

CREEP DAMAGE MECHANISMS IN PARTICLE REINFORCED MAGNESIUM COMPOSITES

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ABSTRACT

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 particles (metal matrix composites – MMCs). The present paper concentrates on this approach and presents the results of creep experiments on two representative magnesium alloys (AZ 91 and QE 22) and their particle-reinforced composites in order to compare directly their creep resistance. The objective of this paper is thus to provide information on the significance of creep strengthening in selected discontinuously reinforced magnesium alloys. It was found that the creep resistance of the AZ 91 alloy is increased through particle reinforcement, however, the creep resistance of the reinforced QE 22 alloy is decreased. Detailed microstructural investigation revealed enhanced precipitation of Nd-rich phases at the SiC/matrix interfaces in the QE 22-SiC composite after T6 heat treatment and during creep. Such precipitation can detrimentally affect 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, poor creep resistance of the QE 22-SiC composite may be explained by 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 microscopic cracks and the debonding of matrix/SiC interface.

1 INTRODUCTION

Although the magnesium AZ 91 and QE 22 alloys reinforced with alumina short fibres exhibit an excellent creep resistance (Sklenicka et al. [1], Sklenicka and Langdon [2]), their use may be hindered in practice because of their high cost. This problem may be avoided by reinforcing the matrix alloy with relatively inexpensive SiC and/or Al₂O₃ particles. Although such particle-reinforced MMCs may give more modest improvements in the creep properties than their fibre-reinforced counterparts, nevertheless they can be formed into useful shapes using conventional metal working processes such as powder metallurgy methods and extrusion and also they exhibit more isotropic properties. Moll et al. [3] reported recently, in an investigation conducted to evaluate the creep behaviour of 15vol.% silicon carbide particle reinforced PM magnesium AZ 91 and QE 22 alloys, 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 AZ 91 alloy is increased through particle reinforcement, the creep resistance of the reinforced QE 22 alloy is decreased with a increase in the particle size. These unexpected results provided a strong motivation for the following more detailed study of the QE 22–15vol.%SiC composite.

2 EXPERIMENTAL MATERIALS AND PROCEDURES

Powder metallurgy was used to fabricate SiC particle-reinforced and unreinforced AZ91 (Mg-9Al-1Zn-0.3Mn, wt.%) and QE 22 (Mg-2.5Ag-2.0Nd rich rare earth-0.6Zr) 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): representative microstructures

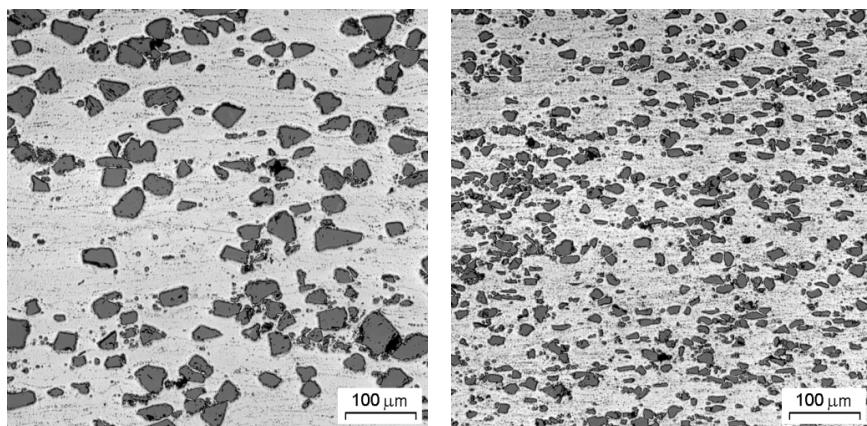


Figure 1: Optical micrographs showing microstructure of the QE 22-15vol.%SiC(p) composite with different SiC particles: (a) 320 BL and (b) 600 BL.

are shown in Fig. 1. Both materials were investigated in an as-received state after extrusion and after a T6 heat treatment, respectively.

Creep tests were performed at 423 and 473 K in purified argon in tensile creep testing machines with the nominal stress maintained constant to within 0.1% up to a true strain of about 0.35. The creep elongations were measured using a linear variable differential transducer and they were continuously recorded digitally and computer processed.

Following creep testing, samples were prepared for examination by transmission electron microscopy (TEM) using ion beam etching technique. Observations were performed using a Philips CM 12 TEM/STEM transmission electron microscope with an operating voltage of 120 kV, equipped with an EDAX Phoenix X-ray microanalyzer. Fractographic details were investigated using either light microscopy or scanning electron microscopy with a Philips SEM 505 microscope.

3 RESULTS AND DISCUSSION

Stress dependences of minimum creep rates at 473 K for the AZ 91 monolithic alloy and the AZ 91-15 vol%SiC composite (Fig. 2a) and stress dependences of minimum creep rates for the QE 22 monolithic alloy and the QE 22-15vol%SiC composite (Fig. 2b) 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 AZ 91 alloy is improved through particle reinforcement, the creep resistance of the reinforced QE 22 alloy is markedly lower than monolithic QE 22 alloy and the particle size influences the creep behaviour significantly. These results confirm earlier results published by Moll et al. [3].

Stress dependences of minimum creep rates for the QE 22 monolithic alloy and its composites with various SiC particle sizes and shapes after two different heat treatments (as received state and T6 heat treatment) are illustrated in Fig. 3 at test temperature of 423 K. Inspection of the creep data in Fig. 3 leads to two observations. First, the unreinforced QE 22 alloy exhibits better creep resistance than the composites both in the as received state (Fig. 3a) and after a T6 heat treatment (Fig. 3b). Second, the T6 heat treatment tends to have a detrimental effect on the creep resistance of both monolithic alloy and its composites.

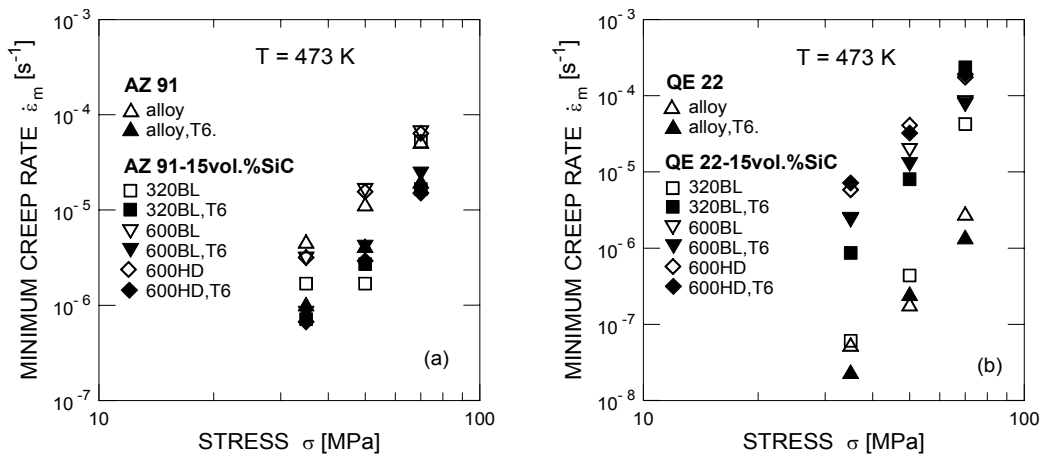


Figure 2: Stress dependences of minimum creep rates at 473 K for (a) the AZ 91 alloy and the AZ 91-15vol.%SiC(p) composite, and (b) the QE 22 alloy and the QE 22-15vol.%SiC(p) composite, in as received state, and after T6 heat treatment.

The microstructure of the QE 22 alloy after the T6 heat treatment is complex (Fig. 4). Large and medium size precipitates of Mg₃(Ag, Nd), medium size rounded precipitates of α Nd (hP4-La, a=0.3658 nm, c=1.179 nm), rod-like precipitates of complex chemical composition including Zr and Ni (Fig. 4a), very small GP zones (Mg-Nd) (Fig. 4b) and MgO particles were presented as the main secondary phases. Two types of Mg₃(Ag, Nd) precipitates were found: large precipitates, 0.2–0.5 μ m in diameter, located mostly at triple points and medium size precipitates, 50–100 nm, located both at grain boundaries and in the grain interiors. The chemical compositions of the large precipitates was determined by EDX as 76Mg - 24Ag (at.%) and the composition of the medium size precipitates was 75Mg-17Ag-8Nd (at.%). Selected area electron diffraction verified that the

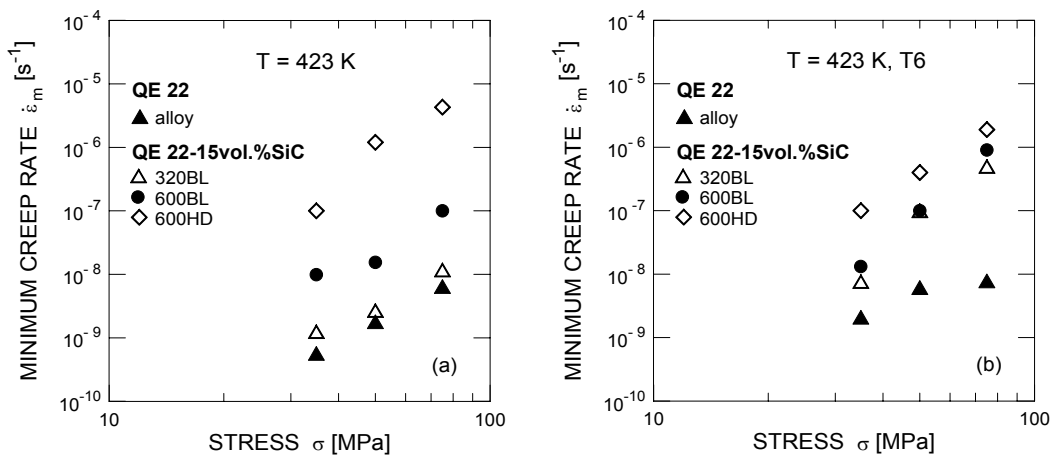


Figure 3: Stress dependences of minimum creep rates for the QE 22 alloy and the QE 22-15vol.%SiC(p) composite at 423 K in (a) as received state, and (b) after T6 heat treatment.

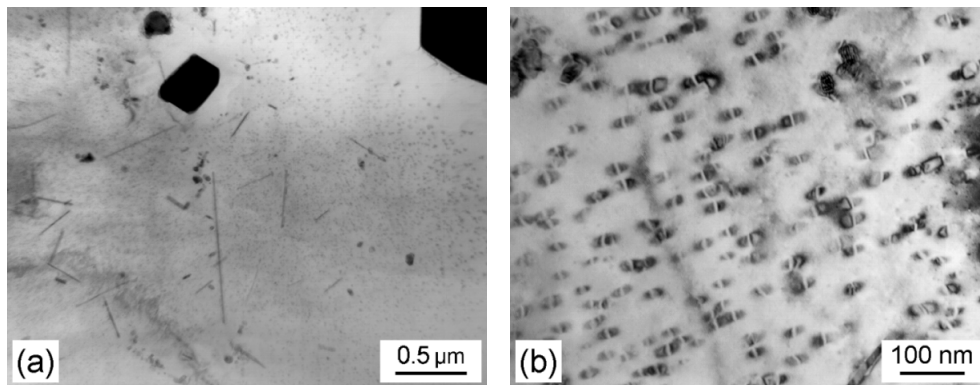


Figure 4: TEM micrographs showing microstructure of QE 22 monolithic alloy after T6 heat treatment: (a) various types of precipitates and (b) detail of GP zones.

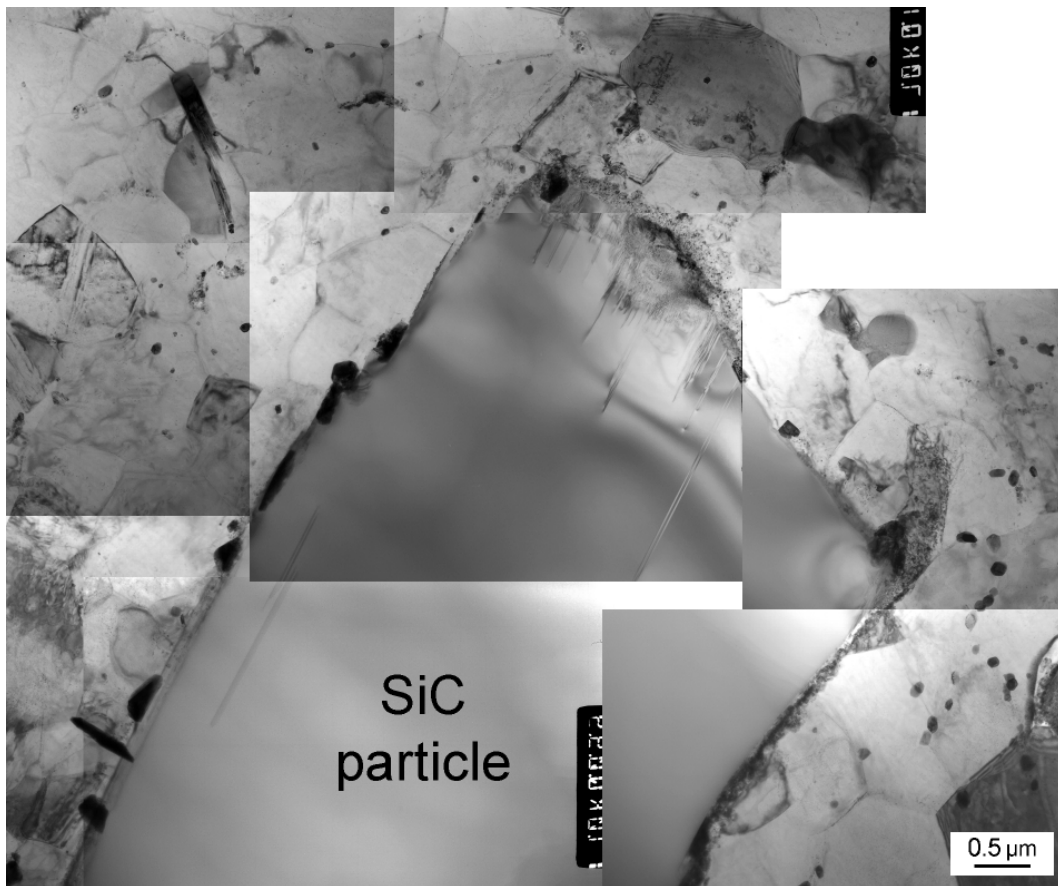


Figure 5: Montage of TEM micrographs showing microstructure of QE 22-15 SiC(p) composite after T6 heat treatment and creep (35 MPa, 473 K).

large precipitates are Mg_3Ag (orthorhombic, $a=1.424$, $b=1.421$, $c=1.466$ nm, sometimes denoted as $Mg_{54}Ag_{17}$). Only one type of the very small GP zones was found and this is responsible for precipitation hardening of the alloy. This is attributed to solid solution depletion of Ag due to the pronounced precipitation of Mg_3Ag .

The matrix of the QE 22-15vol.%SiC composites after a T6 heat treatment contained all phases revealed in the QE 22 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. 5. It is evident that the SiC/matrix interface acts as a nucleation site for this phase. The lack of GP zones in the QE 22-15vol.%SiC composite after T6 heat treatment may be attributed to matrix depletion of Nd due to precipitation of Nd-rich phases at the interfaces. During creep, the precipitates within the grains grow substantially whereas the interfacial precipitates remain unaffected or they are even partially dissolved. An EDX analysis of the particles at the interfaces has not provided conclusive evidence regarding their chemical composition due to the small size of the particles compared to the foil thickness and roughness at the SiC edges. Nevertheless, it was established that these particles are extensively Nd-rich. The average chemical composition of the particles with a negligible Si peak in the X-ray spectra was 91Mg – 9Nd (at.%) which suggests the presence of $Mg_{12}Nd$. However, the particles with the highest measured content of Nd had a composition of 48Mg-22Nd-30Si (at.%), thereby suggesting that another Nd-rich phase forms at the SiC/matrix interface during creep.

Enhanced precipitation of Nd-rich phases at the SiC/matrix interface in the QE 22-15vol.%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. [4] and Sklenicka et al. [5] have proposed that poor creep resistance of the QE 22-15vol.%SiC composite may be explained by taking into account interfacial sliding as an additional creep mechanism acting in 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. Such an explanation has been supported by the experimental observation (Fig. 6). It is generally accepted that intergranular creep cavities are nucleated at the particles due to high local stress concentration caused by grain boundary sliding (Sklenicka [6]). In a similar way necessary stress conditions can be developed on interfacial particles due to interaction of interfacial sliding and interfacial precipitates.

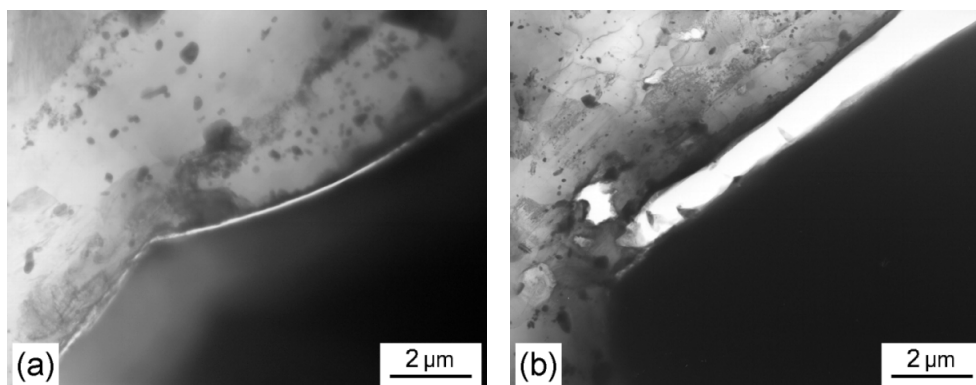


Figure 6: TEM micrographs of showing cracks on SiC particle/matrix interface in QE 22-15SiC(p) composite after creep.

CONCLUSIONS

A different situation occurs when the presence of the ceramic reinforcement affects the matrix microstructure. While reinforcement effects generally produce strengthening, they can also produce weakening, as it was found in the case of QE 22-15 vol.%SiC composite. Thus reinforcement effects can be very complex due to the potential for multiple, competing, or complementary effects. However, knowledge of these effects is critical for understanding of creep and creep fracture in particle reinforced magnesium alloys.

ACKNOWLEDGEMENTS

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