Creep fracture mechanisms in selected discontinuously reinforced magnesium metal matrix composites

Václav Sklenička[a], Květa Kuchařová[b] and Milan Svoboda[c]

Institute of Physics of Materials, Academy of Sciences of the Czech Republic,

Žižkova 22, CZ-616 62 Brno, Czech Republic

[a]sklen@ipm.cz, [b]kucharova@ipm.cz, [c]svobm@ipm.cz

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Abstract. This paper reviews the creep characteristics of the two representative magnesium monolithic alloys (AZ91 and QE22) and their various discontinuous composites. It was found that the magnesium AZ91 and QE22 alloys reinforced with 20 vol.% alumina short fibres exhibit an excellent creep resistance. An investigation conducted to evaluate the creep behaviour of 15 vol.% silicon carbide particles reinforced magnesium AZ91 and QE22 alloys revealed, that the creep resistance of the AZ91 alloy is increased through particle reinforcement; by contrast the creep resistance of the reinforced QE22 alloy is decreased. A comparison between the creep characteristic of unreinforced squeeze-cast AZ91 and QE22 magnesium alloys and their hybrid composites reinforced with 8 vol.% carbon short fibres and 15 vol.% SiC particulates has shown, that the creep resistance of the hybrid reinforced AZ91 alloy was considerably improved by comparison with the unreinforced matrix alloy. No beneficial effect of hybrid reinforcement on the creep resistance of QE22 was resulted.

Introduction

Short fibres, whiskers or ceramic particulates reinforced Mg based composites, which are conventionally known as discontinuous magnesium composites, represent a class of advanced materials that exhibit the attractive properties, like high specific strength and stiffness, low density, excellent castability, etc. [1-5].

The creep resistance of magnesium alloys is rather limited at temperatures above 400 K. However, a marked improvement in the creep properties of magnesium monolithic alloys can be potentially achieved through the production of composite materials where the matrices consist of conventional magnesium alloys which are strengthened through the introduction of non-metallic fibres or particulates (metal matrix composites – MMCs). The present paper concentrates on this approach and presents results of extensive creep experiments on two representative magnesium alloys (AZ91 and QE22) and their various discontinuously reinforced composites in order to compare directly their creep resistance.

Experimental materials and procedures

All of the experimental materials used in the study were fabricated at the Department of Materials Engineering and Technology, Technical University of Clausthal, Germany. Short-fibre reinforced and unreinforced blocks of the most common alloy AZ91 (Mg-9 wt.% Al-1 wt.% Zn-0.3 wt.% Mn) and the high strength silver-containing alloy QE22 (Mg-2.5 wt.% Ag-2.0 wt.% Nd rich rare earths-0.6 wt.% Zr) were produced by squeeze casting. The fibre preform consisted of planar randomly distributed δ-alumina short fibres (Saffil Al_2O_3 fibres ~ 3μm in diameter with varying lengths up to an estimated maximum of ~ 150 μm). The final fibre fraction after squeeze casting in both composites was about 20 vol.%. For convenience, the composites are henceforth designated AZ91+

Saffil and QE22+Saffil. Unreinforced AZ91 and QE22 matrix alloy and their composites were subjected to a T6 heat treatment [6].

Powder metallurgy was used to fabricate SiC particle-reinforced and unreinforced AZ91 and QE22 alloys [7]. The particulate 15 vol.%SiC reinforced AZ91 and QE22 composites were prepared from gas-atomized metal alloy powders of various sizes (ASTM sieve sizes 320 and 600 corresponding to the mean particle diameters of 30 and 10 μm, respectively) and various shapes of the SiC particles (bulky particles-BL rounded particles-HD). All materials were investigated in an as-received state after extrusion and after a T6 heat treatment [7], respectively.

Two various types of the hybrid AZ91 and QE22 composites were produced by liquid infiltration of the fibre-particle preforms by matrix alloy melt via squeeze casting. Details of the compositions and processing techniques of the hybrid composites will be given later on.

The creep tests were carried out at temperatures from 423 to 523 K and at the applied stresses from 10 to 200 MPa in tensile creep testing machines. The creep elongations were measured using a linear variable differential transducer and continuously recorded digitally and computer processed.

Metallographic and fractographic investigations were conducted after creep testing using either transmission electron microscope (TEM Philips CM12) with an operating voltage of 120 kV or scanning electron microscope (SEM Philips 505).

Results and Discussion

Squeeze-cast short fibre composites

Selected creep curves are shown in Fig. 1 in the form of strain, ε, versus time, t plots, for an absolute testing temperature, T, of 423 K and under comparable levels of the applied stress, σ. As demonstrated by the figure, significant differences were found in the creep behaviour of the composite when compared to its monolithic matrix alloy. First, the presence of the reinforcement leads to a substantial decrease in the creep plasticity, which is proved by the values of the total strains to fracture for the composite. Second, the composite exhibits markedly longer creep life than the alloy at the entire stress range used. Third, the shapes of creep curves for the composite and the alloy differ considerably.
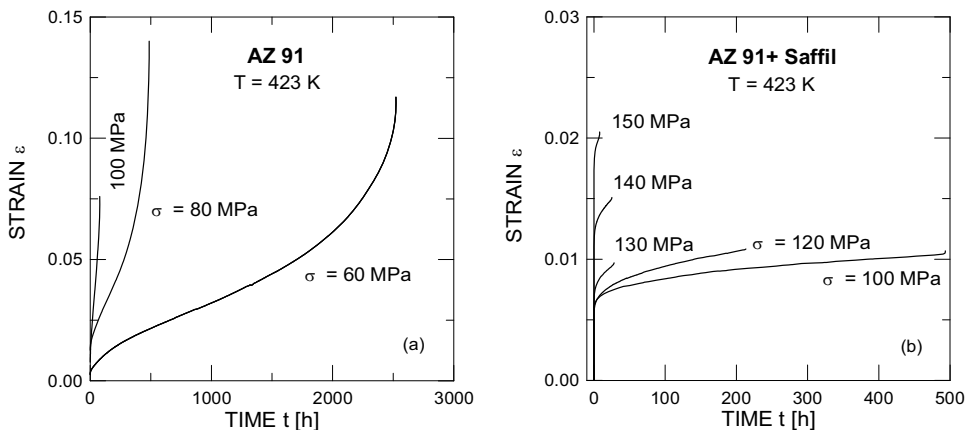

Fig. 1 Creep curves at 423 K for (a) the AZ91 alloy, and (b) the AZ91+ Saffil composite

The creep data for the AZ91 and QE22 alloys and the AZ91+Saffil and QE22+Saffil composites at a testing temperature of 473 K are shown in Fig. 2, where the minimum creep rate $\dot{\varepsilon}_m$ is plotted against the applied stress σ on a logarithmic scale. Inspection of the creep data in Fig. 3 leads to two observations. First, the AZ91+Saffil composite exhibits an improved creep resistance in comparison with the AZ91 monolithic alloy over the entire stress range used at this temperature. Second, the creep resistance of the QE22+Saffil composite is also considerably better than that of the matrix QE22 alloy. All magnesium alloys and their composites exhibit better creep resistance than commercially pure magnesium.

Fig. 3 shows the variation of the time to fracture with the stress for the same specimens tested in Fig. 2. The results for AZ91 alloy and its composite demonstrate the creep life-times of the composite up to one order of magnitude longer than for the monolithic alloy although this difference decreases with increasing applied stress so that ultimately there is very little difference at stresses > 100 MPa.

It is well established, that the presence of a reinforcement leads to a substantial decrease in the overall ductility of matrix alloys. Thus, the values of the strain to fracture ε_f in both composites were only ~ 1-2% and these values were essentially independent of stress and temperature (see Fig. 1b). By contrast, the strains to fracture in the monolithic alloys were markedly higher; typically up to ~ 10-15% in the AZ91 alloy (see Fig. 1a) and up to ~ 30% in the QE22 alloy.

At present it is generally accepted that creep deformation in metal matrix composites is controlled by flow in the matrix materials. This conclusion is supported by recent experimental results on a squeeze-cast AZ91+Saffil composite [8] (an identical material to that used in this work). When the creep data were interpreted by incorporation of a threshold stress σ_0 into the analysis, it was shown that the results are consistent with the behaviour anticipated for a magnesium solid solution alloy, including a true stress exponent of the creep rate $n = (\partial \ln \dot{\varepsilon}_m / \partial \ln \sigma)_T$ close to 3. Such behaviour can be interpreted in terms of a viscous glide process.

Creep behaviour in the composite may be substantially influenced by the development of creep damage and fracture processes. Fractographic investigations of squeeze-cast AZ91 and QE22 composites failed to reveal either substantial creep fibre cracking and breakage or any debonding at
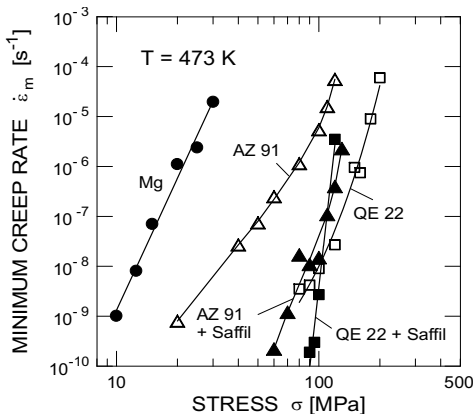

Fig. 2 Minimum creep rate versus stress for the monolithic alloys and their short-fibre composites.
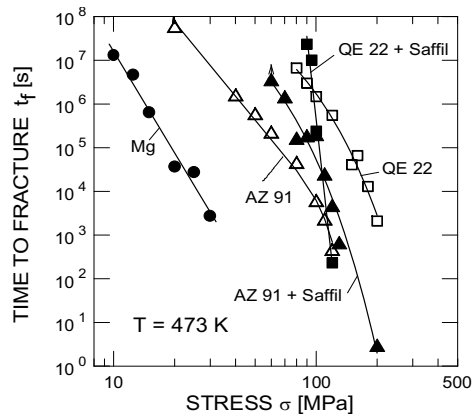
Fig. 3 Time to fracture versus stress for the same materials as in Fig. 2
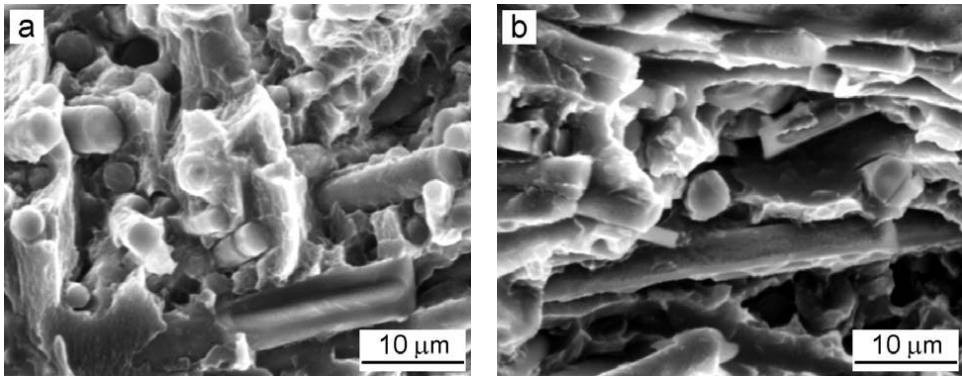
Fig. 4 SEM micrographs of AZ91+Saffil composite taken at two different places, (a) and (b), of the creep fracture surface after testing at 473 K and stress 70 MPa.

the interfaces between the fibres and matrix due to creep. This result is supported by the response observed through acoustic emission monitored during the creep testing of the AZ91+Saffil composite [9]. As can be seen in Fig. 4a, b both transverse fibre cracking and fibre pull-out were revealed at the creep fracture surface of the composite. Reminder of the matrix alloy covers the fibres indicating an ease interface sliding process without any kind of forced fracture.

Particle reinforced composites fabricated by powder metallurgy
Powder metallurgy was used to fabricate SiC particle-reinforced and unreinforced AZ91 and QE22 alloys. The composites were prepared from gas-atomized metal alloy powders of various sizes (ASTM sieve sizes 320 and 600 corresponding to mean particle diameters of 30 and 10 μm, respectively) and various shapes of the SiC particles (bulky particles – BL, rounded particles – HD).

Stress dependences of minimum creep rates at 473 K for the AZ91 monolithic alloy and the AZ91+15vol%SiC composite (Fig. 5a) and stress dependences of minimum creep rates for the QE22 monolithic alloy and the QE22+15vol%SiC composite (Fig. 5b) show that the reinforcing effect of SiC particles on the creep resistance is not uniform and depends strongly on the matrix alloy. While the creep resistance of the AZ91 alloy is improved through particle reinforcement, the creep resistance of the reinforced QE22 alloy is markedly lower than monolithic QE22 alloy and the particle size influences the creep behaviour significantly. These results confirm earlier results published by Moll et al. [10].

The microstructure of the QE22 alloy after T6 heat treatment is very complex and has been extensively studied by Svoboda et al. [11]. The matrix of the QE 22+15vol.%SiC composites after a T6 heat treatment contained all phases revealed in the QE22 monolithic alloy except only that the coherent GP zones were missing. A pronounced precipitation of a Nd-rich phase occurred at the SiC/matrix interfaces, Fig. 6. It is evident that the SiC/matrix interface acts as a nucleation site for this phase.

The enhanced precipitation of Nd-rich phases at the SiC/matrix interface in the QE22+15vol.%SiC composite after T6 heat treatment and during creep can affect detrimentally the creep behaviour in two possible ways. First, matrix depletion due to interfacial precipitation in the composite can produce precipitate inhomogeneity and deficiency in matrix precipitate structure leading to the composite weakening. Second, Moll et al. [10] and Sklenička et al. [12] have proposed that poor creep resistance of the QE 22-15vol.%SiC composite may be explained by
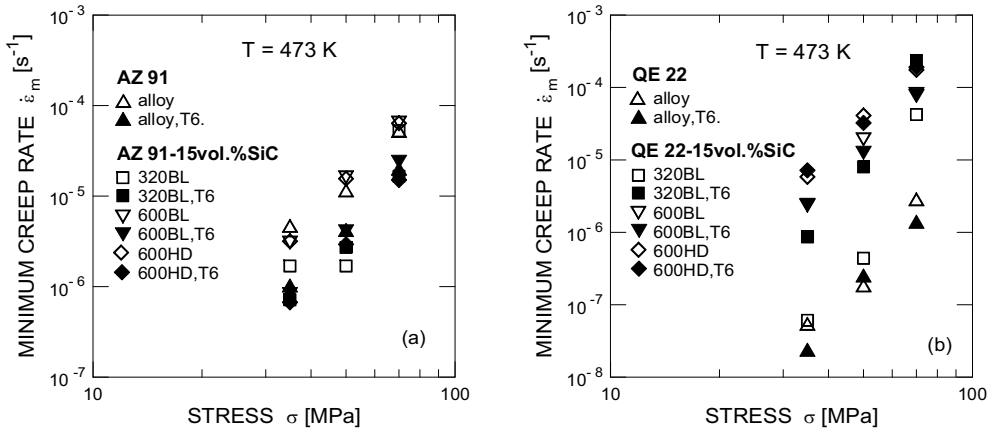
Fig. 5 Stress dependences of minimum creep rates at 473 K for (a) the AZ91 alloy and the AZ91+15vol.%SiC composite, and (b) the QE22 alloy and the QE22+15vol.%SiC composite, in as received state, and after T6 heat treatment.
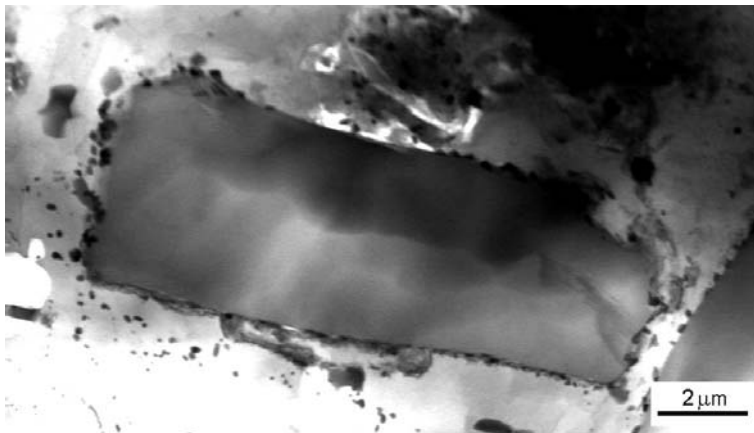

Fig. 6 STEM micrograph showing pronounced precipitation of a Nd-rich phase at the SiC/matrix interfaces in QE22+15 SiC composite after T6 heat treatment and creep test at 35 MPa and 473 K.

taking into account interfacial sliding as an additional creep mechanism acting in the composite. As a consequence of interfacial sliding, many cavities can occur at interfaces giving rise to macroscopic cracks and the debonding of matrix/SiC interfaces. It is generally accepted that intergranular creep cavities are nucleated at the particles due to high local stress concentration caused by grain boundary sliding (Sklenička [13]). In a similar way necessary stress conditions can be developed on interfacial particles due to interaction of interfacial sliding and interfacial precipitates.

Hybrid composites

To demonstrate the effect of the hybrid reinforcements on the creep properties an informative creep study on hybrid magnesium composites was performed. Again, the magnesium AZ91 and QE22 alloys were selected as the matrix alloys of hybrid composites. Hybrid composites produced by such a way that the fibre-particle preform consists of 8 vol.% carbon short fibres and 16 vol.% blocky shaped SiC 600 BL particles. Both monolithic AZ91 and QE22 alloys and their composites were prepared by infiltration of preforms via squeeze casting and investigated in an as-cast state.

The creep tests were carried out at temperature 473 K and at the applied stresses 35, 50 and 70 MPa during a creep exposure, respectively. The creep data of the unreinforced AZ91 alloy and its composite are shown in Fig. 7a. Inspection of the creep data in Fig. 7a leads to following observations. The composite exhibits a better creep resistance than the unreinforced AZ91 alloy over the stress levels used. By contrast, no beneficial effect of the hybrid reinforcement on the creep rate and thus marked improvement of the creep resistance was found for QE22 base composite (Fig. 7b). Metallographic investigation revealed substantial difference in the microstructure of both composites. Whereas no intensive precipitation was found in the SiC/AZ91 matrix interface (Fig. 8), the Nd-rich phases were frequently formed at the SiC/QE22 matrix interfaces during creep. Further, needle shape precipitates containing Mg, Si and Nd were often observed in the close proximity of the MgO/QE22 matrix interfaces (Fig. 9). Fractographic investigation of the crept and fractured specimens revealed another difference between the creep fracture surfaces of the composites. Whereas debonding in the AZ91 composite occurs between the reaction MgO zone [15] at the carbon fibre surface and the matrix it appears that debonding in the QE22 composite is the result of a separation along carbon fibre/reaction zone interface. A strong bonding between carbon fibre surfaces and MgO reaction zones and SiC/matrix interfaces implies very important role of the load transfer in creep strengthening of magnesium alloys. This result confirms the previous conclusion for the particle reinforced QE22+SiC composite prepared by powder metallurgy method and indicates a paramount importance of the choice of the composite matrix alloy and the reinforcement used. The aim of development in this area is to identify an appropriate matrix alloy and fabrication procedure [14].
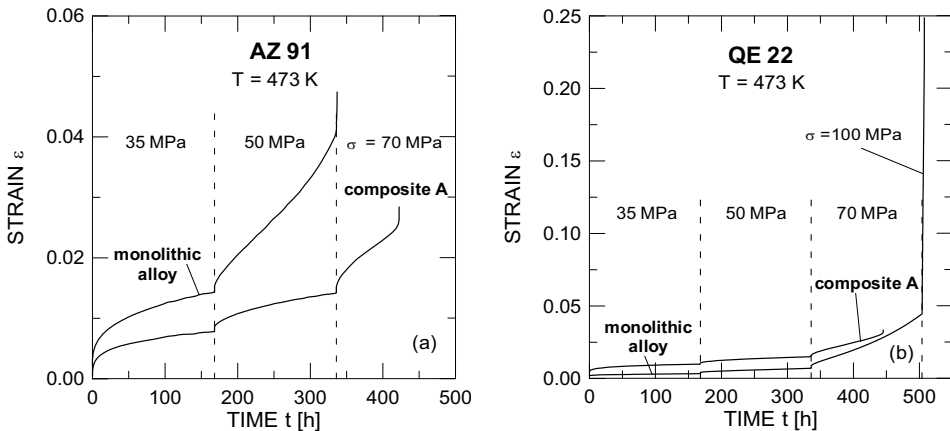

Fig. 7 Creep curves for: (a) the AZ91 alloy and its hybrid fibre-particle composite A and B; and (b) the QE22 alloy and its hybrid composites A and C.
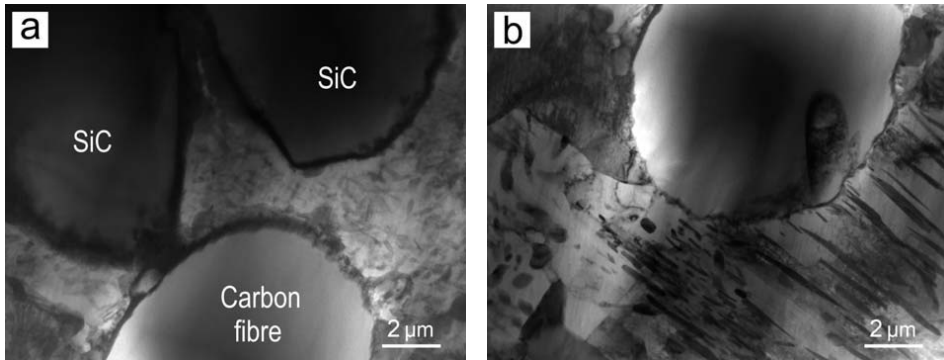
Fig. 8 TEM images of the composite AZ91 showing (a) carbon fibre and SiC particles with MgO envelope and $Mg_{17}Al_{12}$ precipitates at their surfaces and (b) carbon fibre and $Mg_{17}Al_{12}$ precipitates at the fibre surface and in the matrix.
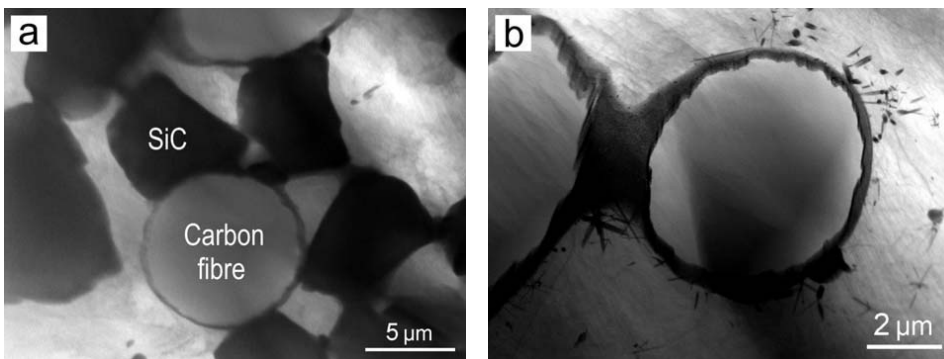

Fig. 9 TEM images of the composite QE22 showing (a) carbon fibres and SiC particles and (b) needle shaped precipitates containing Mg, Si and Nd in the vicinity of MgO/matrix interface.

Conclusions

Both of the squeeze-cast short-fibre reinforced AZ91 and QE22 composites exhibit better creep resistance than their monolithic matrix alloys due to an effective load transfer in which part of the external load within the matrix is transferred to the reinforcement. Indirect composite strengthening may be caused by microstructural effects leading to a threshold stress that increases the creep resistance. However, indirect reinforcement effect can also produce weakening as it was found in the case of particle-reinforced QE22+SiC composite fabricated by powder metallurgy.

Acknowledgements

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