Alexandra Rudajevová[1], **Pavel Lukáč**[2]
Charles University, Ke Karlovu 5, 121-16 Praha 2, Czech Republic

ON THE THERMAL CHARACTERISTICS OF Mg BASED COMPOSITES

The thermal diffusivity, thermal conductivity and thermal expansion were measured to obtaine the information about the behaviour of Mg composites during their heating. The thermal diffusivity indicates precipitation of the β-phase ($Mg_{17}Al_{12}$) in AZ91 alloy and in AZ91 26 vol.% Saffil fibres composite. The thermal conductivity of QE22 alloy and reinforced QE22 alloy with 10, 15 and 25 vol.% SiC particles was obtained in the temperature range of 20÷300°C. The thermal conductivity decreases with increasing concentration of the particles and increasing temeperature. The thermal expansion of Mg-30 vol.% Saffil fibres composite was measured and the results were analysed to obtaine information about the residual and thermal strain.

Key words: **thermal conductivity, thermal expansion, Mg composites**

CHARAKTERYSTYKI CIEPLNE KOMPOZYTÓW NA OSNOWIE STOPÓW Mg

Przedstawiono wyniki badań dyfuzyjności, przewodności oraz rozszerzalności cieplnej kompozytów wytworzonych na osnowie stopu AZ91 i QE22 umacnianych cząstkami SiC oraz włóknami Saffil (Al_2O_3). Badania prowadzono w celu uzyskania informacji o zachowaniu się kompozytów magnezowych podczas ich nagrzewania. Analizy wyników badań dyfuzyjności cieplnej wykazały wydzielanie fazy β ($Mg_{17}Al_{12}$) zarówno w stopie AZ91, jak i w kompozycie AZ91-26% obj. włókien Al_2O_3. Badania przewodności cieplnej samego stopu QE22 oraz kompozytu umacnianego 10, 15 i 25% obj. cząstek SiC przeprowadzono w zakresie temperatur 20÷300°C. Ujawniono, że przewodność cieplna kompozytów malała wraz ze wzrostem udziału objętościowego cząstek oraz ze wzrostem temperatury. Badania rozszerzalności cieplnej kompozytu Mg-30% obj. włókien Saffil pozwoliły natomiast na analizy dostarczające informacji o naprężeniach cząstkowych i cieplnych materiału.

Słowa kluczowe: **przewodność cieplna, rozszerzalność cieplna, kompozyty Mg**

INTRODUCTION

The increasing demand for a reduction weight of materials for aeronautical and automobile applications has been the motivation for the evolution of Mg composites. The reinforcement of Mg alloys with ceramic fibres and particles has led to an improvement of their mechanical properties as well as their thermal stability.

Magnesium alloys are very often reinforced with SiC and/or Al_2O_3. The SiC particles used in composites and produced from inexpensive raw materials exhibit low density of 3 200 kg/m^3, low thermal expansion coefficient of 4.7×10^{-6} K^{-1}, and the thermal conductivity in the range of 80 to 200 Wm^{-1}K^{-1} depending on purity and processing conditions. The Saffil fibres (96÷97% Al_2O_3, 4÷3% SiO_2) are used most frequently for preparation of magnesium-Al_2O_3 composites. The density of this material is 3300 kg/m^3, thermal expansion coefficient is 7.6×10^{-6} K^{-1} and the thermal conductivity at the room temperature is in the range of 30 to 36 Wm^{-1}K^{-1}.

The use of magnesium composites requires a better understanding not only their mechanical properties but also their thermal characteristics. A large difference of the thermal expansion coefficients of the components leads to thermal stresses at the interfaces. Owing to these thermal stresses the composite is deformed at room temperature. Heating the composite up to certain temperature where the free stress state is reached, the strains decrease to zero.

The main aim of this paper is to describe and to analyse the temperature dependences of the thermal diffusivity of AZ91 alloy reinforced with 26 vol.% Saffil fibres, the thermal conductivity of QE22 alloy reinforced with 10, 15 and 25 vol.% SiC particles in the temperature range from 20 to 300°C. The thermal expansion chracteristics of Mg reinforced with 30 vol.% Saffil fibres will be measured and analysed in the temperature range from 20 to 375°C.

EXPERIMENTAL PART

Composites AZ91-26 vol.% Saffil fibres (Mg-9 wt.% Al-0.7 wt.% Zn-0.2 wt.% Mn) and Mg - 30 vol.% Saffil fibres were produced by squeeze casting. The AZ91 composites were studied after heat treatment at 413°C

[1] RNDr., CSc, [2] Prof. RNDr., DrSc

for 18 hours and cooling in air, the Mg composites were studied in the as-prepared state. The QE22-SiC ($d = 9$ µm), composites were prepared by powder metallurgical method. The speciments were investigated in the as-extruded state.

The measurement of the thermal diffusivity was performed in the temperature range from 20 to 300°C in argon atmosphere using the flash method described elsewhere [1]. The thermal conductivity K was calculated using the relation $K = a\rho c$, where a is the thermal diffusivity, ρ is the density and c is the specific heat capacity. The specific heat capacity of the alloys was calculated using the Neumann-Kopp rule, the specific heat capacity of the composites was calculated by the rule of mixtures. The temperature dependence of the density was calculated from the density measured at 20°C by weighing the sample in toluen and from respective values of volume thermal expansion data.

The linear thermal expansion of samples was measured in argon atmosphere using the Netzsch 402E dilatometer, over a temperature range from room temperature to 375°C at heating and cooling rates of 5 K/min. The planes of planar randomly distributed short fibres were parallel to the sample axis.

RESULTS AND DISCUSSION

Thermal diffusivity of the AZ91-26 vol.% Saffil fibres composite

The temperature dependence of the thermal diffusivity of the first, second and third runs for AZ91 with 26 vol.% Saffil is shown in Figure 1.

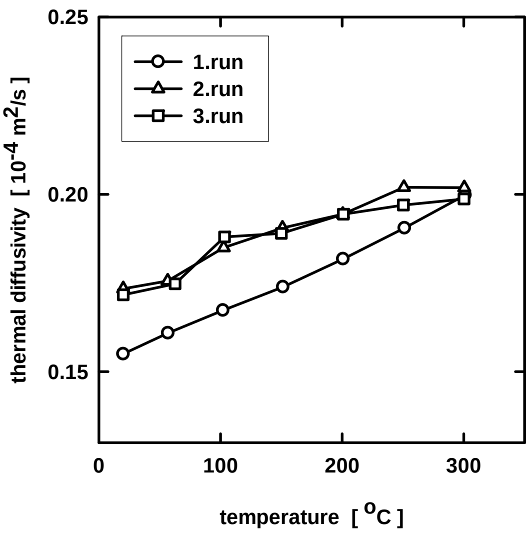

Fig. 1. Temperature dependence of the thermal diffusivity for AZ91 composite

The temperature dependences for the second and third runs are almost the same and the values of the thermal diffusivity are higher than in the first run (similar behaviour as in the case of the unreinforced AZ91 alloy). Changes in the thermal diffusivity Δa (measured at room temperature) between the first and the third run of the measurement for the solution treated (20°C) or isochronally annealed (140 and 300°C) for reinforced and unreinforced AZ91 are presented in Table 1. The differences in the thermal diffusivity between the first and further runs are due to the precipitation process that is connected with the purification of alloy. According to data in the literature [2, 3] the decomposition sequence of a supersaturated solid solution of Al in the AZ91 alloy is very simple, involving only the formation of incoherent particles of the equilibrium β-phase ($Mg_{17}Al_{12}$) without any formation of Guinier-Preston zones or intermediate precipitates. It is supposed that precipitation in AZ91 matrix took places in the first run. Therefore, the values for he thermal diffusivity are lower at the beginning of the first run compared to the others runs. A preceding anneal at 300°C reduced Δa value considerably due to accelerated precipitation in the temperature range from 200 to 300°C. The difference Δa for the reinforced specimens is slightly higher than that for the unreinforced alloy. This fact can be explained by the preferred precipitation of β-phase on the fibres due to heterogeneous nucleation and the Al enrichment of the matrix near the Saffil fibres.

TABLE 1. **Change in the thermal diffusivity Δa between the first and the third run of the measurement for AZ91 specimens, solution treated or isochronally annealed up to 140 and 300°C, respectively**

AZ91	Δa upper temperature of isochronal annealing		
	20°C	140°C	300°C
unreinforced	8%	9%	3%
reinforced	11%	12%	3%

Thermal conductivity of the QE22-SiC composites

The temperature dependencies of the thermal conductivity for the QE22 alloy and three composites are shown in Figure 2. It can be seen that the thermal conductivity at any temperature decreases with increasing volume fraction of SiC particles. Whereas the temperature dependence of the thermal conductivity for the QE22-10 vol.% SiC_p composite is similar to that for the QE22 alloy, the temperature variation of the thermal conductivity for the composites with higher volume fractions of SiC particles is different from the former. The behaviour of three composites may be discussed in the connection to the model of Hasselman and Johnson [4]. They derived the following equation for the effective thermal conductivity of composite, K_c, of a continuous matrix with dilute dispersed volume fraction of spherical inclusions

$$K_c = K_m \frac{\left[2f\left(\frac{K_p}{K_m} - \frac{K_p}{rh} - 1\right) + \frac{K_p}{K_m} + 2\frac{K_p}{rh} + 2\right]}{\left[f\left(1 - \frac{K_p}{K_m} + \frac{K_p}{rh}\right) + \frac{K_p}{K_m} + 2\frac{K_p}{rh} + 2\right]} \quad (1)$$

where K_m is the thermal conductivity of matrix, K_p is the thermal conductivity of particles, r is their radius and h is the interfacial thermal conductance.

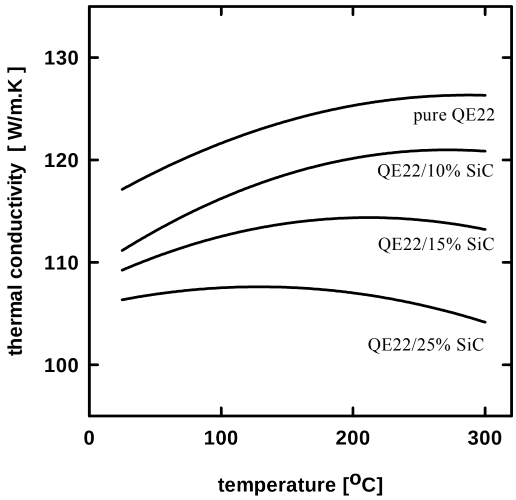

Fig. 2. Temperature dependence of the thermal conductivity for QE22 alloy and QE22 composites

In the alloys reinforced with SiC particles new dislocations (with a high density) in the immediate vicinity of the SiC particles are created due to large differences in the coefficient of thermal expansion between matrix and particles. Flom and Arsenault [5] reported that the resultant dislocation densities at the Al-SiC interfaces could be very high, about $10^9 \div 10^{10}$ cm^{-2}. Similar dislocation densities may be expected at the QE22-SiC interfaces. During heating the composite new dislocations may be generated. With increasing temperature expansion of some defects may occur into matrix. Therefore, the thermal conductivity of the alloy in composite is not the same as that of unreinforced alloy especially at high temperatures.

The thermal conductivity of QE22 matrix in composites could be estimated from measured values of the thermal conductivity of the composites using equation (1). The value $h = 1.10^8$ W/m^{2}K, estimated at room temperature, was used for all composites. The calculated values of the thermal conductivity of the matrix as a function of temperature are plotted in Figure 3. It can be seen good agreement between the calculated thermal conductivity of the QE22 matrix in QE22-10 vol.% SiC in the temperature range investigated here. The thermal conductivity of the QE22 matrix in QE22-15 vol.% SiC and QE22-25 vol.% SiC composites has the same character as unreinforced alloy only up to 130 and 70^{o}C, respectively. Above these temperatures the thermal conductivity of the matrix in both composites is lower than that of pure alloy. The results suggest that the lattice defects, especially newly created dislocations, are localised around interface at low temperatures and do not influence the thermal conductivity of the matrix. With increasing temperature the defects expand into matrix and reduce its thermal conductivity.

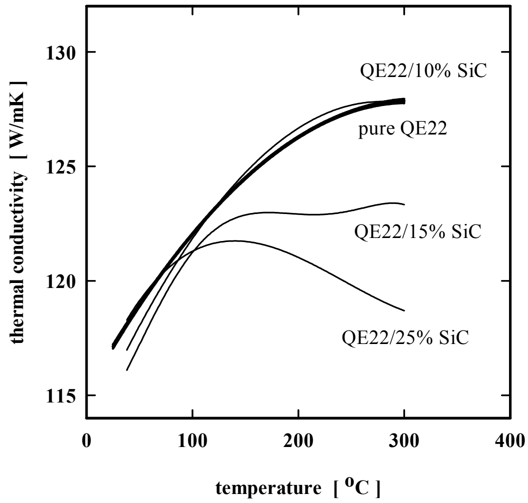

Fig. 3. Temperature dependence of the calculated thermal conductivity for QE22 alloy in QE22 composites

Thermal expansion of the Mg-30 vol.% Saffil fibres composite

The temperature dependencies of the relative elongation of pure Mg and composite Mg with 30 vol.% Saffil (two runs) are shown in Figure 4.

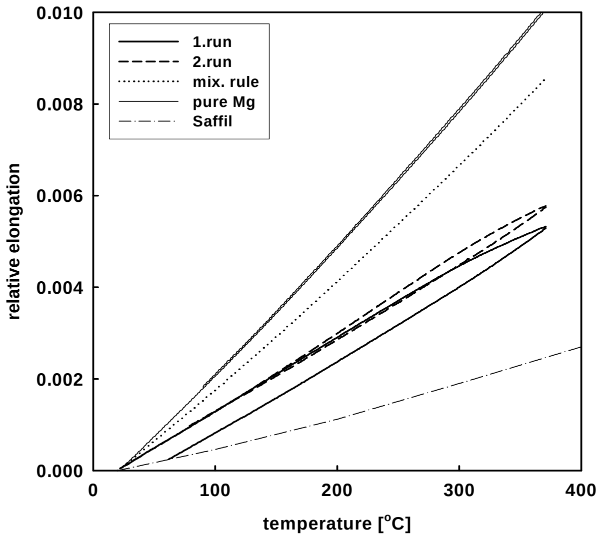

Fig. 4. Temperature dependence of the relative elongation for Mg-30 vol.% Saffil fibres

The dotted curve in this figure was calculated from the relative elongation of Mg and Saffil using the rule of mixtures. The relative elongation of Saffil was obtained from [6]. Figure 4 shows that the residual contraction of the composite, about 30 μm, occurs after the first run. In the second and third runs (the third run had the same course as the second one) no permanent change in the sample length was found. When comparing the relative elongation of the Mg composites with the relative elongation obtained from the rule of mixtures (stress free state), three important effects could be noted for the composite studied here:

- a reduction of the relative elongation of the composite in comparison to the values obtained from the rule of mixtures,
- the different course of the temperature dependences of the relative elongation in the first and the second thermal cycle, and
- the hysteresis.

Figure 5 shows the temperature dependence of the parameter $\Delta d/l_o$ for the Mg-30 vol.% Saffil fibres composite. This parameter was obtained using the following equation:

$$\frac{\Delta d}{l_o} = \left(\frac{\Delta l}{l_o}\right)_{meas.} - \left(\frac{\Delta l}{l_o}\right)_{mix.rule} \quad (2)$$

where $(\Delta l/l_o)_{meas.}$ is the measured relative elongation of where the composite and $(\Delta l/l_o)_{mix.rule}$ is the relative elongation of the composite calculated by the rule of mixtures. The parameter $\Delta d/l_o$ represents the departure of the composite behaviour from the case where no thermal stresses are present.

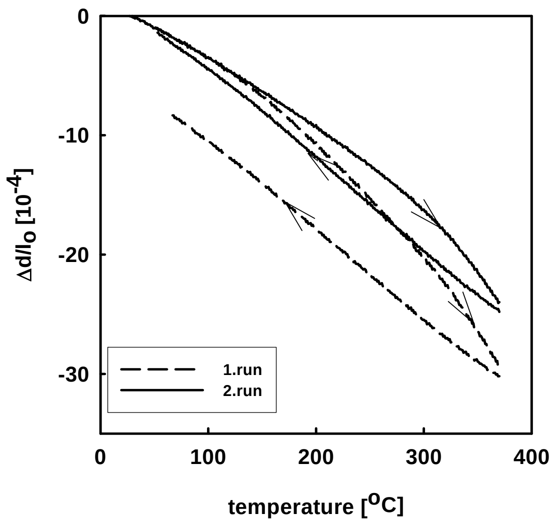

Fig. 5. Temperature dependence of the thermal strain in Mg-30 vol.% Saffil composite

It is well known that in the composites with the components with the various CTEs the thermal stresses occur near the interface between matrix and reinforcement as a consequence of technological cooling at the fabrication of the composite. These stresses and thermal strains related to them increase with decreasing temperature and they are the highest at room temperature if it is the lowest temperature studied. Heating the as-prepared composite, thermal stresses and strains continue to be released again, the free stress state being approached at certain temperature. Figure 5 shows that the release of the thermal strain slips by two mechanisms. The linear dependence of the thermal strain with the temperature is connected with the elastic release of the thermal strain, the non-linear part is connected with plastic mechanism. The more detailed informations about these processes are presented in our paper [7].

CONCLUSIONS

The temperature dependence of the thermal diffusivity of the AZ91-26 vol.% Saffil fibres composite shows that precipitation processes occur above 200°C. The thermal diffusivity increases due to purification of the alloy by the precipitation process.

The temperature and concentration dependence of the thermal conductivity of the QE22-SiC composites show that the dislocations are localised at interface at room temperature. With the increasing temperature the dislocations extend in the matrix and hence, the thermal conductivity decreases.

The Mg-30 vol.% Saffil fibres composite is deformed at room temperature owing to thermal stresses at the interface between matrix and Saffil fibres. With increasing temeperature the thermal strain decreases up to free stress state. The relative elongation of the composite is not linear as in pure components and it shows hysteresis. It is consequence of the elastic-plastic changes which occur at the interface between matrix and reinforcement.

Acknowledgements

The authors acknowledge financial support from the Grant Agency of the Academy of Sciences under Grant A2112302 and from the Grant Agency of the Czech Republic under Grant 106/03/0901.

REFERENCES

[1] Rudajevová A., Mat. Res. Bull. 1991, 26, 1363.
[2] Bettles C.J., Humble P., Nie J.F., Proceedings of the 3rd International Magnesium Conference, ed. G.W. Lorimer, The Institute of Materials, London 1997.
[3] Polmear I.J., Magnesium Alloys and Their Applications, ed. B.L. Mordike, F. Hehmann, DGM, Oberursel 1992.
[4] Hasselman D.P.H., Johnson L.F., J. Comp. Mater. 1987, 21, 508.
[5] Flom Y., Arsenault R.J., Mater. Sci. Eng. 1986, 77, 191.

[6] Chang S., Lin S., Flemings M.C., Metall. Mater. Trans. 2000, 31A, 291.
[7] Rudajevová A., Lukáč P., Acta Mater. 2003, 51, 5579.

Recenzent
Edward Fraś