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# **Effect of particulate content on the thermal cycling behaviour of the magnesium alloy based hybrid composites**

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## **Abstract**

The thermal cycling behaviour of a creep resistant magnesium alloy AE42 and its composites containing various volume fractions of saffil short fibres and SiC particulates has been examined. It is found that the plastic deformation due to internal thermal mismatch stresses above 250°C increases and coefficient of thermal expansion decreases with increasing volume fraction of SiC particulates. The only microstructural change observed after thermal cycling is an increased precipitation in the matrix.

Keywords: A. Hybrid; A. Metal matrix composites; B. Thermal properties B. Hysteresis; B. Residual strain; D. Thermal analysis; E. Liquid metal infiltration

## **Introduction**

The beginning of 1990s has marked the renaissance of magnesium as a structural material, the demand mainly coming from automotive industry owing to environmental concerns, increasing safety and comfort levels, a significant improvement in the corrosion resistance of high purity magnesium alloys, rising fuel prices and lowering of prices of primary magnesium metal. For powertrain components, a creep-resistant magnesium alloy AE42 has been developed in 1980s by Dow Chemical Company, USA,

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which has suitable properties up to 150°C [1]. However, magnesium alloys reach their limit at about 200°C, above which metal matrix composites (MMCs) have to be developed. Particulate reinforced Mg-MMCs might actually deteriorate the creep properties [2,3]. Therefore, short fibre reinforced MMCs have to be developed for these applications. However, they are expensive and have anisotropic properties. Hybrid composites provide the answer. The partial replacement of expensive short fibres by cheap particulates reduces the cost as well as anisotropy. The addition of particulates also helps in keeping the fibres apart, which improves mechanical properties [4,5]. Friend et al. observed that the partial replacement of saffil short fibres by SiC particulates in 7039 Al alloy-based hybrid MMCs did not affect the strength and elastic moduli but improved fracture toughness [6]. Park observed an improvement in wear resistance of 6061 Al alloy based hybrid MMC reinforced with Al<sub>2</sub>O<sub>3</sub> short fibres and SiC whiskers than that in MMCs reinforced either with Al<sub>2</sub>O<sub>3</sub> short fibres or SiC whiskers alone [7]. However, there have been only few studies on Mg alloy-based hybrid MMCs [8-10].

Thermal cycling studies are very important for metal matrix composites being developed for engine components, as they are subjected to fluctuating temperature environment in these applications. In the present study, thermal cycling behaviour of creep-resistant AE42 alloy, AE42 alloy reinforced with 20 vol% saffil short fibre as well as various volume fractions of saffil short fibre and SiC particulate has been studied.

### **Experimental procedure**

A preheated preform containing saffil short fibres (>95 vol%  $\delta$ -Al<sub>2</sub>O<sub>3</sub>, <5 vol% SiO<sub>2</sub>) or saffil short fibres and SiC particulates was infiltrated with AE42 alloy (Mg-4wt% Al-

2.5wt%RE-0.2wt%Mn) by squeeze casting. During squeeze casting a vertical hydraulic press squeezes the superheated melt into the perform with an upper ram speed of 10 m/s. The part solidifies under pressure within 150 seconds. The solidified composite contains a dense microstructure with saffil fibres having a maximum length of about 200  $\mu\text{m}$  and a diameter of about 8  $\mu\text{m}$  in a planar random array and SiC particulates with a maximum diameter of about 40  $\mu\text{m}$ .

Thermal cycling was done in a NETZSCH dilatometer DIL 402 C between a programmed temperature of 30°C and 350°C with 15 minutes of isothermal holdings at the maximum and minimum temperatures at a ramp rate of 5°C/min for 10 cycles. Pure platinum with a dimension of 4 mm diameter and 25 mm length was used as reference standard. The dimension of the test sample was 6 mm diameter and 22 mm length. It has been shown earlier that a slight difference in dimensions between reference standard and test sample does not influence the thermal cycling curves [11]. The composite specimens were tested in the longitudinal direction, i.e., the plane containing random fibre orientation was parallel to the axis of the test sample.

## **Results and discussion**

### **Residual strain and hysteresis**

The AE42 alloy is not expected to exhibit any residual strain and hysteresis, as is indeed observed in the thermal cycling curves in Fig. 1. This shows that the platinum standard serves as a good reference standard for magnesium alloy and the residual strain and hysteresis imparted by the system is completely avoided, as discussed earlier in ref. [11]. For the composites, compressive plastic deformation due to internal thermal mismatch

stress between matrix and reinforcement starts at about 250°C, as exhibited by a decrease in instantaneous CTE values ( $\alpha_T$ ) in Fig. 2 (a-d). This leads to a net residual strain and hysteresis in thermal cycling curves. The temperature at which plastic deformation starts (about 250°C) is the same as that observed earlier for QE22 alloy reinforced with 20 vol% saffil short fibres [12,13]. The maximum plastic deformation occurs in the first heating cycle, which is also in accordance with the previous results [12].

For the fibres, the thermal stresses produced by a temperature change  $\Delta T$  in a metal matrix composite are given by Carreno et al. [14]

$$\sigma_{ts} = \frac{E_f \cdot E_M}{(E_f f + E_M (1 - f))} \cdot f \Delta \alpha \cdot \Delta T$$

where  $E_f$  and  $E_M$  are the Young's Moduli of the reinforcement and of the matrix respectively,  $f$  is the reinforcement fraction and  $\Delta \alpha$  is the difference in the coefficients of thermal expansions of matrix and reinforcement. Based on these calculations, a temperature of 250°C for plastic deformation was justified for QE22 alloy reinforced with 20 vol% saffil short fibres [12]. Since AE42 alloy has similar yield strength as QE22 alloy, similar temperature for plastic deformation of AE42 alloy reinforced with 20 vol% saffil short fibres in the present case is expected. With particulate reinforcement, the global yielding by long-range dislocation glide is not expected with hydrostatic stress field. However, as the thermal stresses are not hydrostatic in every local region [15], plastic deformation by local secondary dislocation generation and motion is possible. Rudajevova et al. have observed plastic deformation at 240°C for QE22 alloy reinforced

with 25 vol%SiC particles and at 255°C for QE22 alloy reinforced with 15 vol%SiC particles. Since SiC has lower CTE value ( $4.5 \times 10^{-6} / ^\circ\text{C}$ ) as compared to saffil ( $7.7 \times 10^{-6} / ^\circ\text{C}$ ), a greater amount of plastic deformation, hence greater residual strain and hysteresis, is observed for hybrid composites in Fig. 2(a-d), when saffil fibres are partially replaced by SiC particulates.

### **Coefficient of thermal expansion**

Table 1 gives the standard average CTE values ( $\alpha_m$ ) for magnesium [16]. Fig. 1 shows the experimental average and instantaneous CTE values ( $\alpha_m$  and  $\alpha_T$ ) for all the ten heating cycles of the AE42 alloy. Since only  $\alpha_m$  values are available for magnesium and since the composites exhibit a decrease in CTE above 250°C due to plastic deformation, we shall take  $\alpha_m$  values in the temperature range 20-200°C for comparisons. An average value of  $28.5 \times 10^{-6} / ^\circ\text{C}$  from all the ten cycles in the temperature range 20-200°C is obtained for  $\alpha_m$  of the AE42 alloy from Fig. 1. For magnesium, the standard value of  $\alpha_m$  in this temperature range is  $27.1 \times 10^{-6} / ^\circ\text{C}$ . The CTE values of AE42 alloy are not expected to be different from magnesium. Therefore, the experimental  $\alpha_m$  value is found to be  $1.4 \times 10^{-6} / ^\circ\text{C}$  higher than the standard value. The same observation was also made for pure aluminium [11]. Hence, a correction of this magnitude would be made for the experimental  $\alpha_m$  values of the composites, as well.

For the AE42+20%saffil composite an average value of  $\alpha_m$  for all the ten cycles in the temperature range 20-200°C is obtained as  $19.6 \times 10^{-6} / ^\circ\text{C}$  from Fig. 2a. Subtracting a

correction factor of  $1.4 \times 10^{-6} / ^\circ\text{C}$ , this value is reduced to  $18.2 \times 10^{-6} / ^\circ\text{C}$ . This can be compared with the theoretical value of CTE obtained from Shapery's model for long fibres [17], as given below

$$\alpha_L = \frac{E_f \alpha_f f + E_m \alpha_m (1-f)}{E_f f + E_m (1-f)}$$

where,  $\alpha_L$  is the CTE value of the composite in the longitudinal direction,  $\alpha_f$  ( $7.7 \times 10^{-6} / ^\circ\text{C}$  for saffil fibres) and  $\alpha_m$  ( $27.1 \times 10^{-6} / ^\circ\text{C}$  for AE42 alloy) are the CTE values of the fibre and matrix,  $E_f$  (285 GPa for saffil fibres) and  $E_m$  (45 GPa for AE42 alloy) are Young's moduli for fibre and matrix, and  $f$  (0.2) is the volume fraction of fibre. Thus,  $\alpha_L$  is obtained as  $15.2 \times 10^{-6} / ^\circ\text{C}$ , which is  $3 \times 10^{-6} / ^\circ\text{C}$  higher than the corrected experimental value. About the same magnitude of difference was also observed for QE22+20% saffil fibres [12]. This is expected as Schapery's model considers long fibres aligned in the longitudinal direction, whereas the composite under investigation contains short fibres having planar random orientation as shown in Fig. 3 (a and b).

For the hybrid composites, the average value of  $\alpha_m$  for all the ten cycles in the temperature range 20-200°C are obtained as  $19.7 \times 10^{-6} / ^\circ\text{C}$ ,  $19.0 \times 10^{-6} / ^\circ\text{C}$  and  $15.4 \times 10^{-6} / ^\circ\text{C}$  for AE42+15% saffil+5% SiC, AE42+10% saffil+10% SiC and AE42+10% saffil+15% SiC from Fig. 2(b-d) respectively. After subtracting a value of  $1.4 \times 10^{-6} / ^\circ\text{C}$ , the corrected  $\alpha_m$  values are obtained as  $18.3 \times 10^{-6} / ^\circ\text{C}$ ,  $17.6 \times 10^{-6} / ^\circ\text{C}$  and  $14.0 \times 10^{-6} / ^\circ\text{C}$  respectively. Thus, the replacement of 5% saffil short fibres by 5% SiC particulates does not cause a change in CTE value and the replacement of 10% saffil

short fibres by 10% SiC particulates even reduces the CTE value. A further addition of 5% SiC particulates decreases the CTE value sharply to  $14.0 \times 10^{-6} / ^\circ\text{C}$ . Thus, the hybrid composites have better properties than the short fibre reinforced composite from CTE point of view.

There is no model available for the evaluation of CTE of hybrid composites containing fibres and particulates. The available models deal either with hybrid composites containing different types of short fibres [18] or with hybrid composites containing particulates and voids [19]. For the theoretical estimation of CTE, composites containing fibres and particulates, we proceed in the following way.

The CTE values for 20%, 15% and 10% saffil fibres reinforcement are obtained from Schapery's model as  $15.2 \times 10^{-6} / ^\circ\text{C}$ ,  $16.9 \times 10^{-6} / ^\circ\text{C}$  and  $19.1 \times 10^{-6} / ^\circ\text{C}$  respectively. The CTE of particulate reinforced composites can be calculated from Turner [20], Kerner [21] or Schapery [17] models. However, if the matrix modulus is much smaller than that of reinforcement, as in the present case, the CTE of the particulate reinforced composites can be calculated by a rule of mixture [22], i.e.,

$$\alpha_C = \alpha_M \cdot V_M + \alpha_P \cdot V_P$$

where,  $\alpha_M$  ( $27.1 \times 10^{-6} / ^\circ\text{C}$  for Mg) and  $\alpha_P$  ( $4.5 \times 10^{-6} / ^\circ\text{C}$  for SiC) are the CTE values, and  $V_M$  and  $V_P$  are the volume fractions of matrix and particulates respectively. Thus, the CTE values for 5%, 10% and 15% SiC particulate reinforcement are obtained as  $26.0 \times 10^{-6} / ^\circ\text{C}$ ,  $24.8 \times 10^{-6} / ^\circ\text{C}$  and  $23.7 \times 10^{-6} / ^\circ\text{C}$  respectively. Thus the addition of 5%, 10% and 15% SiC causes a decrease of  $1.1 \times 10^{-6} / ^\circ\text{C}$ ,  $2.2 \times 10^{-6} / ^\circ\text{C}$  and  $3.4 \times 10^{-6} / ^\circ\text{C}$  respectively in the CTE value of the matrix. Now, the CTE values of the hybrid



composites are estimated by a rule of mixture. For example, the CTE of the AE42+15% saffil fibres has been evaluated above from Shapery's model as  $16.9 \times 10^{-6} / ^\circ\text{C}$ . The addition of 5% SiC particulates would cause a further decrease in CTE of  $1.1 \times 10^{-6} / ^\circ\text{C}$ . Thus, the CTE of AE42+15% saffil+5% SiC is estimated as  $15.8 \times 10^{-6} / ^\circ\text{C}$  ( $16.9 \times 10^{-6} / ^\circ\text{C} - 1.1 \times 10^{-6} / ^\circ\text{C}$ ). In the same way, the theoretical CTE value of AE42+10% saffil+10% SiC is estimated as  $16.9 \times 10^{-6} / ^\circ\text{C}$  ( $19.1 \times 10^{-6} / ^\circ\text{C} - 2.2 \times 10^{-6} / ^\circ\text{C}$ ) and the CTE value of AE42+10% saffil+15% SiC is estimated as  $15.7 \times 10^{-6} / ^\circ\text{C}$  ( $19.1 \times 10^{-6} / ^\circ\text{C} - 3.4 \times 10^{-6} / ^\circ\text{C}$ ). These results are summarised in Table 2. From this estimation, it would appear that the replacement of saffil short fibres by SiC particulates would increase the CTE values. However, the opposite is observed experimentally. Therefore, it appears that the CTE of the hybrid composites is not governed by a simple rule of mixture, but the short fibres and particulates interact in a more complex way, and the reduction of CTE by the addition of particulates in a short fibre reinforced composite is greater than that estimated by rule of mixture.

Fig. 3(a and b) show the microstructure of the AE42+10% saffil+10% SiC composite as a typical example before and after thermal cycling. As can be seen, the only microstructural change observed is an increased precipitation in the matrix. Powell et al. have reported that the as-cast microstructure in AE42 alloy contains predominantly  $\text{Al}_{11}\text{RE}_3$  phase, which decomposes above  $150^\circ\text{C}$  into  $\text{Al}_2\text{RE}$  and Al forming  $\text{Mg}_{17}\text{Al}_{12}$  phase [23].

## Conclusion

The replacement of saffil short fibres by SiC particulates in the hybrid composites reduces CTE. The saffil short fibres and SiC particulates interact in a complex way and the reduction in CTE is greater than that expected by a simple rule of mixture. The replacement of saffil fibres by SiC particulates causes greater plastic deformation above 250°C arising from thermal mismatch stresses. The only microstructural change observed on thermal cycling is an increased precipitation in the matrix.

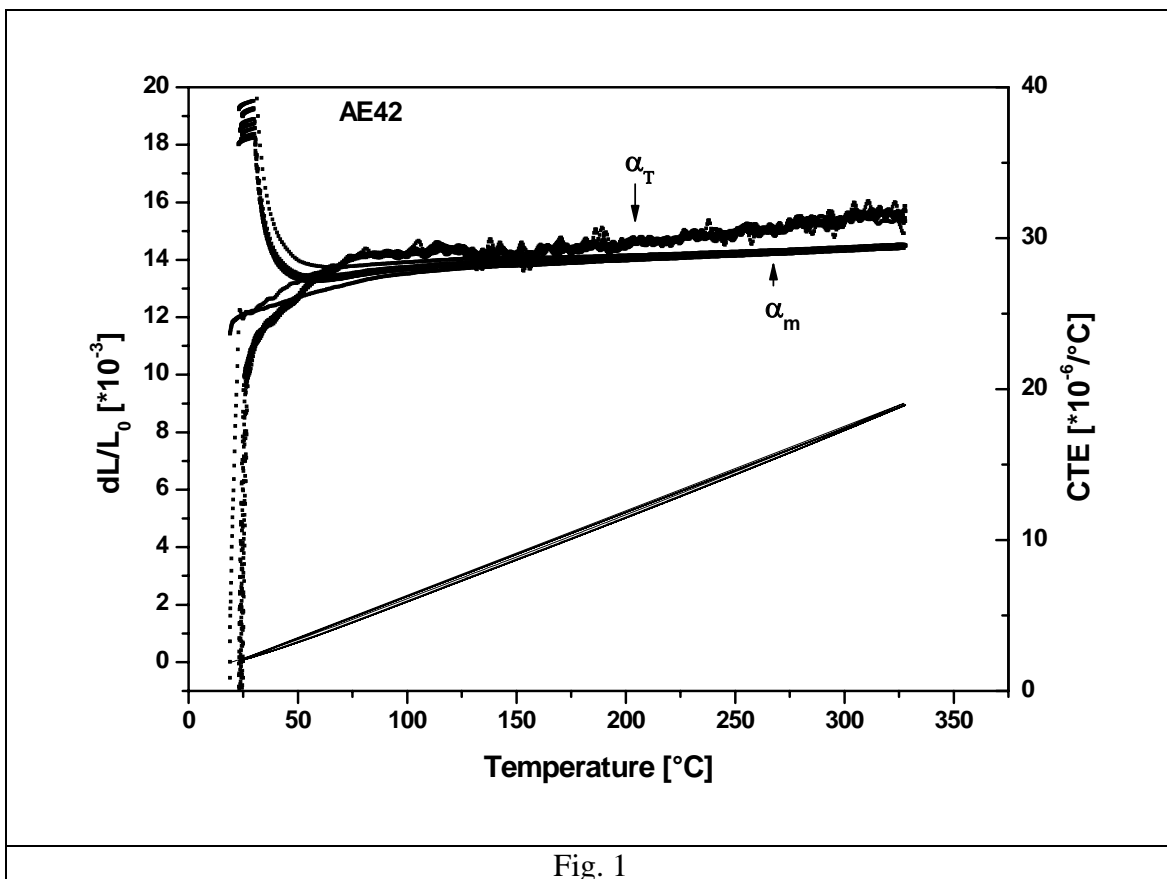


Fig. 1

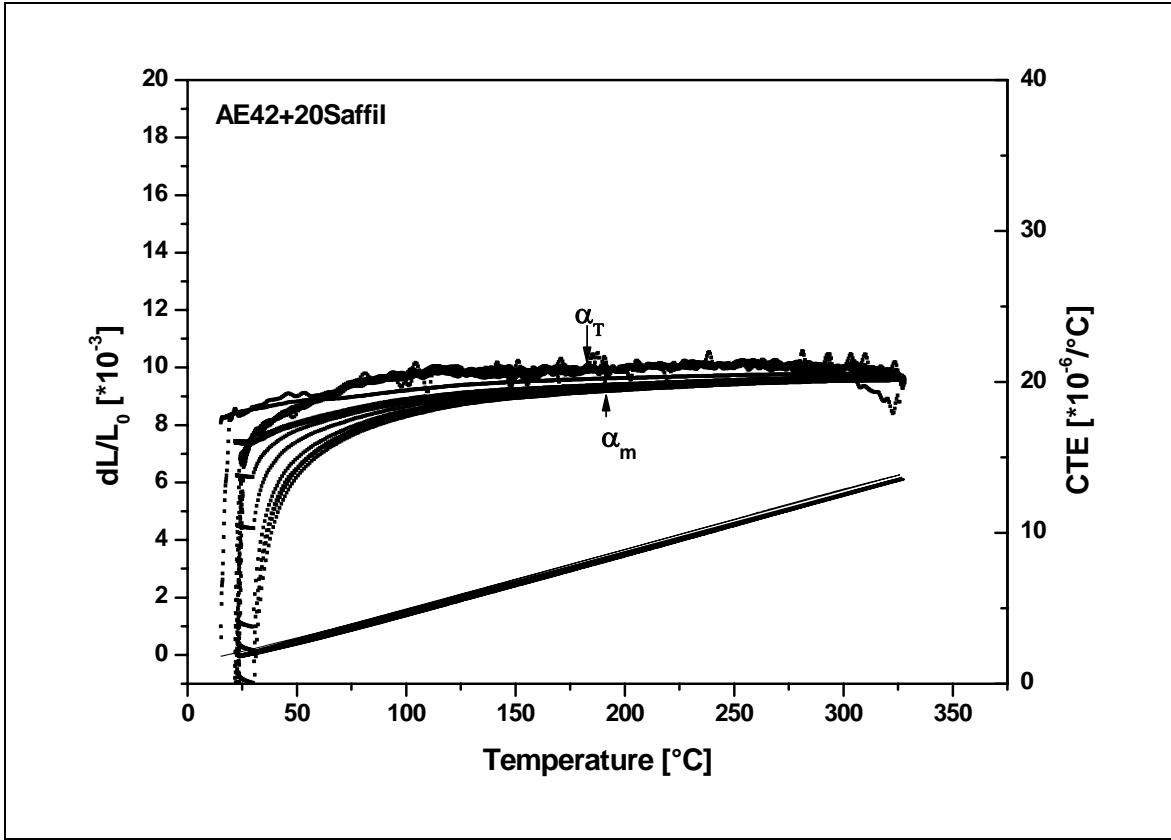


Fig. 2a

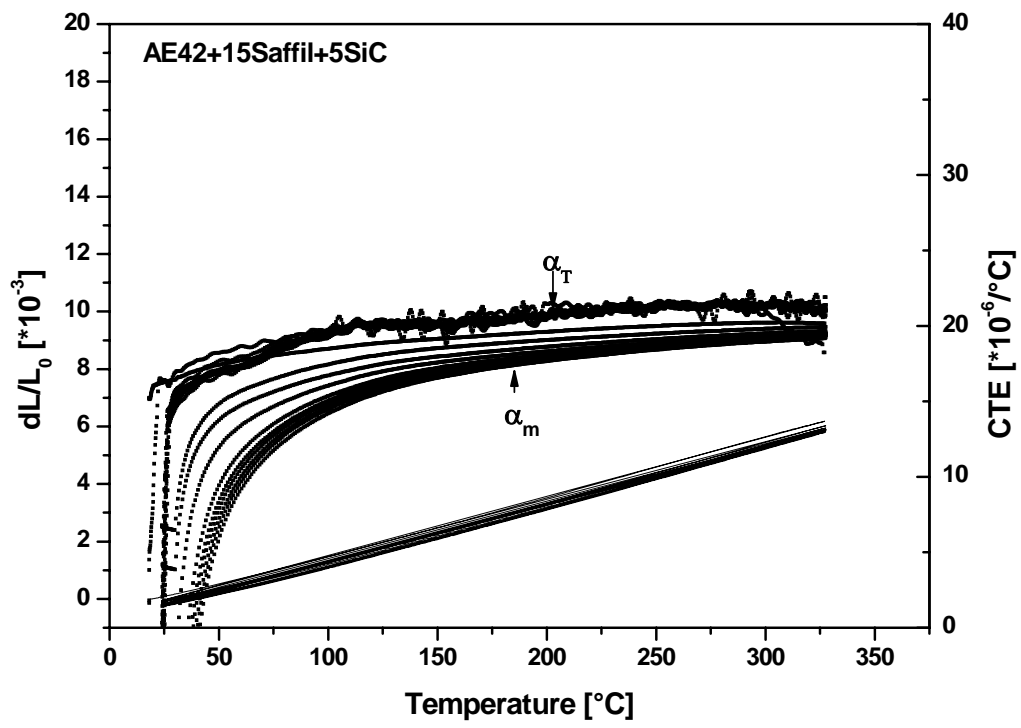


Fig. 2b

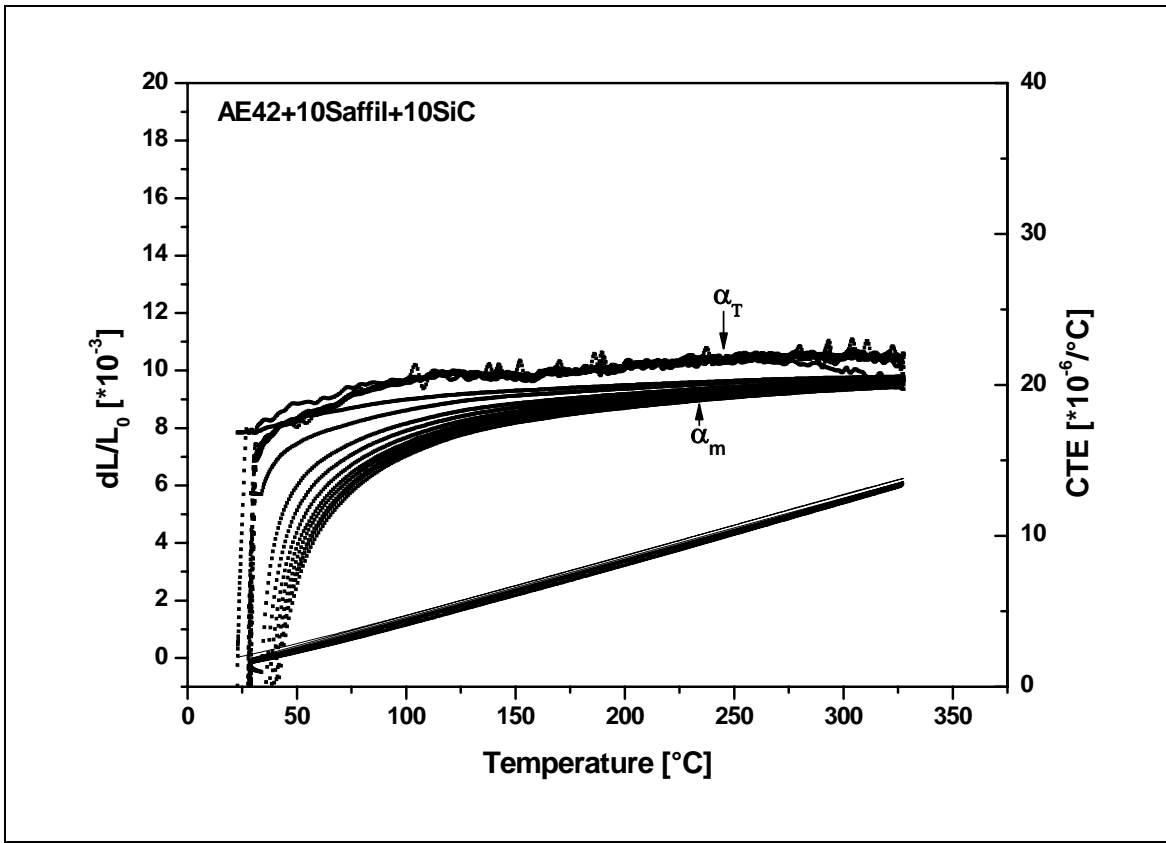


Fig. 2c

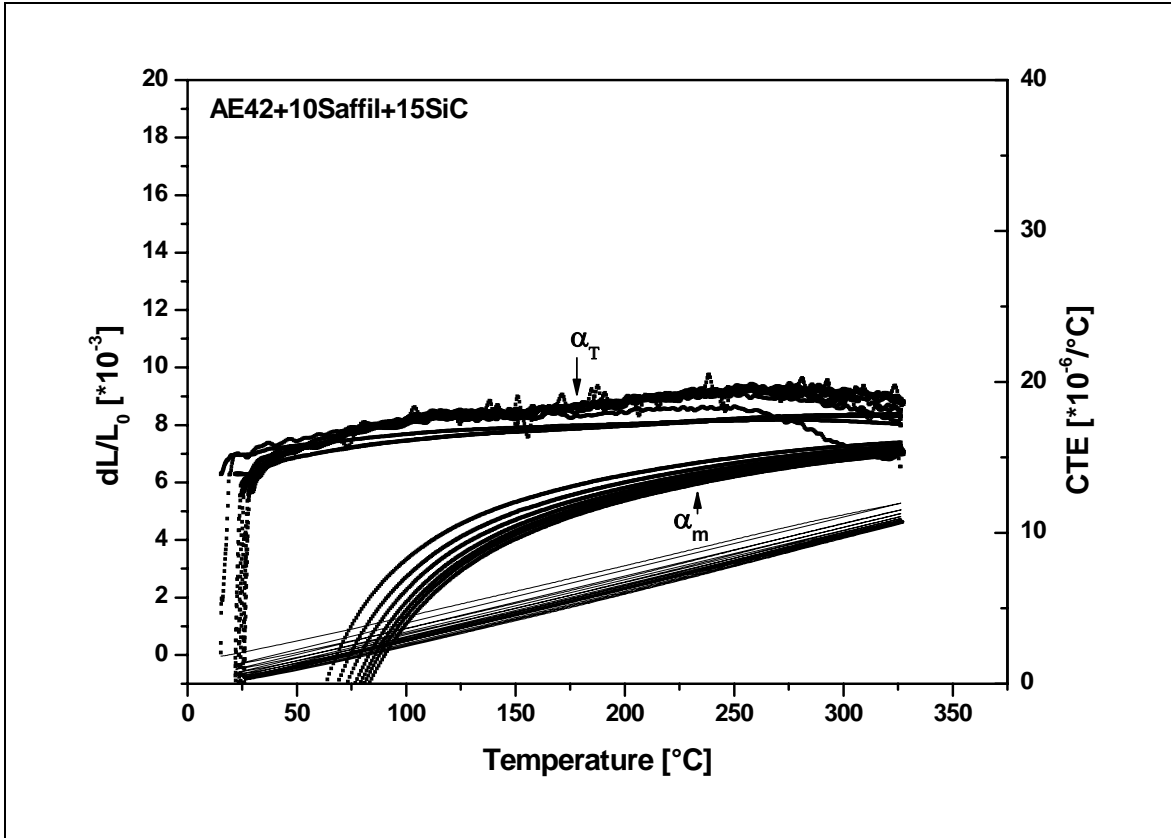


Fig. 2d

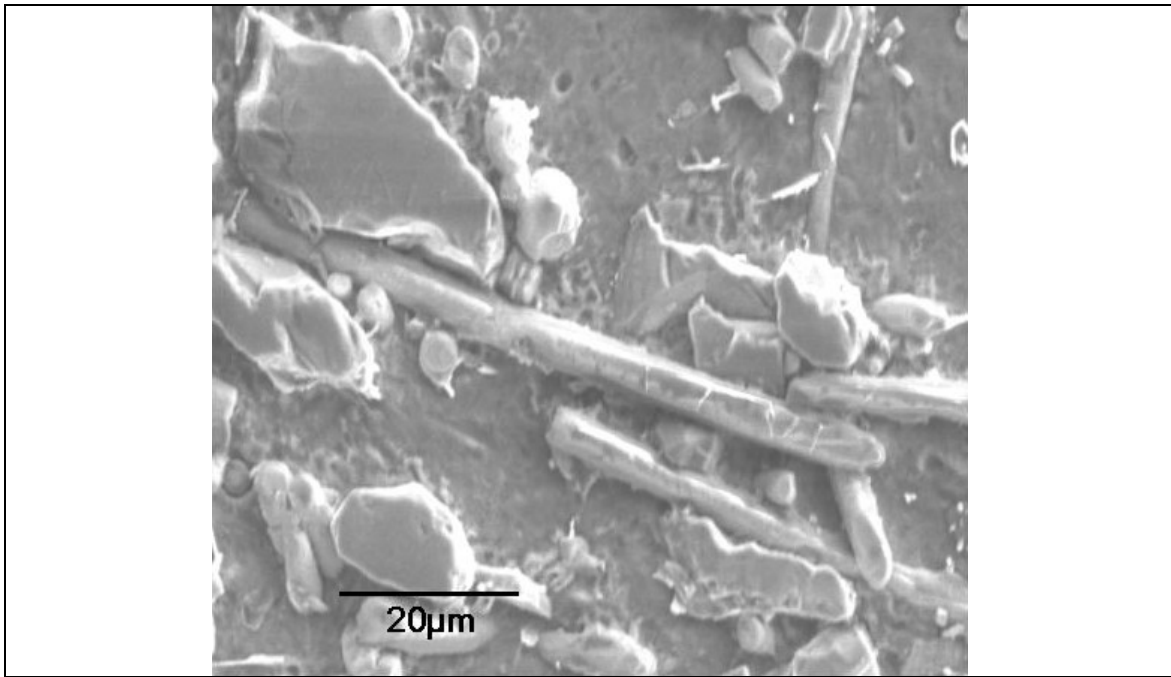
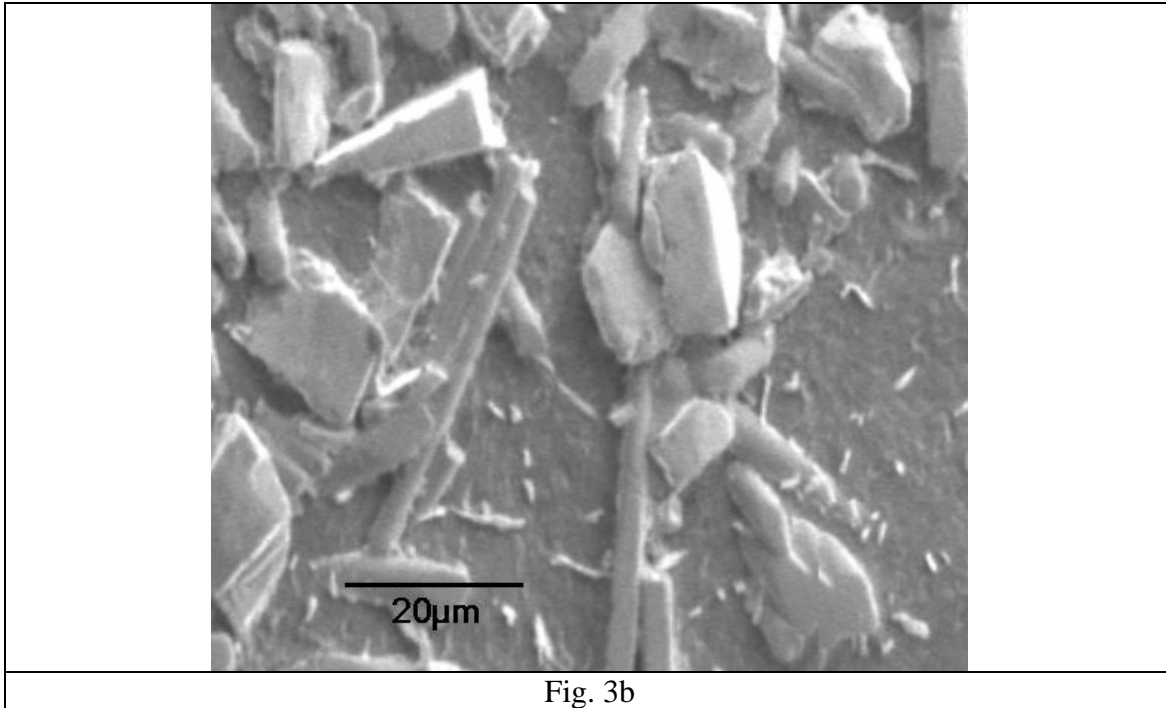


Fig. 3a



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Table 1: Standard average CTE values ( $\alpha_m$ ) for magnesium after ref. (16)

Temperature range (°C)	CTE ( $\times 10^{-6} / ^\circ\text{C}$ )
20-100	26.1
20-200	27.1
20-300	28.0
20-400	29.0
20-500	29.9

Table 2: Experimental and theoretical average CTE values ( $\alpha_m$ ) of the AE42 alloy and composites in the temperature range 20-200°C

Composition of the alloy and composites	Experimental CTE ( $\times 10^{-6} / ^\circ\text{C}$ )	Corrected CTE ( $\times 10^{-6} / ^\circ\text{C}$ )	Theoretical CTE ( $\times 10^{-6} / ^\circ\text{C}$ )
AE42	28.5	27.1	27.1
AE42+20% saffil	19.6	18.2	15.2
AE42+15% saffil+5% SiC	19.7	18.3	15.8
AE42+10% saffil+10% SiC	19.0	17.6	16.9
AE42+10% saffil+15% SiC	15.4	14.0	15.7

**Figure Captions:**

- Fig. 1 Thermal cycling curves and experimental average and instantaneous CTE values ( $\alpha_m$  and  $\alpha_T$ ) for AE42 alloy
- Fig. 2 Thermal cycling curves and experimental average and instantaneous CTE values ( $\alpha_m$  and  $\alpha_T$ ) for AE42 alloy reinforced with
- a) 20 vol% Saffil
  - b) 15vol% Saffil and 5vol% SiC
  - c) 10vol% Saffil and 10vol% SiC
  - d) 10vol% Saffil and 15vol% SiC
- Fig. 3 SEM micrograph of AE42 + 10vol% Saffil + 10vol% SiC
- a) before thermal cycling
  - b) after thermal cycling