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# The effect of mix ratio of dissimilar aluminium chips on microstructure and property of solid-state recycled alloy

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## Abstract

To overcome the restriction that the solid-state recycled processes can only be performed for the single brand of aluminium alloy chips, the feasibility of solid-state recycling of ADC12 and 6005A aluminium chips at varying ratios was investigated. The chips were first mixed by ball milling and then hot compacted before hot extrusion. The results revealed that ball milling can effectively mix and refine the chips, the chips gradually refined as the milling speed increased, in contrast, the milling time and the ratio of mixed chips had little effect on the morphology and average size of the chips. Microscopic examination showed that the recycled alloy with a mixing ratio of 9 : 1 between ADC12 and 6005A alloy achieved the best bonding quality, with the oxide layer and Fe-rich phases crushed and dispersed in the Al matrix, which improved the strength of the recycled alloy through the dispersion strengthening mechanism. The tensile test results also showed that the recycled alloy with the ratio of 9 : 1 between ADC12 and 6005A alloy has prominent mechanical properties, with the ultimate tensile strength, yield strength, and elongation of 271 and 151 MPa, and 12 % respectively. The investigation in this work provides a novel and effective method for the recycling of aluminium alloy chips and contributes to the sustainable resource utilization of the aluminium industry.

Key words: solid-state recycled, dissimilar aluminium chips, microstructure, mechanical properties

## 1. Introduction

In various industries, the production of primary aluminium stands out as highly energy-intensive and a major source of pollutants, accounting for approximately 3 % of the global industrial energy consumption and about 4 % of total $CO_2$ emissions. In stark contrast, the energy consumption and greenhouse gas emissions of recycled aluminium amount to a mere 3–5 % and 5.3 % of those associated with primary aluminium production [1–3]. Therefore, for response to the urgent challenge of global warming, driving technological progress in the aluminium industry, especially in the area of aluminium alloy recycling, is of the utmost importance. At present, conventional remelting and solid-state recycling (SSR) are the dominant methods for recycling aluminium alloys. Remelting, however, requires heating aluminium alloys beyond their melting point, which still resulting in highly metal loss, energy consumption and substantial $CO_2$ emissions [4–7]. Furthermore, when dealing with machining chips, this process unavoidably causes significant metal loss. By comparison, solid-state recycling involves mechanical processing at temperatures ranging from 300 to 500 ℃, which not only saves 40 % of materials, 26–31 % of energy, and 16–60 % of labor, but also improves material re-utilization rate [8]. Consequently, this approach is emerging as a practical and viable alternative to remelting and other traditional recycling methods.

Current solid-state recycling techniques include hot extrusion, field-assisted sintering techniques, fric-

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Table 1. Chemical composition of ADC12 and 6005A alloy (wt.%)

| Element | Si | Fe | Cu | Mg | Mn | Zn | Al |
|---|---|---|---|---|---|---|---|
| ADC12 | 10.56 | 0.85 | 1.91 | 0.21 | 0.28 | 0.55 | Bal. |
| 6005A | 0.88 | 0.53 | 0.20 | 0.70 | 0.27 | 0.16 | Bal. |

tion stir extrusion and so on. Compared with other solid-state recycling methods, hot extrusion stands out for its ability to produce more complex and precise metal profiles with excellent mechanical properties, and it is also can offer high material utilization and relatively simple process. Furthermore, by optimising the hot extrusion parameters, it can destroy the oxide layers on the aluminium chips by induces sufficient plastic deformation to achieve the better bonding and a more homogeneous dispersion of oxides in the aluminium matrix [9–17], thus obtain a more uniform and stable product. Therefore, the hot extrusion method has received extensive attention in aluminium alloy recycling research. Lela et al. [11] developed a mathematical model that relates extrusion parameters to the mechanical properties of aluminium chips, and conducted hot extrusion experiment on ENAW2011 aluminium chips using response surface methodology (RSM) and Box-Behnken experimental design. The results showed that the extrusion temperature had the greatest impact on the mechanical properties, while the influences of chip size and compression force were minimal. Finally the recycled alloy obtained the maximum tensile strength and yield strength under the conditions of extrusion temperature of 358 ℃, cutting depth of 1.75 mm for chips, and compression force of 200 kN. Aal et al. [13] and Mahmoud et al. [14] both investigated the effects of extrusion ratio and temperature on the microstructure and mechanical properties of the recycled aluminium chips and found that increasing the extrusion ratio and temperature significantly enhanced the bond quality and tensile properties of recycled alloys. Under the conditions if extrusion ratio of 12.8 and a temperature of 500 ℃, the most uniform structure and highest mechanical performance were obtained. In addition, recycling aluminium chips containing reinforcing particles is expected to further improve the performance of recycled alloys [18–21]. Fogagnolo et al. [18] investigated the cold-press combine hot extrusion technique for recycling $Al_2O_3$-reinforced AA6061 chips and found that the recycled aluminium alloy exhibited higher ultimate tensile strength (UTS) and hardness than the primary alloy.

Although extensive research has been conducted on the solid state recycling methods of aluminium alloy chips by numerous researchers at present [22–25], the large-scale intrusion and accumulation of impurity elements in the recycled alloys has emerged as a new great challenge [26–28]. It is worth nothing that the specific combinations of mixed chips can reduce defects such as compositional inhomogeneity and non-metallic inclusions, while reducing impurity levels to promoting more efficient and higher-quality recycling, and potentially enhancing the overall performance of recycled alloys. However, most existing research has focused on recycling same-type alloy chips, with less attention given to mixed chips. Thus, this work investigated the feasibility of recycling mixed aluminium chips via hot extrusion to meet the demand for high-performance materials and advance aluminium sustainability. The effects of ball milling parameters and chip mixing ratios on the microstructures and mechanical properties of the solid-state recycled alloys were systematically investigated.

## 2. Experimental details

A die-casting alloy ADC12 and a wrought alloy 6005A, which commonly used in automobiles production, were employed as the raw materials in this study, the chemical composition of the two alloys were tested by an M5000 Metal Analyzer CCD direct-reading spectrometer, as listed in Table 1. And their chips were milled from the automotive blanks manufactured by Harbin Jixing Mechanical Engineering Co., Ltd., as shown in Fig. 1. The ADC12 chips had lengths of 4.30 ± 0.50 mm, widths of 1.70 ± 0.30 mm, thicknesses of 0.30 ± 0.03 mm, while the 6005A chips had lengths of 8.40 ± 0.50 mm, widths of 4.90 ± 0.40, thicknesses of 0.79 ± 0.07 mm, respectively. This difference in chip size can be attributed to the higher microhardness of the 6005A alloy (127 HV) compared to the ADC12 alloy (80–90 HV). The increased hardness of the 6005A alloy makes it more resistant to deformation, resulting in the formation of larger chips.

Then the ADC12 and 6005A alloy chips with a mixing ratio of 1:1 were mixed by A vertical Planetary Ball Mill (XQM-4) under different milling speed and milling time, as listed in Table 2, to investigate the effect of milling conditions on the refinement and uniformity of the mixing chips. Based on the results investigated above, the optimal ball milling process parameters were proposed, and the chips of ADC12 alloy and 6005A alloy were mixed with different ratios of 1 : 1, 3 : 1, 5 : 1, 7 : 1, 9 : 1, 11 : 1 (and the corresponding recycled alloys were designated as L1, L2, L3, L4, L5, and L6), by using the optimal ball milling process.

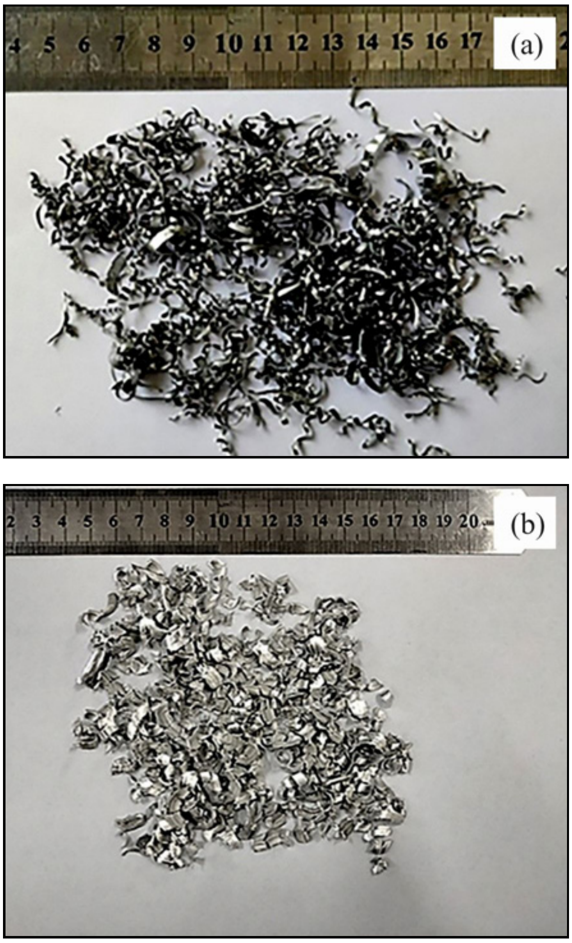

Fig. 1. Macroscopic feature of aluminium chips: (a) ADC12, and (b) 6005A.

Table 2. Ball milling and solid-state recycling process parameters

| Parameters | Value |
|---|---|
| Ball-to-Powder ratio | 3 : 1 |
| Milling time | 30 min, 60 min |
| Milling speed | 100–600 rpm |
| Milling temperature | Room temperature |
| Pre-compacted mold size | ø 40 mm × 110 mm |
| Pre-compacted temperate | 250 ℃ |
| Pre-compacted pressure | 640 MPa |
| Pre-compacted holding time | 45 s |
| Extrusion ratio | 25 : 1 |
| Extrusion temperate | 400 ℃ |
| Extrusion pressure | 864 MPa |
| Extrusion speed | 1 m min$^{-1}$ |

After that, the mixed chips were recycled by solid-state recycling process, including the pre-compacted and hot extrusion, the detailed solid-state recycling process parameters were also listed in Table 2.

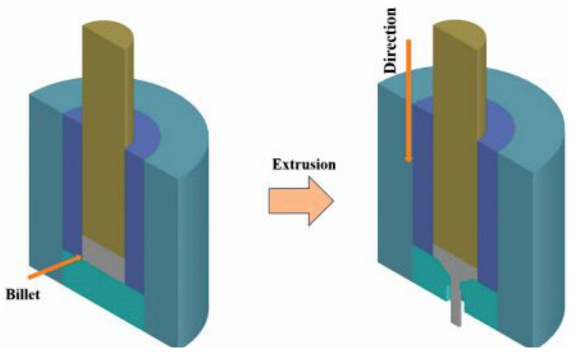

Fig. 2. The schematic diagram of the compaction and extrusion process.

To enhance the integrity and densify of the billets, the mixed chips of ADC12 and 6005A alloy were loaded into an H13 steel mold with 40 mm inner diameter and 110 mm height, and the mold was pre-coated with a thin layer of graphite to reduce friction during compaction. The axial pressure was applied to compact the chips into billets with preheated temperature of 250 ℃ and pressure of 640 MPa, as illustrated in Fig. 2a. After compaction process, the billets were placed into an extrusion die with 40 mm inner diameter and preheated to 400 ℃ for 30 minutes. Then extrusion was implemented under the extrusion ratio of 25 : 1, pressure of 864 MPa and extrusion speed of 1 m min$^{-1}$ , as shown in Fig. 2b.

The as-extruded specimens for microstructural analysis were ground, polished, and etched by 1 % hydrofluoric acid (HF) and subsequently observed by an optical microscope (Leica DM ILM) and a scanning electron microscope (Thermo scientific Apreo C), and the phase composition of the specimens was analyzed by XRD (Ultima IV). Tensile specimens were tested by a universal testing machine (UTM5305H) at a constant strain rate of 1 mm min$^{-1}$ at room temperature until fracture occurred. All tensile specimens were machined along the extrusion direction with a gauge length of 8 mm, as shown in Fig. 3. Additionally, each specimens was tested at least five times for the average value calculation. Finally, the fracture morphology of the specimens was analyzed by the same scanning electron microscope employed for the microstructural analysis.

## 3. Results and discussion

### *3.1. Effects of ball-milling on mixed chips*

The morphology and average size of the mixed ADC12 and 6005A alloy chips with mixed ratio of 1 : 1 after ball milling are presented in Fig. 4 and Table 3, respectively. It indicated that the surface mor-

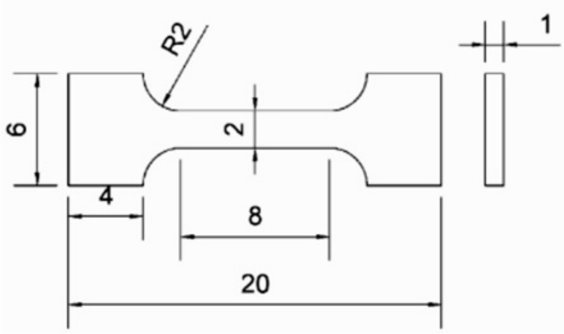

Fig. 3. Dimensions of tensile specimens (all in mm).

phology of the mixed chips experienced greater disruption, and different chips were mixed more uniformly with increasing milling speed. The size of both ADC12 and 6005A chips barely changed at a milling speed of 100 rpm, when the milling speed increased to 300 rpm, the deformation of the mixed chips became more pronounced. Notably, the ADC12 aluminium chips began to transition into a powder-like form, while the 6005A aluminium chips taken on smooth-edged, small block shapes with an average size of 5.5 mm × 4.0 mm × 0.71 mm, as depicted in Fig. 4c. When the milling speed reached 600 rpm, the ADC12 chips were fully transformed into powder, while the 6005A chips showed only a slight reduction in size compared to 300 rpm, with an average size of 5.3 mm × 4.0 mm × 0.58 mm. This difference can be attributed to the significant disparity in mechanical properties between the two aluminium

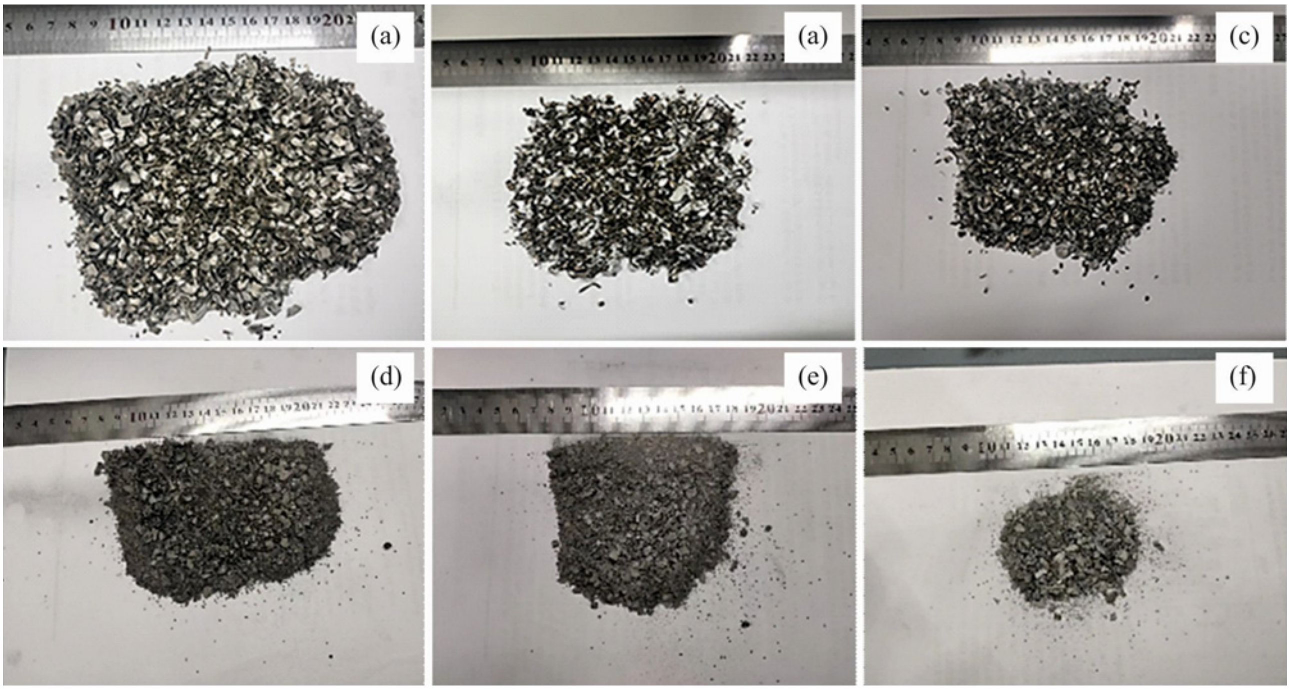

Fig. 4. The morphology of the mixed chips at different milling speeds: (a) 100 rpm, (b) 200 rpm, (c) 300 rpm, (d) 400 rpm, (e) 500 rpm, and (f) 600 rpm.

Table 3. The average size of mixed chips at different milling speeds at a ball-milling time of 30 min (mm)

| | 6005A | | | ADC12 | | |
|---|---|---|---|---|---|---|
| | Length | Width | Thickness | Length | Width | Thickness |
| As-received | 8.4 ± 0.5 | 4.9 ± 0.4 | 0.79 ± 0.07 | 4.3 ± 0.5 | 1.7 ± 0.3 | 0.30 ± 0.03 |
| 100 rpm | 6.9 ± 0.5 | 4.1 ± 0.3 | 0.79 ± 0.05 | 4.1 ± 0.3 | 1.3 ± 0.3 | 0.25 ± 0.02 |
| 200 rpm | 6.2 ± 0.4 | 4.1 ± 0.3 | 0.77 ± 0.05 | 3.9 ± 0.3 | 0.8 ± 0.3 | 0.21 ± 0.02 |
| 300 rpm | 5.5 ± 0.4 | 4.0 ± 0.3 | 0.71 ± 0.05 | 3.3 ± 0.3 | 2.6 ± 0.3 | 0.18 ± 0.01 |
| 400 rpm | 5.5 ± 0.4 | 4.0 ± 0.3 | 0.61 ± 0.04 | 2.9 ± 0.2 | 1.0 ± 0.2 | 0.17 ± 0.01 |
| 500 rpm | 5.4 ± 0.4 | 4.0 ± 0.2 | 0.60 ± 0.04 | 2.7 ± 0.2 | 0.8 ± 0.2 | 0.16 ± 0.01 |
| 600 rpm | 5.3 ± 0.4 | 4.0 ± 0.2 | 0.58 ± 0.04 | 2.6 ± 0.2 | 0.8 ± 0.2 | 0.14 ± 0.01 |

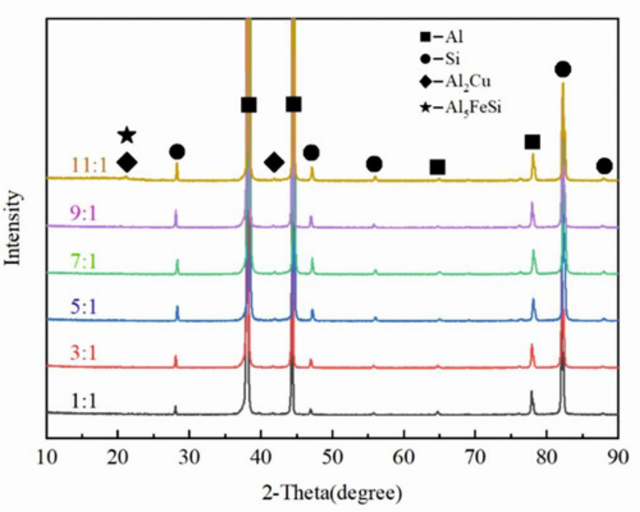

Fig. 5. XRD patterns of recycled alloys at different mixing ratios.

alloys, ADC12 as-cast alloy with higher silicon content offered a greater strength but lower ductility, while 6005A as-wrought alloy has better plasticity. As a result, the more brittle ADC12 aluminium chips are more likely to be broken during the high-speed ball milling process. The results demonstrated that a higher milling speed can achieve a uniform mixing of different chips. Hence, the chips of ADC12 alloy and 6005A alloy with six mixed ratios were ball milled at 600 rpm for 30 minutes, and the results suggested that the mixed ratio does not impact the mixed chips size under the same ball milling parameters. Additionally, the milling time was increased to 60 minutes at the same speed for all mixing ratios of the chips, it was found that no significant differences between the two milling times in terms of chip morphology and average size. Therefore, the optimal ball milling parameters were determined to be a ball milling speed of 600 rpm and a ball milling time of 30 minutes, which were applied to the chip treatment before solid-state recycling process of aluminium chips.

### *3.2. Microstructure evolution*

Figure 5 shows the XRD patterns of six kinds of recycled alloys, the results indicated that all solid-state recycled alloys consist of Al, Si, $Al_2Cu$, and $Al_5FeSi$ phases, as well as ADC12 alloy, and the diffraction peak intensity of Si increased with the increase in ADC12 content. To evaluate the bonding qualities between different aluminium chips, the microstructure of recycled alloys with different mixing ratios obtained by solid-state recycling process was shown in Fig. 6. It can be observed that the 6005A alloy chips are elongated along the extrusion direction, and there is a distinct boundary between ADC12 and 6005A alloy chips,which also indicates poor bonding between the chips. For the recycled alloys with relatively low mixing ratio, the distribution of Si phase particle size is heterogeneous. As shown in Figs. 6a,b, the coarse Si phase indicated by the yellow arrow is caused by uneven internal stress in the billet during the extrusion process. With the increase of ADC12 alloy chips content, the content of Si phases also increase accordingly, while the size of Si phases decreased and became more uniform. This is attributed to the fragmentation of Si phases during the extrusion process, and the effect becomes more pronounced with the increase in ADC12 alloy chips content.

The better bonding can be achieved when the mixing ratio above 9 : 1, as shown in Fig. 6e. This is owing to more ADC12 chips were ground into powder during the ball milling process, which filled the gaps between the 6005A chips and promoted better bonding. Additionally, high content and uniform distribution of harder Si phases within the ADC12 alloy chips promoted the deformation of the 6005A chips through migration during extrusion process, also facilitating the better bonding.

To further investigate the interface bonding between the two kinds of chips, SEM characterization and EDS analysis were performed on the solid-state recycled alloys, as shown in Fig. 7. The results indicated that hot extrusion did not induce significant component diffusion, due to the low solid-state diffusion, and the elemental distribution remained consistent with the type of alloy used. There was a distinctly poor bonding interface between ADC12 and 6005A alloys chips in recycled alloys with a low mixing ratio, as shown in Figs 7. The width of the defective bonding regions of recycled alloys L1, L2, and L3 were approximately 2–5 µm, with an accumulation of O and Fe. These findings indicate that the presence of an oxide layer and Fe-containing phases both hindered bonding between the chips, which had a negative impact on the quality of the recycled alloys. Notably, Cu enrichment was observed within the ADC12 regions; combined with XRD analysis, this phase was identified as $Al_2Cu$. As the content of ADC12 alloy chips increased, the width of the poor bonding regions at the interface decreased. Therefore, the L5 and L6 recycled alloys exhibited the best bonding with no obvious microcracks. Additionally, although the O content at the interface was still slightly enriched, the distribution of Fe elements within the alloy was uniform. This is due to the inconsistent deformation capabilities of the two alloy chips during the extrusion process, and the oxide layer at the interface of the chips has not been completely broken. In contrast, the increase in the chip content of ADC12 alloy can better fill the gaps between the 6005A chips and facilitated load transfer, further promoting the fracture of the oxide layer and the bonding between the

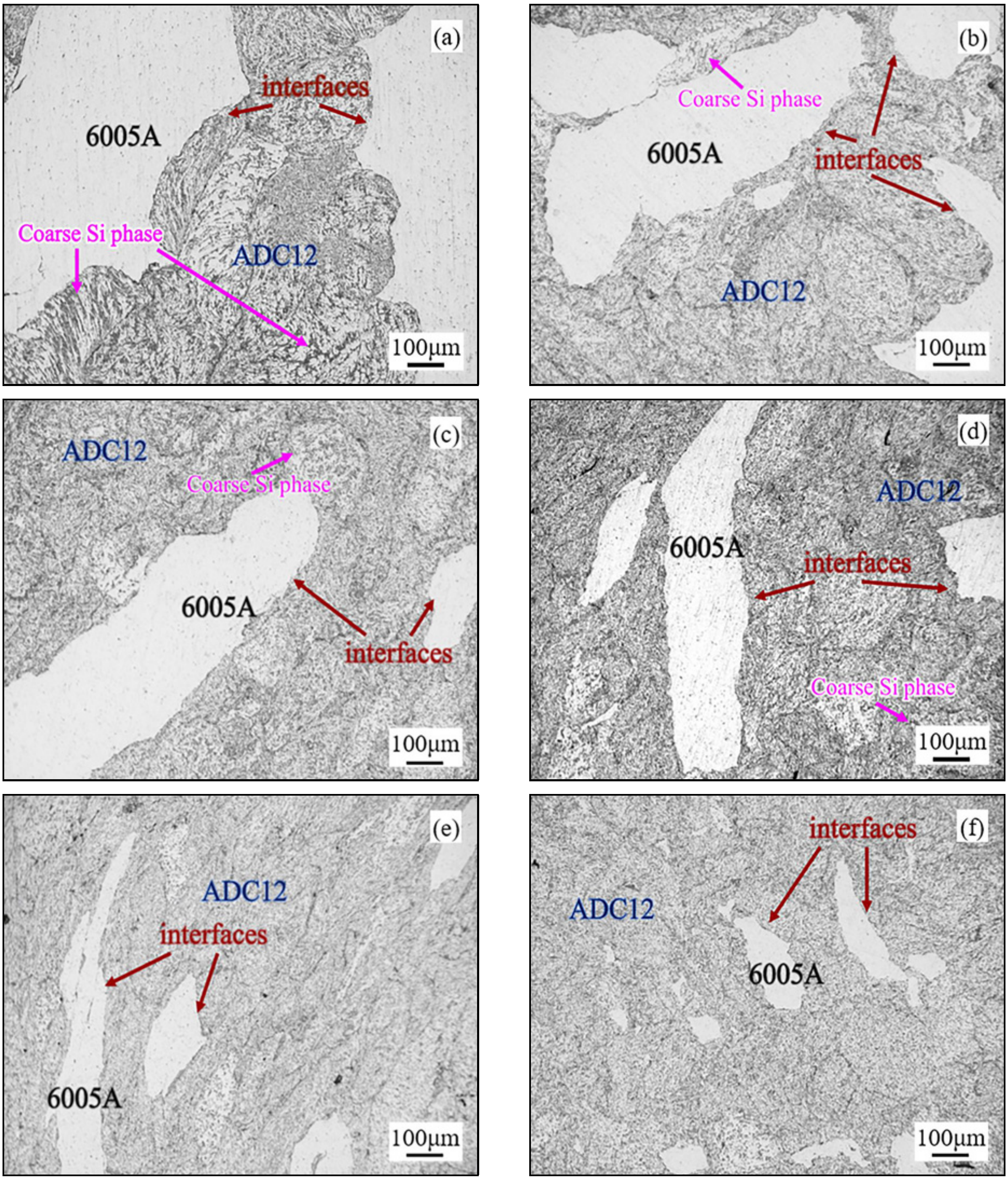

Fig. 6. The microstructures of recycled alloys at different mixing ratios: (a) L1, (b) L2, (c) L3, (d) L4, (e) L5, and (f) L6.

chips, enhancing the performance of the recycled alloy.

### 3. 3. *Tensile properties*

The tensile properties of the solid-state recycled alloys with different mixing ratios were shown in Fig. 8. The results indicated that the increase in content of ADC12 aluminium alloy chips can significantly enhance the mechanical properties of the solid-state recycled alloy, the tensile strength, yield strength, and elongation of the recycled alloys improved from 18.83, 29.91, and 55.62 %, as the mixing ratio increased from 1 : 1 to 11 : 1, respectively. And the tensile strength of all recycled alloys is higher than that of as-cast ADC12 and 6005A aluminium alloys, with the tensile strengths of the as-cast alloys being approximately 220 and 175 MPa [26, 29]. On the one hand, the size of the eutectic Si and Fe-rich phase are reduced due to fragmentation during the mechanical process, mean-

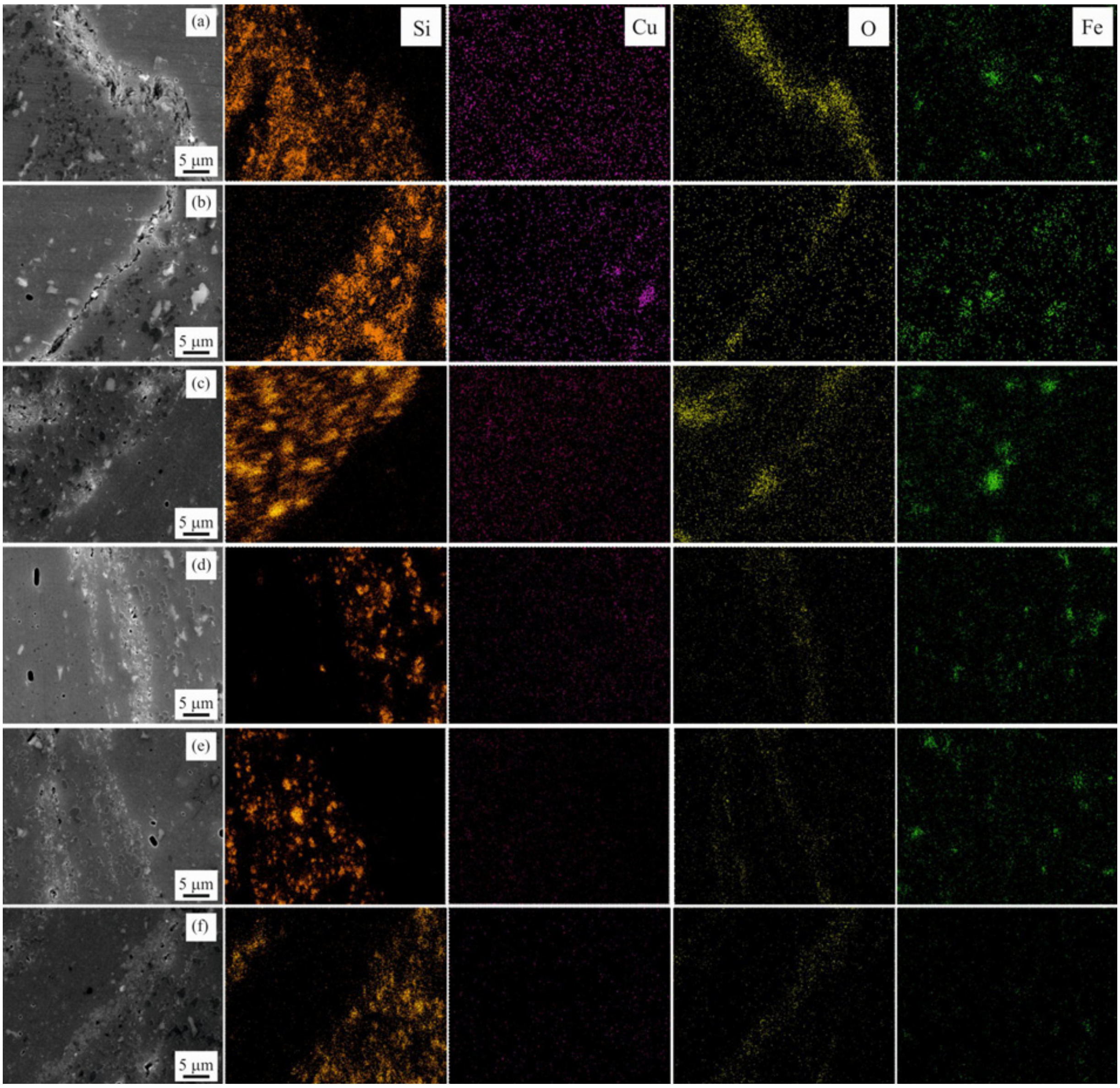

Fig. 7. The SEM and EDS results of solid-state recycled alloys at different mixing ratio: (a) L1, (b) L2, (c) L3, (d) L4, (e) L5, and (f) L6.

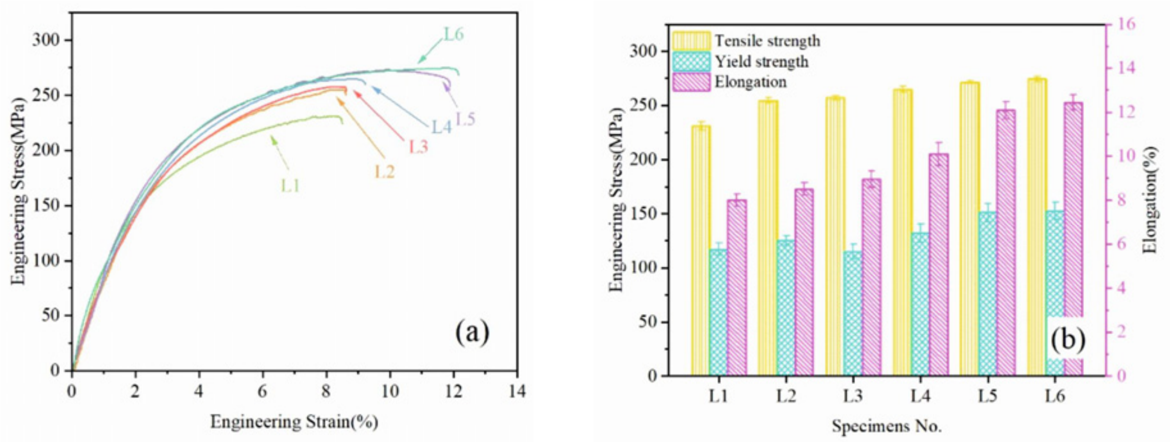

Fig. 8. The mechanical properties of solid-phase recycled samples at different heterogeneity ratio: (a) engineering stress-strain curves and (b) comparison of mechanical properties.

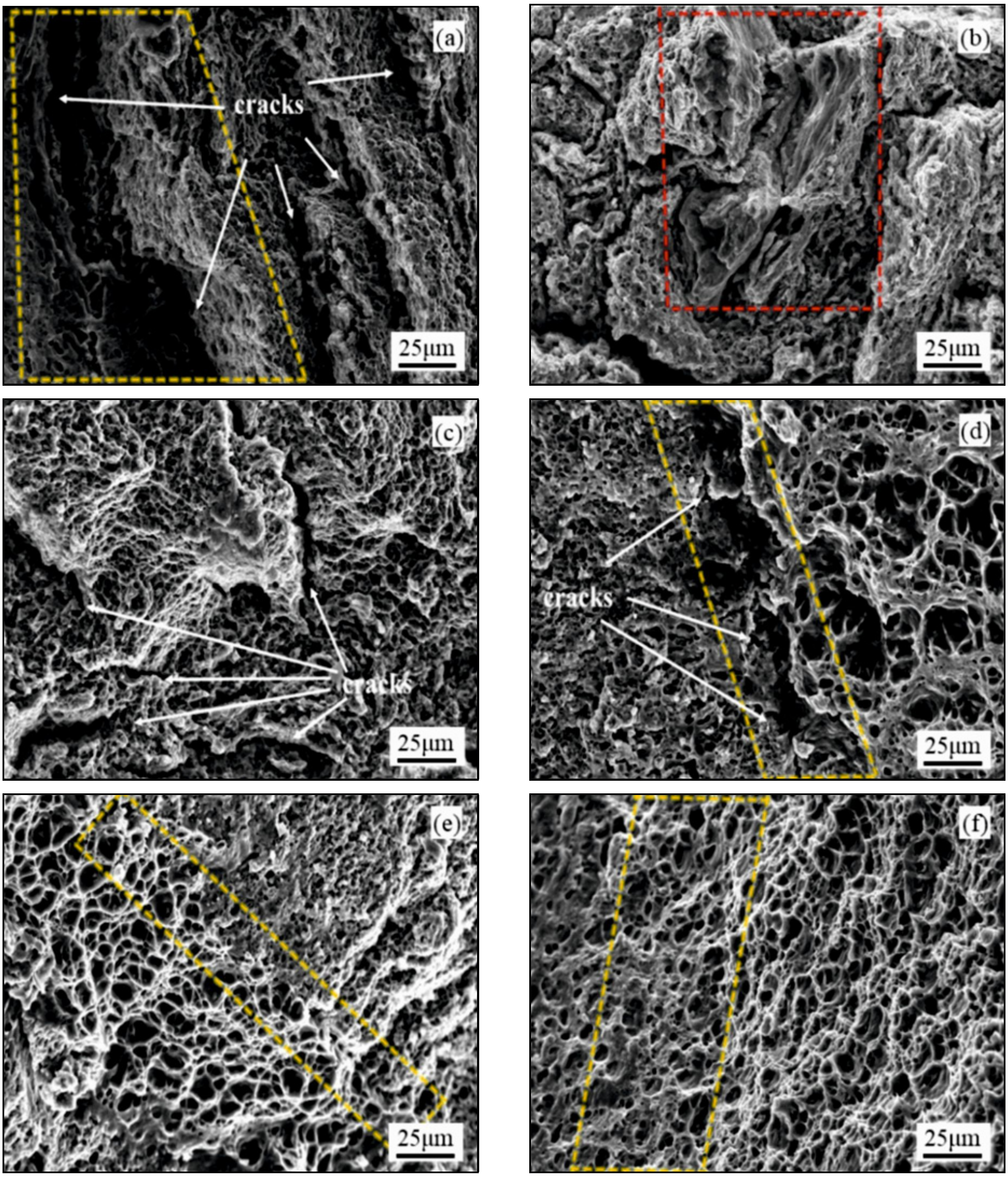

Fig. 9. Fracture morphology of solid-phase recycled samples at different heterogeneity ratio: (a) L1, (b) L2, (c) L3, (d) L4, (e) L5, and (f) L6.

while alumina and secondary phases are further broken and dispersed uniformly in the matrix during the extrusion, which is beneficial in increasing the strength of the alloy [30]. On the other hand, the higher ADC12 content induced an increase volume fraction of Si phases within the recycled alloy, consequently enhanced the load transfer and induced deformation in the 6005A alloy chips. This promoted better bonding between the two types of chips, and hence improved the mechanical properties of the recycled alloys. Notably, the mechanical properties of the recycled alloy with a mixing ratio of 9 : 1 were similar to those of recycled alloy with a mixing ratio of 11:1. Considering the impact of 6005A alloy content on reducing the internal impurity of the recycled alloys, it can be concluded that the mechanical properties of the recycled alloy with a mixing ratio of 9 : 1 are optimal, with a yield strength of 151 MPa, an ultimate tensile strength of 271 MPa, and an elongation of 12 %.

Figure 9 presents the tensile fracture morphology of recycled alloys with different mixing ratios. The results showed that as the content of ADC12 alloy chips increased, the number of dimples increased, while the micro-cracks decreased. Evidently, this is due to the improved bonding quality at the interfaces of recycled alloys with the increase in ADC12 alloy chips content, as indicated by the yellow dashed lines in the Fig. 9. The fracture surfaces of the L1 and L2 recycled alloys (with low ADC12 chip content) exhibited a large number of cracks, particularly at the interfaces between the different alloys, where larger cracks were observed, as shown in Figs. 9a,b, moreover, the pit-like features by red dashed lines marked in Fig. 9b were also observed in the L2 recycled alloy, which may be induced by debonding of the 6005A alloy chips during the tensile process. This suggested that at low mixing ratios, the poor bonding between the chips promoted the initiation and propagation of cracks, leading to brittle fracture of the recycled alloys. With the increase of ADC12 content, the bonding quality between the chips improved, thus, the smallest cracks and the varying sizes of dimples in two alloys chips were observed on the fracture surfaces of L5 and L6 recycled alloys, predominantly exhibiting a ductile fracture mode. Overall, adding a small amount of 6005A alloy chips to ADC12 alloy chips can effectively improve the bonding quality of solid-state recycled alloy, and resulting in a product with higher strength and better ductility.

## 4. Conclusion

In this study, mixed ADC12 and 6005A aluminium alloy chips were successfully recycled via hot extrusion. The effects of ball milling process parameters and mixing ratios on the microstructure and mechanical properties of the solid-state recycled alloy were investigated. And the following conclusions are made:

(1) Milling speed is a primary factor influencing particle size reduction and morphological changes during the ball milling process, while the mixing ratios and milling time had minimal impact. The optimal ball milling parameters were determined as a milling speed of 600 rpm and milling time of 30 minutes, which effectively transformed ADC12 chips into a powder form and reducing the size of 6005A chips by approximately 62.2 %.

(2) The interface bonding quality of the recycled alloys improved with the increase in mixing ratio, and the best bonding is achieved at a mixing ratio of 9 :1. This is due to the powdered ADC12 chips effectively fill the gaps between the 6005A chips, facilitates load transfer and induces deformation of the 6005A alloy chips.

(3) Due to the effect of load transfer, the oxide layer and Fe-rich phases are fragmented and evenly distributed in the matrix, and the bonding quality between different chips is improved with the increase in ADC12 content, which significantly enhances the mechanical properties of the recycled alloy. Considering that 6005A chips can reduce impurities in the recycled alloy to a certain degree, the optimal mechanical properties are reached with mixing ratio of 9 :1, with the yield strength, ultimate tensile strength, and elongation of 151 MPa, 271 MPa, and 13 %, respectively.

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