An evaluation of the creep characteristics of an AZ91 magnesium alloy composite using acoustic emission

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Abstract

Creep experiments were undertaken to determine the potential for using acoustic emission (AE) to monitor the high temperature deformation of an unreinforced AZ91 magnesium alloy and a similar alloy reinforced with short alumina fibers after a conventional T6 heat treatment. Samples of each material were tested to failure and the fracture surfaces of selected specimens were examined using scanning electron microscopy. The results show there is an improved creep resistance in the composite by comparison with the unreinforced alloy. It is demonstrated that the use of AE provides a sensitive procedure for monitoring the nature of the creep deformation. © 2002 Published by Elsevier Science B.V.

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1. Introduction

Magnesium alloys are attractive for use in many structural applications, especially in the automotive industry, because of their superior strength-to-weight ratios and their high impact resistance [1,2]. Furthermore, the introduction of short ceramic fibers into magnesium matrices provides an opportunity to produce metal matrix composites (MMCs) having a low density combined with a high specific stiffness and excellent creep strength. Despite the interest in these materials, and the extensive development of creep-resistant magnesium alloys based, most recently, on the MgSc and MgGd ternary systems [3], only limited information is available on the creep properties of Mg-based MMCs although this is a critical prerequisite for any successful utilization of these materials in applications at elevated temperatures.

There are a number of reports describing the creep behavior of Mg-based MMCs reinforced with short fibers and using AZ91 [4–10], AS41 [5] or QE22 [4] as the matrix alloys. The present investigation was initiated to provide more information on the creep of an MMC with an AZ91 matrix alloy and especially to critically evaluate the potential for making use of acoustic emission (AE) as a monitoring tool for characterizing creep deformation and the development of creep damage during testing. Because very slow strain rates are an inherent feature of long-term creep investigations, a series of specific tests was judiciously selected and undertaken to provide information on three separate issues: (i) the ability of fiber reinforcement to provide additional creep strengthening by comparison with the unreinforced alloy, (ii) the testing conditions required to reveal a meaningful response using AE monitoring and (iii) the effect on the AE response of testing at different temperatures.

When an MMC is subjected to mechanical loading, creep occurs in the matrix alloy through the generation and motion of dislocations and, in addition, there may be structural damage at the higher stress levels through...
debonding at the fiber–matrix interfaces or fracturing of the individual fibers. The process of AE denotes the generation of transient elastic waves within a material due to sudden irreversible structural changes. It is reasonable to anticipate that AE will provide a meaningful and measurable response to some deformation and damage mechanisms [11] and/or flow changes. This suggests the potential for using AE measurements to characterize the flow and damage processes occurring within the material during testing. This paper describes the use of AE to evaluate the deformation and fracture characteristics of an AZ91 alloy reinforced with 20 vol.% of alumina fibers and tested at temperatures of 473 and 583 K which are within the range anticipated for many structural applications at high temperatures.

2. Experimental procedure

An AZ91 alloy, having a composition (in wt.%) of Mg–9% Al–1% Zn, was reinforced with 20 vol.% Saffil® δ-Al2O3 short fibers by squeeze casting whereby the molten AZ91 alloy is infiltrated under pressure into short fiber pre-forms having planar isotropic fiber distributions. The fibers were 97% Al2O3 and 3% SiO2 and they had diameters of ~3–5 μm and varying lengths up to a maximum of ~150 μm. For comparison purposes, an identical unreinforced alloy was also prepared by squeeze casting. After production, parts of the batches of the reinforced and unreinforced materials were given a standard T6 heat treatment involving a solution treatment for 24 h at 685 K followed by air cooling and subsequent ageing for 16 h at 450 K. Metallographic examination of the composite in the as-cast condition revealed a two-dimensional array of fibers with the fibers arranged essentially randomly within this plane. A typical optical micrograph of the as received state is shown in Fig. 1.

Tensile specimens were machined with gauge lengths of 25.4 mm and cross-section 3 mm × 3.2 mm. The longitudinal specimen axes were within the plane containing the long axes of the fibers. All of the creep testing was conducted under conditions of constant stress using a creep machine equipped with a contoured lever arm. Tests were performed at stresses in the range from 40 to 95 MPa and at temperatures of either 473 or 583 K with the temperature controlled during each test to ±2 K. Some tests were conducted at a single stress until failure and others used progressive loading in which the stress was increased at selected time intervals until ultimate failure.

The AE signal was monitored during each creep test using a steel waveguide of cylindrical shape that fitted tightly to the specimen. This waveguide was coupled to the sample surface using heat-resistant silicon paste and steel springs, and a computer-controlled DAKEL-LMS-16 AE facility was then used to record the AE counts. A highly sensitive LB10A transducer was coupled to the conical end of the waveguide using a cyanoacrylate glue: this transducer had a sensitivity of 85 dB ref. 1 V ms⁻¹, a flat response from 100 to 500 kHz and a built-in preamplifier giving a gain of ~30 dB. As described in an earlier report [12], the DAKEL AE facility applies a two-threshold level of detection and evaluation of the AE signals. The total gain at the lower threshold level (count N C1) was about 90 dB and the total gain at the higher level (burst count N C2) was about 70 dB.

Surfaces of selected specimens were examined after fracture using a TESLA BS 343 scanning electron microscope operating at an accelerating voltage of 15 kV. Fracture surfaces were cleaned in acetone before making any observations.

3. Experimental results and discussion

3.1. Creep behavior of the unreinforced and reinforced materials

To evaluate whether additional creep strengthening is introduced through the presence of the reinforcement, Fig. 2 shows the initial portions of the creep curves for tests conducted on the unreinforced AZ91 alloy and the composite material at a stress of 40 MPa and a temperature of 473 K; both of these materials were tested in the T6 condition. It is apparent from Fig. 2 that, when the applied stress is the same on each specimen, the creep rate is substantially slower in the composite due to an apparent strengthening effect arising from the presence of the alumina fibers.

In practice, however, care must be taken in making a comparison of this type because there is experimental evidence for a threshold stress, σ0, when creep testing the same MMC as in this work [6–8]. This means in
practice that the effective stress, $\sigma_e = \sigma - \sigma_0$, acting on the composite may be significantly lower than in the unreinforced material where there is no threshold stress and $\sigma_0 = 0$. In the MMC used in these experiments, an earlier analysis suggested the occurrence of load transfer [8] whereby part of the external load is transferred to the reinforcement. It is instructive to note that experiments on numerous Al alloys and Al-based MMCs have often revealed slower creep rates in the composites even when creep data are compared at the same values of the effective stress [13–15].

3.2. The nature of the AE response in unreinforced and reinforced materials

Tests were conducted on the unreinforced alloy and on the MMC at 473 K and at the same initial level of the applied stress of 40 MPa. In each test, the stress was increased progressively at selected time intervals. The results are shown in Fig. 3 for (a) the unreinforced AZ91 alloy and (b) the composite material: again, both materials were in a T6 condition. The count rate for AE is documented along the lower axes.

Inspection of Fig. 3 shows there is a well-defined AE response throughout the total lifetime of the unreinforced alloy whereas creep of the MMC is not accompanied by a measurable AE except only at the instances associated with the stress increases and at the very highest stress increment of 80 MPa. Several AE pulses in the MMC are observed at the applied stress of 70 MPa. It is important to note the creep lifetime of the unreinforced alloy and the MMC. There is also a large difference between the count rate associated with the stress increases during creep of the unreinforced alloy and the MMC. The AE count rate change due to the stress increase in the MMC is several times lower than for the unreinforced alloy.

The presence of a measurable AE response must be associated either with the creep deformation occurring within the material or with the advent of creep damage in the form of debonding or breaking of the fibers. However, there are no fibers in the unreinforced alloy and, since there is an AE response throughout the creep lifetime of this material, it must be associated with the flow mechanism occurring within the alloy. There are several experiments showing a stress exponent of $n = 3$ for the unreinforced AZ91 alloy [9,16,17] and this suggests that a viscous glide process is dominant whereby the movement of dislocations is impeded by the presence of aluminum solute atom atmospheres. In addition, there have been similar reports of $n = 3$ in Mg solid solution alloys containing 0.8% Al [18] and 3% and 5% Al [19], respectively.

If viscous glide is the rate-controlling flow process in the unreinforced AZ91 alloy with $n = 3$, it is reasonable to assume there will be no AE response: but an AE response is anticipated at higher stress levels when the dislocations are able to break away from their solute atom atmospheres [20]. Therefore, in order to explain the experimental results documented in Fig. 3, it is...
necessary to demonstrate that flow occurs in the unreinforced alloy within the region of dislocation breakaway at stress levels as low as 40 MPa.

The breakaway stress, \( \sigma_b \), in solid solution alloys is given by an expression of the form [21]

\[
\sigma_b = \frac{W_m c}{5b^3 k T}
\]

where \( c \) is the concentration of the solute, \( b \) is the magnitude of the Burgers vector, \( k \) is Boltzmann’s constant, \( T \) is the absolute temperature and \( W_m \) is the binding energy between the solute atom and the dislocation which may be written as

\[
W_m = -\frac{1}{2\pi} \left( \frac{1 + \mu}{1 - \mu} \right) G|\Delta V_a|
\]

where \( \mu \) is Poisson’s ratio, \( G \) is the shear modulus and \( \Delta V_a \) is the difference in volume between the solute and the solvent atoms.

Eq. (1) may be solved through the use of \( \mu = 0.34 \) and \( \Delta V_a = 8.2 \times 10^{-30} \text{ m}^3 \) [22] for aluminum atoms in magnesium and with \( b = 3.2 \times 10^{-10} \text{ m} \) and \( G = ((1.92 \times 10^4) - 8.6T) \text{ MPa} \) [23] for pure magnesium and noting that the initial concentration of aluminum in the alloy is \( \sim 9 \text{ wt.\%} \) which corresponds to \( \sim 8 \text{ at.\%} \) so that \( c \approx 0.08 \). Following this procedure, the value of the breakaway stress is estimated as \( \sigma_b \approx 110 \text{ MPa} \) and this is higher than any of the applied stresses used to obtain the data in Fig. 3(a). In practice, however, it has been shown that Eq. (1) tends to overestimate the magnitude of \( \sigma_b \) by a factor of \( \sim 2 \) because it fails to include the variation in the interaction energy with the solute concentration and the influence of the different spacings between the solute atoms and the line of the dislocation [24]. The introduction of these improvements reduces the value of \( \sigma_b \) to \( \sim 55 \text{ MPa} \), and in practice an additional reduction is necessary because the T6 heat treatment leads to a fine dispersion of \( \text{Mg}_17\text{Al}_{12} \) precipitates [25, 26] which serves to deplete the concentration of aluminum atoms remaining in solid solution within the matrix and thereby it reduces the value of \( c \) in Eq. (1). The total extent of this aluminum depletion is not known at the present time but it is reasonable to conclude that all of the stresses used in Fig. 3(a), including the lowest applied stress of 40 MPa, are within the region of dislocation breakaway and therefore the occurrence of breakaway accounts for the AE response which is clearly visible throughout the test. This conclusion is consistent also with experimental creep data on the composite material where it was shown that breakaway occurred, and \( n \) increased above a value of 3, at an effective stress level of \( \sim 14 \text{ MPa} \) at the slightly higher temperature of 573 K [6].

By contrast, no AE response is visible in the composite material shown in Fig. 3(b), at least below 70 MPa, although these tests were conducted at the same temperature of 473 K and with the same lowest applied stress of 40 MPa using material subjected to the same T6 heat treatment. This difference arises because there is a significant threshold stress in the composite, and, strictly, this requires comparing the unreinforced and the reinforced materials at the same values of the effective stress acting on dislocations. No detailed information is available on the magnitude of the threshold stress, \( \sigma_0 \), in the composite material at a testing temperature of 473 K but scattered results from tests conducted at this temperature under shear conditions [6] suggest the threshold stress is of the order of \( \sigma_0 \approx 30 \text{ MPa} \). Practically the same value of the threshold stress may be obtained from results reported in other work [8]. If it is now assumed that the lowest applied stress level of 40 MPa used for the unreinforced alloy in Fig. 3(a) is probably close to the lowest stress for a breakaway condition in the AZ91 matrix alloy at a temperature of 473 K, it follows that breakaway in the composite will require an effective stress, \( \sigma_e \), of \( \sim 40 \text{ MPa} \) which is equivalent, when incorporating the threshold stress, to an applied stress of the order of \( (\sigma_e + \sigma_0) \approx 70 \text{ MPa} \). These calculations are therefore consistent with Fig. 3(b) and with the occurrence of a significant AE response in the composite only at the highest applied stress of 80 MPa.

Fig. 4 shows the appearance of the fracture surface of the composite material after testing through the progressive loading shown in Fig. 3(b). An optical micrograph showing the distribution of fibers in the perpendicular plane near the crack surface is shown in Fig. 5. It is seen that the majority of fibers are perpendicular to this plane. However, some fibers lie also in this plane. It is also seen that the distribution of fibers is not homogeneous in the volume of the specimen. Some areas of the volume are almost free of fibers and, on the other hand, in some areas the volume content of fibers is much higher than the nominal volume of 20%. A scanning electron microscopy image of the fracture surface (Fig. 4) shows no clear evidence for fiber breaking nor fiber pull-out. Some fibers lying in the perpendicular plane might indicate fiber fracture. Their breaking, however, was caused during squeeze casting of the composite material. Drozd [27] has observed similar features in non-deformed AZ91 composite.

### 3.3. Effect of increasing the applied stress and the test temperature

Identical tests were conducted on the composite material using a temperature of 473 K and an applied stress of 70 MPa for samples after the T6 heat treatment. One can expect that breakaway in the composite will occur under this applied stress. The result is shown in
Fig. 6. The character of the AE count rate changes drastically. It is apparent from Fig. 6 that the AE response is significantly greater for the sample crept at 70 MPa than after creep at 40 MPa. There is a large increase in the AE activity in the latter stage of tertiary creep immediately prior to fracture, indicating failure. The decrease in the level of the aluminum solute due to the precipitation of Mg₁₇Al₁₂ during the T6 heat treatment combined with the increase in the applied stress serve to increase the potential for dislocations to break away from their solute atmospheres and to give an increase in the AE response.

An increase in the test temperature should result in a decrease of the effective stress necessary for dislocations to break away from their solute atmospheres. Tests were
conducted on the composite at 583 K with an initial applied stress of 40 MPa after a T6 heat treatment. Fig. 7 shows a faster creep rate and a larger AE response throughout the creep lifetime of the MMC by comparison with the results obtained at 473 K. In Fig. 8 showing the fracture surface, there is a pull-out of the fibers corresponding to local debonding and the large AE response observed prior to failure in Fig. 7. The distribution of fibers in the perpendicular plane near the crack surface was similar to that shown in Fig. 5.

4. Summary and conclusions

(1) The introduction of short alumina fibers into an AZ91 magnesium alloy improves the creep resistance because of the introduction of a threshold stress that serves to reduce the effective stress acting on the material.

(2) The creep deformation may be monitored using AE. There is little or no AE response when deformation occurs by the viscous glide of dislocations with $n = 3$, but an AE response is visible at stress levels which are sufficiently high that the dislocations are able to break away from their solute atom atmospheres. There may be also a very large AE response immediately prior to failure due to a local debonding around the fibers, which is visible as a pull-out of fibers at the fracture surface.

Fig. 7. Creep curve and the AE response for the composite tested at 583 K with an initial applied stress of 40 MPa after a T6 heat treatment.

Fig. 8. Fracture surface (scanning electron micrograph) of the composite after testing at 583 K with an initial applied stress of 40 MPa after a T6 heat treatment.
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References