Aluminium composites prepared by laser cladding assisted by friction stir processing

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Abstract

A laser cladding and friction stir processing hybrid method was employed to produce an Al matrix composite layer. The microstructure, phase composition, microhardness and conductivity of the composites were investigated. A laser cladding layer with a thickness of approximately 200 μm was prepared on a 1060 aluminium plate and it was broken up and distributed on the Al matrix after friction stir processing. The particle/Al interfaces exhibited extremely good interfacial integrity. Microstructural observations revealed that an obvious in situ reaction occurred at the particle/Al interfaces, which effectively improved the bonding between the reinforcement phase and the matrix. TEM analysis and selected area diffraction enabled the identification of the intermetallic compounds and confirmed them to be Al₅Fe₂ and Al₃Fe. The average microhardness values of the friction stir processed composites reached approximately 85 HV. The electrical resistivity of the friction stir processed composites is slightly higher than that of the aluminium matrix.

1. Introduction

Aluminium alloys are the most widely used alloys in the mechanical, transportation vehicle, portable electronics, aerospace, steamship and other industries due to their advantages of low density, high specific strength, and good electrical and thermal conductivities [1–3]. However, a relatively low hardness and poor wear resistance greatly limit their application. The preparation of a surface-reinforced composite layer is a promising method to effectively improve the surface hardness and wear resistance while maintaining good toughness and ductility inside the aluminium matrix [4, 5]. Many surface modification technologies have been used to improve aluminium surface performance, such as thermal spraying [6], vapor deposition [7], laser cladding [8], friction stir processing [9] and so on. The laser cladding (LC) of aluminium alloys has been extensively studied by many researchers [10–12]. Large residual stressed and cracks at the cladding layer/aluminium interface easily occurred during the laser rapid melting/solidification process due to the large difference of the thermal expansion coefficient between the aluminium and the cladding layer [13]. Therefore, excellent bonding between the cladding layer and aluminium matrix has been difficult to obtain. In addition, laser cladding can very easily yield metallurgical defects, especially solidification cracks and brittle precipitation phases, with a network distribution.

Friction stir processing (FSP), a new material modification method, has become a research hotspot for aluminium surface composites [14–16]. The surface hardness and wear resistance can be markedly improved by particle reinforcement [17, 18] and microstructure refinement [19, 20]. Stationary shoulder tool in FSP was proposed and it can develop tiny gradient in the microstructure refinement across the thickness attributed to the less heat input and small temperature gradient [21–23]. Friction stir processed composites with a good distribution of the reinforcing material have been reported for aluminium matrix composites (Al/SiC [24–27],
Al/TiO$_2$ [28], Al$_2$O$_3$ [17, 29], Al/NiTi [30], Al/B$_4$C [31], Al/CNT [32, 33], and Al/ Ni [34]). Generally, the process involves incorporating the reinforcement into a groove or holes on the surface of the aluminium matrix plate and then applying FSP to mix the two. However, there are some problems, such as an easy extrusion of hard particles, an uneven distribution of the reinforcing phase and poor binding of the reinforcing phase with the Al matrix during friction stir processing. Liu et al [35] used LC and FSP composite technology to prepare an Al-Si composite coating on the surface of an AZ31B magnesium alloy. The surface layer was obviously refined, homogenized and densified. Mukherjee et al [36] indicated that FSP can significantly refine the microstructure, crush carbide particles and eliminate the metallurgical defects in LC layers. Tacikowski et al [37] also showed that LC and FSP hybrid surface treatment is an effective method that can obviously improve the performance of the surface composite layer.

In this paper, a LC and FSP hybrid method is proposed to prepare an in situ reaction particle-reinforced aluminium-based surface composite layer on a 1060Al substrate. The microstructure, phase composition, microhardness and conductivity of the composite layer were analysed. It is hoped that this investigation can serve as a reference for processes that improve the performance of the aluminium alloy plate surfaces.

2. Materials and method

2.1. Materials
The commercially available 1060-H14 pure aluminium alloy plates were cut into rectangular sheets with a size of 120 mm $\times$ 60 mm and a thickness of 4 mm. A mixed Fe-Co-Ni-Mo powder was used in this study. The nominal chemical composition (at%) of the Fe-Co-Ni-Mo mixed powder was Fe:80, Co:6, Ni:7 and Mo:7.

2.2. Composite layer fabrication by LC and FSP
The main preparation process of the aluminium-based surface composite material layer is shown in figure 1. The specific steps are shown as follows:

(1) The surface of the aluminium plate was processed with sandpaper to remove the oxide film on the surface, and the surface of the plate was cleaned with an acetone solution.

(2) A laser cladding diagram and surface of a clad layer are shown in figure 1(a). The Fe-Co-Ni-Mo mixed powder was stirred with ethylene glycol until colloids were formed, and then it was uniformly coated on the polished surface of the aluminium plate. Then, the sample was placed in a dry and ventilated place and air dried for 24 h. A semiconductor laser (SISTAN3000) was used for cladding, where the spot diameter was 5 mm, the focus was located on the coating surface, the laser power was 2 kW, the scanning speed was 280 mm min$^{-1}$, and high-purity argon was used as the protection gas.
The FSP was conducted using a modified XA5032 vertical milling machine with homemade fixtures. Friction stir processing was performed on the cladding area, as shown in figure 1(b). The stir tool was made of W18Cr4V high-speed steel with a columnar shaped shoulder (with a diameter of 20 mm) and a screw thread tapered pin (the root Φ6 mm, the tip Φ4 mm, and the length 3 mm). The main process parameters were as follows: the rotation speed was 950 rpm, the forward speed was 95 mm min$^{-1}$, and the number of stirring passes were 2, 4, and 6. In this experiment, the cladding layer (approximately 200 μm) was broken and dispersed in the aluminium matrix during the FSP process, the abrasion of the stir tool was very slight and negligible.

2.3. Characterization and properties evaluation
After LC and FSP, cross-sections of the samples perpendicular to the travelling direction were cut and polished. Microstructural changes from the base metal to the stirred zone with different numbers of FSP passes were examined by optical microscopy (OM), scanning electron microscopy (SEM) energy dispersive spectroscopy (EDS), and transmission electron microscopy (TEM). The microhardness measurements were performed by using the TUKON2100 Vickers microhardness tester. Samples with dimensions of 5 mm × 5 mm × 2 mm from the friction stir processed composites with different numbers of passes and original base material were used for testing the electrical resistivity, which was evaluated using a standard four-wire connection method.

3. Results and discussion
3.1. The microstructure of the laser cladding layer on the Al surface
A laser cladding layer with a thickness of approximately 200 μm was prepared on a 1060 aluminium plate. The typical microstructure of the laser cladding layer is shown in figure 2. Many cracks formed in the cladding layers, as shown in figures 2(a) and (c). In the process of laser cladding, the rapid heating and melting of the cladding material by a high-energy laser beam results in a large temperature gradient. The cracks occurred in the cladding layers and at the cladding layer/Al interface due to a large internal stress caused by the large difference in the linear expansion coefficients between the cladding material and the Al substrate and the large temperature gradient. The chemical compositions in different regions of the coating determined with EDS are shown in figures 2(b) and (d). It was found that there were differences in the elemental distributions in the different regions. The chemical compositions of region A were close to the original powder composition. The composition of region B showed obvious atomic diffusion with the aluminium matrix. The distribution of elements across the Al substrate to the cladding layer showed an obvious metallurgical bonding interface.

Figure 2. The typical microstructure of the laser cladding layer showing (a) the laser cladding layer, (b) the distribution of elements, (c) magnified view of the interface, (d) the composition, (e) the cladding layer surface and (f) a magnified view.
between the coating and the Al base material, and the bonding interface was wavy. The surface topography of the cladding layer showed a large number of obvious shrinkage pores, similar to the network distribution, as shown in figure 2(f). These results indicated that there were obvious cracks and shrinkage holes in the laser cladding layer, and there were obvious cracks at the cladding layer/Al interface after the laser cladding process.

Figure 3. OM images of the cross-sections showing as: (a) 2 passes, (b) 4 passes and (c) 6 passes.

Figure 4. SEM micrographs of the friction stir processed composites produced by different numbers of passes (a) 2 passes, (b) 4 passes and (c) 6 passes.
3.2. Microstructure investigations of the composites after FSP

Optical microscopic examinations revealed the cross section of the samples, as shown in figure 3. There are different regions including unaffected base metal, heat affected zone (HAZ), thermo-mechanically affected zone (TMAZ), friction stir zone (SZ). The retreating side was on the left while the advancing side was on the right of the image. The SZ was generated due to the direct interaction of the tool pin and the base material. With the increase of the number of stir passes, the size of the SZ increased slightly. The low magnification OM images of the cross-section cannot show the distribution of the cladding layer particles from different stir passes. Detailed particle distribution of the samples perpendicular to the travelling direction was investigated by SEM.

Figure 4 shows the cross-sectional morphology (as-polished condition) of the friction stir processed composites produced by different numbers of passes. The laser cladding layer coated on the Al matrix was broken up and distributed on the Al matrix after FSP. The original laser cladding was broken up into irregular particles of varying shapes ranging from \( \sim 200 \ \mu m \). The distribution was characterized by a large number of small particles around some larger particles. It can be seen that the uniformity of the particle distribution in the stirred area increased with an increase in the number of stir passes. In addition, it is worth noting that particles were incorporated into the substrate at the advancing side in the 2-pass friction stir processed composite, as shown in figure 4(a). As the number of FSP passes increased, additional particles were brought to the stirred nugget and the fragmentation increased, as shown in figure 4(c). This was attributed to additional plastic deformation and thorough mixing caused by the accumulated plastic strain and the repeated thermal exposure. Nevertheless, no significant change in the size and morphology of the alloy particles was observed as the number of FSP passes increased.

The details of the distribution in the aluminium matrix and the degree of in situ reaction between the particles and Al matrix are shown in figure 5. Figure 5(a) shows the multiscale particle distribution in the 4-pass friction stir processed composite layer. Magnified views of regions A and B are shown in the figures 5(b) and (c), respectively. The particle/Al interfaces exhibit extremely good interfacial integrity with no micropores. This can be attributed to the fact that the plasticized Al wet the entire surface of the alloy particles (especially the major Fe elements) and thereby prevented the formation of micropores and kiss bonding at the particle/Al interface. SEM images revealed obvious formation of a reaction layer at the particle/Al interfaces. Under SEM backscattering imaging, the greyscale of these small particles was significantly different from that of the original large particles, revealing that their composition may have changed. Figures 5(d)–(f) shows the degree of in situ reaction between particles and the matrix. The shape of the particles and their reaction with the aluminium matrix are clearly shown in the figures. Figure 5(d) shows particles with size between 1 ~ 10 \( \mu m \). It can be seen that the particles of this size had different grey distributions, revealing that they had an obvious reaction with the matrix, but the reaction was not complete, and there were still bright areas in the particles. In figure 5(e), most particles (approximately less than 2 \( \mu m \)) completely reacted with the aluminium matrix, and their colour became a
uniform light grey. This result indicated that there was a significant in situ reaction between the reinforcement particles and the aluminium matrix, which effectively improved the bonding between the reinforcement phase and the matrix.

To determine the distribution of the elements in the particles and near the particle/Al interface, element analysis and scanning of the 4-pass friction stir processed composite were carried out. Figure 6 displays a typical SEM image of a particles, EDS elemental maps and line scanning results. The SEM image of the particles and the corresponding EDS mapping of the elements are shown in figure 6(a). It can be seen that the Fe and Al elements experienced obvious mutual diffusion, as part of the Al atoms entered into the inside of the particle, and the Fe atoms also entered the aluminium matrix nearby the particle. Other elements with lower contents (Ni, Mo, and Co) showed no obvious distribution characteristics. Figures 6(b) and (c) show the line scan analysis results across the particle marked as a green line with blue circles at both ends. The element distribution curves revealed that there was a diffusion layer (from approximately 2 ~ 8 μm width) at the particle/Al interface. The interface reaction layer provided effective metallurgical bonding to the particle/Al interface and allowed the load to transfer effectively from the matrix to the stronger alloy particles to enable plastic deformation.

The composition of the grey reaction layer was analysed by EDS. The position where the analysis of the reaction particles occurred is shown in figure 7. Particles with a different contrast show different compositions. The composition of these reaction layers was analysed, and the EDS spot analysis results are shown in table 1.
The atomic ratio of Al:Fe at point 1 in figure 7(a) was close to 5:2. It can be speculated that the possible phase was Al$_3$Fe$_2$. The EDS analysis at point 2 in figure 7(a) shows that it consisted mainly of Al and Fe atoms and a small amount of Ni, Co and other elements. The original cladding layer underwent metallurgical reaction with the aluminium matrix after being crushed by friction stir processing, but the original elements comprising the alloy particles still existed inside the particles. At a high magnification, as shown in figure 7(b), the Al content in particles smaller than 2 μm was generally high, indicating that the in situ reaction between the particles and the Al matrix was relatively sufficient. In the reaction layer, a chemical profile was measured with a plateau from approximately 74–77 at.% Al, which could correspond to Al$_3$Fe, as shown in table 1. However, due to the small differences in the Al and Fe contents of the known Al$_3$Fe$_x$ phases, it was not possible to distinguish them by EDS. The phases present at the particle/Al interface had to be identified by selected area diffraction in the transmission electron microscope.

TEM analysis with selected area diffraction assisted by EDS permitted the identification of the intermetallic compound (IMC). Figure 8 shows a TEM micrograph of the interfacial area between the Al and alloy particles. It can be seen that the interface between the particles and the aluminium matrix is well bonding. Electron diffraction patterns performed on several precipitates confirmed them to be Al$_3$Fe (monoclinic phase) and Al$_3$Fe$_2$ (orthorhombic phase), as shown in figures 8(b) and (c). The Al$_3$Fe precipitates contained a high density of microtwin boundaries (figure 8(b)), as observed by Agudo et al [38]. The electron diffraction pattern of the Al$_3$Fe$_2$ phase is very close to that of the regular hexagon, but it is not strictly a regular hexagon, so it belongs to the orthorhombic system.

Based on the above analysis, it can be seen that the in situ metallurgical reaction mainly occurred between Al and Fe atoms with a relatively high content. In general, the Al-Fe phase diagram shows two different phases: (a) three Al-rich IM phases, namely, Al$_3$Fe, Al$_5$Fe$_2$, and Al$_3$Fe$_3$; and (b) Fe-rich intermetallic phases that include AlFe and AlFe$_2$ [39]. According to reports in the literatures [40–42], Al$_5$Fe$_2$ and Al$_3$Fe IMCs are more likely to be formed in the temperature range from 700 °C–900 °C, and IMCs with low Al composition, such as AlFe and AlFe$_2$, can only be formed at a higher temperature of over 1000 °C. Springer et al [43] reported that a thick IMC layer was formed and Al$_3$Fe$_2$ was the major constituent in the temperature range from 600 °C–800 °C in both solid/solid and solid/liquid interdiffusion experiments. In all the above findings, the effect of pressure and high rate deformation on the formation of IMCs were not considered. FSP is a typical thermal-mechanical coupling process in which temperature, pressure and deformation act together. It is reported that an increase in the pressure and deformation can cause the formation of an IMC layer at relatively low temperatures [44, 45].

![Figure 8. TEM micrographs of the intermetallic compound at the particle/Al interface showing (a) the interface, (b) selected area diffraction pattern of the Al$_3$Fe micrograph (monoclinic phase) and (c) SADP of the Al$_5$Fe$_2$ (B = [−101]) (orthorhombic phase).](image)

**Table 1.** EDS point analysis results of location in figure 7.

| Points | Al (at%) | Fe (at%) | Ni (at%) | Co (at%) | Mo (at%) | Possible phase |
|--------|---------|---------|---------|---------|---------|----------------|
| 01     | 70.94   | 20.50   | 5.40    | 3.16    | —       | Al$_3$Fe$_2$   |
| 02     | 45.05   | 37.13   | 10.67   | 5.51    | 1.64    | solid solution |
| 03     | 79.83   | 15.79   | 4.39    | —       | —       | Al-rich solid solution |
| 04     | 73.20   | 20.55   | 6.25    | —       | —       | Al$_3$Fe      |
| 05     | 77.75   | 22.25   | —       | —       | —       | Al$_3$Fe      |
| 06     | 75.52   | 19.09   | 5.39    | —       | —       | Al$_3$Fe      |

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The effect of temperature, pressure and deformation on the friction stir processed composites layer can be characterized by the effective temperature, as shown in figure 9(a). The material corresponds to the effective temperature, which combines the temperature, pressure and deformation, although the actual temperature is relatively low (the actual temperature near the stir pin was approximately 450 °C). Thus, the formation of IMCs, such as Al₃Fe and Al₅Fe₂, is quite likely at FSP temperatures, pressures and deformations, but for the formation of other IMCs, such as AlFe, higher temperatures are required. An illustration of IMC formation at the Al/Fe interface during FSP is shown in figure 9(b).

3.3. The performance investigations of the composites

The microhardness of the composites was measured using a TUKON2100 Vickers microhardness tester. The loading was 10 g, and the dwell time was 10 s. Figure 10 shows a cross-section with the test location and the microhardness values of the friction stir processed composites with different numbers of passes. Figure 10(a) shows the outline of the friction stir processed composites region. The approximate location of the hardness test is indicated. The transverse and longitudinal hardness distributions are shown in figures 10(b) and (c), respectively. The microhardness of the friction stir processed composites was higher than that of the base metal (41 HV). The width of the high hardness zone corresponded to the width of the compound mixing zone. The microhardness in the SZ increased significantly. No obvious softening phenomenon was found in the HAZ. The average microhardness values of the friction stir processed composites reached approximately 85HV, excluding...
the high hardness points corresponding to individual alloy particles. Moreover, it should be noted that the hardness distribution increased in uniformity with an increase in the number of stirring passed.

The increase in the microhardness was attributed to three factors. One is the grain refinement strengthening owing to the fine grain size of the Al matrix. The microstructure refinement in the FSPed Al alloys and Mg alloys have been reported in many literatures [17, 19–23]. FSP is characterized by intense plastic and frictional deformation. This severe plastically deformed material at elevated temperature induces the occurrence of dynamic recrystallization phenomenon in the SZ followed by fine equiaxed grain size microstructure. The comparison of grain size between the 1060Al base material and the SZ was shown in Figure 11. The average grain size of the 1060Al was approximate 30 ~ 60 μm and the grain shape was elongated, as shown in figure 11(a). The grains in the SZ were obviously equiaxed and most of the grain size was ~5 μm, as shown in figure 11(b).

Another factor is the Orowan mechanism due to the presence of particle reinforcements [46]. Compared with adding high-hard ceramic particles to achieve particle strengthening, the in situ reaction particles can achieve better reinforcement effect. The interface reaction layer provided effective metallurgical bonding to the particle/Al interface and allowed the load to transfer effectively from the matrix to the stronger alloy particles. In addition, thermal expansion dislocation strengthening resulting from the generation of dislocations due to the difference in the thermal contraction coefficients of the particles and Al matrix.

Figure 12 shows the electrical resistivity of the Al base and friction stir processed composites with different numbers of passes at room temperature. The electrical resistivity of the friction stir processed composites is higher than that of the aluminium matrix. It should be noted that increasing the numbers of FSP passes resulted
in a slight elevation of the electrical resistivity of the composites. The enhanced electrical resistivity may be attributed to the following two factors. First, the dispersed alloy particles and the IMCs in the friction stir processed composites increased the electrical resistivity. The dispersed particles led to an increased volume fraction of particle/Al interfaces, which increased the electrical resistivity due to the high electrical resistivity of alloy particles and the IMCs. Second, the electrical resistivity was affected by the grain size, and the grain boundary resistivity can be enhanced by reducing grain size. Thus, the friction stir processed composites with notably refined grains had a slightly higher electrical resistivity than the aluminium matrix.

4. Conclusion

LC and FSP hybrid surface modification methods were proposed to prepare an Al aluminium base surface reinforcement layer. The microstructure, phase composition, microhardness and conductivity of the composite layer were analysed. The following conclusions can be drawn:

(1) A laser cladding layer with a thickness of approximately 200 μm was prepared on a 1060 aluminium plate. An obvious metallurgical bonding interface between the cladding layer and the Al base material was formed. There were obvious cracks and shrinkage holes in the laser cladding layer and on the cladding layer/Al interface.

(2) The laser cladding layer coated on the Al matrix was broken up and distributed on the Al matrix after FSP. There was a significant in situ reaction between the reinforcement particles and the aluminium matrix, which effectively improved the bonding between the reinforcement phase and the matrix. TEM analysis with selected area diffraction permitted the identification of IMCs and confirmed them to be Al5Fe2 and Al13Fe.

(3) The average microhardness values of the friction stir processed composites reached approximately 85HV, which is higher than that of the base metal. The hardness distribution increased in uniformity with an increasing number of stirring passes. The electrical resistivity of the friction stir processed composites is slightly higher than that of pure aluminium.

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Conflicts of interest

The authors declare that they have no conflicts of interest.

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