever the boundary was temporarily caught up on them.

The similarity of the deformation markings in the DS lead to the soft fatigue slip bands in pure metals suggests that the work-hardening stage may not have developed to a significant extent because a high level of work-hardening would tend to suppress further dislocation activity. A low level of hardening appears to be consistent with the steady extrusion of material from these bands and with the bands being favoured paths for crack propagation.

The change from an intercrystalline mode of failure of specimens fatigued in air to the mixture of transcrystalline cracking and plastic tearing in the specimens fatigued in vacuum suggests that the difference in energy between intercrystalline and transcrystalline paths is small in vacuum and large in air at atmospheric pressure. This change in failure mode is similar to that of pure lead under corresponding environmental conditions [10].

The observations suggest that the stability of the dispersion of oxide particles is a very important factor in the high fatigue resistance of DS lead. The observations also indicate the likely importance of stress concentrations at defects in the material and of inhomogeneities in the dispersed oxide since these may increase the likelihood of grain growth with consequent loss of fatigue resistance.

5. Conclusions

(i) Dispersed-oxide-strengthened lead had a greater fatigue resistance than pure lead in air and in vacuum environments. The improved fatigue resistance was mainly due to the mechanical strengthening effects of the dispersed oxide.

(ii) The dispersed-oxide-strengthened lead was liable to fatigue owing to oxygen corrosion. The ratio of the life in vacuum to that in air was ~ 8.5 at a strain amplitude of $\pm 0.145\%$.

(iii) In both the vacuum and air-fatigue tests, crack initiation was associated with grain growth which occurred without a marked redistribution of oxide particles.

(iv) Fatigue crack propagation occurred along an intercrystalline path in specimens fatigued in air and by a mixture of transcrystalline cracking and plastic tearing in the specimens fatigued in vacuum.

(v) The vacuum-tested specimens exhibited numerous sheet-like slip-band extrusions.

(vi) The stability of the dispersion of oxide particles and the grain structure appear to be important factors in the high fatigue resistance of DS lead.

Acknowledgements

The author would like to thank Dr S. Radtke of the International Lead Zinc Organisation for arranging the supply of material and Professor M. E. Hargreaves and Professor H. W. Worner for the provision of laboratory facilities. He would also like to express his appreciation to the Broken Hill Associated Smelters Pty Ltd for their financial support of the work.

References

1. K. U. SNOWDEN, *Acta Metallurgica* **11** (1963) 675.
2. *Idem*, *J. Mater. Sci.* **2** (1967) 324.
3. D. H. ROBERTS, N. A. RATCLIFF, and J. E. HUGHES, *Powder Metallurgy* (Autumn 1962) p. 132.
4. K. U. SNOWDEN, *J. Sci. Instr.* **41** (1964) 470.
5. *Idem*, *Phil. Mag.* **6** (1961) 321.
6. H. K. WORNER and H. W. WORNER, *J. Inst. Mat.* **66** (1940) 45.
7. P. J. E. FORSYTH, "The Physical Basis of Metal Fatigue" (Blackie and Son Ltd, 1969).
8. J. A. LUND, E. G. VON TIESENHOUSEN, and D. TROMANS, Proc. Second International Conference on Lead (1965), p. 7. Pergamon Press; Oxford.
9. M. F. ASHBY and I. G. PALMER, *Acta Metallurgica* **15** (1967) 420.
10. K. U. SNOWDEN, *Phil. Mag.* **14** (1966) 1019.

Received 15 March and accepted 13 May 1971.