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Microstructure Changes in Magnesium Alloys Base Composites Studied by Internal Friction

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When a metal matrix composite (MMC) is subjected to temperature changes thermal stresses arise at the interfaces between the matrix and the reinforcement owing to a considerable mismatch of the thermal expansion coefficient of the matrix and that of the reinforcement. Even moderate temperature changes may produce thermal stresses that exceed the matrix yield stress, consequently, generating new dislocations at the interfaces causing thermal fatigue (microstructure changes, matrix plastic deformation and irreversible shape changes).

The strain dependencies of the logarithmic decrement and stress relaxation have been measured for magnesium alloys AZ91 (9 % Al – 1 % Zn – balance Mg), ZC63 (6 % Zn – 3 % Cu – balance Mg) and ZE41 (4 % – 1 % RE – balance Mg), QE22 (2 % Ag – 2 % RE – balance Mg) reinforced by short Saffil fibres. Prior to the measurements, the samples were subjected to thermal cycling with increasing upper temperatures. The response of the composite on thermal loading has been characterised by measurements of internal friction.

1. INTRODUCTION

Several magnesium-based metal matrix composites (MMCs) have been developed and manufactured over the last decade for potential use as light-weight high-performance materials in a wide range of applications including, for example, the automotive industry [1]. In practice, it is well established that the mirostructures and the mechanical properties of these composites are strongly affected by the nature of the interfaces between the matrix and the reinforcement. The standard operating condition for most MMCs will generally include some form of thermal

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loading and, of necessity, this will introduce internal stresses because of the significant mismatch in the thermal expansion coefficients between the matrix and the reinforcement. It follows that even minor temperature changes may lead to structural changes, plastic deformation within the matrix and significant microstructural damage.

When the coefficients of thermal expansion (CTE) of the two phases of composite are different, thermal stresses are generated upon temperature change in both matrix as well as in the reinforcing phase. This effect has been studied experimentally using etch pits [2] or transmission electron microscopy [3]. The dislocations formed during cooling from an elevated temperature below a certain temperature T_C, can glide inducing plastic deformation in the matrix. An increase in the dislocation density near reinforcement has been calculated as [4]

$$\varrho = \frac{Bf\,\Delta\alpha\,\Delta T}{b(1-f)}\frac{1}{t}, \tag{1}$$

where f is the volume fraction of the reinforcing phase, t its minimum size, b the magnitude of the Burgers vector of dislocations, B a geometrical constant, $\Delta\alpha$ the absolute value of the CTE difference between the matrix and the reinforcement, ΔT the temperature difference. Microplastic zones around the fibres with high density of mobile dislocations are developed during cooling.

Understanding of thermally induced plasticity in metal matrix composites is important since dislocations produced by thermal cycling from manufacture temperature influence many mechanical and physical properties of materials. In this paper we present indirect experimental evidence of dislocation emission in composite materials subjected to thermal loading.

If a material containing dislocations is submitted to a harmonic stress $\sigma = \sigma_0 \sin \omega t$ with an angular frequency $\omega = 2\pi f$, one can define the mechanical loss factor η by the following relation

$$\eta = \frac{1}{2\pi}\frac{\Delta W_{diss}}{W_{\max}} \tag{2}$$

where ΔW_{diss} is the mechanical energy dissipated in one cycle of the applied stress, and $W_{\max}$ is the maximum mechanical energy stored on it. The lost energy ΔW_{diss} depends only on the anelastic strain. In the case of an anelastic dislocation strain

$$\Delta W_{diss} = \int \varepsilon_d d\sigma\,. \tag{3}$$

The maximum stored energy can be well approximated by the maximum elastic stored energy

$$W_{\max} = \int_0^{\sigma_0} \sigma d\varepsilon_{el} = \frac{1}{2} J_{el}\sigma_0^2, \tag{4}$$

where J_{el} is the elastic compliance related to the shear modulus $G^{-1} = J_{el}$. Mechanical loss factor due to the presence of dislocations in the material may be written as

$$\eta = \frac{1}{\pi J_{el} \sigma_0^2} \int \varepsilon_d d\sigma . \tag{5}$$

The logarithmic decrement, δ, as another damping quantity, is given as

$$\delta = \frac{1}{n} \ln \frac{A_i}{A_{i+n}}, \tag{6}$$

where A_i and A_{i+n} are the amplitudes of the i-th cycle and (i+n)th cycle, respectively separated by n periods of the free vibrations of the specimen. Between these damping quantities the following relationship holds $\pi\eta = \delta$ (for small δ).

The aim of the present work was to investigate the influence of thermal treatment on the internal friction and from this behaviour to deduce changes in the dislocation structure of magnesium alloys reinforced by short Saffil fibres.

2. EXPERIMENTAL PROCEDURE

The commercial Mg alloys AZ91, QE22, ZC63 and ZE41 were reinforced by δ-alumina short fibres (Saffil consisting 96 – 97 % Al_2O_3, 4 to 3 % SiO_2, 3 μm in diameter with the mean length of about 87 μm) applying the squeeze cast technology. Composites based on AZ91, ZC63 and QE22 alloys were exposed to the T4 heat treatment (AZ91: 413 °C/18 h; ZC63: 440 °C/8 h; QE22: 520 °C/5.5 h), closely to the recommendation for the Mg alloys by Polmear [5]. Damping measurements were carried out on bending beams (80 mm long with thickness of 3 mm) in vacuum (about 30 Pa) at ambient temperature. The damping was obtained by the measurement of the logarithmic decrement of free vibrations by a vibrating apparatus. The beam was fixed on one side and a permanent magnet was fastened on the opposite side. The beam was excited to resonance vibrations by a sinusoidal current flowing in a field coil. After reaching a definite amplitude the coil current was switched off by a PC and the declining mechanical vibration was stored in the PC via an ADC measuring the voltage induced by the moving permanent magnet. Using these data the decrement δ of the free vibrations was determined by the PC according to exponential law

$$A(t) = A_0 \exp\left(-\delta t/T\right). \tag{7}$$

The samples were sequentially cycled (1 cycle at each temperature) at increasing upper temperatures up to 400 °C for 15 min. and then quenched into water of ambient temperature.

3. RESULTS AND DISCUSSION

For many metallic materials, the dynamic strain dependence of the damping capacity can be divided into a strain independent and a strain dependent component. In case of the logarithmic decrement (6) expresses this experimental finding:

$$\delta = \delta_0 + \delta_{\mathrm{H}}(\varepsilon), \tag{8}$$

δ_0 is the amplitude independent component, found at low amplitudes. The component δ_H depends on the strain amplitude and it is usually caused by dislocations in the material.

Fig. 1a shows the plots of the logarithmic decrement against the strain for AZ91 + 14.6 % of Saffil composite before and after thermal cycling between

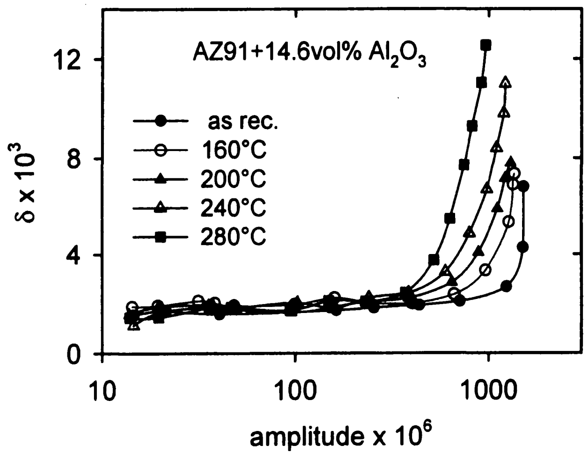

Fig. 1a. Amplitude dependence of decrement obtained for lower tempratures of thermal treatment.

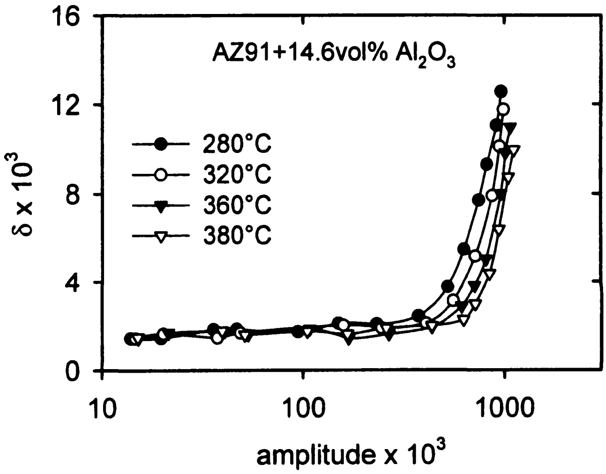

Fig. 1b. Amplitude dependence of decrement obtained for higher temperatures.

room temperature and an increasing upper temperature of the thermal cycle. Fig. 1b shows results obtained for higher temperatures. From Fig. 1a and Fig. 1b it can be seen that the strain dependencies of the logarithmic decrement exhibit two regions in good accord with (8). The values of the δ_H in the strain amplitude dependent region increase very strongly with increasing upper temperature up to 280 °C and then, above 280 °C, the values of δ_H decrease with the upper temperature. Similar results were found for other composites based on ZC63 alloy (Fig. 2a, b), ZE41 alloy (Fig. 3a,b) and QE22 (4a,b).

The course of all these dependencies has common features. The decrement increases with increasing upper temperature of the cycle up to a certain tempera-

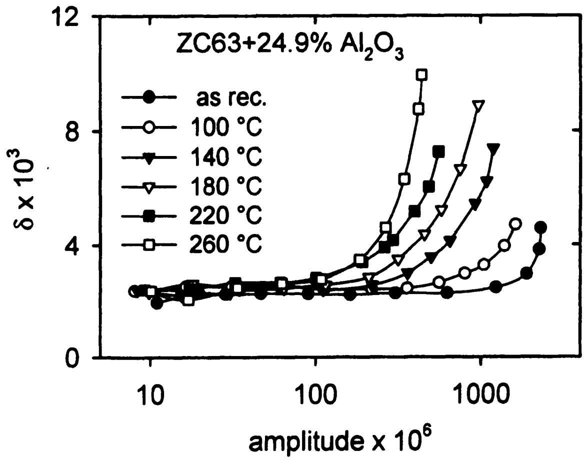

Fig. 2a. Amplitude dependence of decrement obtained for lower temperatures of thermal treatment.

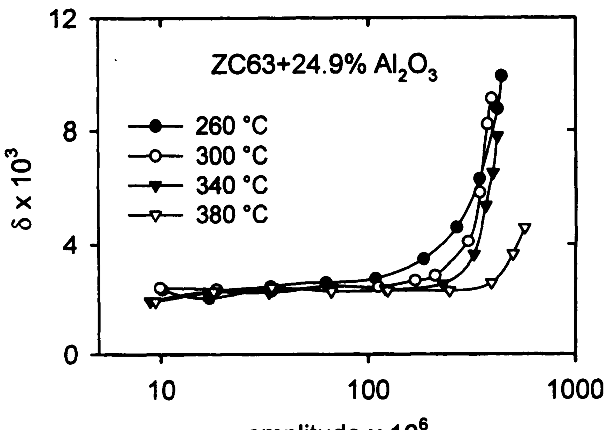

Fig. 2b. Amplitude dependence of decrement for composite with ZC63 matrix, treated at increasing temperature.

ture. Thermal cycling at temperatures higher than this critical temperature has an opposite influence. The decrement decreases with the upper temperature of the cycle. This critical temperature was found to be for $AZ91/Al_2O_3$ 280 °C, $ZC63/Al_2O_3$ 260 °C (temperature 280 °C was not measured), $ZE41/Al_2O_3$ 300 °C, for QE22 the critical temperature about 360 °C.

In the first region, for lower strain amplitudes, the decrement is only weakly dependent on the strain amplitude. In the second region, for higher strains (stresses), the decrement increases strongly with increasing strain amplitude. A number of possible damping mechanisms can be identified in metal matrix composites (MMCs). Some of them are connected with the interface, which can be very effective source

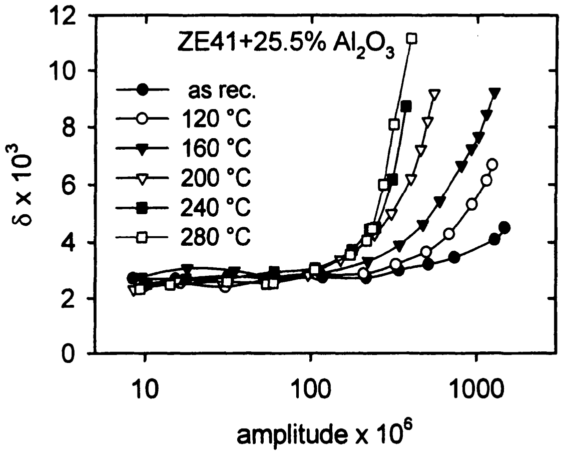

Fig. 3a. Amplitude dependence of decrement for composite with ZE41 matrix, treated at increasing temperatures.

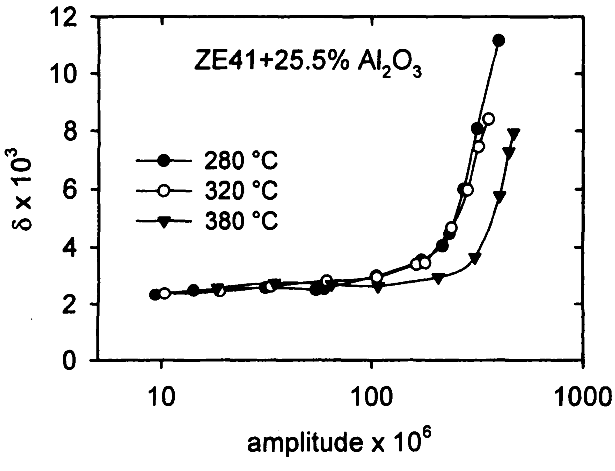

Fig. 3b. Amplitude dependence of decrement obtained for higher temperatures.

of internal friction (interfacial frictional sliding, local dissipative interfacial processes, interfacial diffusion). Grain boundary sliding can occur in the same way as in the unreinforced materials, but the presence of a finer grain size in most MMCs means that there may be rather more scope for this process to occur.

On the other hand, it is evident that, for ceramic reinforcements (with the little probability of internal dislocation motion or grain boundary sliding), the damping effect from these mechanisms will be reduced in proportion to the remaining volume fraction of the matrix in MMC. All these effect may influence the amplitude independent δ_0 component, while the amplitude dependent component δ_H is caused by dislocation vibrations in the material.

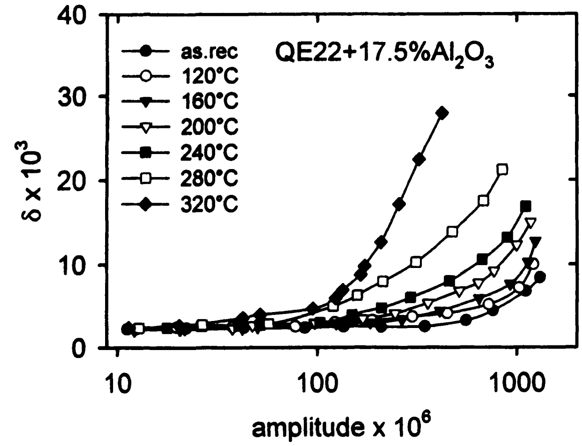

Fig. 4a. Amplitude dependence of decrement for composite with QE22 matrix, thermally cycled at increasing temperature.

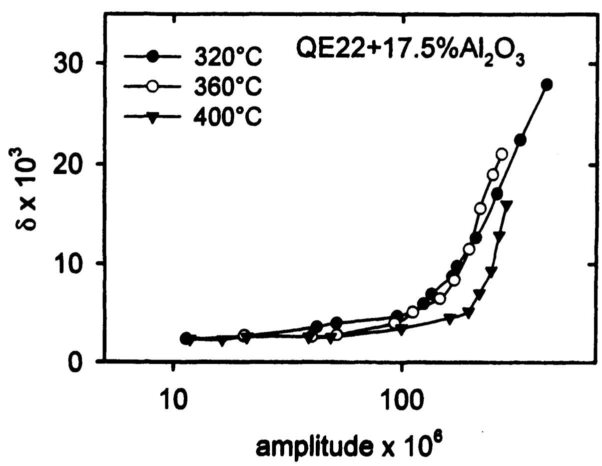

Fig. 4b. Amplitude dependence of decrement measured for higher temperatures of thermal cycling.

The presented experimental data indicate microstructure changes in the sample. An increase of internal stresses due to the difference in CTE is very probably responsible for these changes. The internal stresses produced by thermal loading of composites can be reduced by various relaxation mechanisms: creation of dislocations, their glide, by decohesion or sliding of the matrix-reinforcement interface, by diffusion of solute atoms in the matrix. It is expected that the dislocation density near the reinforcement is significantly higher than that elsewhere inside of the matrix. The dislocations formed during cooling from an elevated temperature below a certain temperature T_C, can glide inducing plastic deformation in the matrix.

The strain amplitude dependence of the logarithmic decrement suggests dislocation unpinning processes. The differences in the damping behaviour of specimens thermally cycled to various upper temperatures can be attributed to the interaction between dislocations and point defects including small clusters of foreign atoms and to changes in the dislocation density. Observations presented here are consistent with there being more, but less mobile, dislocations present in composites.

The strong strain dependence of the logarithmic decrement of composites studied shown in Fig. 1 to Fig. 4 may be explained using the Granato-Lücke theory of dislocation damping. In the Granato-Lücke theory [6] the dislocation structure is assumed to consist of segments of L_N along which weak pinning points are randomly distributed. The mean distance between two weak points is ℓ with $\ell \ll L_N$. The mean total density of dislocations is ϱ. The periodic stress $\sigma = \sigma_0 \sin \omega t$ is applied. At $T = 0$ K and at sufficiently high stress the dislocation is able to break-away from the weak pinning point. The longer segments L_N are assumed to be unbreakable pining points. The stress required for break-away of dislocations is determined by the largest double loop in a segment and it is strongly dependent on the distribution of the pinning points. With increasing temperature the stress is decreased because the break-away process is thermally activated [7]. At higher temperatures the break-away can occur at lower stresses than possible for double loop, but higher activation energies are required because the break-away is simultaneous from several neighbouring pinning points. In the high temperature and low frequencies approximation the stress dependence of the decrement component δ_H can be expressed as [7]

$$\delta_H = \frac{\varrho L_N}{6} \frac{v}{\omega} \left(\frac{3\pi \mathrm{k}T}{2U_0}\right)^{1/2} \left(\frac{\ell^3 \sigma_0^2}{U_0 G}\right)^{1/2} \exp\left[-\frac{4}{3} \frac{U_0}{\mathrm{k}T} \left(\frac{U_0 G}{\ell^2}\right)^{1/2} \frac{1}{\sigma_0}\right] \quad (9)$$

here G is the shear modulus, σ_0 is the amplitude of the applied stress and ω its frequency, v the dislocation frequency, U_0 is the activation energy, kT has its usual meaning. It can be seen that this relationship has a similar form as the original formula given by Granato and Lücke [6]. The σ_H component depends exponentially on the stress amplitude. With increasing upper temperare of the thermal cycle the decrement component δ_H increases too. The observed behaviour may be explained if we consider that during cooling and also during thermal cycling new dislocations are created due to the difference in the CTE and/or that new pinning points on existing dislocations are formed by reactions between the matrix and the reinforcement. Number of free foreign atoms or their small clusters can be modified by thermodynamic processes in the matrix. A redistribution of solute atoms may be studied by electrical resistivity measurents. Residual resistivity ratio RRR ($RRR^{-1} = \varrho_e$ (77 K)/ϱ_e(293 K)) measured for step by step annealed sample at increasing temperature is introduced in Fig. 5 for ZC63/Al_2O_3 MMC. A sharp drop in RRR^{-1} detected in the temperature interval from 160 to 240 °C is due to solute redistribution. This decrease of the resistivity reflects very complex processes, precipitates of various types can be formed in the matrix. The absence of the

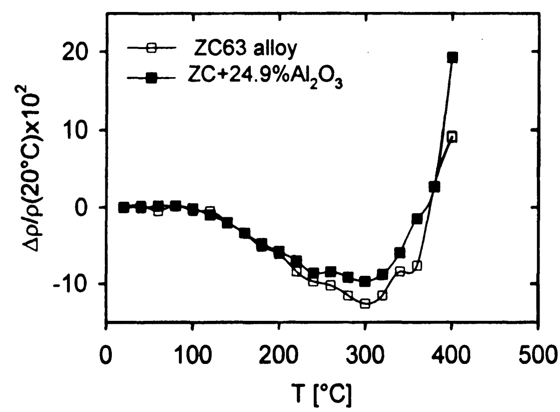

Fig. 5. Temperature dependence of residual resistivity ratio measured for alloy and composite.

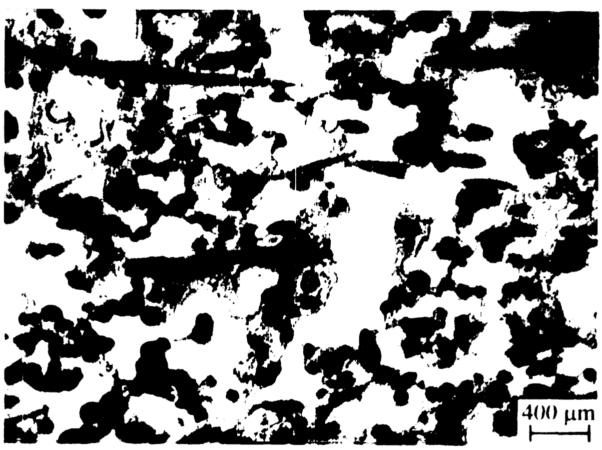

Fig. 6. Microstructure of AZ91 MMC.

expected ageing effect might be due to te massive CuMgZn consuming a high amount of alloying elements. Electrical resistivity measurements have shown that the precipitation processes occur in all Mg based MMCs studied approximately between 200 and 300 °C [8, 9]. Similarly as in the case of ZC63 MMC, complex precipitates are formed in the matrix [10]. $Mg_{17}Al_{12}$ particles of β-phase arise in AZ91 MMC [8] and MgZnRE pseudoternary phases in ZE41 MMC [10] or (Al_xMg_{1-x}) Nd cubic particles and tetragonal $Mg_{12}Nd$ in QE22 composite [11]. Microstructure of the AZ91 MMC is introduced in Fig. 6. Saffil fibres as well as intermetallic phase Al_2O_3 decorating α grains are visible. The reduction in solute content of the matrix in the temperature range between approximately 200 and 300 °C leads to an increase of the mean distance between two weak pinning points ℓ. It seems that the main weak pinning points are the solute atoms or their small clusters.

The stress which is necessary for a thermal break-away of dislocation loops σ_T at a finite temperature is given by [7]

$$\sigma_T = \sigma_M \left[1 - \left(\frac{kT}{U_1} \ln A \right)^{2/3} \right] \tag{10}$$

with

$$A = \frac{2}{3} \frac{\nu}{\omega} \frac{\sigma_M}{\sigma_0} \left(\frac{kT}{U_1} \right)^{2/3}. \tag{11}$$

σ_M is assumed to be break-away stress in pure mechanical process. For a double loop with the loop lenght ℓ_1 and ℓ_2 it occurs at the stress

$$\sigma_M = \frac{2F_m}{b(\ell_1 + \ell_2)}. \tag{12}$$

Here F_m is the maximum force between the dislocation and the pinning point. $U_1 = 4/3\,(F_m^3/\Phi)^{1/2}$, where Φ is a constant. All experiments were performed at ambient temperature. Then, we can assume that the quantities A and U_1 are, in the first approximation, independent of thermal treatment. After a thermal treatment shorter dislocation segments becomes longer because of the enhanced dislocation density due to temperature cycling and due to the lower number of solute atoms. Then, the critical stress according to (12) that is indirect proportional to ℓ should decrease with the increasing upper temperature of the cycle.

The experimental data were analysed using (9) in the form $\delta = \delta_0 + C_1\varepsilon \exp(-C_2/\varepsilon)$. Values of C_2 parameter are introduced in Fig. 7 to Fig. 10. Good correlation with the theory presented was found only for temperatures higher than 100 °C. Very probably Granato and Lücke theory which was constructed for single crystals of pure metals may be applied only for newly created dislocations that are relatively free only slightly pinned by solute atoms.

From Fig. 7 to Fig. 10 it follows that the C_2 constant decreases with increasing upper temperature of the thermal cycle. This tendency stopped at tempertures between 200 – 300 °C (depending on the matrix alloy) and then it again increases with temperature. Thermal stresses produced at the matrix/ceramic fibre interfaces are accommodated by the formation of plastic zones.

The radius of this plastic zone is given by the following approximate relationship [12]

$$r_{\text{plz}} = r_{\text{f}} \left[\frac{4\, \Delta a E_M\, \Delta T}{(5 - 4\nu)\, \sigma_{\text{y}}} \right]^{1/2}, \tag{13}$$

where E_{M} are Young's moduli of fibres and matrix, respectively, ν is Poisson

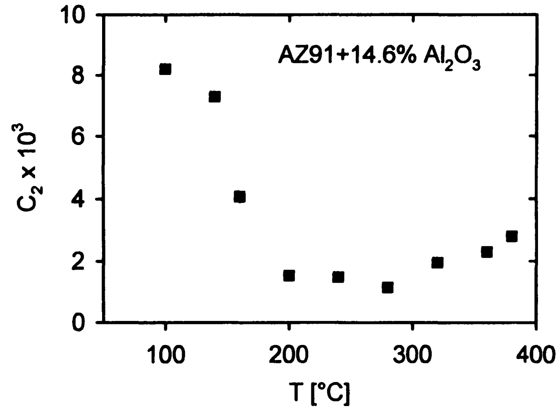

Fig. 7. Dependence of C_2 parameter on the upper temperature of the thermal cycle for AZ91 alloy based composite.

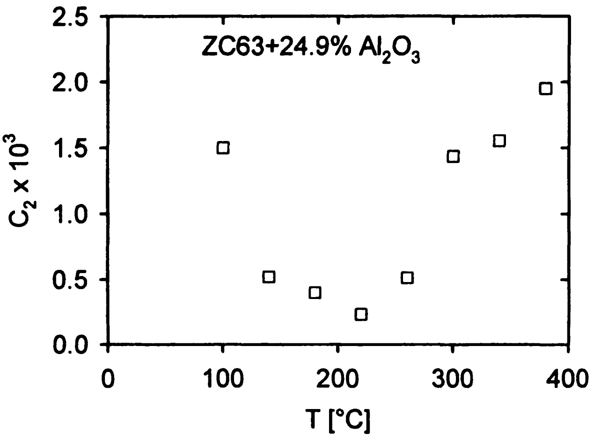

Fig. 8. Dependence of C_2 parameter on upper temperature of the thermal cycle for ZC63 alloy based composite

constant and σ_y the yield stress in the matrix, r_f is radius of fibres. Similarly it is possible to express the volume fraction of the plastically deformed matrix [13]

$$f_{\rm plz} = f\left[\frac{4\,\Delta\alpha E_{\rm M}}{(5-4\nu)\sigma_y}\cdot\Delta T - 1\right]. \tag{14}$$

If the volume fraction increases above a certain value the plastic zones can overlap. The dislocation density can increase only up to the moment when the plastic zones in the matrix begin to overlap. Consequently the dislocation loops formed near the interfaces have the opposite sign on the both sides of the fibre. At higher temperatures the yield stress in the matrix is lower than internal stress and at

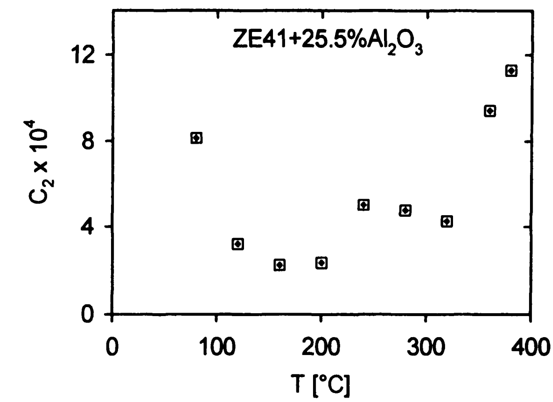

Fig. 9. Dependence of C_2 parameter on upper temperature of the thermal cycle for ZE41 alloy based composite

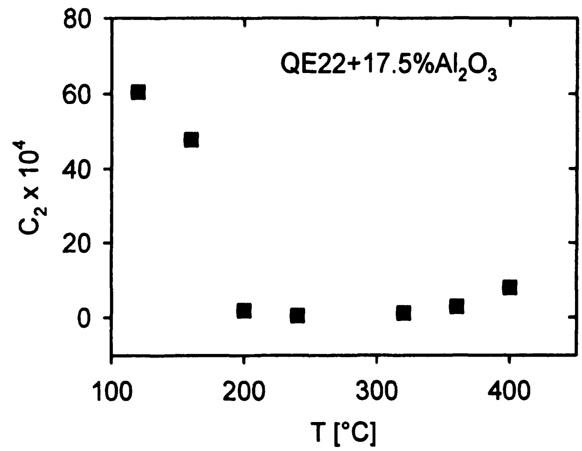

Fig. 10. Dependence of C_2 parameter on upper temperature of the thermal cycle for QE22 alloy based composite

temperatures higher than 260 – 340 °C the tensile stresses change to compression ones. At this moment dislocations move in the plastic zones and annihilation of dislocations can occur and hence the dislocation density decreases.

It has been shown that, under conditions similar to those used in these experiments, more than 1000 thermal cycles are needed in order to produce any measurable damage in the samples [14]. Since the MMCs were fabricated by squeeze casting at an elevated temperature, the composites contain thermal residual stresses at room temperature due to the mismatch in the thermal expansion coefficients between the matrix and the renforcement [15] and the magnitude of these stresses is of the order of the minimum stress required for creep in the matrix. In practice, the matrix in a MMC experiences tensile stresses whereas the fibre reinforcements experiences compressive streses. Therefore, when the MMC is heated, the internal tensile stress acting on the matrix reduces to zero and, on further heating, there is a build up of compressive stresses; whereas on cooling, the internal stresses behave in the opposite sense. It is anticipated these stresses will be concentrated near the matrix-fibre interfaces and at the ends of the reinforcing fibres. These thermal stresses may also exceed the matrix yield stress within discrete temperature ranges and relaxation will the occur trough the generation of new dislocations and plastic deformation within the matrix.

Chmelík et al. [15, 16] have measured acoustic emission of samples thermally cycled at increasing upper temperature of the thermal cycle. Permanent elongation of the sample was detected at elevated temperatures from 200 °C (in the case of AZ91 MMC) to 280 °C, then rapid shortening of the sample after cycling at temperatures higher. These temperatures were different for various magnesium alloys used as the matrix alloys, but in each case the critical temperature at which the sample became to be shorter accord with the critical temperature obtained in the internal friction experiment. Decrease of the amplitude dependent component of the decrement is observed in the region, when the sample is plastically deformed by thermal stresses. Acoustic emission signal was detected only in the cooling parts of the thermal cycle, while high temperature strain of the sample was completely noiseless.

The model of longitudinal strain response of the unidirectional reinforced composite during thermal cycling was developed by Garmong [17]. It was assumed that the fibre remains completely elastic throughout the whole temperature range whereas the matrix can elasto-plastic deform by creep. Further, it was assumed that there is no sliding on the interface between matrix and fibres during heating and the effect of transverse stress due to Poisson ratio differences and CTE mismatch along the transverse direction between the matrix and the fibres were ignored.

The plastic deformation may occur as dislocation glide, as twinning or possibly as grain boundary sliding at higher temperatures depending upon the testing temperature and the crystallographic structure of the matrix. In general, it is reasonable to ancicipate that the compressive deformation that appears on heating will give some form of diffusion-controlled high temperature creep whereas the tensile deformation appearing on cooling will lead to dislocation glide and twinning. Thus, and in support of the experimental observations, acoustic emission (AE) was indicated during thermal treatment of the MMC sample [16, 17]. A larger AE is observed during cooling at the lower temperatures. The stress for dislocation glide in matrix increased and then the creep strain at higher temperatures in the heating part of the cycle has a higher value than dislocation deformation in the cooling part of the cycle. This assymetry is not significant and it is compensated by tensile strain at higher upper temperatres of the cycle.

4. CONCLUSIONS

The thermal cycling response of four magnesium alloys reinforced by short Saffil fibres is characterised by thermal stresses which can relax by anelastic as well as elastic strain. New dislocations created in the vicinity of fibres ends can be detected by internal friction measurements.

From the internal friction measurements it can be concluded that changes in the microstructure occur at temperatures above about 200 °C. These changes are very probably connected with migration of solute atoms and precipitation processes. Thermal internal stresses generated at temperatures higher than 220 – 280 °C are high enough to invoke motion of new dislocations. Thermal cycling at temperatures higher than $\sim$ 300 °C causes movement and annihilation of new dislocations in the matrix, under appearing compressive internal stresses, which leads to a cedrease of the decrement.

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