Effect of heat treatment on the tribological properties of Al–Cu–Mg/nanoSiC composites

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In this study, the effect of heat treatment on the tribological properties of Al–Cu–Mg alloy reinforced with 4 wt.% SiC particles with 650 nm average particle size has been investigated. The age hardening process consists of solution treatment at 540 °C for 6 h, followed by water quenching and ageing at different temperatures of 175, 200 and 225 °C with soaking times of 3, 6 and 9 h. Hardness measurements were applied to monitor the precipitation effect and the aged samples were then subjected to wear tests under dry sliding conditions against steel and alumina counterfaces. The results showed that the reinforced material exhibits an enhanced ageing response compared to the unreinforced material in the same heat treatment conditions. The rate of ageing increases with increasing temperature; however, ageing at 200 °C and 225 °C for more than 6 h resulted in over-ageing. The best combinations for the enhanced tribological properties for the composite material were selected as 6 h ageing at 225 °C. The precipitation effect for this alloy can be enhanced by the small addition of SiC nanoparticles. Having a small amount of nanoSiC particles with fine precipitates inside the matrix further increases the hardness and wear properties.

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1. Introduction

The requirement for strong, light and stiff materials has extended the interest in metal matrix composites (MMCs). During the past two decades, MMCs have received substantial attention because of their improved strength, high elastic modulus and increased wear resistance over conventional base alloys. The widespread introduction of MMCs has been increasing simultaneously with the technological development [1]. Aluminium, magnesium, titanium and their alloys are commonly used metallic matrices for the production of MMCs. The reinforcements being used are fibres, whiskers and particulates [2,3]. Among these materials aluminium matrix composites have been emerged as advanced materials such as automotive, space, aircraft, defense and other engineering applications due to their high specific strength and enhanced wear resistance as compared to the alloy irrespective of applied load and sliding speed [4,5]. There has been extensive investigation of aluminium MMCs reinforced with hard ceramic phases in an effort to understand and improve the tribological properties of aluminium [6,7].

The properties of some Al alloys and their composites can significantly increase with ageing treatment, especially 2xxx and 6xxx series alloys. In the investigation of age-hardening kinetics, it has been shown that the addition of ceramic particles to age-hardenable Al alloys has different effects on the precipitation of the composite compared with unreinforced alloy. It has been proposed that the addition of reinforcing particles accelerates the ageing kinetics; on the other hand, there have been some explanations that it decreases or causes very little alteration in the ageing kinetics. It has been shown that the age-hardening kinetics of Al–Cu–Mg alloy–10 wt.% SiC fibre composite was enhanced by the presence of the reinforcement during heat treatment [8], which is explained by the plastic deformation induced during heat treatment due to the difference between the coefficients of thermal expansion (CTE) of the matrix and reinforcement. It was stated that in powder metallurgy (PM) aluminium alloy 2124/SiCp composites, the presence of reinforcing particles facilitates the nucleation of precipitates which results in the reduction of the time required to achieve peak hardness. 6061 aluminium alloy reinforced with SiC particles was studied by some researchers [9]. They showed that the precipitation sequence of the composite was similar to that of the unreinforced 6061 alloy, but the ageing kinetics was altered. Ageing was accelerated because solute diffusivity increased as dislocation density increased too. However, some research [10] has shown that in the Al–Cu–Mg alloy composite reinforced with different percentages of SiC particles, the presence of reinforcing particles led to a deceleration of the age-hardening kinetics. This behaviour was attributed to a lower concentration of vacancies, inadequate dislocation density and extensive interfacial segregation of alloying elements. Similar observations of the effect of the ceramic particles on the ageing kinetics were previously made for Al–Al2O3 composites.
It was also reported that there is no obvious difference between the age-hardening kinetics of the 6061 Al alloy and its composite reinforced with 10 and 20 wt.% SiC aged at 175 °C [13]. Although it is accepted that the ageing behaviour depends on the type of reinforcement and its volume fraction, alloy composition, heat treatment and other processing parameters [9,14], there is still a lack of information about the ageing behaviour of Al alloys and their composites.

Most of the studies show that increasing hardness of the material with heat treatment increases wear resistance too. Lee et al. found the lowest abrasive wear rate in a peak-aged AA6061–SiCp composite compared to the as-solutionized and over-aged materials [15]. Also Zongyi et al. found that the abrasive wear resistance of an AA6061–SiCp composite increased with increasing matrix hardness and reached the highest value for the peak-aged condition [16].

In the present work, the tribological behaviour of an Al–Cu–Mg alloy reinforced with nanoSiC particles, both in the as-cast and aged conditions, was studied under dry sliding conditions against AISI 52100 steel and alumina balls. Microstructures and hardness values and coefficients of friction data for specimens with different heat treatments were investigated for this purpose.

2. Experimental details

The chemical composition of the aluminium matrix alloy used in this study is 93.09Al–4.01Cu–1.36 Mg–0.43Mn–0.52Fe–0.42Si–0.13Cr–0.03Zn–0.01Ti (wt.%). The Al–Cu–Mg alloy reinforced with 4 wt.% SiC particles, with an average diameter of 650 nm, was produced by casting. The melt was frequently mixed to provide homogeneous distribution of the SiC particles inside the aluminium matrix. After solidification of the alloy, samples were cut and solution heat treated at 540 °C for 6 h, followed by a water quench (25 °C) and then artificially aged. Three different temperatures (175, 200 and 225 °C) and three different ageing times (3, 6 and 9 h) were selected for the thermal ageing of the quenched samples. Metallographic specimens were polished in the usual manner with final polishing carried out using 3 µm diamond. Specimens were etched with Keller’s reagent (1% HF + 1.5% HCl + 2.5% HNO3 + 95% H2O) to reveal the microstructures clearly. Microstructures of the specimens were investigated using a light microscope (Zeiss Axios- tech) and a scanning electron microscope (Jeol JSM 6060) equipped with energy dispersive X-ray spectroscopy (EDX).

The ageing behaviour of the samples was monitored using hardness measurements. Vickers hardness tests were performed using a Future-Tech type Vickers hardness tester under 3 kg load and 10 s of loading duration. All reported hardness values are based on the average of five measurements. To show the effect of heat treatment on the tribological properties of the alloy, wear tests were conducted at room temperature under dry sliding conditions according to ASTM: G99-05. A Nanovea MT/60/NI type pin-on-disc tribometer (Fig. 1), which permitted rotation of a flat specimen against a stationary pin or ball, was used in the wear tests. All wear tests were carried out under 20 N normal load using AISI 52100 steel and alumina (Al2O3) balls (5 mm in diameter) as counterfaces. Sliding speed and sliding distance were kept constant at 0.13 m/s and 500 m respectively for all tests. During the wear tests, friction coefficients were recorded continuously. The specimens were thoroughly cleaned with alcohol after the wear tests, and then dried with a hot air blower. The weight loss of the alloys was measured using an AND GR200 type microbalance with a resolution of 0.1 mg. The following equation was used to obtain the wear rate of the specimens: \( W = M/D \), where \( W \) is the wear rate (mm³/m), \( M \) denotes mass loss (g), and \( D \) (m) are the density and sliding distance respectively [17]. The worn surfaces of the specimens after wear tests were also investigated using scanning electron microscopy.

3. Results and discussion

3.1. Microstructural investigations

The optical microscope image and EDX analysis results of the phases in the microstructure of the as-cast sample before heat treatment are given in Fig. 2. It can be observed from the figure that the SiC particles are seen as dark phases distributed mainly in the interdendritic areas while a small amount of the particles are seen inside the primary aluminium dendrites. According to the EDX analysis results of the other phase with grey contrast in the interdendritic regions, the chemical composition of the phase is expected to be Al2Cu intermetallics [18,19].

Fig. 3a presents an optical micrograph of the solution-treated sample showing almost full dissolution of the phases. The microstructure consists of large equiaxed grains. Partially undissolved
second phases are also located at the grain boundaries due to their stability at high temperatures. These second phase precipitates were examined by EDX analysis and the results are given in Fig. 3b. The results showed that alloying elements form intermetallics in the grain boundaries of $\alpha$-Al grains. Most of the second phases were dissolved into the matrix as shown in Fig. 3, while fine SiC particles were arranged along the grain boundaries.

Fig. 4 shows optical micrographs of the composite material aged in different conditions. As can be seen in the figure, increasing the ageing temperature causes a greater amount of precipitates in short ageing times (3 h). But when the ageing time is 9 h, the amount of precipitates increases and the precipitate becomes more coarse. This phenomenon is called over-ageing, which causes a dramatic drop in the hardness and strength of the material. The coarse particles formed during 9 h of ageing can be seen in the microstructures of the specimens, and over-ageing also causes particle-free zones with no precipitates. In these areas, dislocation movement is easy and this causes a decrease in the strength and hardness.

3.2. Hardness measurements

After the ageing process, to understand the effect of the heat treatment on the hardness, the samples were subjected to Vickers hardness measurements. Fig. 5 shows the variation of hardness
with ageing times of 3, 6 and 9 h at different temperatures. The hardness was increased with increasing ageing temperature due to higher mobility of the atoms, which forms the strengthening phases at elevated temperatures. There is an obvious age-hardening effect for the temperatures of 200 and 225°C. At these temperatures, the hardness of the composite increases to the peak value and then tends to decrease. This result corresponds to the over-ageing. For the samples aged at 175°C, the hardness continuously increased with ageing temperature and over-ageing was not observed.

The hardness of the as-cast sample was 110 HV, as shown in Fig. 6. After ageing, the lowest hardness of 121.3 HV was obtained at 175°C for 3 h (Fig. 5). The hardness values of the samples were recorded as 130.2 and 134.8 HV when the ageing times were increased to 6 and 9 h respectively. The maximum hardness value of 147.2 HV was obtained at 225°C for 6 h. These results are compatible with Mousavi Abarghouie and Seyed Reihani [8]. Longer ageing times at 200 and 225°C caused over-ageing, resulting in a decrease in the hardness of the material. Fig. 6 also shows the effect of SiC addition on the hardness. For a comparison, the heat treatment procedure which results in highest hardness value was applied to the sample without SiC addition. The hardness of the material without SiC addition in the as-cast condition was 90 HV. After ageing for 6 h at 225°C the hardness of this material could only reach 129 HV. It is evident that the addition of SiC to the aluminium alloy increases the hardness of the alloy.

3.3. Wear properties

Fig. 7 shows the comparison of the friction coefficient of the alloy used in this study with and without added SiC under an applied load of 40 N for the distance of 500 m. It is clear that with the addition of SiC particles to the alloy, the material exhibits a lower friction coefficient, as a result of an increase in the hardness of the alloy. At the initial stage of the wear test, due to the high hardness of the oxide layer on the surface of the material, the friction coefficient is high. This oxide layer can easily be broken and removed.
by the direct contact with the counterfaces, after which the friction coefficient tends to decrease and reaches a steady value.

Fig. 8 shows the wear rate of the aluminium composites as a function of ageing times and temperatures. For both counterfaces, a slight decrease in the wear rate was determined with increasing ageing temperature, and the material showed the minimum wear rate at 225 °C for the duration of 6 h as a result of high hardness. Gavgali et al. showed similar behaviour for AA 6063 alloy [20]. Comparing the two counterfaces, the composite exhibited higher wear resistance against the alumina ball due to the change of the wear mechanism from abrasive to adhesive. The adhesion of aluminium matrix on the alumina ball changes the wear mechanism. After the adhesion, the two aluminium surfaces start to slide over each other, which causes a drop in the wear rate of the specimen.

Fig. 9 shows SEM images of steel and alumina balls after wear tests showing the coarse adhesive layer on the surface. This is because under the same load with a higher contact temperature for the alumina ball due to its low heat transfer characteristic, the matrix material becomes softer and transfers through the alumina ball. It is evident that the amount of aluminium composite transferred onto the ceramic ball was higher than with the steel ball.

Fig. 10 shows the worn surface of the composite with the highest wear ratio for two counterfaces. The alloy against the steel ball presents large grooves, while the material against the alumina ball exhibits narrow wear grooves. Therefore it is clear that the main wear mechanism was determined as abrasive and delamination wear.

For specimens aged at higher temperature, the worn surfaces exhibited similar characteristics, showing high displacement of material from the centre to the edge in the wear track, as shown in Fig. 11. This mechanism is both similar to microploughing wear, due to the accumulation of material along the edges of the worn surface, and similar to microcutting, due to the chip formation. The main process can be determined by the transfer of worn material from the centre to the edge, which causes oxidation and fracturing. The SEM observations show no effect of SiC on wear mechanisms. This may be a result of nanosize of the SiC reinforcements.

4. Conclusions

In this study, the effect of heat treatment on the tribological properties of Al–Cu–Mg/SiC alloy has been determined. The following conclusions are drawn:

- For ageing temperatures of 200 and 225 °C, an over ageing effect is reported and maximum hardness was obtained at 6 h. For 175 °C, no over ageing is reported because of the lower ageing temperature. The hardness of the as-cast sample increased from 110 HV to 147.2 HV with heat treatment.
- Addition of SiC to the alloy increases the hardness of the material but does not change the wear mechanism.
- An increase in the hardness causes a decrease in the wear rate. The specimen aged for 6 h at 225 °C has the lowest wear rate for both counterfaces.
The steel ball causes abrasive wear whereas the alumina ball causes adhesive wear on the composite surface.

- Adhesion of aluminium on the alumina ball decreases the wear rate during the wear test against the alumina ball.

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**References**


