

Thermal expansion and dimensional stability of unidirectional and orthogonal fabric M40/AZ91D composites

SONG Mei-hui(宋美慧), WU Gao-hui(武高辉), CHEN Guo-qin(陈国钦), YANG Wen-shu(杨文澍)

School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China

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Abstract: Unidirectional (60%, volume fraction) and orthogonal (50%, volume fraction) M40 graphite fibre reinforced AZ91D magnesium alloy matrix composites were fabricated by pressure infiltration method. The coefficients of thermal expansion (in the temperature range of 20–350 °C) and dimensional stability (in the temperature range of 20–150 °C) of the composites and the corresponding AZ91D magnesium alloy matrix were measured. The results show that coefficients of thermal expansion of the composites in longitudinal direction decrease with elevating temperature. The coefficients of thermal expansion (CTE) for unidirectional M40/AZ91D composites and orthogonal M40/AZ91D composites are $1.24 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ and $5.71 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ at 20 °C, and $0.85 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ and $2.75 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ at 350 °C, respectively, much lower than those of the AZ91D alloy matrix. Thermal cycling testing demonstrates that the thermal stress plays an important role on residual deformation. Thus, a better dimensional stability is obtained for the AZ91D magnesium alloy matrix composites. More extreme strain hysteresis and residual plastic deformation are observed in orthogonally fabric M40 reinforced AZ91D composite, but its net residual strain after each cycle is similar to that of the unidirectional M40/AZ91D composite.

Key words: composites; coefficient of thermal expansion; dimensional stability; thermal cycles

1 Introduction

Magnesium alloys have wide applications in many areas due to their high specific strength, high specific stiffness, excellent damping capacity and thermal conductivity[1]. However, they suffer from several inherent deficiencies, such as high thermal expansion and poor dimensional stability, which currently prevent their applications for high-precision aerospace system. Fortunately, graphite fibre reinforced magnesium matrix composites (Gr/Mg) could overcome these deficiencies[2–4]. These composites have been identified as potential candidate materials for application in high-precision space-based systems[5–6], such as mirror substrates of space station, high precision airborne radar, precise navigation and optical measurement systems[7–8].

At present, the unidirectional fibre reinforced magnesium alloy matrix composites have been widely investigated[9–11]. However, the research of fibre reinforced composites is still focused on mechanical properties, while only a few investigations on their thermal expansion behaviours and dimensional stability

were reported[12–14]. The properties of graphite fibre reinforced magnesium alloy matrix composites are greatly influenced by temperature due to the large difference of coefficient of thermal expansion (CTE) between fibre and magnesium alloy and the fibre arrangement. The strain hysteresis and residual plastic strains during thermal cycling have a complex influence on thermal expansion behavior, resulting in instantaneous and cumulative dimensional instability[15]. Moreover, the thermal expansion behaviours are also affected by the interface. Therefore, in this work, unidirectional (60%, volume fraction) and orthogonally fabric (50%, volume fraction) M40 graphite fibre reinforced magnesium alloy AZ91D composites are fabricated by pressure infiltration method, and their microstructure is studied. Meanwhile, their thermal expansion behavior and dimensional stability are investigated by coefficient of thermal expansion (CTE) measurement and thermal cycling testing, respectively.

2 Experimental

2.1 Materials

The M40 graphite fibres were used to reinforce

AZ91D alloy by pressure infiltration method. Fibre volume fractions in unidirectional M40/AZ91D composite and orthogonally fabric M40/AZ91D composite were about 60% and 55%, respectively. Table 1 lists the nominal compositions of AZ91D alloy; and the physical properties of M40 fibre and AZ91D are listed in Table 2.

Table 1 Chemical compositions of AZ91D magnesium alloy (mass fraction, %)

Al	Zn	Mn	Si	Fe	Cu	Ni	Mg
8.5–9.5	0.45–0.90	0.17–0.40	0.05	0.004	0.025	0.001	Bal.

Table 2 Physical properties of M40 fibre and AZ91D alloy

Material	E_L / GPa	E_T / GPa	α_L / ($10^{-6} \text{ }^\circ\text{C}^{-1}$)	α_T / ($10^{-6} \text{ }^\circ\text{C}^{-1}$)	ν
M40	377	10	−1.1	8	0.25
AZ91D	45	45	28	28	0.35

L—Longitudinal direction; T—Transverse direction; ν —Poisson ratio

2.2 Testing

The morphology of the composites was observed by ZEISS 40MAT optic microscope. The interface characteristics of the composites were investigated by PhilipsCM-12 transmission electron microscope (TEM). The morphology of composite was observed by S-4700 scanning electron microscope (SEM), and energy disperse X-ray (EDS) was employed. The CTE measurement and thermal cycling test were both carried out on a Dilatometer 402C. The diameter of cylinder sample was 4 mm and the length was 25 mm. During the CTE measurement, the temperature was heated from 20 $^\circ\text{C}$ to 350 $^\circ\text{C}$ at a rate of 5 $^\circ\text{C}/\text{min}$. The helium atmosphere was maintained at a flow rate of 50 mL/min to ensure the equilibrium of temperature and prevent the oxidation of samples. In this work, the CTE data were determined between 20 $^\circ\text{C}$ and 350 $^\circ\text{C}$, with 50 $^\circ\text{C}$ intervals extending up to 350 $^\circ\text{C}$.

The samples were cycled between 20 $^\circ\text{C}$ and 150 $^\circ\text{C}$ for 15 complete cycles with heating and cooling rates of 8 $^\circ\text{C}/\text{min}$. The helium atmosphere was maintained at a flow rate of 50 mL/min and liquid nitrogen was used as coolant. Before each cycle, the samples were maintained at 20 $^\circ\text{C}$ for 25 min to ensure the uniformity of temperature.

3 Results and discussion

3.1 Microstructure

Fig.1 shows the microstructure of the polished and etched M40/AZ91D composites. M40/AZ91D composites are well infiltrated with good fibre dispersion. Pores or significant casting defects are not observed. The diameter of single fibre is about 6.5 μm . Fig. 2 displays

the microstructure of the casting AZ91D alloy. The grain size of cast AZ91D alloy is about 300 μm , and $\text{Mg}_{17}\text{Al}_{12}$ precipitates and discontinuous blocky β precipitates are observed on the grain boundary[16–17]. It is reported that large grains may deteriorate the dimensional stability of alloy matrix.

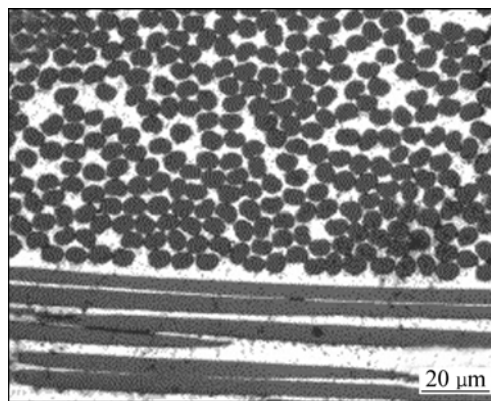


Fig.1 Optic micrograph of M40/AZ91D composite

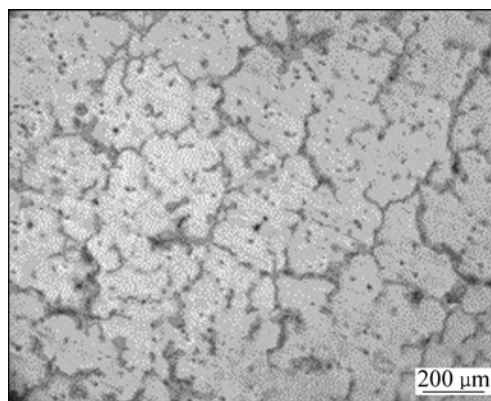


Fig.2 Optic micrograph of AZ91D alloy

It is well established that the properties of composites are associated with their matrix, reinforcement and interface; and especially, efficient load transfer to reinforcement is dependent on the nature of the interface between the matrix and the reinforcement. The interface of M40/AZ91D composite was revealed by TEM (Fig.3). The combination between Mg alloy and fibres is good, and no interfacial debonding is observed by TEM observation. It should be noted that discontinuous needle-shaped precipitate (Fig.3(a)) and blocky precipitate (Fig.3(b)) are evident at the interface, which are indicated to be Al_4C_3 and $\text{Mg}_{17}\text{Al}_{12}$ by electron diffraction analysis, respectively. Furthermore, finely dispersed precipitate is also observed, and it is reported to be $\text{Mg}_{17}\text{Al}_{12}$ or MgO [18]. The temperature of fibre surface is lower than that of liquid magnesium alloy during cooling from the casting temperature, and the fiber surface becomes the solidification front, which results in the segregation of Al at interface (Fig.4), and then Al_4C_3 is formed by reaction between carbon fiber

and Al. Furthermore, fibre surface acts as nucleation centres where equilibrium metallic phases ($\text{Mg}_{17}\text{Al}_{12}$) are formed due to their high surface energies, and grow to blocky shape eventually. Moreover, MgO precipitates result from the chemical reaction between oxygen element adsorbed by fibre and liquid magnesium alloy. Fortunately, both interfacial reactant and precipitate can improve the interfacial bonding, and then impose high mechanical restraints on thermal expansion of matrix, which is beneficial for composite properties[19].

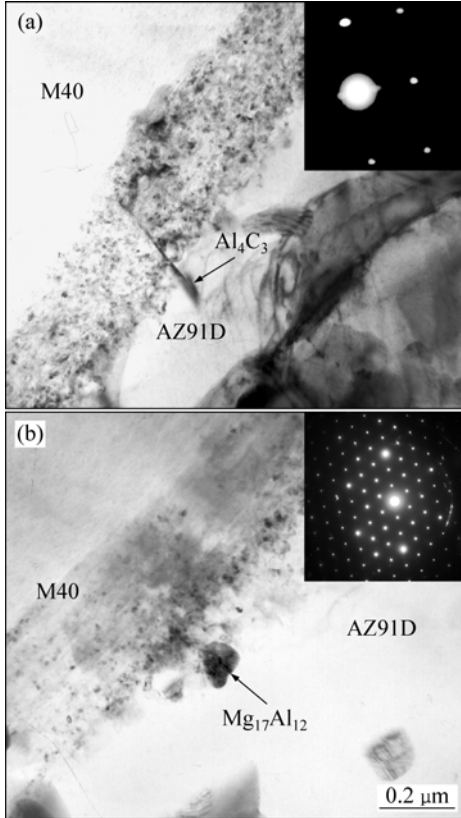


Fig.3 TEM images showing Al_4C_3 (a) and $\text{Mg}_{17}\text{Al}_{12}$ (b)

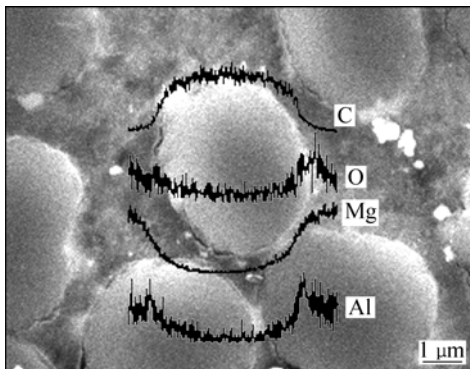


Fig.4 EDS analysis results of M40/AZ91D composite

3.2 Thermal expansion behavior of unidirectional M40/AZ91D composite

The thermal expansion coefficient of unidirectional M40/AZ91D composite is nearly zero, which is much lower than that of the AZ91D alloy matrix under the

same condition, as shown in Fig.5(a). The M40 fibre (CTE: $-1.1 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$) imposes strong restraints on thermal expansion of the matrix in the longitudinal direction when heating, resulting in a much lower CTE for unidirectional M40/AZ91D composite than the matrix, as shown in Fig.5(b). It should be noted that, CTE — temperature relation of unidirectional M40/AZ91D composite is different from that of the AZ91D alloy. The CTE of AZ91D alloy increases with temperature increasing, while the CTE of the composite changes little under the same condition. The difference should also be due to the mechanical restraints imposed by fibres on thermal expansion of AZ91D alloy[20]. In this work, CTE of unidirectional M40/AZ91D composite changes from approximately $1.24 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ at 20 $^\circ\text{C}$ to $0.85 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ at 350 $^\circ\text{C}$, much lower than that of the AZ91D alloy ($28 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$), and achieves near-zero thermal expansion.

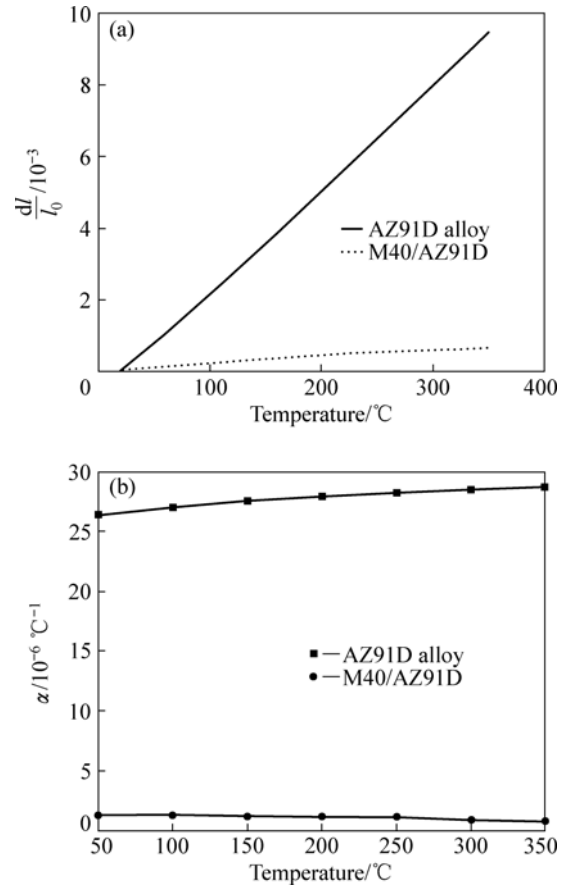


Fig.5 Temperature dependence of relative linear length change (a) and CTE (b) for unidirectional M40/AZ91D composite and AZ91D alloy

Based on the theory of equivalent strain during thermal expansion[21], the thermal strain of the composite can be calculated by the following model[15]:

$$\varepsilon_{c,L} = \frac{\sigma_{f,L}}{E_{f,L}} + \alpha_{f,L} \Delta T = \frac{\sigma_m}{E_m} + \alpha_m \Delta T + \varepsilon_m^p \quad (1)$$

where σ and ε represent stress and strain, respectively; α and E are coefficient of thermal expansion and elastic modulus, respectively; ΔT is the change from the reference temperature; and subscripts m, f and c refer to the matrix, fibre and composite, L refers to longitudinal direction, and p denotes plastic.

Then, the CTE is the derivative of the Eq.(1). DUTTA[22] calculated the thermal strain of carbon fibre reinforced aluminium composite during heating, and reported that the slope of strain curve decreased with increasing temperature, which indicated CTE decreased at elevated temperature. The large CTE difference between fiber and matrix induces large internal thermal stress at the interface during composite fabricating, and fibres endure residual compressive stress while Mg matrix endures residual tensile stress at the initial stage of testing[23–24]. The thermal stress induced by thermal expansion at elevated temperature eliminates the residual tensile stress and ultimately transforms into compressive stress. Plastic deformation occurs in the matrix when the compressive stress exceeds the yield strength of the matrix, which eliminates the compressive stress. The coordination of the strain of fibre and matrix is realized by interface sliding[4, 25–26], which results in fiber protruding significantly from the matrix, as shown in Fig.6. The change of stress mentioned above varies with temperature, and eventually results in the CTE decreasing with increasing temperature[20]. However, there will be many obstacles to calculate CTE of composite.

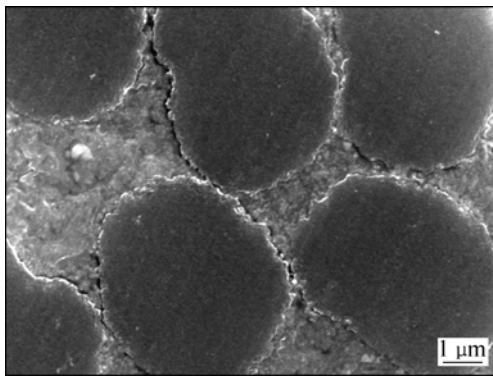


Fig.6 SEM image showing fiber protruding significantly from matrix after testing

In the prediction of the thermal expansion of unidirectional fibre reinforced composites, many models, such as ROM, Turner and Schapery model are usually employed[27–28]. Without consideration of stress transfer at interfaces, the CTE of fibre reinforced composites can be simply predicted by the ROM. However, for more precise predictions in usual cases, the stress interaction at the interfaces may not be neglected and ROM is thus no longer valid. The hydrostatic

pressure assumption is adopted in Turner model, and it neglects the residual stress generating during composite fabrication. Thus, the real stress of composites cannot be precisely predicted by Turner model. Based on energy considerations, Schapery model considers the stress interaction between fibre and matrix, and it is usually in a good accordance with experiments. When matrix alloy in composite is in the elastic stage, Schapery model may be expressed as

$$\alpha_{c,L} = \frac{\varphi_f E_{f,L} \alpha_{f,L} + \varphi_m E_m \alpha_m}{\varphi_f E_{f,L} + \varphi_m E_m} \quad (2)$$

where α , φ and E are referred to the CTE, the volume fraction and the elastic modulus, respectively. The properties of the composite, fibre and matrix are denoted by subscripts c, f and m, respectively.

In this work, CTE and elastic modulus are $-1.1 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ and 377 GPa for M40 fibre, and $28 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ and 45 GPa for AZ91D matrix alloy, respectively. Theoretical CTE of unidirectional M40/AZ91D composite calculated by Eq.(2) is $1.3 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, in a good accordance with experiment ($1.24 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ at 20–150 $^\circ\text{C}$).

3.3 Dimensional stability of unidirectional M40/AZ91D composite

Thermal strain dL/L_0 of unidirectional M40/AZ91D composite reduces greatly over the AZ91D alloy in the same temperature range due to the lower CTE of composite. The residual relative length change (Δ) is calculated from strain—time curves (Fig.7) by following equation:

$$\Delta = \frac{l_i - l_{20}}{l_{20}} \quad (3)$$

where l_{20} is the original sample length at 20 $^\circ\text{C}$; and l_i refers to the sample length at the same temperature after i cycles. The magnitude of Δ represents the accumulative residual length change after i cycles.

Fig.8 reveals the variation of the residual relative length change (Δ) with cycling number for unidirectional M40/AZ91D composite and AZ91D alloy. Both materials present a progressive increase in residual length change with cycling number. Nevertheless, the temperature cycling causes a lower Δ for unidirectional M40/AZ91D composite than matrix alloy. This demonstrates a better dimensional stability of unidirectional M40/AZ91D composite.

Based on the above results, another parameter, the net residual relative length change (Δ_i) could be obtained by

$$\Delta_i = \frac{l_i - l_{i-1}}{l_{20}} \quad (4)$$

where l_{i-1} and l_i refer to the sample length at 20 $^\circ\text{C}$ after $i-1$ and i cycles, respectively. Certainly, i is in the range

of 1–15. The magnitude of Δl_i represents the residual length change in i cycles. Fig.9 shows the results of variation of Δl_i with cycling number.

The first cycle behavior of the unidirectional M40/AZ91D composite is different from that of cycles 2–15. In the high volume fraction composite, strain hysteresis and residual strain generation are most extreme

over the first cycle. Once again, residual plastic strain reduces significantly after the first cycle; however, hysteresis remains to a similar extent over the cycles presented. Residual tensile stress generated during composite fabrication and thermal stress generated during thermal cycle testing would surpass yield strength of the matrix and result in the generation of large residual

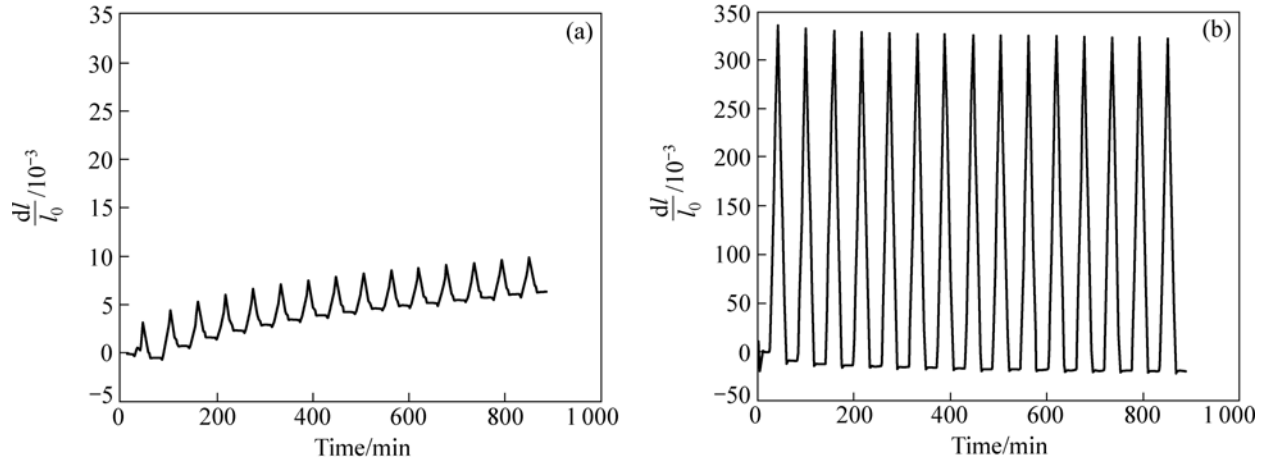


Fig.7 Time dependence of relative length change for unidirectional M40/AZ91D composite (a) and AZ91D alloy (b)

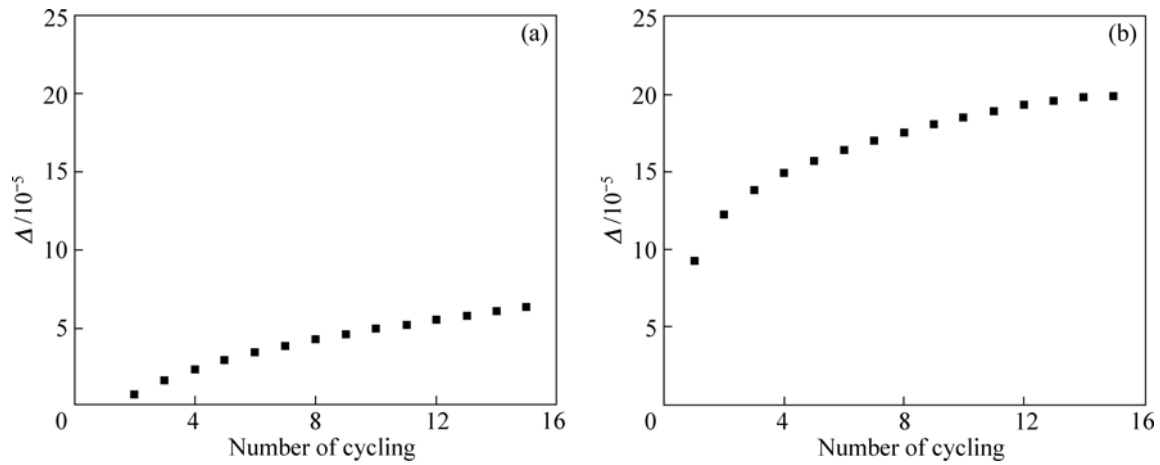


Fig.8 Residual relative length change vs number of cycling for unidirectional M40/AZ91D composite (a) and AZ91D alloy (b)

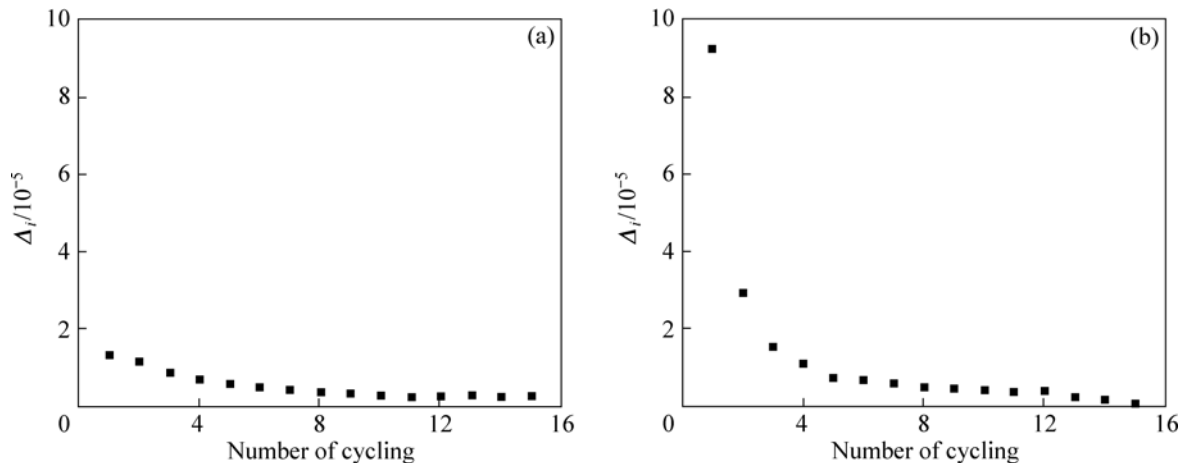


Fig.9 Net residual relative length change vs number of cycling for unidirectional M40/AZ91D composite (a) and AZ91D alloy (b)

stain[22, 29–30]. However, the relaxations of residual tensile stress generated during composite fabrication result in lower plastic deformation in subsequent cycles than the first cycle. Fig.9(a) shows that Δ_i is stable after 10 cycles, which reveals the elimination of residual tensile stress in composites[31]. Nevertheless, incorporating M40 graphite fibres with a negative CTE in longitudinal direction in AZ91D matrix with a large positive CTE can result in the generation of large thermal stress with only modest temperature variations, which causes matrix plastic deformation. Unfortunately, the deformation cannot be eliminated with thermal cycle, and the deformation accumulation has a great influence on dimensional stability [15, 32–33]. Value of Δ_i decreases to be stable eventually with cycling, but is still higher than zero. The residual plastic strain of unidirectional M40/AZ91D composite is much lower than that of the AZ91D matrix after the first cycle; and this reveals a better dimensional stability for the unidirectional M40/AZ91D composite.

3.4 Thermal expansion behavior and dimensional stability of orthogonal fabric M40/AZ91D composite

Fig.10 reveals the relative linear length change and CTE with the variation of temperature of orthogonally fabric M40 reinforced AZ91D composite. Similar to unidirectional M40/AZ91D composite, the CTE of orthogonal fibre reinforced AZ91D composite decreases at elevated temperature, from $5.71 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ at $20 \text{ }^\circ\text{C}$ to $2.75 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ at $350 \text{ }^\circ\text{C}$.

The strain—time curve, Δ and Δ_i with the variation of temperature of orthogonal fibre reinforced AZ91D composite are shown in Fig.11. Compared with Fig.7(a), Fig.8(a) and Fig.11(a), it is obvious that strain hysteresis and residual plastic stain are more extreme in orthogonal fabric M40 reinforced AZ91D composite. However, the

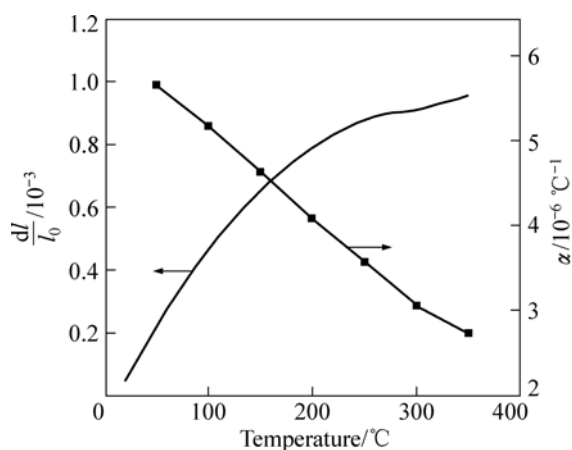


Fig.10 Temperature dependence of relative linear length change and CTE for orthogonally fabric M40/AZ91D composite

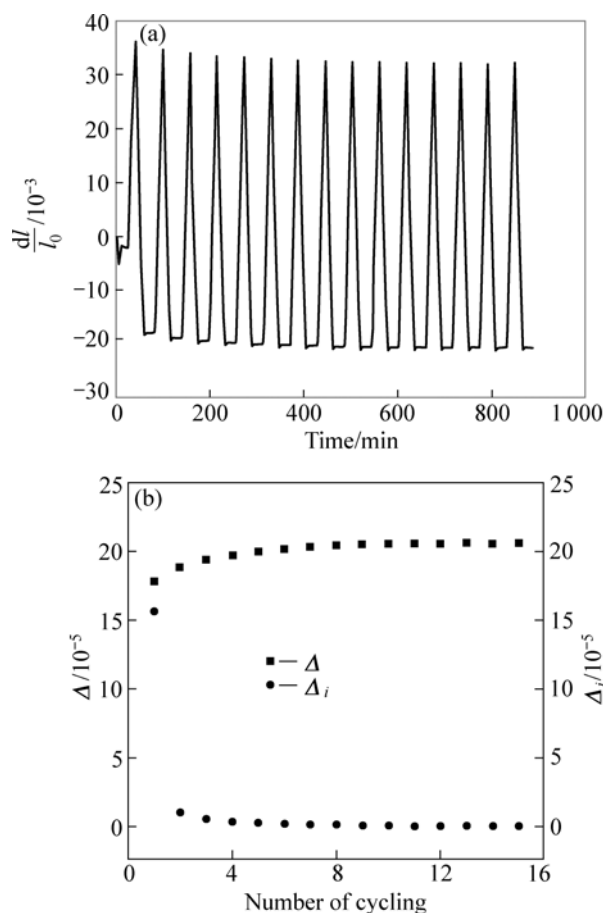


Fig.11 Time dependence of relative length change (a) and Δ and Δ_i vs cycling numbers (b) for orthogonally fabric M40/AZ91D composite

values of Δ_i after each thermal cycle are basically same as unidirectional M40/AZ91D composite.

4 Conclusions

1) The combination between Mg alloy and M40 graphite fibres is good, and Al segregated at the interface mainly exists in the form of interfacial reactant Al_4C_3 and precipitate $\text{Mg}_{17}\text{Al}_{12}$.

2) CTE of unidirectional M40/AZ91D composite changes from approximately $1.24 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ at $20 \text{ }^\circ\text{C}$ to $0.85 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ at $350 \text{ }^\circ\text{C}$, in a good accordance with Schapery model. The CTE of orthogonal fibre reinforced AZ91D composite decreases from $5.71 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ at $20 \text{ }^\circ\text{C}$ to $2.75 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ at $350 \text{ }^\circ\text{C}$.

3) The thermal expansion behavior of orthogonal fibre reinforced composite is similar to that of unidirectional composite. The residual relative length change of the composite increases with thermal cycle, still far lower than that of the AZ91D matrix. Net residual relative length change of the M40/AZ91D

composite of the first cycle is different from that of subsequent cycles, and reaches its maximum in the first cycle. The residual plastic strain of M40/AZ91D composite decreases to a stable value eventually with thermal cycle.

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