Microstructure and tribological properties of in-situ formed Al$_3$Zr/A356 composite

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Keywords: aluminum matrix composites, friction and wear properties, in-situ generation, reinforced phase

Abstract

Al$_3$Zr/A356 Composite was prepared by in-situ reaction of K$_2$ZrF$_6$ powder and cast aluminum A356 melt at different temperatures (710 °C, 750 °C, 770 °C, 790 °C). The effect of different melting temperature on the morphology of Al$_3$Zr particles was studied, and the sliding friction and wear properties of the composites were studied by wear test. It can be seen from the x-ray diffractometer (XRD) that the prepared composite material consists of Al$_3$Zr and α-Al, and also has a small part of the aluminum-silicon eutectic phase; SEM analysis shows that the particles of in-situ reinforced phase are fine. With the increase of temperature, the morphology of Al$_3$Zr reinforced phase changed from block to needle and strip, and the particle distribution of the reinforced phase was uniform and well dispersed in the matrix at 750 °C. TEM experiments show that the reinforced phase exists at 750 °C and has a good combination with the matrix, which plays a very good role in particle reinforcement. Friction and wear experiments show that the different preparation temperature results in different phase morphology. The reinforced phase particles existing on the surface of composites at 710 °C and 750 °C bear most of the friction, so the friction coefficient of the composites is larger at these preparation temperature, and the main wear modes are oxidation wear and abrasive wear. The friction coefficient of the composites prepared at 770 °C and 790 °C is small, and the wear modes are mainly delamination wear and oxidation wear. When the preparation temperature is 750 °C, the wear resistance of the composites is the best.

1. Introduction

In recent years, particle reinforced aluminum matrix composites have been widely concerned because of their high specific strength, high specific stiffness, excellent wear resistance and so on. There are broad application prospects in the fields of automobile, aerospace, optical instrument and sports equipment. Therefore, particle-reinforced aluminum-based composites are becoming more and more highly valued and become a hot spot for researchers at home and abroad [1–8]. At present, powder metallurgy, spray deposition and infiltration are the common methods for the preparation of Particle Reinforced Aluminum Matrix Composites [9–11]. Although the composite prepared by powder metallurgy has good properties, its process flow is long, the procedure is complex and the cost is high. At the same time, the size of the products is limited [12–14]. The pressure infiltration method is to try to make the particle reinforced phase into the preform block, and infiltrate the melt into the gap of the preform under the pressure. The volume percentage of the particle reinforced phase in the composite material can reach 50%. The problems of the process are: the prefabricated block is easy to deform under pressure, the microstructure of the composite is uneven, the grain size is also relatively large, and the harmful interface reaction is difficult to control [15–18]. In situ generation is a widely concerned technology. It can replace powder and in-situ synthesis by adding particles into the melt. The composite material has the
advantages of small particle size, good interface combination, no pollution on the particle surface and not easy to produce brittleness [19–21]. The size and distribution uniformity of the reinforced particles play an important role in improving the mechanical properties and friction and wear properties of the composite, so it is very important to control the distribution of the reinforced particles in the solidified structure [22–25]. In this paper, the reinforcement phase Al₃Zr was produced by in-situ reaction method, and the (A356 + Al₃Zr) composite was prepared by fully reacting K₂ZrF₆ with A356 melt under mechanical agitation. XRD and SEM were used to analyze the phase and morphology of Al₃Zr reinforced phase. The friction and wear properties of (A356 + Al₃Zr) composites were studied by friction and wear experiments.

2. Experimental

The materials tested are cast aluminum A356 (trace element chemical content shown in table 1) and potassium fluorozirconate K₂ZrF₆ powder. The K₂ZrF₆ powder was heated to 200 °C in a drying oven, kept for about 2 h to remove the crystallized water contained therein and ground to a powder of <200 μm. The A356 was heated in a crucible resistance furnace to the set reaction temperature of 710 °C, 750 °C, 770 °C and 790 °C. In order to avoid the floating of salt, the salt mixture was introduced into A356 melt by bell graphite immersion method to react with 3 wt% salt powder [26–29]. The reaction equation is:

\[
3\text{K}_2\text{ZrF}_6 + 13\text{Al} \rightarrow 3\text{Al}_3\text{Zr} + 2\text{K}_3\text{AlF}_6 + 2\text{AlF}_3
\] (1)

The temperature of the melt in the reaction process was measured by thermocouple thermometer, and the graphite rod was used for mechanical stirring to make it react fully with A356 melt. After 10 min of reaction, the AlF₃ generated by the reaction will float to the molten liquid surface due to its light weight, and then be removed after slag extraction. And finally, the aluminum matrix composite reinforced by Al₃Zr particles was obtained by pouring into the metal model.

The phase composition of the sample was measured by the x-ray diffractometer (BRUKER D8 ADVANCE XRD) after the sample was polished. The microstructure of the prepared samples was observed by scanning electron microscope (Carl Zeiss Merlin Copact) and the composition of the reinforced phase was analyzed by EDS energy spectrum. The dry sliding wear test at room temperature was carried out at the sliding speed 200 rpm and at a load of 6 N using the UMT-2 friction tester.

3. Experimental results and discussion

3.1. Phase composition of in-situ formed aluminum matrix composites

Figure 1 shows the x-ray diffraction patterns at different preparation temperatures of 710 °C, 750 °C, 770 °C and 790 °C. It can be seen from this x-ray diffraction pattern that the prepared materials are mainly composed of three phases of AlSiAl₃Zr reinforcing phase. Because the higher the reaction temperature is, the more active the atoms are, and the more parts of Al and Si are replaced, there are a few AlSi eutectic phases on the Al matrix. Compared with the diffraction peaks at four different temperatures, the position of the diffraction peaks shifts slightly to the left with the increase of temperature. This is because with the increase of temperature, the atoms become more active, the formation of Al₃Zr particles increases, the volume of enhanced particles increases, the cell parameters become larger, the crystal plane spacing becomes larger, and more AlSi eutectic phases will be formed, which makes the position of diffraction peak slightly shift to the left, which also leads to the weakening of Al diffraction peak.

Figure 2 shows the SEM images of the composites prepared at different melting temperatures of 710 °C, 750 °C, 770 °C and 790 °C. As can be seen from figures 2(a) and (b) (figure 2(b) is the enlarged image of figure (a)) the reinforcing phase of the material prepared at 710 °C is uniformly distributed as a whole, and there are a few agglomerations, but there are many defects such as pores and holes. This is because the reaction temperature is low, and it is just the peak of gas generation at this time, there will be gas in the material which is difficult to overflow to generate pores, and the existence of large reinforcement phase limits the fluidity of the material, which makes the material performance reduce, resulting in defects. As can be seen from figures 2(c) and (d) (figure 2(d) is the enlarged image of figure (c)), The composite material prepared at 750 °C has dense structure,
the particle morphology is mainly short rod-shaped and block-shaped, and a small number of long-strip particles will be produced, the size is about 3–8 μm, the distribution of the enhanced phase is relatively uniform, without cracks, pores and other defects, the component segregation of the enhanced phase is not serious, and the overall distribution is appropriate. As can be seen from figures 2(e) (g), the distribution and agglomeration of the reinforced phase prepared at 770 °C and 790 °C are serious and have some defects. From figure 2(e) the circle part of the amplification figure 2(f), while the enhancement phase distribution is more, but the reunion phenomenon is serious, particle morphology are long strips, acicular, size around 7–10 μm, It can also be seen from figures 2(f) (h) that with the increase of the preparation temperature, the gain rate of the reinforced phase increases, the long strip particles increase, and the reinforced phase particles become coarser.

The morphology of reinforced phase of composites at different preparation temperatures is different. The shape of Al3Zr is sensitive to reaction temperature. When the melt temperature is 710 °C and 750 °C, most of the strengthening phases are short rod and bulk. The long and needle-like reinforcing phases increased greatly and the particles became coarser. This is because with the increase of preparation temperature, the atomic diffusion coefficient increases, the nucleation rate increases, and the grain grows. Therefore, with the increase of preparation temperature, the enhanced particles grow spontaneously.

Through the TEM image of the aluminum matrix composite prepared at 750 °C in figure 4, it can be clearly found that the difference between the enhancement phase and the aluminum matrix is translucent. Figure 4(c) is the diffraction spot of the reinforcement phase, which can be proved that there is the reinforcement phase Al3Zr in the aluminum matrix composite. The particles of Al3Zr reinforcement phase are black block and short rod, distributed in the aluminum matrix. From figures 4(a) and (b), it can be observed that the reinforcing phase is well combined with the aluminum matrix, the surface is seamless, complete and clean, and the size is about 400 ~ 600 nm. Therefore, combined with the previous SEM images, it can be concluded that at the preparation temperature of 750 °C, the particles of the enhanced phase Al3Zr are fine and dispersed in the aluminum matrix.

3.2. Mechanical properties of in-situ formed aluminum matrix composites
The macrohardness of Al3Zr/Al composites formed in situ by salt-coated K2ZrF6 at different preparation temperatures is shown in figure 5. The hardness of the composites prepared at 750 °C is the highest, and the hardness of the composites decreases slightly at 770 °C and 790 °C. This is because with the increase of reaction temperature, the particle distribution of the reinforced phase is heavily segregated, and the morphology changes, and gradually there are blocks, and the rod shape becomes a long strip, which weakens the dispersion of the particles and is easy to form agglomeration. At 750 °C, the fine particles are distributed in the collective, which plays the role of fine grain strengthening and hindering the slip of dislocations, but when the particles increase, the fine grain strengthening and dislocation pinning will weaken, and the hardness of the materials will decrease.
3.3. Friction and wear properties of *in-situ* formed aluminum matrix composites

The friction coefficient curves of aluminum matrix composites at different preparation temperatures using UMT-2 high temperature friction and wear testing machine are shown in figure 6. The friction process...
parameters employed are, load 6 N, speed 200 r/min, and the time is 30 min. It can be seen from the figure that the friction coefficient of the composites prepared at 710 °C and 750 °C decreases and becomes flat with the increase of the friction time. The friction coefficient of aluminum matrix composites at 770 °C and 790 °C fluctuates greatly and has a sharp peak value. This is due to the shedding of fragments and particles of Al3Zr particle reinforced aluminum matrix composites during wear, and the cracks caused by interfacial stress misalignment between matrix and \textit{in situ} particles due to different thermal expansion coefficients, which leads to the increase of surface roughness and friction coefficient. It can be found from the diagram that the friction coefficient of each preparation temperature is not much different, and the average friction coefficient of 750 °C is the largest, this is due to the fact that the mechanical properties of the composite at 750 °C are strengthened.

**Figure 3.** The EDS energy spectrum of composites prepared at 750 °C.

**Figure 4.** TEM image and diffraction spot of aluminum matrix composite at 750 °C, (a) (b) transmission image of aluminum matrix composites, (c) diffraction spots of the enhanced phase Al3Zr.
and the crack growth is hindered due to the uniform distribution of particles in the reinforcement phase, and the massive and Short rod-like Al3Zr reinforced phases were found. The wear debris and hard particles are pressed into the surface under the action of shear stress in the friction process, which makes the friction coefficient increase.

Observe the wear marks with SEM. As shown in figure 7, there are furrows and flake peeling pits on the worn surface, and plastic deformation occurs on the worn surface of the hard particles. Combined with EDS energy spectrum, it can be found that there are a large number of oxygen elements on the worn surface. It can be seen that there is an oxide film on the worn surface, which is due to the reason is that the grinding material Al2O3 produces a lot of heat and partial oxidation under the cutting action of contact rotating moment with the wear pattern. As can be seen from figures 7(a) (b), there is a large area of flaking on the worn surface due to the inherent voids in the composites prepared at 710 °C. Under cyclic contact stress, a crack is formed on the surface of the composite material, and the crack grows and falls off to form wear debris. The wear mechanism can be considered as abrasive wear and fatigue wear. Figures 7(c) (d) in addition to obvious furrows, there are pits left by debris. The furrow is formed by the hard particles adhering to the surface of Al2O3 ceramic ball and ploughing the surface of composite material. The wear mechanism can be understood as oxidation wear and abrasive wear. Due to the cyclic loading, the surface layer of the composite materials with the temperature of 770 °C and 790 °C produces shear plastic deformation and accumulates continuously. Because of the serious segregation of the materials at these two preparation temperatures, the crack propagation is hindered less, the expansion is faster, which leads to more serious wear and larger debris shedding. When the crack extends to a certain length, the material between the crack and the surface will peel off in the form of flake wear debris [30–37], so the wear mechanism at this time is peeling wear.

From figure 7, the average wear area of composites with different preparation temperatures of 710 °C, 750 °C, 770 °C and 790 °C was measured to be $21373.16 \times 10^{-6} \text{ mm}^2$, $20971.55 \times 10^{-6} \text{ mm}^2$, $25096.10 \times 10^{-6} \text{ mm}^2$, $27124.80 \times 10^{-6} \text{ mm}^2$ by laser confocal microscope. According to formula $V = CS$, the wear
volume of 750 °C composite can be obtained. This is due to the fact that the reinforced phase particles of the temperature material prepared at 750 °C are block and short stick, uniformly distributed in the matrix, and analyzed by EDS energy spectrum. The oxygen content of the material at 710 °C, 750 °C, 770 °C and 790 °C is

Figure 7. SEM diagram of wear marks of composites at four preparation temperatures (a) (b) Preparation of al-based composites at 710 °C; (c) (d) Preparation of al-based composites at 750 °C; (e) (f) Preparation of al-based composites at 770 °C; (g) (h) Preparation of al-based composites at 790 °C.
29.54%, 36.66%, 36.98% and 38.03%, respectively. The oxygen content of the material at 750 °C is the lowest, and the oxygen content of the material at 790 °C is the highest. Because of the high temperature resistance and wear resistance of the grinding material Al₂O₃ ceramic ball, the ceramic ball has the characteristics of high temperature resistance, wear resistance and so on. Therefore, this part of O element is unlikely to come from Al₂O₃ ceramic ball, mainly from Al₂O₃ formed by surface oxidation of composite material during wear. The atoms on the surface of the material are usually unbalanced during friction, which is easy to act with the surrounding medium to form an oxide film. The surface film can reduce the friction coefficient of the material. Similar to the lubricating film, the surface film can make the atomic binding force or ion binding force between the friction pairs be replaced by the weak van der Waals force, thus reducing the surface molecular force. In addition, the mechanical strength of the surface film is lower than that of the matrix material, and the shear resistance is smaller when sliding, so the friction coefficient at 790 °C is lower.

The wear marks of different kinds of composites were observed and measured by laser confocal microscope, and the cross sections of different positions and different wear marks were selected. Three wear marks were selected at each position to measure the cross section area of wear marks and take their average value. With a formula \( K = \frac{CS}{FL} \) get its wear rate. (C is circumference, S is cross section area, F is load, L wear mark length.) The wear rate of the composites prepared at 710 °C is \( 0.5937 \times 10^{-6} \) (mm³/N × mm); The wear rate of the composites prepared at 750 °C is \( 0.7534 \times 10^{-6} \) (mm³/N × mm); The wear rate of the composites prepared at 770 °C is \( 0.6971 \times 10^{-6} \) (mm³/N × mm). It can be seen that the wear rate is the lowest at 750 °C, which is due to the uniform dispersion and fine particles in the matrix, which strengthens the wear resistance of the matrix.

4. Conclusion

(1) Al₃Zr/Al composites were prepared by chemical in situ reaction process. The results of XRD show that the composites are composed of Al matrix, AlSi eutectic phase and Al₃Zr reinforced phase. Scanning electron microscope (SEM) analysis shows that the microstructure of the material prepared at 750 °C is dense, there are no obvious cracks, pores and other defects, and the reinforcement phase is evenly distributed. TEM analysis shows that Al₃Zr, as the reinforcement phase, has a good combination with aluminum matrix, and the particles are relatively small, which plays a good role in particle reinforcement.

(2) With the increase of melting temperature, the particle morphology of the reinforced phase of Al Matrix composites prepared at 710 °C and 750 °C is mainly short stick and block, and the size is about 3 ~ 8um; At 770 °C and 790 °C, a large number of long-strip and needle-like particles with sizes of about 6 ~ 10 um began to appear.

(3) The wear experiments show that the wear time of the composites prepared at 710 °C and 750 °C increases, the wear amount becomes larger and the fluctuation is small, which is mainly abrasive wear and oxidation wear, and there are some delamination wear. The composites prepared at 770 °C and 790 °C have serious segregation. With the increase of wear time, there will be occasional peak, which shows delamination wear and oxidation wear; The friction coefficient of the materials prepared at 750 °C was the highest, about 0.45, and the friction properties of the materials prepared at 710 °C, 770 °C and 790 °C were relatively stable at 0.35.

Acknowledgments

(1) This research was financially supported by the National Natural Science Foundation of China, No. 51605206.
(2) Postgraduate Research&practice Innovation Program of Jiangsu Province, No.KYCX19_1669.
(3) Jiangsu Province key Laboratory of High-end structural Materials, No.hsm1806.

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