

## TEMPERATURE DEFORMATION OF THE AX41 COMPOSITES STUDIED BY THE DILATOMETER MEASUREMENTS

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### Abstract

Magnesium alloy AX41 (Mg-4Al-1Ca in wt. %) reinforced with 15 vol.% of short Saffil fibres and AX41 hybrid composite with 5 vol.% of Saffil fibres and 15 vol.% of SiC particles were used in this study. Thermal expansion was measured over a wide temperature range from 22 up to 400 °C in four runs. The thermal expansion coefficient was estimated during heating and cooling. The Young's modulus of the fibre composite was estimated and the modulus defect was compared with the relative change of the thermal expansion coefficient. Dilatation characteristics of the pre-deformed hybrid composite were determined in the same temperature range as in the case of the fibre composite.

### 1 Introduction

Light alloys reinforced with short fibres or particles have unique and desirable thermal and mechanical properties [1]. When compared with monolithic metals and alloys, metal matrix composites (MMCs) have higher strength, Young's modulus, wear resistance, fatigue resistance, and lower thermal expansion [2]. Investigations of mechanical and physical properties of light metals composites (among them magnesium alloys based composites) are important not only for applications but also for better understanding of the processes responsible for their behaviour. An attractive attribute of MMCs is the ability to tailor the thermal conductivity and the coefficient of thermal expansion (CTE). This can be achieved by careful control of the low expansion, reinforcement volume percent, particle size, and particle packing characteristics [3-4].

It is known that in composites, there is a large difference in the coefficients of thermal expansion between the matrix and the ceramic reinforcement. When a metal matrix composite is cooled from a higher temperature to room temperature, misfit strains occur because of different thermal contraction at the interfaces. These strains induce thermal stresses that may be higher than the yield stress of the matrix. Therefore, the thermal stresses may be sufficient to generate new dislocations at the interfaces between the matrix and the reinforcement. Accordingly, after cooling the composite, the dislocation density in the matrix is higher than in unreinforced matrix. An increase in the density of newly created dislocations near reinforcement fibres has been calculated as [5-6]

$$\Delta\rho = \frac{Bf\Delta\alpha_c\Delta T}{b(1-f)t}, \quad (1)$$

where  $f$  is the volume fraction of the reinforcement,  $t$  is its minimum size,  $b$  is the magnitude of the Burgers vector of dislocations,  $B$  is a geometrical constant,  $\Delta\alpha_c = \alpha_f - \alpha_m$  is the difference in the CTEs of the reinforcement and the matrix, and  $\Delta T$  is the temperature change. The yield stress of MMC decreases with increasing temperature. At a certain temperature, the values of thermal stresses at the interfaces may exceed the yield stress of the matrix, and plastic flow can occur. A permanent elongation as well as contraction of the sample length during thermal exposition was observed by several authors [7–8]. The sample exhibited a residual contraction (or elongation) upon cooling to room temperature, while the temperature dependence of the thermal expansion coefficient was measured. The value of the observed permanent (residual) change of the sample length after the measurement of thermal expansion depends on the matrix, volume fraction of reinforcement, and the maximum temperature of the thermal cycle. The thermal expansion behaviour may be characterised by the temperature dependence of the thermal strain parameter  $(\Delta L/L_0)_{th}$  defined by the following equation [9]:

$$\left(\frac{\Delta L}{L_0}\right)_{th} = \left(\frac{\Delta L}{L_0}\right)_{exp} - \left(\frac{\Delta L}{L_0}\right)_{rm}, \quad (2)$$

where  $(\Delta L/L_0)_{exp}$  is the measured strain of the composite sample,  $(\Delta L/L_0)_{rm}$  is the strain of the composite sample calculated by the rule of mixtures and  $L_0$  is the original length of the sample. The rule of mixtures is a function of the volume fraction of the reinforcement and the strain of the matrix and the reinforcement. Parameter  $(\Delta L/L_0)_{th}$  represents the departure of the composite behaviour from the case where no thermal stresses are present. It should be noted that  $(\Delta L/L_0)_{exp}$  is determined by dilatometer in the axial direction of the sample.

## 2. Experimental

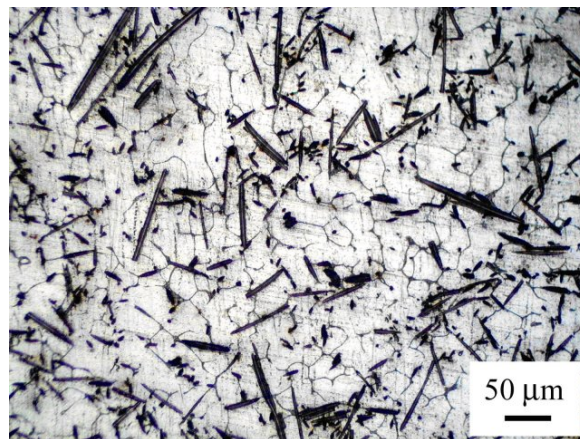
A commercial magnesium alloy AX41 (4wt%Al, 1wt%Ca, balance Mg) was used as the matrix. The alloy was reinforced with 15 vol.% of  $\delta$ -Al<sub>2</sub>O<sub>3</sub> short fibres (Saffil<sup>®</sup>) – in this paper we call this composite as fibre composite, or 5 vol.% of Saffil fibres and 15 vol.% of SiC particles (hybrid composite). The short fibres showing a planar isotropic distribution had the mean diameter of 3  $\mu$ m and their length was 78  $\mu$ m (measured after squeeze casting). The diameter of SiC particles was 5  $\mu$ m approximately.

Cylindrical samples for the thermal expansion measurements had a length of 50 mm and a diameter of 6 mm. The planes of planar randomly distribute fibres were parallel to the longitudinal axis of the samples. Figure 1 shows the microstructure (light micrograph) of as prepared fibre composite taken from the fibres plane.

The linear thermal expansion of the composite samples was measured in an argon atmosphere, using the Netzsch 410 dilatometer, over a temperature range from room temperature to 400 °C for heating and cooling rates of 0.9 °C/min. The accuracy of the apparatus was controlled by measuring of the coefficient of thermal expansion of pure Al and comparing it with the literature data. The agreement between measured and tabled data was in the range  $\pm 1\%$ . The thermal expansion curves of composites were measured in four thermal (heating and cooling) cycles (runs). The results obtained in the third and fourth thermal cycles were practically the same as those in the second cycle. The relative changes in the thermal

expansion coefficient  $\Delta\alpha/\alpha=(\alpha(T)-\alpha(25^\circ\text{C}))/\alpha(25^\circ\text{C})$  were calculated for the fibre composite.

The Young's modulus  $E$  for fibre composite was estimated after each thermal cycle by measurement of the resonant frequency of free vibration of the sample at ambient temperature [10]. Thermal cycles between room temperature and an increasing upper temperature were performed step by step up to 430 °C. The temperature step was 20 °C and duration of the sample at each temperature 15 minutes. The modulus defect  $\Delta E/E=(E-E_0)/E_0$  (where  $E_0$  is the modulus value without thermal treatment) was estimated.

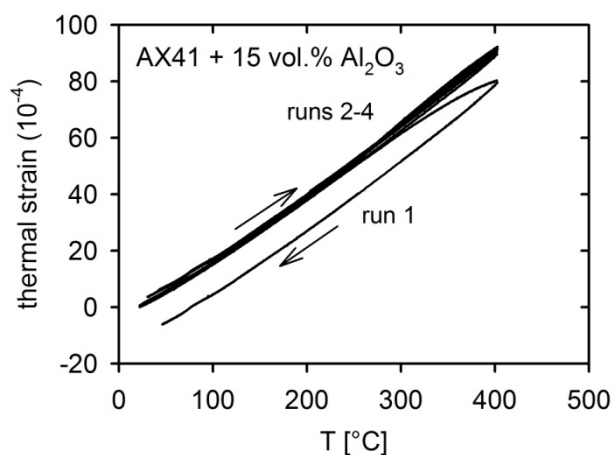


**Figure 1.** Microstructure of the sample of the investigated material.

### 3. Results

#### 3.1 Fibre composite

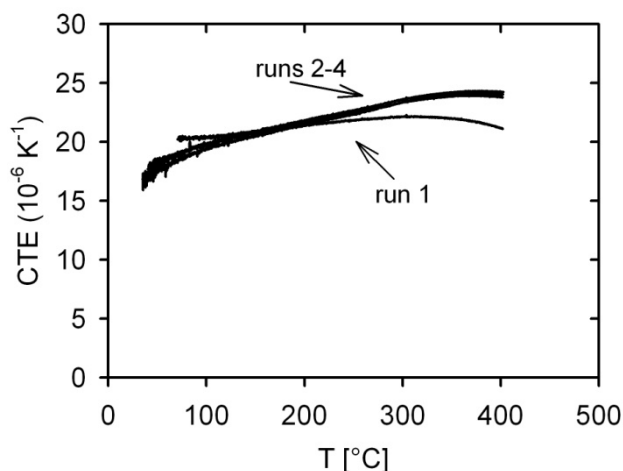
Figure 2 shows the temperature dependences of the thermal strain obtained during the four thermal cycles.



**Figure 2.** Temperature dependence of the thermal strain obtained during 4 thermal cycles (fibre composite).

From the figure can be seen that the curves obtained in the second, third and fourth run are practically the same. Also the estimated difference between the heating and cooling part of the dependence is in the second and following cycles smaller. The temperature dependences of the CTE for heating, measured for four thermal cycles, are shown in Fig. 3. The addition of Saffil fibres decreases the CTE from  $25 \times 10^{-6} \text{ K}^{-1}$  estimated for the alloy to  $20 \times 10^{-6} \text{ K}^{-1}$  for the

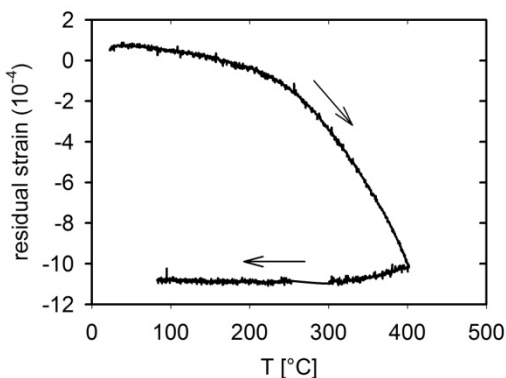
composite at room temperature. Further thermal cycling led to the additional decrease of the CTE to approximately value of  $16 \times 10^{-6} \text{ K}^{-1}$ .



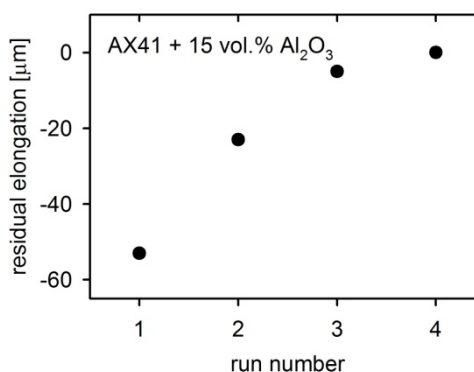
**Figure 3.** Temperature dependence of the CTE of the composite under heating.

The dilatation characteristics are also influenced by the residual strain, which is connected with a permanent change of the sample length. The temperature dependence of the residual strain was obtained by subtracting the relative elongation obtained in the first run and the relative elongation in the second thermal cycle. It is introduced in Fig. 4.

The permanent changes in the sample length (residual contraction) measured after individual thermal cycles are shown in Fig. 5.



**Figure 4.** Residual strain in fibre composite.

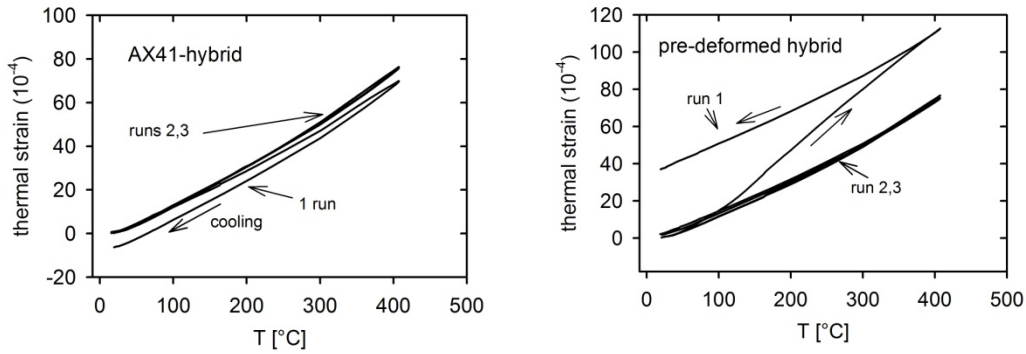


**Figure 5.** Residual elongations (fibre composite).

### 3.2 Hybrid composite

The linear thermal expansion of the hybrid composite was measured using a Netzsch 410 dilatometer. The first thermal cycle was measured for the sample pre-deformed in compression in a direction of the longitudinal axes of the sample in an INSTRON testing machine (room temperature, strain rate of  $3.3 \times 10^{-5} \text{ s}^{-1}$ , plastic deformation of 1%). Non-deformed sample was used for the comparison purpose.

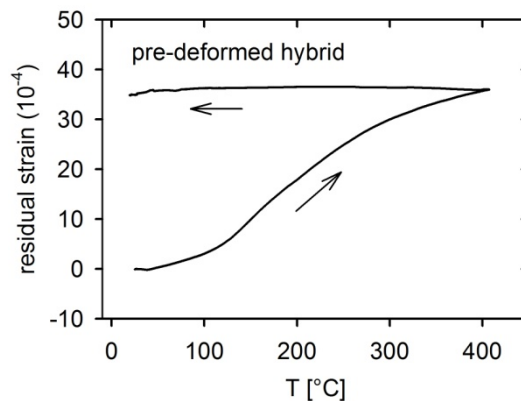
The temperature dependences of the thermal strain obtained during three thermal cycles (runs) for the hybrid composite are introduced in fig. 6a,b. (For both, the pre-deformed and comparative, samples.)



**Figure 6a, 6b.** Temperature dependences of the thermal strain obtained for hybrid composite and pre-deformed hybrid composite.

Residual strains were also calculated for hybrid composite. It can be seen from figure 7 that the residual strain achieve quite high value for the pre-deformed sample. (The residual dilatation of the sample was 180  $\mu\text{m}$  after the first thermal cycle.)

The possible explanation of this observation is in the large amount of twins arising in the sample during the plastic pre-deformation. These twins are removed by a heating cycle in the dilatometer. The removal of twins is a thermal activated process and it takes place during heating [11].



**Figure 7.** Temperature dependence of the residual strain in hybrid composite measured after pre-deformation in compression (1% of plastic strain).

#### 4. Discussion

Using the Grüneisen theory of thermal expansivity [12], it follows  $(C_p - C_v) = (\alpha^2 TV_0)/K$ , and from isotropic elasticity,  $K = 3(1 - 2\nu)/E$ . Hence, the following relationship between the thermal expansion coefficient and the Young's modulus can be written:

$$\alpha^2 = \frac{(C_p - C_v)3(1 - 2\nu)}{TV_0E}, \quad (3)$$

here  $C_p$  and  $C_v$  are the specific heats at constant pressure and volume,  $V_0$  the molar volume,  $K$  the compressibility modulus and  $\nu$  the Poisson ratio. The term  $(C_p - C_v) \cdot 3 \cdot (1 - 2\nu) / V_0$  is a constant (at certain temperature  $T$ ). So the relationship between the modulus defect and relative change of the thermal expansion coefficient is:

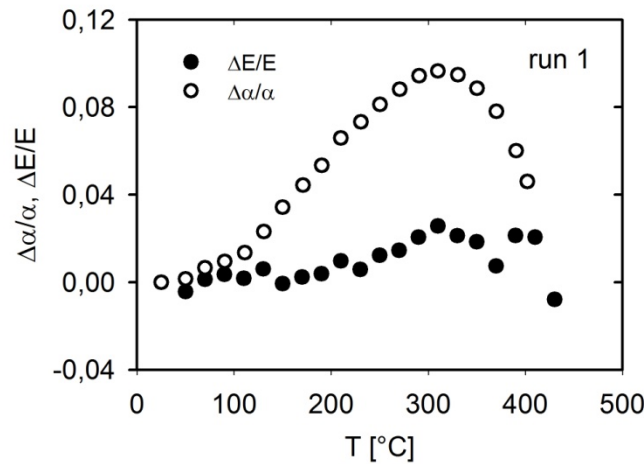
$$\frac{\Delta\alpha}{\alpha} = -\frac{1}{2} \frac{\Delta E}{E} \quad (4)$$

The modulus defect  $\Delta E/E$  ( $= (E-E_0)/E_0$ , where  $E_0$  is the modulus value without thermal treatment) is given by the following equation

$$-\Delta E / E = \gamma \rho \ell^2, \quad (5)$$

where  $\gamma$  is a constant,  $\rho$  is the dislocation density and  $\ell$  the length of dislocation segments between weak pinning points (solute atoms, point defects).

The temperature dependences of the  $\Delta\alpha/\alpha$  and the modulus defect  $\Delta E/E$  are given in Fig. 8.



**Figure 8.** Temperature dependences of the relative changes of the CTE and the  $E$ -modulus defect.

Note, that the modulus defect is plotted against the upper temperature of the thermal cycle. It can be seen that both dependences exhibit a maximum at a temperature of 310 °C.

Temperature changes during the thermal cycle invoke thermal stresses in the matrix which may reach its yield stress and generate new dislocations in plastic zones. The radius of the plastic zone is given by the following approximate relationship [13]

$$r_{plz} = r_f \left[ \frac{4\Delta\alpha E_M}{(5-4\nu)\sigma_y} \Delta T \right]^{1/2}, \quad (6)$$

where  $E_M$  is Young's modulus of the matrix,  $\nu$  is Poisson constant,  $\sigma_y$  the yield stress in the matrix and  $r_f$  is the radius of fibres. Similarly, it is possible to express the volume fraction of the plastically deformed matrix [14]

$$f_{plz} = f \left[ \frac{4\Delta\alpha E_M}{(5-4\nu)\sigma_y} \Delta T - 1 \right], \quad (7)$$

where  $f$  is volume fraction of the reinforcing phase. Fresh dislocations in plastic zones, produced by different expansion behaviour of the matrix and the reinforcements due to temperature changes, are only slightly pinned and contribute substantially to modulus defect

[13]. Plastification of the matrix is due to newly created dislocations. An increase in the dislocation density near reinforcement can be calculated according the equation (1). Considering that the number of pinning points is constant, the effective length of dislocation segments increases with increasing dislocation density. The similar situation can be considered in the sample thermally cycled in the dilatometer. Although the thermal expansion coefficient was determined on the specimen heated in the dilatometer, the increase of the dislocations density may be considered due to thermal stresses generated in the matrix. It is suggested that, in addition to the regular lattice temperature expansion, the extension of mobile dislocation loops, which are a result of the accommodation of the thermal stresses, causes localized increases in volume and an increase in the apparent expansion coefficient. An increasing tendency of the modulus and CTE defects stopped at a temperature of 310 °C. Thermal loading up to higher temperatures decreases the both defects. The radius of the plastic zones in the vicinity of fibres was estimated according to relationship (5) to be about 11 µm. The volume fraction of plastic zones exhibits after thermal treatment at 310 °C about 82 % of the matrix volume (substituting into relationship (6) for  $\Delta\alpha=20\times 10^{-6} \text{ K}^{-1}$ ,  $r_f=3 \text{ }\mu\text{m}$ ,  $E_M=45 \text{ GPa}$ ,  $\nu=0.35$ ,  $\sigma_y=45 \text{ MPa}$  [15]). The plastic zones in the matrix may overlap and dislocations may annihilate. Thermal stresses in the AX41 alloy matrix which are originally tensile convert to compression ones [16]. Dislocations moving under arising compression stresses in the inverse direction interact with dislocations of opposite sign and may annihilate. Relatively high maximum strain amplitude renders possible movement of free dislocation segments in the slip plane. Solute atoms and their small clusters are obstacles for the dislocation motion. After loading at higher temperatures, increasing concentration of vacancies interacts with dislocations producing jogs on the dislocation lines. Thermally activated motion of dislocation segments is restricted and the both defects decrease. A deviation from the temperature dependence of the CTE is observed. Values of the CTE decrease with temperature above about 310 °C (Fig. 3). The yield stress of the matrix decreases with increasing temperature. The internal stress state in the matrix is tensile. It is decreasing during heating and the state becomes compressive at some temperature. At a certain temperature plastic deformation in the matrix occurs. This deformation induces changes in the internal stress, which modify the elastic properties in the matrix. The specimen length is reduced under compression stresses. This leads to a decrease in the CTE, which is observed. Thus, decreasing dislocation density reduces the additional thermal expansion coefficient.

## 5. Conclusions

The thermal expansion of the AX41 fibre and hybrid composites was measured over a wide temperature range from ambient temperature up to 400 °C in four runs. The addition of Saffil fibres decreases the CTE. This decrease is the maximum in the first run; in the second and other runs the value of the CTE is practically the same. The fibre composite was thermally cycled between room temperature and increasing upper temperature of the thermal cycle up to 430 °C. After each thermal cycle the resonant frequency of the free vibrations of the sample was measured. The modulus defect  $\Delta E/E$  (calculated from the resonant frequency) and the relative change of the CTE  $\Delta\alpha/\alpha$  exhibit the similar temperature dependence with a local maximum at 310 °C. Considering formation of plastic zones as a consequence of thermal stresses invoked in the matrix, newly created dislocations are the reason for the observed changes. Thermal loading at temperatures higher than 310 °C yields a new situation: Thermal internal stresses generated at temperatures higher than 310 °C are high enough to invoke motion of new dislocations. Thermal cycling at temperatures higher than 310 °C causes movement and annihilation of new dislocations in the matrix, under appearing compressive internal stresses, which leads to a decrease of the  $\Delta\alpha/\alpha$  and the absolute value of the modulus

defect. Heating of the hybrid composite probably leads to recovery of twins which were created in the sample during compressive pre-deformation.

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