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Investigations on thermal fatigue of aluminum- and magnesium-alloy based composites

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Abstract

Both the KS1275[®] piston and AE42 alloys and their composites have realistic and/or potential applications as engine components in the automotive industry. Used as engine components, the dimensional stability is of great concern. Thermal cycling experiments can simulate the service conditions of the materials and give an evaluation how the dimension changes during their service in the changing temperature environments. The present paper investigates the thermal fatigue of the short fiber reinforced KS1275[®] piston and AE42 alloys, with an emphasis on the changes in the strain and hardness before and after thermal cycling. The effects of fiber orientation and composition, and subsequent heat treatment, on the thermal strain were discussed. It is shown that the thermal strain was affected by experimental condition of the thermal cycling and the strength of matrix. After thermal cycling, the hardness decreases due to the occurrence of the matrix overageing and recovery.

Keywords: Metal matrix composite; Thermal fatigue; Thermal cycling; Heat treatment; Microstructure

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1. Introduction

Metal matrix composites (MMCs) offer increased specific strength and stiffness and low thermal expansion coefficient (CTE) than monolithic materials [1,2]. However, due to the mismatch in CTEs between the matrix and ceramic reinforcement, any fluctuation in environmental temperature can introduce large thermal stresses that are possible to cause the local elastic or plastic deformation in the matrix near the reinforcement, leading to an instability in dimension of the component and possible degradation in mechanical properties [3,4]. The resulting increase in dislocation density near the interface can accelerate the ageing and consequently the overageing of matrix easily occurs [5-7].

Thermal cycling experiments in a specified temperature range were extensively used to evaluate the thermal fatigue in MMCs because it can simulate the service conditions and give an evaluation of their thermal fatigue behavior [3,4,8-12]. Due to the yielding of matrix thermal cycling can lead to the appearance of strain hysteresis between the heating and cooling segments and also the permanent residual strain. Most of previous investigations were carried out on the continuous fiber reinforced composites. In contrast, investigations on the short fiber and particulate reinforced composites received fewer attentions. Furthermore, in the previous works, the investigations on the evolution of thermal strain and its related physical mechanisms received considerable interests, but less was done on the investigations of mechanical properties after thermal cycling.

To understand the phenomena occurred during thermal fatigue is useful for the composition design of MMCs. The present work investigates the thermal fatigue of KS1275[®] piston and AE42 alloy based composites, including the changes in dimension, microstructure and hardness after thermal cycling. The KS1275[®] alloy developed by the Toyota Company and Kolbenschmidt-Pierburg AG can be used as piston components in diesel engines [13-15]. The AE42 alloy was developed from non-aluminum magnesium alloys in which rare earth elements were shown to increase creep resistance. It is one of the potential magnesium alloys for automobile applications particularly in engine components [16].

2. Materials and experimental procedures

Table 1 lists the properties of the materials used for producing the MMCs. The KS1275[®] alloy has a composition of 11~13.5 Si, 0.5~1.3 Cu, 0.8~1.3 Mg, 0.5~1.3 Ni, ~0.25 Zn, ~0.1 Cr, balance Al (unless stated, all compositions are given in wt.%). The AE42 alloy has a composition of ~4 Al, ~2.5 RE, ~0.2 Mn, balance Mg. The reinforced composites with the addition of 20% Saffil[®], 20% Maftech[®] or 20% Kaowool[®] short fibers were produced by direct squeeze casting with the reinforcement predominately and randomly oriented in the plane of the disc.

The specimens in both the transversal and longitudinal directions used for thermal strain response investigations, were of rectangular shape with a length of 10 mm and a cross-section area of 5 mm × 5 mm for the KS1275[®] alloy and its composites, and of cylinder shape with a length of 22 mm and a diameter of 6 mm for the AE42 alloy and its composites. The samples of the KS1275[®] alloy and its composites were cycled up to 47 cycles in a temperature range from 58±2 to 287±5°C at a heating and cooling rate of 10

°C/min using an Al₂O₃ tube dilatometer Netzsch Thermische Analyse DIL 402C equipped with TASC 414/3 controller. The samples of the AE42 alloy and its composite were cycled up to 20 cycles in a temperature range from 30 to 340°C with a heating and cooling rate of 5 °C/min. The residual thermal strain after each thermal cycling was evaluated according to:

$$\text{Thermal strain } (\epsilon) = \frac{L - L_0}{L_0} \quad (1)$$

Where L_0 is the original sample length and L is the sample length after each thermal cycling.

The microstructures of composites were investigated in both the longitudinal and transversal samples. The unreinforced alloy was etched in a solution of 20 ml distilled water, 100 ml ethanol, 6 ml acetic acid and 12 g picric acid before optical observation. SEM observations were performed on a low vacuum scanning microscope JEOL 5310 equipped with X-ray spectrometer using an accelerating voltage of 15kV. To observe the interfacial microstructure in the composites, TEM specimens were ground mechanically using tripod machining technique and then thinned using ion-milling machine. The interface was investigated by a transmission electron microscope (TEM) JEOL 2000 equipped with an energy dispersive X-ray analysis (EDAX) system operating at 200 kV.

The microhardness was measured on the transversal section before and after thermal cycling. Hardness tests were carried out using a HVM 2000 machine with a load of 19.6 N and a holding time of 10 s. Ten points were evaluated for each specimen.

3. Experimental results

3.1. Microstructural observations

The unreinforced KS1275[®] alloy shows a typical dendritic structure (Figure 1 (a)). However, this is not the case in the composites due to the restricted free dendritic growth by the reinforcements. The gray phase was identified as silicon. Figure 1(b) shows the optical microstructure of as-cast Saffil[®] fiber reinforced KS1275[®] alloy. The fibers remain predominantly in a planar-random arrangement with relatively few fibers parallel to the disc axis. The aspect ratio of short fiber was measured to be about 4.5. There is no large difference in the microstructure among the composites.

Figure 2 shows the optical microstructures of as-cast unreinforced AE42 alloy and its Saffil[®] short fiber reinforced composite. Eutectic phase can clearly be observed near the grain boundaries. This interdendritic phase was reported to be Al₁₁RE₃ [17]. In the composite, the fibers remain predominantly in a planar-random arrangement, as observed in the KS1275[®] alloy-based composites (Figure 2(b)).

The interface is clean in the Saffil[®]-fiber reinforced KS1275[®] composite. Very few and thin reaction layer was observed (Figure 3(a)). However, a relative thick reaction layer was observed on all investigated fibers in the Kaowool[®]-fiber reinforced composite (Figure 3(b)). Its thickness is in the range from 400 to 600 nm. The size of reaction product is in nanometer order with a value of about 10 to 30 nm. The EDX analysis shows the magnesium and silicon enrichments in the reaction layer. Further selected area diffraction shows the characteristics of polycrystalline diffraction which is

identified as MgAl_2O_4 phase (spinel phase). The interfacial reaction was also observed in the Saffil[®] fiber reinforced magnesium alloy composites. The reaction product is MgO with a layer thickness of about 300 nm [18,19].

3.2. Thermal strain evolution

The residual strain largely increases with the thermal cycling proceeding in the transversal samples of as-cast unreinforced KS1275[®] alloy and Kaowool[®]-fiber reinforced composite (Figure 4(a)). In the composites reinforced with the Saffil[®] or Maftech[®] fibers, it largely changes only at the beginning of thermal cycling. After then it tends to stabilize. The residual strain in the transversal direction is higher than that in the longitudinal direction in the composites. Figure 4(b) shows the residual strain as a function of the time and thermal cycles for the aged alloys. It is shown that the residual strain of the unreinforced alloy is not the highest. It is less than that of the longitudinal samples of both the Saffil[®]- and Maftech[®]-fiber reinforced alloys. After the thermal cycling, it decreases a little. Compared with the as-cast composites, the residual strain changes in a different way at the beginning of the thermal cycling. It increases for the longitudinal samples, but decreases for the transversal samples.

During the whole thermal cycling, the residual strain of the unreinforced AE42 alloy changes less compared to its Saffil[®] short fiber reinforced composite (Figure 5(a)). In the composite, the residual strain increases with the thermal cycling proceeding in the longitudinal sample, but decreases in the transversal sample. It largely changes at the early stage and tends to stabilize at the later stage in both the longitudinal and transversal samples. The subsequent heat treatment has an effect on the residual strain evolution (Figure 5(b)). The sample with T6 treatment has the lowest residual strain. The residual strain of the sample with T4 treatment is the highest before the 7th cycle, but after then, it is less than that of the as-cast sample. As described above, for all samples, the residual strain increases largely at the early stage and tends to be stable at the later stage.

3.3. Hardness

Cycling of unreinforced alloy and its composites results in a decrease of hardness (Table 2). With the thermal cycles increasing, the hardness decreases. For example, the hardness decreases from 139.3 to 118.9 in the Saffil[®]-fiber reinforced KS1275[®] alloy after 15 cycles. If the thermal cycles are increased to 47 times, the hardness further decreases to 108.4. The hardness difference before and after 47 cycles for the unreinforced alloy is not much lower than that for the Saffil[®]- and Maftech[®]-fiber reinforced alloys. It is 21.3, 30.9 and 30.5 for the unreinforced alloy, Saffil[®]-fiber reinforced and Maftech[®]-fiber reinforced composites, respectively.

Figure 6 compares the hardness before and after thermal cycling for the samples with different heat treatments [20]. In order to be comparable, the samples have the same state before the thermal cycling. Then one group of the samples was performed with the thermal cycling and another group not. As observed in Table 2, the thermal cycling results in a decrease in the hardness. The ageing peak can be observed for the materials without thermal cycling, but not for the materials with thermal cycling. After thermal cycling, the hardness of each material is not affected by the ageing time.

4. Discussion

4.1. Evolution of thermal strain in the KS1275[®] alloy and its composites

If the thermal stress exceeds the yield strength of matrix, the matrix will experience the permanent plastic deformation and usually the composite has a residual strain after thermal cycling. In this case, the strain hysteresis loop between the heating and cooling cycles can be observed [21]. If the thermal stress is less than the yield strength, the deformation of matrix remains within the elastic range, and it cannot experience plastic deformation. The strain and temperature follow the same path on cooling as on heating. Both the hysteresis loop and residual strain are absent. Therefore, the thermal strain response depends on those factors affecting not only the thermal stresses but also the yield strength of matrix. Such factors include cycling temperature range, heating and cooling rates, and microstructure.

The composites reinforced with the Saffil[®], Maftech[®] and Kaowool[®] fibers have a comparative fiber composition (Table 1). By comparing their residual strain, it is possible to conclude the effect of fiber composition on the residual strain. The transversal sample with a higher SiO₂ content has a larger change in the residual strain (Figure 4(a)). The CTE of SiO₂ is larger than that of the other ceramics [3]. The internal thermal stresses resulting from its CTE mismatch with the matrix should relatively be small. The unexpected large residual strain could be explained by the occurrence of microstructural changes due to the existence of SiO₂. The SiO₂ can promote the interfacial reactions and deplete the strengthening agent magnesium (Figure 3 and Equation 1).

It can therefore be expected that the amount of the major strengthening phase Mg₂Si decreases in the matrix. The strengthening mechanism is the precipitation strengthening in the KS1275[®] alloy. Now the decrease in the precipitates favors the dislocation glide and the plastic deformation proceeds easily.

After peak-ageing treatment, the thermal strain changes a little during the whole thermal cycling. This heat treatment can result in an increase the yield stress of matrix [5]. As a consequence, the plastic deformation of matrix becomes difficult. The difference in the evolution of residual strain at the beginning of thermal cycling between the as-cast and aged alloys could be due to the different initial stress states. In the as-cast composites, the internal stresses can be generated during both the squeeze casting and subsequent cooling. The pressure of squeezing casting gives a superimposition to the internal stress and increase the complexity of stress. After solution and ageing treatments, the internal stresses generated during casting are released and new internal stresses produce again during the subsequent cooling.

4.2. Evolution of thermal strain in the AE42 alloy and its composite

The difference in the evolution of residual strain between the unreinforced alloy and its composite can be explained based on the difference in their microstructures. In the composite, the reinforcement introduces large internal stresses and results in a larger deformation. The monotonous evolution of residual strain at the early stage of thermal cycling (Figure 5(a)) is possibly related to the accumulation of internal thermal stresses. The interfaces between the matrix and reinforcement are well bonded, at least under the present thermal cycling conditions. The internal thermal stress is mainly released by the

plastic deformation of matrix rather than by the interfacial debonding. Otherwise, the monotonous increase in the residual strain should not be observed in the longitudinal sample.

The evolution of thermal strain is also affected by the heat treatment in the AE42 alloy based composite. Under the as-cast condition RE (rare earth elements)-containing intermetallics in lamellar colonies are predominately situated near the grain boundaries. During the T4 heat treatment, the content of aluminum and RE increases in the matrix, and the lamellae become spherodized [16]. On the subsequent T6 treatment, precipitation of small intermetallic phases occurs. The residual strain is lower in the samples with T6 treatment (Figure 5(b)) because the distribution and size/shape of precipitates results in a lower residual strain. With these precipitates the yield stress of matrix can be improved. The residual strain of the as-cast and T4 samples show a similar evolution at the early stage of cycling. During thermal cycling the residual strain of the T4 sample approaches to the sample with T6 treatment because ageing can occur in this sample at the present cycling temperature.

4.3. Deterioration of mechanical properties

After thermal cycling, the deterioration in mechanical properties probably results from the interface debonding, fiber fracture, matrix recovery, creep and overageing etc. The previous investigations show that the interface debonding, fiber fracture and matrix creep can be excluded during the thermal cycling up to 47 cycles [3]. For the unreinforced KS1275[®] alloy, the reduction in hardness should be related to the relaxation of initial thermal stresses or overageing. During thermal cycling (47 cycles), the time for the temperatures ranging from 180 to 287°C, in which the ageing can proceed, is up to about 15 hours and 40 minutes. The previous investigation indicated that the time to reach the peak ageing only needs about 3 hours and 45 minutes when solution treated at 510°C for 8 hours followed by annealing at 200°C [3]. Therefore, the overageing should happen during thermal cycling. The larger reduction of the hardness in the composites can be explained as follows: firstly, the ageing is accelerated due to the increase of dislocation density. The overageing is much severer in the composites than that in the unreinforced alloy; secondly, more thermal stresses have been released in the composites because the composites have a higher initial thermal stress. Therefore, it is reasonable to conclude that the larger reduction in microhardness is caused by much severer overageing and matrix recovery rather than by the interface debonding. Slight decrease in the strength of a thermal cycled composite is due to metallurgical transformation of matrix, such as ageing and recovery, rather than due to physical composite damage was also reported by Hall and Patterson [22]. The decrease in hardness caused by matrix recovery can further be illustrated by the comparison of hardness before and after thermal cycling for the Kaowool[®]-fiber reinforced composite. In this composite, there is no precipitation due to the depletion of alloying element magnesium caused by the interfacial reaction (see above discussion). The decrease of hardness (Table 2) should be attributed to the matrix recovery rather than to the overageing.

Compared to the as-cast materials, the hardness of the peak-aged materials decreases more after thermal cycling. For example, the hardness of the as-cast unreinforced KS1275[®] alloy decreases from 98.3 ± 3.7 to 77.0 ± 1.3 after 47 cycles. In the peak-aged unreinforced KS1275[®] alloy, it decreases from 130.5 ± 2.9 to 56.5 ± 1.5 . The precipitating may not be completed during cast due to the rapid solidification. In the subsequent

thermal cycling, the precipitating is possible to be continued and the materials can be strengthened at the early stage of thermal cycling. With thermal cycling continuing, the overageing occurs and then the hardness decreases. In the peak-aged materials, no precipitating happens and the coarsening of precipitates immediately occurs in the subsequent thermal cycling. Therefore, the overageing is much severer in these samples and correspondingly the hardness decreases more.

5. Conclusions

- (1). In the KS1275[®] based composites, the thermal strain response of the longitudinal samples is different from that of the transversal samples. The residual strain can be affected by heat treatment and fiber composition. The transversal samples with a high SiO₂ content have a larger residual strain. A stable state cannot be reached during the present thermal cycling because the stress relaxation can easily proceed in these samples.
- (2). In the AE42 alloy based composites, the residual strain is also affected by the fiber orientation. The subsequent ageing treatment results in a decrease of the residual strain due to the increase in the yield stress of matrix.
- (3). Under the present thermal cycling, the thermal stresses are mainly released by the plastic deformation of matrix in both the KS1275[®] and AE42 based composites.
- (4). Thermal cycling can result in the deterioration of mechanical properties. The decrease in the hardness is attributed to the matrix overageing and recovery.

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Figure captions:

- Figure 1 Optical microstructure, (a) as-cast KS1275[®] alloy and (b) as-cast KS1275[®]+Saffil[®] composite, longitudinal direction.
- Figure 2 Optical microstructure, (a) as-cast AE42 alloy and (b) as-cast Saffil[®] short fiber reinforced AE42 composite, longitudinal direction, without etching.
- Figure 3 TEM image showing the interfacial microstructure in (a) as-cast Saffil[®]-fiber reinforced KS1275[®] composite, (b) as-cast Kaowool[®]-fiber reinforced KS1275[®] composite. The circle shows the zone where the composition is analyzed by EDX. The number shown inside a rectangle is the atomic percent of alloying elements. The interfacial reaction product was identified as MgAl₂O₄.
- Figure 4 Residual strain as a function of thermal cycles and time for the KS1275[®] alloy and its composites, (a) as-cast, TD is transversal direction and LD longitudinal direction; (b) with peak-ageing treatment [3].
- Figure 5 Residual strain as a function of thermal cycles for the AE42 and its composite, (a) as-cast; (b) with different heat treatments.
- Figure 6 Microhardness as a function of ageing time in the unreinforced KS1275[®] alloy and its composite. The thermal cycles is 47. The solution treatment was carried out at 510°C for 8 hrs for all specimens. The ageing treatment was performed at 200°C with different time [20].

Table 1 Properties of materials used for casting MMCs

Components	Compositions (wt.%)	Density (g/cm ³)	Fiber diameter (μm)	Vol. (%)	CTE (×10 ⁻⁶ ,K ⁻¹)	E (GPa)	Tensile strength (MPa)
KS1275 [®]	11~13.5 Si, 0.5~1.3 Cu, 0.8~1.3 Mg 0.5~1.3 Ni, ~0.25 Zn, ~0.1 Cr Balance Al	2.7		0	23.6	70	250
AE42	4.0 Al, 2.5 RE 0.2 Mn, Balance Mg	1.9		0	29.0	45	230
Saffil [®]	>95 δ-Al ₂ O ₃ , <5 SiO ₂	3.3	6.8±2.7	20	7.7	285	2000
Maftech [®]	~72 δ-Al ₂ O ₃ , ~28 SiO ₂	3.0	5.7±1.9	20	10.3	220	2000
Kaowool [®]	~47% δ-Al ₂ O ₃ , ~53% SiO ₂	2.8	6.2±2.8	20		210	1300

Table 2 Microhardness of the as-cast KS1275[®] alloy and its composites before and after thermal cycling.

Materials	Microhardness (HV)		
	As cast	After 15 cycles	After 47 cycles
Unreinforced	98.3 ^a ±3.7 ^b	NA ^c	77.0±1.3
+Saffil [®] fiber	139.3±6.3	118.9±2.3	108.4±3.0
+Maftech [®] fiber	132.4±5.8	117.4±7.6	101.9±7.4
+Kaowool [®] fiber	131.2±5.6	98.9±4.4	NA

^a average value, ^b standard deviation, ^c not available

Figure 1 Optical microstructure, (a) as-cast KS1275[®] alloy and (b) as-cast KS1275[®]+Saffil[®] composite, longitudinal direction.

Figure 2 Optical microstructure, (a) as-cast AE42 alloy and (b) as-cast Saffil® short fiber reinforced AE42 composite, longitudinal direction, without etching.

Figure 3 TEM image showing the interfacial microstructure in (a) as-cast Saffil[®]-fiber reinforced KS1275[®] composite, (b) as-cast Kaowool[®]-fiber reinforced KS1275[®] composite. The circle shows the zone where the composition is analyzed by EDX. The number shown inside a rectangle is the atomic percent of alloying elements. The interfacial reaction product was identified as MgAl_2O_4 .

Figure 4 Residual strain as a function of thermal cycles and time for the KS1275[®] alloy and its composites, (a) as-cast, TD is transversal direction and LD longitudinal direction; (b) with peak-ageing treatment [3].

Figure 5 Residual strain as a function of thermal cycles for the AE42 and its composite, (a) as-cast; (b) with different heat treatments.

Figure 6 Microhardness as a function of ageing time in the unreinforced KS1275[®] alloy and its composite. The thermal cycles is 47. The solution treatment was carried out at 510°C for 8 hrs for all specimens. The ageing treatment was performed at 200°C with different time [20].