Creep Fracture Processes in Magnesium Metal Matrix Composites

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1. 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. [1-3].

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.

2. 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 [4].

Powder metallurgy was used to fabricate SiC particle-reinforced and unreinforced AZ91 and QE22 alloys [5]. 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 [5], respectively.

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 selected hybrid composites will be given later on.

The creep tests were carried out at temperatures from 423 and 473 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).

3.Results and discussion

3.1 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.

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. 2 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.

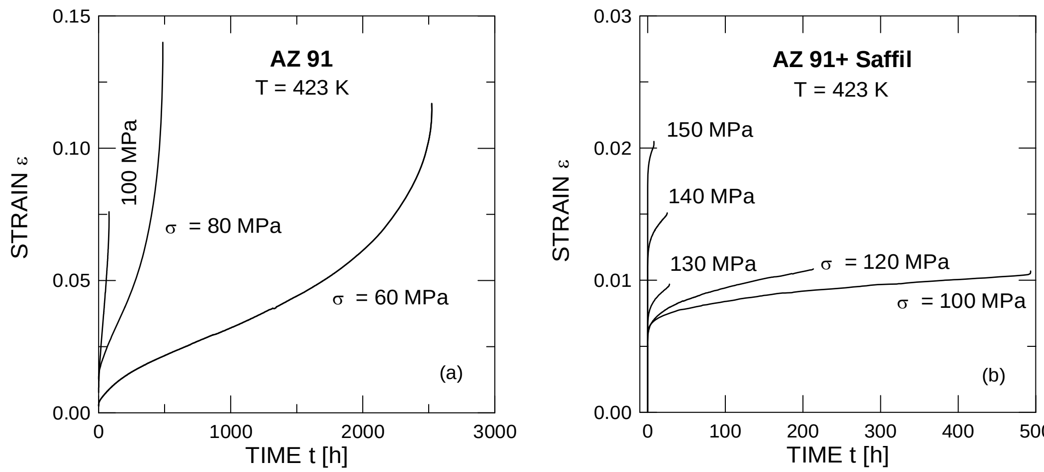

Fig. 1 Creep curves at 423 K for (a) the AZ91 alloy, and (b) the AZ91+ Saffil composite

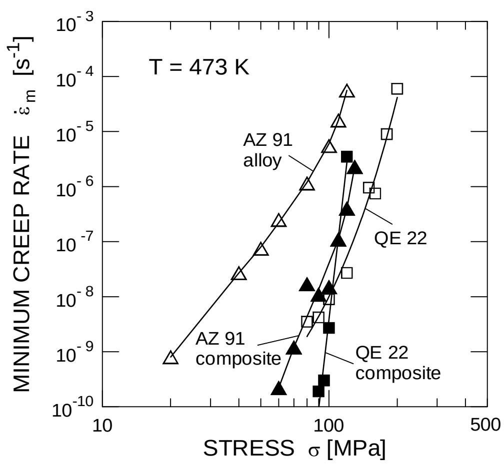

Fig. 2 Minimum creep rate versus stress for the monolithic alloys and their short-fibre composites.

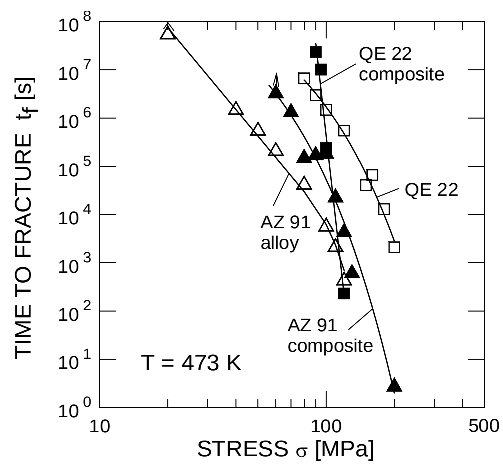

Fig. 3 Time to fracture versus stress for the same materials as in Fig. 2

Fig. 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 are 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 strengthening of the composite may occur by direct strengthening due to a load transfer from the matrix to the reinforcement. Thus, load transfer is accompanied by a redistribution of stresses in the matrix and this reduces the effective stress for creep. In the presence of load transfer, the creep data may be successfully reconciled by taking, for the same loading conditions, the ratio of the creep rates of the composite, $\dot{\varepsilon}_c$, and the matrix alloy, $\dot{\varepsilon}_a$, equals to a factor that is given by $(1-\alpha)^n$, where α is the load-transfer coefficient having values lying within the range from 0 (where there is no load transfer) to 1 (where there is full transfer of the load) and n is the appropriate value of the stress exponent [7]. Thus, taking n = 3 and using the experimental data for the AZ 91+Saffil composite in Fig. 2, the values of α in the present investigation are estimated to lie within the range of ~ 0.8, see Table 1.

Table 1. The values of load transfer coefficient α for the AZ 91+ Saffil composite at 473 K and n = 3, according the relation $\dot{\varepsilon}_c / \dot{\varepsilon}_a = (1-\alpha)^n$.

T[K]	σ[MPa]	$\dot{\varepsilon}_c \left[s^{-1}\right]$	$\dot{\varepsilon}_a \left[s^{-1}\right]$	α
473	50	7.50×10^{-11}	8.61×10^{-8}	0.90
	70	1.36×10^{-9}	5.48×10^{-7}	0.86
	90	1.18×10^{-8}	2.18×10^{-6}	0.82
	100	2.94×10^{-8}	3.89×10^{-6}	0.80

It is possible to check the validity of this approach by estimating a theoretical value for α by using the modified shear-lag model [8] and considering the load-transfer effect at the end of short fibres for various reinforcement geometries and arrangements (for example, the volume fraction of the fibres, their average aspect ratio, etc.) [8]. The values of α predicted theoretically from this model, assuming 20 vol. pct of short-fibre reinforcement and an experimentally observed fibre aspect ratio given by a length/diameter value of ~ 50, are of the order of ~ 0.8. It follows, therefore, that the predicted values of α are in excellent agreement with the experimental values inferred from the present analysis.

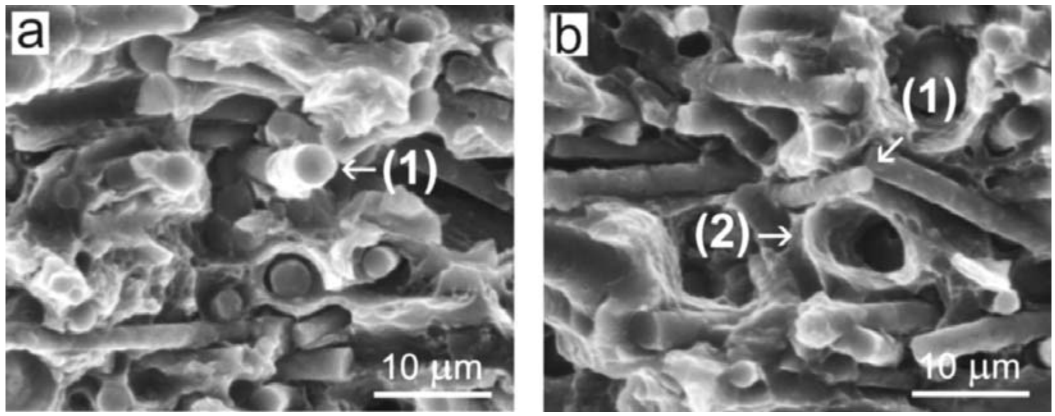

Fig. 4 SEM micrographs of AZ91+Saffil composite taken at two different places, (a) and (b), of the creep fracture surface after testing at 473 K and 70 MPa; (1) transverse fibre cracking, (2) fibre pull-out.

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 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 Figs. 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.

3.2 Particle reinforced composites fabricated by powder metallurgy

Powder metallurgy was used to fabricate SiC particle-reinforced and unreinforced AZ91 and QE22 alloys. 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.

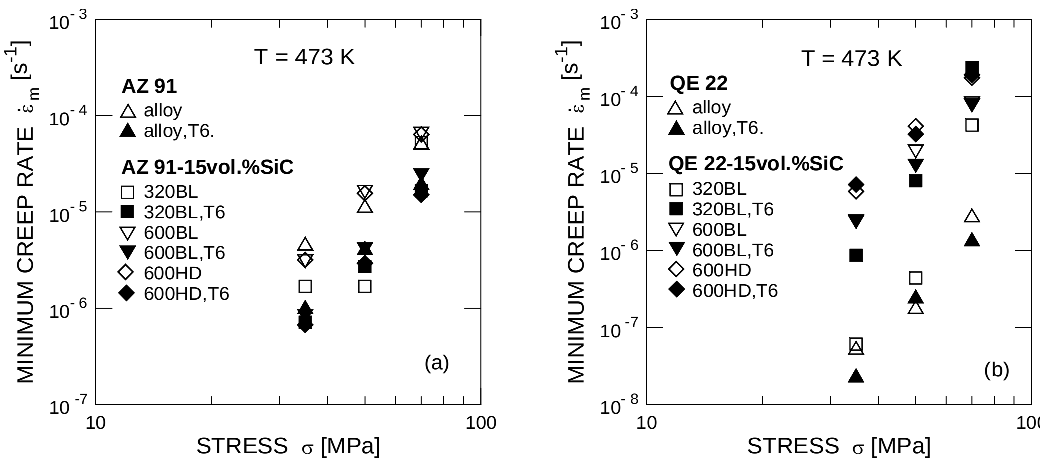

Fig. 5 Stress dependences of minimum creep rates at 473 K for (a) the AZ91 alloy and the AZ91+15vol.%SiC composite, and (b) the QE22 alloy and the QE22+15vol.%SiC composite, in as received state, and after T6 heat treatment.

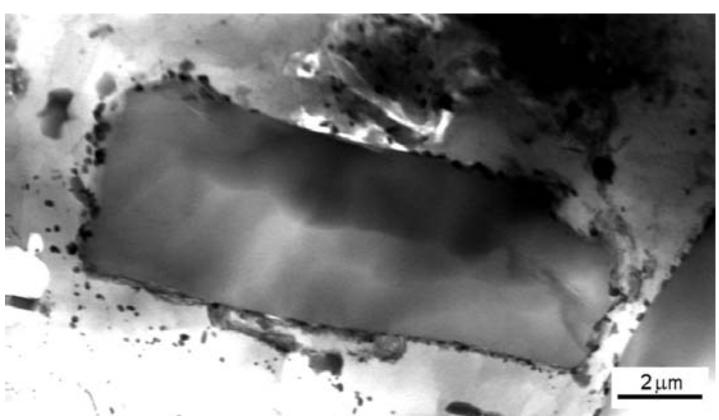

Fig. 6 STEM micrograph showing pronounced precipitation of a Nd-rich phase at the SiC/matrix interfaces in QE22+15vol.%SiC composite after T6 heat treatment and creep test at 35 MPa and 473 K.

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 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.

3.3 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. These composites will be denoted as composites A. 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 [14] 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 [15].

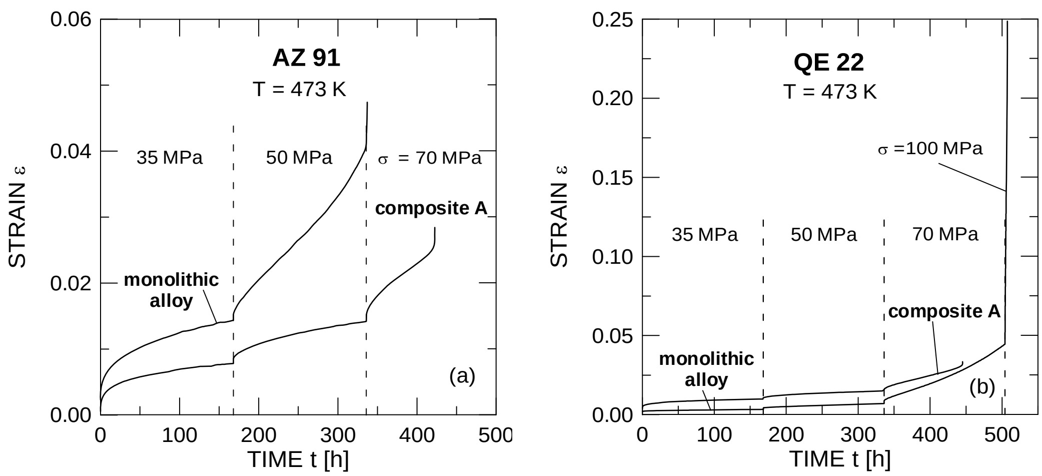

Fig. 7 Creep curves for: (a) the AZ91 alloy and its hybrid fibre-particle composite A; and (b) the QE22 alloy and its hybrid composite A.

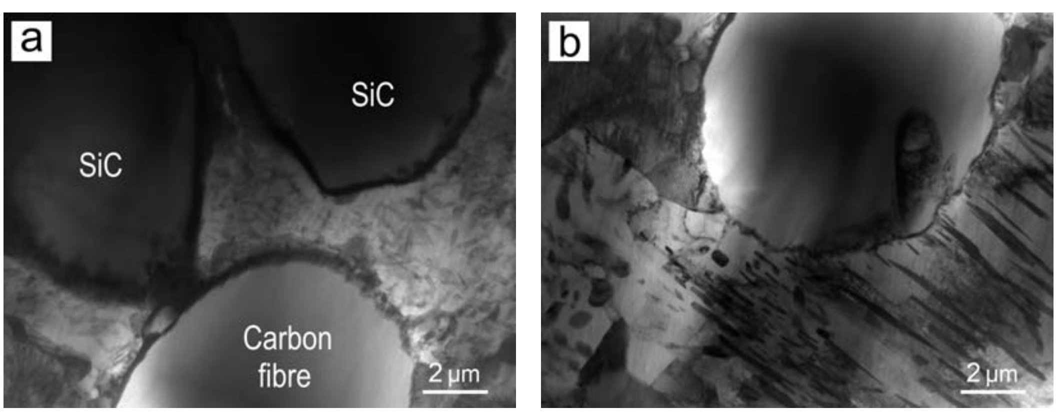

Fig. 8 TEM images of the composite AZ91 showing (a) carbon fibre and SiC particles with MgO envelope and $Mg_{17}Al_{12}$ precipitates at their surfaces and (b) carbon fibre and $Mg_{17}Al_{12}$ precipitates at the fibre surface and in the matrix.

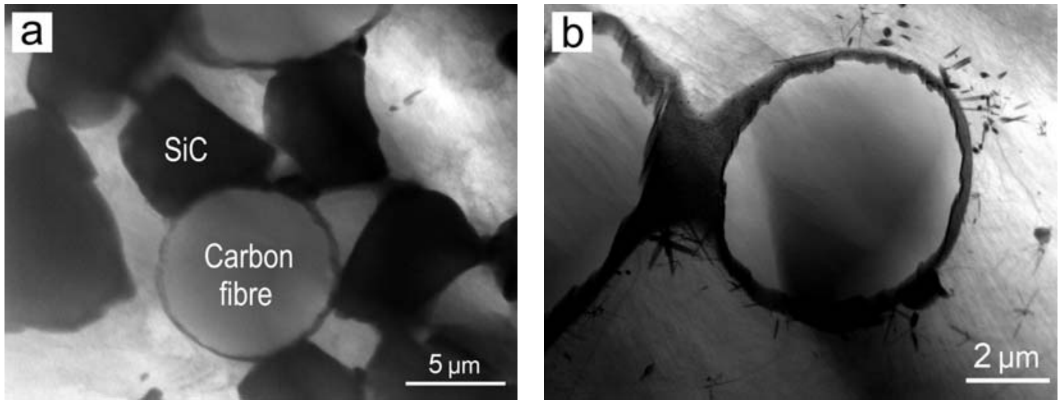

Fig. 9 TEM images of the composite QE22 showing (a) carbon fibres and SiC particles and (b) needle shaped precipitates containing Mg, Si and Nd in the vicinity of MgO/matrix interface.

3. 4 Concluding remarks

The microstructural considerations, the experimental data analysis conducted, and the reasonably good fit with model prediction of the values of load transfer coefficient α (Table 1) support the relevance of load transfer to explain the improved creep resistance of magnesium metal matrix composites. It is frequently suggested that the creep strengthening in the composite arises also from the existence of a threshold stress [6, 16 - 23]. Very recently, extensive analysis of the creep studies in the literature on discontinuously reinforced aluminium metal matrix composites was reported by Fernández and Gonzáles–Dancel [21]. From this analysis it follows that the improved creep strength relies mainly on microstructural factors and a load transfer mechanism. The importance of the threshold stress included in various models and the creep equations of metal matrix composites to rationalize the apparent high values of the stress exponent of the minimum creep rate n and the activation energy for creep Q_c has been criticized [21]. In addition, the most recent works on the creep of metal matrix composites do not discuss the possible origins of the threshold stress [20 - 22].

It should be stressed, that damage processes occurring at the interfaces between the reinforcement and matrix and arising from reactions during composite processing and loading have to be included in on overall predictive model of the composite creep behaviour. However, the characteristics of these processes make a relevant analytical treatment of damage for any model of creep in metal matrix composites very difficult.

4. 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.

5. Acknowledgements

The authors acknowledge funding for this work by the Academy of Sciences of the Czech Republic under the Institutional Research Plan AV0Z20410507. We would like to thank Prof. K. U. Kainer, Prof. P. Lukáč, Prof. T.G. Langdon and Dr. F. Moll for introducing the subject and for valuable discussions.

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