Microplasticity and fatigue of some magnesium-lithium alloys

Part 1 Tensile microplasticity

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The tensile microstrain behaviour of a series of magnesium-lithium alloys, with lithium contents up to 12.5 wt %, has been studied. The strain hardening exponents in the microstrain region were found to depend on the lithium content, the increase in which led to a change in crystal structure from hcp to bcc. The onset of cross-slip in the bcc alloys was found to sharply reduce the strain hardening capacity of the materials.

1. Introduction

Plastic strain occurs in most materials at stresses below the accepted engineering yield stress. It is also known that the magnitude of the cyclic plastic strain which is responsible for fatigue failure at lives in excess of 10⁵ cycles is very small and is comparable in magnitude with these preyield plastic strains. This suggests that study of the pre-yield plasticity of a metal may provide information concerning the processes which contribute to its failure in fatigue. In order to explore this idea, a study has been made of the unidirectional and cyclic plastic strain behaviour of a series of magnesium-lithium alloys at the microstrain level. This paper reports the unidirectional behaviour, the cyclic behaviour being reported elsewhere [1].

Throughout this work, microplastic strains are defined as those plastic strains which are between 10^{-6} and 10^{-3} . The value of stress at which plastic strain is first detected is a function of the strain sensitivity of the strain measuring technique. It is for this reason that in this work the term microyield stress (MYS) is used as a reference point, being the stress to produce 1×10^{-6} plastic strain.

Microplastic strains are produced by the movement of relatively few dislocations within a material, the driving force for the dislocation motion being the resolved shear stress on the slip plane. The amount of dislocation activity will, in part, depend on the nature of the slip planes available which, in turn, is a function of the crystallographic structure of the metal. This implies that the microplastic behaviour of metals will be structure-dependent.

An opportunity for studying this aspect of microplasticity is presented by the magnesiumlithium alloy system. The addition of small amounts of lithium to magnesium has the effect of increasing its ductility owing to an increase in the number of active slip systems, whilst still maintaining an hcp structure. However, for lithium concentrations in excess of 10 wt % the structure changes from hcp to bcc, the latter giving ductile materials with many more slip systems available. A series of four alloys was made up ranging from 1.2 to 12.5 wt % lithium, two of which had an hcp structure, one a mixed hcp and bcc structure and one a bcc structure. Together with pure magnesium, these provided a range of unidirectional and cyclic stress-strain behaviour.

2. Materials and experimental procedure 2.1. Materials

The magnesium and magnesium-lithium alloys were supplied in the form of 0.375 in. diameter extruded rod, 99.5% pure. The compositions of the alloys are given in Table I, and the equilibrium diagram for the magnesium-lithium system is given in Fig. 1.

The 8.4% lithium alloy in the cast state would consist of a primary beta structure in a eutectic of alpha + beta. However, during extrusion, this structure was very heavily worked and, after annealing, resolved itself into a simple structure of elongated alpha and beta grains.

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Figure 1 The equilibrium diagram for the magnesium-lithium alloy system.

| Wt % lithium | Annealing temperature (°C) | Grain diameter (mm) | Vickers hardness | Specific gravity | Crystal structure |
|-----------------|-------------------------------|------------------------|---------------------|------------------|-------------------|
| 0 | 400 | 0.05 | 36 | 1.74 | hcp |
| 1.2 | 300 | 0.03 | 36 | 1.71 | hcp |
| 4.2 | 300 | 0.03 | 37 | 1.59 | hcp |
| 8.4 | 300 | 0.03 | 45 | 1.48 | hcp + bcc |
| 12.5 | 100 | 0.14 | 36 | 1.35 | bcc |

TABLE I Data for magnesium-lithium alloys

There was no eutectic structure present.

Specimens 50 mm \times 6.25 mm diameter for tensile testing were machined from the as-received rod, the final cuts never exceeding 0.025 mm. All the specimens were longitudinally polished with fine emery to remove the machining marks and then given a brief chemical polish in 10%nitric acid for magnesium and 15% hydrochloric acid for the magnesium-lithium alloys. They were then sealed in pyrex tubes under vacuum and annealed for 2 h. The annealing temperatures, together with the Vickers hardness, grain size and specific gravity, are given in Table I. After annealing, the specimens were again chemically polished to remove traces of oxide which may have formed. Owing to the fact that the grain size of the magnesium-12.5% lithium alloy was 0.14 mm in the as-received state, this alloy was only given a stress relieving anneal at 100°C to avoid the possibility of further grain growth.

2.2. Test procedure

All the tensile tests were carried out in a vertically mounted Monsanto tensile machine, using the standard spherically mounted specimen grips. Strain gauges were bonded to the load measuring beam of the machine to give an electronic readout of the load. The machine was calibrated by dead loading and was found to be linear to better than 0.1%.

Strain was measured using metal foil strain gauges, temperature compensated for magnesium alloys, which were bonded to the surface of the specimen with Eastmann 910 adhesive. Two gauges were bonded to each specimen, diametrically opposite to each other, and were wired in parallel to reduce the effects of any bending during loading.

Taking into account the possible errors in the load and strain measuring systems the maximum total error in the stress and strain values could be approximately 2%, but in practice is likely to be much less than this. Despite this possible error the system gave stress-strain results which were reproducible to better than 1% using a steel dummy specimen.

3. Results

3.1. Macroscopic stress-strain curves

Stress-elongation curves to fracture were plotted for all the alloys and the results are shown in Fig. 2. From these curves it can be seen that for magnesium and the 1.2% lithium alloy the effect of adding a small amount of lithium to magnesium gives the expected increase in yield strength (0.1% offset) and rate of strain hardening which are known to accompany solid solution alloying. However, as more lithium is added to reach 4.2% the yield is apparently reduced and the rate of strain hardening is also lowered, the stress-strain curve being below that for pure magnesium. This is in agreement with the work of Hauser et al [2] who found that increasing the lithium content led to the introduction of prismatic slip in addition to the basal slip which occurs in pure magnesium. The presence of lithium reduces the critical resolved shear stress for prismatic slip and this in turn reduces the offset yield by allowing larger amounts of plastic strain to occur at lower stresses. The change in structure from hcp to bcc on going to the 8.4 and 12.5% lithium alloys was accompanied by an increase in ductility. A discontinuous yielding (Portevin LeChatelier) effect was found to occur in the 4.2% lithium alloy and is shown by a broken line in Fig. 2. The values of ultimate tensile strength and the elongation to fracture for the alloys are given in Table II.

3.2. Stress and strain in the microstrain region

In an attempt to demonstrate differences in the behaviour for the alloys in the microstrain region, stress-strain curves were plotted for each material to a strain sensitivity of 10^{-6} . Each curve was condensed on to one sheet of paper (10 in. \times 13 in.) by using the back-off facility on the X-Y recorder. Fig. 3 shows a typical set of experimental plots, reduced for reproduction. Proceeding from left to right, the stress-strain curve is built up by adding the bottom of each successive curve to the top of the previous one. All the curves were plotted at a strain rate of 10^{-5} in. in.⁻¹ sec⁻¹. For each plot, S_m represents



Figure 2 Stress-elongation curves for the magnesiumlithium alloys. The elongation to failure for magnesium-8.4% lithium was 33% and for magnesium-12.5% was 45%.

the stress at which 10^{-6} plastic strain was observed (MYS). It can be seen from these curves that for magnesium and the 1.2% lithium alloy, the MYS is low and plastic strain builds up progressively. On the other hand, the 4.2, 8.4 and 12.5% lithium alloys have a higher MYS and show a well-defined yield behaviour.

Table II gives the microyield stress values derived from these curves together with the stress increment (above the MYS) which is required to develop 10^{-3} plastic strain.

The MYS for magnesium-4.2 % lithium may be influenced by the Portevin LeChatelier effect which was found to be present in this alloy. The existence of this effect implies that dislocations are capable of being pinned by the solute atoms and will require additional stress to release them. A sudden release of dislocations could lead to

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|-----------------|------------------------------|----------------------------|--|--|--|
| Wt % lithium | UTS (MN m ⁻²) | Elongation to fracture (%) | Microyield stress (MN m ⁻²) | Stress for 10 ⁻³ plastic strain (MN m ⁻²) | Stress increment for 10 ⁻³ plastic strain (MN m ⁻²) |
| 0 | 172 | 4.5 | 3.5 | 69 | 66 |
| 1.2 | 186 | 14.0 | 10 | 110 | 100 |
| 4.2 | 159 | 7.5 | 48 | 74 | 26 |
| 8.4 | 131 | 33.0 | 35 | 90 | 55 |
| 12.5 | 110 | 45.0 | 41 | 66 | 24 |

TABLE II Mechanical properties of the magnesium-lithium alloys



Strain

Figure 3 High sensitivity stress-strain plots for the magnesium-lithium alloys. Beginning in the left hand bottom corner of each plot, the stress-strain curve is built up by adding the lower end of the second curve to upper end of the first.

the large amount of strain per unit stress following the MYS.

It is seen from the plots in Fig. 3 that for a given external strain-rate, there is a transition in the rate at which plastic strain occurs. This suggests that there are two regions in the stress-strain curves for the magnesium alloys at strains below 10^{-1} , the transition from one region to the other occurring between 10^{-3} and 10^{-2} total strain. The first of these is the microstrain region.

It was found that the microstrain region of the stress-strain curves could be expressed in the form

$$S - S_{\rm m} = k(e_{\rm p})^n \tag{1}$$

where S is the stress, S_m the microyield stress, e_p the plastic strain and k and n are constants. The values of k and n were obtained by plotting log $(S - S_m)$ against log (e_p) . Fig. 4 shows a typical example of the result of such a plot for the alloys. Table III gives the values of the constants k and n obtained for the alloys, each value being the mean of several determinations. Over the range of plastic strain for which magnesium and 1.2% lithium alloy have a single value of n, there are two values for the other alloys as seen in Table III. At stresses just above the MYS the k and n values are high, reducing to a lower value on increasing the stress even further.



Figure 4 The stress in excess of the MYS $(S-S_m)$ plotted against the plastic strain (e_p) for the magnesium-lithium alloys.

TABLE III Constants from Equation 1

| Wt% lithium | k(MN m ⁻²) | n |
|-------------|------------------------|------|
| 0 | 2 340 | 0.51 |
| 1.2 | 3 450 | 0.50 |
| 4.2 | 28 300 | 0.70 |
| | 138 | 0.23 |
| 8.4 | 131 000 | 0.80 |
| | 240 | 0.23 |
| 12.5 | 9 700 | 0.66 |
| | 83 | 0.22 |

3.3. Anelasticity

The anelastic behaviour of the magnesiumlithium alloys was studied by plotting the initial portion of the stress-strain curve for each material, the specimen being unloaded and reloaded at intervals throughout the test, the results being shown in Fig. 5.

For magnesium, both the loading and unloading lines are curved, the unloading portion indicating that anelastic strain occurs during loading. This anelastic effect softens the material in terms of the next loading half cycle, as seen by the fact that the material is able to achieve the same strain with respect to the origin for a lower stress than previously. It was found that the envelope of the tips of the loading-unloading loops lay on the continuously loaded stress-strain curve provided that the loops were plotted at stress intervals of not less than 7 MN m⁻². Under these conditions the material behaved in a way which did not reflect its previous stress cycling history.

The behaviour of the 1.2% lithium alloy was very similar to that of magnesium, the only differences being that the amounts of strain for



Figure 5 Load-unload plots for annealed magnesiumlithium alloys.

given stress levels were lower than for magnesium. This is a result of the solid solution strengthening which occurs on adding small amounts of lithium to magnesium.

The mechanism which gives rise to the Portevin LeChatelier effect in the 4.2% lithium alloy appears to influence its anelastic behaviour. After unloading and then reloading, the stress had to be increased in excess of the previous maximum before flow could be once again induced in the material. Even when unloading from large plastic strains very little anelastic strain was observed as shown by the loops in Fig. 5. This suggests that once pre-strained, this alloy is very resistant to further dislocation motion.

The plots for the 8.4 and 12.5% lithium alloys are very similar in nature, except that a small upper yield was observed on reloading the 12.5% lithium alloy as shown in Fig. 5. As with the magnesium-4.2% lithium alloy, a very small amount of anelasticity was observed in these materials at total strains of less than 10^{-2} .

3.4. Tensile hysteresis

Tensile hysteresis loops were plotted for magnesium and each of the magnesium-lithium alloys in the microstrain region. Magnesium showed a very marked hysteresis behaviour and was studied in detail. Difficulties were encountered with the 4.2, 8.4 and 12.5% lithium alloys owing to the very small amounts of anelasticity observed in the microstrain region.

On loading an annealed specimen of magnesium to stresses less than 3.0 MN m⁻², no detectable plastic strain resulted as shown by the fact that the subsequent unloading line coincided with the loading line and no residual strain was observed. The microyield stress, which is defined as the stress to give 10⁻⁶ residual strain on unloading an annealed specimen, may sometimes be preceded by hysteresis. Residual strain will not be observed if the hysteresis loops are closed for the annealed material, as in the case of beryllium studied by Bonfield [3]. However, the value of MYS obtained by this method agrees with the value found by measuring the deviation from linearity of the stressmicrostrain curve in Fig. 3.

On first loading and unloading to a stress in excess of the MYS, residual strain was always observed. However, on repeating the load-unload sequence there was found to be a stress above the MYS from which, when unloading, there was no residual strain at a strain sensitivity of 10⁻⁶. In other words, a closed hysteresis loop existed. On going to higher stresses and unloading, the loops failed to close and residual strain was once again present in the material. The highest stress at which a closed loop was observed was a function of the previous maximum strain. This means that closed loops could not be produced in magnesium unless the specimen was pre-strained. This finding is in agreement with the work of other authors [12].

The anelastic limit (S_a) is defined as the highest stress level at which a closed hysteresis loop is formed during stress cycling. Closure was considered to have occurred when the residual strain in any load-unload cycle was less than 10^{-6} . Fig. 6 is a plot of the anelastic limit for magnesium plotted against the plastic part of the pre-strain. It is seen that initially there is no increase in the anelastic limit up to pre-strains of 10^{-3} , but thereafter it increases approximately linearly with pre-strain.

In Fig. 6, the anelastic limit for the 1.2%lithium alloy is also plotted. There is an initial rise in the value of S_a with pre-strain up to 5×10^{-4} after which it is constant until a prestrain of 2×10^{-3} , when it tends to increase once again. The plateau and the following rise in the plot are very similar to the curve for magnesium. The beginning of the increase in S_a at a plastic strain of 2×10^{-3} again corresponds



Figure 6 The anelastic limit (S_a) plotted against the plastic part of the pre-strain for magnesium and the 1.2% lithium alloy.

to the transition into the macrostrain region of the tensile stress-strain curve. This suggests that the process which is responsible for the increase in anelasticity with pre-strain may be connected with the change in slip characteristics of the material as the pre-strain increases.

Considerable problems were experienced when trying to establish values of the anelastic limit for the 4.2, 8.4 and 12.5% lithium alloys and accurate results were not obtained. At the prestrains at which measurable anelasticity was expected, the incidence of strain gauge failures was very high. This was owing to the very marked yield behaviour of these alloys when compared with magnesium and the 1.2% lithium alloy.

4. Discussion

The experiments carried out and reported here were designed to compare the behaviour of the alloys under as near as possible identical testing conditions. No attempt has been made, therefore, to use the results to formulate quantitative mechanical deformation mechanisms to explain their behaviour.

It should be pointed out that the microyield stress (MYS) used in the present experiments is neither the elastic limit nor the friction stress used by other workers [3]. It is merely the stress to produce an arbitrary plastic strain of 10^{-6} .

Whilst no fundamental significance can be placed on the values of the MYS obtained in these tests, they do give an indication of the difficulty of initiating and propagating dislocations. Taking magnesium-1.2% lithium as an alloy representing an hcp structure and magnesium-12.5% lithium a bcc structure, Table II shows that the MYS for the 12.5% lithium alloy is a factor of 4 greater than that for the hcp alloy. This means that mobilizing sufficient dislocations to give 10^{-6} plastic strain (the MYS) is more difficult in the bcc alloy. However, Table II also shows that to produce 10^{-3} plastic strain in excess of the MYS requires 4 times the stress increment in the case of the 1.2% lithium alloy (hcp). The implication is that creating the first few active dislocations is difficult in the bcc material, but once created propagation and multiplication are very much easier than for the hcp alloy. The other alloys follow this trend.

The logarithmic plots of stress against plastic strain for the microstrain regions of the alloys again show the differences in behaviour. Below 10⁻³ plastic strain there are two stages in the microstrain response for the 4.2, 8.4 and 12.5%lithium alloys whereas magnesium and the 1.2%lithium alloy have only one stage. In the case of the last two, the slope of the line is approximately 0.5 as predicted by the Brown and Lukens [4] theory. Their derivation of a parabolic relationship between stress and strain in the microstrain region was based on the idea of exhaustion hardening in which the grain boundaries limit the amount of slip from each dislocation source. They further assume that there is a homogeneous distribution of active dislocations with a single activation shear stress. However, a number of workers have produced evidence to show that the dislocation distribution within a material under microstrain conditions is far from homogeneous [5-8]. In contrast, Billelo and Metzger [9] carried out a similar analysis assuming a non-uniform dislocation distribution with a range of activation shear stresses and still arrived at a parabolic stressstrain relationship. The fact that two completely different approaches lead to the same result renders measurements of the slope of doubtful value in formulating possible deformation mechanisms in the absence of additional information from electron microscopy. Nevertheless, the microstrain regions of the stress-strain curves for beryllium [5], iron [10], nickel [10], iron-3% silicon [10], rolled silver [11], copper [4], and zinc [4] have been fitted to a parabolic relationship.

Perhaps more significant than the parabolic relationship for magnesium and the 1.2% lithium alloy is the difference between these materials and the other alloys. The two-stage microstrain region found by Bonfield and Li [5] in beryllium, and Billelo and Metzger [9] in

copper, was also found for the 4.2, 8.4 and 12.5% lithium alloys in this experiment. Bonfield and Li concluded that the onset of the second stage coincided with the initiation of cross-slip which they observed with an electron microscope. The present experiments on the magnesium-lithium alloys support this idea in that the two-stage behaviour occurs in those alloys in which cross-slip is more easily induced.

The materials studied here appear to be divided into two behavioural groups, the first containing magnesium and the 1.2% lithium alloy and the second the remaining alloys. This is shown by the results of the load-unload, tensile hysteresis and anelasticity experiments described above.

5. Conclusions

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1. A series of magnesium-lithium alloys was prepared with lithium concentrations up to 12.5%. Magnesium and the low lithium alloys had an hcp structure and the higher alloys a bcc structure.

2. Tensile microstrain and hysteresis experiments were carried out on these materials to strains not exceeding 10^{-3} . The strain hardening rates were determined in this region.

3. The results show that for the hcp alloys in which cross-slip is limited, there is a uniform hardening rate throughout the microstrain region. For the bcc alloys, in which cross-slip is possible, a two-stage microstrain behaviour was observed in which there is a sudden lowering of the strain hardening capacity, the transition being attributed to the onset of cross-slip. 4. Tensile hysteresis experiments show that in the bcc alloys the anelastic strain observed on unloading stressed specimens is considerably reduced for those materials in which cross-slip occurred.

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References

- 1. R. E. LEE and W. J. D. JONES, J. Mater. Sci. 9 (1974) 476.
- 2. F.E. HAUSER, C.D. STARR, L. TIETZ and J.E. DORN, Trans. ASM. 47 (1955) 102.
- 3. W. BONFIELD and C. H. LI, Acta. Metallurgica 13 (1965) 317.
- 4. N. BROWN and K. F. LUKENS, *ibid* 9 (1961) 106.
- 5. W. BONFIELD and C. H. LI, *ibid* 12 (1964) 577.
- 6. J. MAN, M. HOLZMANN and B. VLACH, *Phys. Stat.* Sol. 19 (1967) 543.
- 7. J. C. SUITS and B. CHALMERS, Acta. Metallurgica 9 (1961) 854.
- 8. P. J. WORTHINGTON and E. SMITH, *ibid* **12** (1964) 1277.
- 9. J. C. BILLELO and M. METZGER, *Trans. Met. Soc.* AIME 245 (1969) 2279.
- 10. W. D. BRENTNALL and W. ROSTOKER, Acta. Metallurgica 13 (1965) 187.
- 11. W. BONFIELD, ibid 13 (1965) 551.
- 12. J. M. ROBERTS and D. E. HARTMANN, *Trans. Met.* Soc. AIME 230 (1964) 1125.

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