Pseudoelastic behaviour of cast magnesium AZ91 alloy under cyclic loading–unloading

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Abstract

Large stress–strain hysteresis loops are observed under cyclic loading after a small plastic prestrain. Loops have been observed in sand-cast material in a variety of tempers under tension or compression, and in high-pressure die-cast material with different cross-section thickness tested in tension. The loops are first observed after a nucleation strain of between 0.001 and 0.01% and grow to a maximum width after 1–2% plastic strain, becoming slightly narrower afterwards. When fully developed, the loops add a large (0.3–0.45%) pseudoelastic strain to the elastic strain, effectively decreasing the elastic modulus of the alloy by up to 70%. In sand-cast material of a given temper, the effects tend to be more pronounced in compression than in tension. Further, the effect is slightly larger in die-cast or aged sand-cast as compared to as-cast sand-cast material. The phenomenon is discussed in terms of the partial reversal of \{1 0 \bar{1} 2\} twins upon unloading.

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1. Introduction

Pseudoelastic behaviour associated to elastic twinning and leading to large hysteresis loops has often been observed in polycrystalline metals cycled in tension or compression after being plastically deformed [1–4]. Jones and Munro [1] reported hysteresis loops in uranium, ascribing the effect to reversible twinning. Reed-Hill et al. [2] observed similar loops in zirconium prestrained at low temperature, ascribing them to reversible movement of \{1 1 \bar{1} 2\} twin boundaries. Gharghouri et al. [4,5] used in situ neutron diffraction to show that the anelastic loops in pure Mg and a Mg–8%Al alloy tested in cyclic tension and compression are due to \{1 0 \bar{1} 2\} twins, (with some contribution from \{1 0 \bar{1} 1\} twins in cyclic tension due to the strong texture of their materials), which grow as the material is stressed, and partially revert upon unloading.

In the most general sense [6], any non-linearity in the unloading curve can be referred to as pseudoelasticity. Upon reloading, the non-linearity
leads to a hysteresis loop. Pseudoelasticity may recognise several origins such as reversible movement of dislocations [3], twinning, and stress induced phase transformations [6,7]. Twinning pseudoelasticity is caused by the reversible movement of twin boundaries. The position of the twins in the deformed state is not stable, and a driving force causes them to return back upon unloading [6]. In the ordered alloys Ti$_{50}$Ni$_{47}$Fe$_{3}$ and Ti$_{49}$Ni$_{51}$, under certain conditions, mechanical twinning accompanied by extensive shuffling of atoms [6,7], often referred to as pseudo-twinning, results in hysteresis loops upon loading–unloading. Another related phenomenon, but in a much larger scale, is that of superelasticity of shape memory alloys. Superelasticity is observed when the material is mechanically tested within a critical temperature range in which martensite plates are made stable with the application of a stress, but become unstable when the stress is removed [8,9].

The development of a large pseudoelastic strain when the material is plastically deformed poses a problem to engineers trying to base designs on a constant value of the elastic modulus. If the pseudoelastic effect is large, conventional formulae relating stress and strain may become invalid, and the use of finite elements may be required [6,10]. The fatigue response at high stress [11], as well as the energy absorption characteristics [2,3,11], may also be affected by the cyclic movement of twin boundaries. Thus, understanding the strain dependence of the loading–unloading behaviour may be of interest from the point of view of both static and dynamic mechanical design. This seems especially relevant in the case of safety related components which may suffer repeated small over-loadings, such as steering wheels in motor vehicles, or when the fabrication process involves plastic deformation.

The main objective of this work is to describe the development of the cyclic loading–unloading hysteresis loops in cast AZ91 alloy under uniaxial tension or compression in detail. The effect of the microstructure on the development of the hysteresis loops is considered through the evaluation of sand-cast material in different tempers and of high-pressure die-cast material with different section thicknesses. A preliminary study of the phenomenon in pure Mg has been published elsewhere [12].

2. Method

2.1. Materials

Commercial AZ91 alloy was used for the study. High-pressure die-cast (hpdc) specimens were cast-to-shape using a 250 tonne cold-chamber machine. The sand-cast (sc) specimens were machined out of cast plates produced as described in another study [13]. The grain size of the sc material was ~130 µm [13]. The sc specimens were solution heat treated for 20 h at 413 °C and quenched in water (T4 temper). Subsequently, some of these specimens were aged for 16 h or 120 h at 165 °C (labelled T6 and 120 h, respectively). The T6 temper is recommended by the ASM Metals Handbook [14]. Note, however, that as peak ageing in this alloy does not occur until after 100–150 h at this temperature [13], the material aged for 120 h is closer to peak strength. The hpdc specimens were tested as-cast.

2.2. Mechanical testing

Flat tensile specimens with a rectangular cross-section of 4 × 5 mm$^2$ and a 25 mm gauge length were used for most of the tensile testing of sc material. For compression testing, and some of the tensile testing, cylindrical specimens, of gauge length 25 mm and 6 mm in diameter, were used. Compression was limited to 4% strain to avoid buckling effects. The hpdc specimens were tested only in tension and had a gauge length of 80, 12 mm wide, and 1, 2 or 6 mm thick. Testing was carried out in a screw-driven machine at a cross-head speed of 1 mm/min. A knife-edge extensometer was attached to the specimen’s gauge length and digital output files of the flow curves were stored for analysis.

2.3. Metallographic observations

Small bars 50 mm long and 3 × 10 mm$^2$ cross-section were polished to 1 µm finish on one of the
narrow sides and deformed in a small three point bend rig mounted on the stage of an optical microscope for observation in situ. The bars were loaded until twins first appeared and then slightly unloaded to observe the reversal. The load was repeatedly cycled to observe the successive expansion and reversal of existing or newly formed twins as the peak load was increased.

3. Observations

3.1. Mechanical testing

Fig. 1 shows hysteresis loops in hpdc AZ91 alloy tested in cyclic tension. If the specimen is unloaded after some applied plastic strain, the unloading strain is much larger than expected under linear elastic behaviour. When the specimen is reloaded, the flow curve describes a closed loop whose width increases with the amount of plastic prestrain. Figs. 2 and 3 show similar cycles for sc specimens.

Figs. 1–3 show that once fully developed, the loops are generally similar in size and shape for
all materials, tempers or loading direction. The loops are reversible for a small number of cycles. This is shown in Fig. 2 on the specimen labelled T4, which was subject to multiple loops after a strain of 3.5%. Little change in the width of the loops is observed during the first loading–unloading cycles, although after some 50 cycles, the width decreases to about half. This resembles the behaviour of shape memory alloys in which the size of the hysteresis loops decreases with the number of stress–strain cycles [15,16]. Fig. 2 also shows that cycling does not affect the overall plastic behaviour in comparison with the monotonic flow curve. Fig. 3 shows that the loops are slightly wider in compression for sc material in given temper. Fig. 4 compares the monotonic tensile flow curves of some of the materials studied.

In Fig. 5, the secant or “apparent” elastic modulus (\(E_{\text{ap}}\) defined in Fig. 1) is plotted as a function of the applied (tensile or compressive) plastic strain, for all the materials tested. The experimental values have been normalised to the initial (elastic) slope of the stress–strain curve for each specimen and a GPa scale was added to the right hand y-axis such that the normalised \(E_{\text{ap}} = 1\) corresponds to the nominal value for AZ91 alloy [17], \(E = 45\) GPa.

The normalising was done to eliminate variations of up to ±1.5 GPa in the sc material caused by the different tempers, and a systematic difference of about 2.5 GPa between the sc and hpdc materials, due to the effect of porosity in the latter. A detailed study of the influence of microstructural effects on the elastic modulus of the alloys of this work has been published elsewhere [17]. Fig. 5 shows that the \(E_{\text{ap}}\)-value decreases with the plastic strain, reaching a minimum at 1–2% strain, slightly increasing afterwards. Note that hpdc materials develop the effect at a lower strain. The stronger materials, i.e. hpdc and aged sc, show the smallest decrease in \(E_{\text{ap}}\), to about 26 GPa. For given temper, the modulus of sc material systematically drops to a lower value in compression, a consequence of the loops being wider in compression (Fig. 3).

Fig. 6 shows the anelastic strain (\(e_{\text{a}}\), defined in Fig. 1) as a function of the (true) applied plastic strain for the materials of Fig. 5. The anelastic strain develops after an applied strain of about 0.001% (hpdc) or 0.02% (sc material), and saturates at between 0.3% and 0.45% after a plastic strain of 1–2%, slightly decreasing at larger strains. The effect is more pronounced at low strains in hpdc material due to the lower nucleation strain,
but the saturation value is larger (~0.45%) in sand-cast material tested in compression, a consequence, again, of the loops being wider in compression. For given loading sense, the nucleation strain is slightly smaller and the saturation anelastic strain somewhat larger in aged (T6 and 120 h) sc material. It is noteworthy that for hpdc material at low strains, the anelastic component of the strain is larger than the applied plastic strain, as can be seen from the line $\varepsilon_a = \varepsilon_p$ in Fig. 6.

Fig. 7 shows the $E_{ap}$-value as a function of the applied stress (the $E_{ap}$ values have been normalised as explained for Fig. 5). Although the overall behaviour is generally similar for all materials, the effect spreads over a range of stresses, reflecting the relative strengths of the individual materials (Fig. 4). As for Fig. 5, the decrease in $E_{ap}$ is less for the stronger materials, i.e. the sc material aged for 120 h and the 1 and 2 mm thick hpdc, while, for given temper in sc material, the effect is more pronounced in compression.

3.2. Observations in situ

The micrographs of Fig. 8 show twins in sc and hpdc specimens deformed in bending. Some of the
twins partially reverted when the applied load was slightly decreased. Twins that reverted became either slightly narrower or shorter with the decrease in applied load. In the case of very thin twins, complete reversal seemed to occur, but upon reloading, the twin reappeared on the same location. Generally speaking, reversal was observed to occur through reversible movement of the twin boundaries, as described by Reed-Hill et al. [2] for zirconium.

4. Discussion

Several features of the phenomenon suggest that its magnitude is largely controlled by the applied strain, namely (Fig. 6): the hysteresis loops develop only after a nucleation plastic strain, the anelastic strain increases monotonically with the applied strain, and the maximum value of the anelastic strain is observed at the same applied strain (1–2%), for all the materials and for tension or compression, despite the wide range of flow strengths shown in Fig. 4.

For the given strain, the phenomenon appears to be stress controlled: unloading and reloading (without further plastic strain) cause the same amount of pseudoelastic strain (repeated cycles in Fig. 2); the loops are wider, hence the effects are generally larger in compression, indicating an intrinsic load asymmetry of the phenomenon.

In addition, if one considers that the grain size is much smaller in hpdc than in the sc material, the smaller nucleation strain and the generally larger anelastic strain observed for hpdc material (Fig. 6) suggests that the pseudoelastic effects are enhanced by a small grain size.

Following Gharghouri et al. [4,5], these observations will be discussed assuming that the effect is caused by partial reversal of {1 0 1 ̅2} twins.

4.1. Nucleation and saturation strain

Reed-Hill et al. [2] pointed out that the plastic prestrain acts as a nucleating step for the reversible {1 1 2 1} twins observed in Zr. Once nucleated, the twins expand laterally on the rather undisturbed matrix, accompanied by a small amount of dislocation plasticity, which relaxes the local accommodation stresses at the extremities of the twins, enabling further growth. Reed-Hill et al. [2] also ascribed the levelling-off of the hysteresis loop size after a plastic strain of about 1% to the combined effect of two factors: (1) saturation in the number of twin nuclei, (2) decreased mobility of the {1 1 2 1} twin boundaries due to increased dislocation activity and formation of {1 0 1 ̅2} twin nuclei. This description is generally consistent with the observations made here and accounts for the observed nucleation strain, as well as for the increase, saturation and subsequent decrease of the anelastic strain (i.e. the width of the hysteresis loops) with increasing applied strain shown by Fig. 6.

4.2. Role of the strength of the material

Reed-Hill et al. did not study alloys with a range of strengths as in the present work, but the results of Figs. 5 and 7, where the lowest $E_{ap}$-value is observed for the materials with lower flow stress, i.e., in T4 and T6 tempers, can be understood within the same context. From Fig. 1,

$$E_{ap} = \frac{E}{1 + \varepsilon_a(E/\sigma_F)}$$  \hspace{1cm} (1)

Eq. (1) shows that $\sigma_F$ is only a scale factor relating the truly elastic with the anelastic behaviour, and implies that the anelastic strain, $\varepsilon_a$, is independent of the strength of the material (as already evident from Fig. 6). Since $\sigma_F$ measures the resistance to the movement of dislocations, the lack of sensitivity of $\varepsilon_a$ to $\sigma_F$ is consistent with the idea that twinning and not dislocation plasticity is responsible for the hysteresis loops.

4.3. Load asymmetry

Due to the polar nature of twinning, in a randomly oriented polycrystalline aggregate, the fraction of grains having their c-axes favourably oriented for {1 0 1 ̅2} twinning is smaller than the fraction favourably oriented for {1 0 1 ̅2} twinning in compression [19]. Thus, any effect that depends on the activation of {1 0 1 ̅2} twins is expected to
be larger in compression [19,20], as indeed observed in Figs. 3 and 5–7.

4.4. Grain size effects

Fig. 6 indicates that the nucleation strain is smaller, and the anelastic strain generally larger, in hpdc material, whose grain size is very small (d=10 µm [18]) in comparison with sc material (d=130 µm). It is worth mentioning that similar experiments performed in pure Mg grain refined with Zr also show a generally stronger pseudoelastic effect, and from smaller strains, for small grain size [18]. This seems to contradict the generally accepted observation that twinning is more difficult in the small grained material [21]. This apparent contradiction can be solved considering the effect a small grain size may have on the number and stability of twins.

A smaller grain size normally reduces the amount of twinning [21,22] as observed in metallographic sections of predeformed samples. However, unless the grain size is so small so as to completely suppress twinning (e.g. in pure Zn [23] twinning is not observed at grain sizes below 1 µm), twins will still be observed for small grain size, although they will be limited in size, as Fig. 8 shows. For a given strain in a random polycrystalline aggregate of Mg, the total number of twin nuclei can be expected to be proportional to the number of grains favourably oriented for twinning. The latter is obviously larger for small grain size. The grain boundary area, where twins often nucleate, is also larger for small grain size, and this will also tend to increase the number of twins. On the other hand, the interfacial energy per unit volume of twinned material is likely to be larger for smaller twins, and small twins will require less accommodation at their ends by dislocation slip, with its concomitant stabilising effect. Both effects make smaller twins less stable. Thus, for a given strain, a small grain size can be expected to result in an increased number of small twins that are more prone to revert upon unloading, hence the magnified pseudoelastic strain. The parallel with shape memory alloys can be extended further by mentioning that the superelasticity of some alloys is magnified [24] with a reduction in grain size, as is the high strain amplitude fatigue resistance [25].

5. Conclusions

The development of hysteresis loops under cyclic loading has been studied in sand-cast AZ91 alloy heat-treated to different tempers, in tension and compression, and in high-pressure die-cast AZ91 alloy with different section thicknesses, in tension.

The hysteresis loops progressively develop after a plastic strain of between 0.001% and 0.02%, the smaller value observed for die-cast material. Loops reach a maximum width, as measured by the anelastic strain upon unloading, at a plastic strain of about 1–2%, slightly decreasing in width at larger strains. At constant plastic strain, the phenomenon is fully reversible for small number of loading–unloading cycles.

The maximum anelastic strain, measured as the strain in excess of the linear elastic strain upon unloading, is 0.3–0.45%. The larger values are observed in high-pressure die-cast material, and aged sand-cast material tested in compression.

When the loops are fully developed, the anelastic strain causes a decrease of up to 70% in the effective elastic modulus of the alloy. This effect is less marked in materials with higher strength.

The observed effects can be understood in terms of the partial reversal of \{10\bar{1}2\} twins upon unloading.

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References
