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Effect of rapid cooling on the microstructure and properties of fine-grained 7075 aluminium alloy under friction stir welding

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Abstract

In this study, 7075 aluminium alloy plates with a fine-grained structure were produced by four-pass equal-channel angular pressing and subsequently joined at room temperature (297 K) using carbon dioxide (CO2)-assisted cooling friction stir welding. Electron backscatter diffraction, x-ray diffraction, Thermocouple measuring instrument, optical microscopy and microhardness testing were used to investigate the microstructural and mechanical characteristics of friction stir welded joints. The results indicated that the maximum temperatures of welded joints at room temperature and CO2-assisted were 673 K and 568 K, macroscopic surfaces of the welded joints under CO2-assisted cooling were smoother than those created under room temperature; this was because the rapid cooling of liquid CO2 inhibited the growth of grains and, following dynamic recrystallisation, the grain size was finer (~2.9 μm). Compared with the fine-grained base material (BM), the proportion of large-angle grain boundaries in the stir zone region of the welded joints increased under both conditions, and the anisotropy was weakened. The precipitation hardening of the joints was obvious; the welded joints appeared to soften at room temperature, while the hardness of the joints was the same as that of the fine-grained BM under the CO2-assisted cooling condition.

1. Introduction

As lightweight structural materials, 7075 aluminium alloys have high strength and hardness and are widely used in aviation, aerospace and military applications. With the ongoing expansion of industrial demands, higher requirements are placed on the strength, hardness and plastic deformation capacity of traditional 7075 aluminium alloys [1]. Equal-channel angular pressing (ECAP) is one of the most effective severe plastic deformation methods for the preparation of bulk fine-grained materials. In this process, metal grains are refined through the pure shear deformation between two channels, thereby changing the mechanical properties of the material [2–4].

Fine-grained materials have become an important area of study in terms of materials and their preparation [5–7]. However, as research on the topic has developed, welding technology has become a limiting factor in the application of these materials. When applying conventional welding to fine-grained materials, due to excessive heat input, the grain growth process loses its original characteristics [8].

As a new solid-state joining technology, friction stir welding (FSW) has a significant inherent effect on the joining of fine-grained materials and low-melting-point metals, such as aluminium and copper [9–11]. Compared with a traditional welding method, FSW has the advantages of low heat input, small welding deformations, high production efficiency and low production costs; additionally, it is environmentally safe and can limit the growth of base metal grains by altering the welding environment [12].

The principle of FSW is illustrated in figure 1. Through the high-speed rotation of the stirring pin and the shoulder force, the heat friction between the weldment and the stirring pin creates local softening, thereby
enabling severe plastic deformation to occur. As the stirring pin moves along the direction of the combined line of the two specimens, the metal runs through the plastic flow to form a dense weld [13–15].

According to an existing study [16], fine-grained materials have inferior thermal stability compared with general materials. Even when the heat input is low, the grain size will still be enlarged when FSW is conducted on these materials [17]. Liu et al [18] applied FSW to a 6061 aluminium alloy with a grain size of 700 nm after cold rolling; the grain size in the stir zone (SZ) region increased to ∼2 μm. Wang et al [19] conducted underwater FSW on an ultrafine-grained 2017 aluminium alloy after ECAP; the grain size in the SZ region increased slightly (from 0.4 to 0.7 μm) from that of the base material (BM). Sato et al [20] carried out FSW on an 1100 aluminium alloy with a 0.42 μm grain size after accumulative rolling; the grain size in the SZ region increased slightly to 0.46 μm after welding.

In recent years, to optimise the microstructure and properties of the FSW joints of fine-grained materials, rapid cooling technology has been developed to reduce the thermal cycle of welded joints. For example, in underwater FSW, liquid carbon dioxide (CO₂) or a dry ice environment can inhibit the grain growth of joints and prevent them from softening [21–24]. Liquid carbon dioxide (197 K) has the advantages of low price, simple equipment required for implementation, high practicability and convenience, and has application value for industrial production. Moreover, the liquid carbon dioxide can be used to cool the weld more accurately, which can effectively improve the cooling rate of the weld [25, 26]. Therefore, this experiment uses liquid carbon dioxide for forced cooling.

In the present work, a fine-grained 7075 aluminium alloy was prepared using four-pass ECAP, and FSW was carried out at room temperature and under a liquid CO₂-assisted condition, respectively. The resulting microstructure and mechanical properties of the alloy were subsequently reviewed.

2. Experiment

2.1. Experimental materials

The initial material used in this experiment was a rolled 7075 aluminium alloy, the composition of which is shown in table 1. The 7075 aluminium alloy was processed into a 303183170 mm block sample. The sample was heat-treated at 750 K for 30 min before rapidly being cooled in water. The surface was smoothed using a milling machine and ECAP deformation was carried out.

2.2. The experimental process

Before ECAP deformation took place, a lubricant comprising graphite powder, oil and other components to the alloy. At room temperature, four-pass ECAP deformation was carried out in C mode, because the size of this study can only be applied to C and A modes, the performance of C mode is better than that of the two modes.
The angle of the die was 135°, and stress relief annealing was conducted at 573 K between passes. Following ECAP deformation, the sample was processed into a 3031834 mm sized plate; then, FSW welding was carried out at room temperature and under a CO₂-assisted condition, respectively. The pin was made of H18 steel, and its shoulder diameter was 18 mm; the maximum diameter of the pin was 5 mm, the minimum diameter was 3 mm, the welding speed was 600 r min⁻¹, and the welding speed was 50 mm min⁻¹. During CO₂-assisted welding, liquid CO₂ was sprayed onto the dense weld resulting from the high-speed rotating stirring pin and onto the subsequent weld so that the welding temperature rapidly decreased. In the welding process, measurement of weld temperature by K thermocouple and thermocouple thermometer, the specific welding process is shown in figure 1. The sample was corroded with Keller reagent, and the etched time was ~30 s. The microstructure of the joint was observed using an Olympus-GX51 metallographic microscope and further analysed using a Nordlys Nano electron backscatter diffraction detector. For the electrolytic polishing, the polishing solution comprised a 10% perchloric acid alcohol solution. The polishing temperature was 243 K, the voltage was 25 V, and the current was 0.4–0.5 A; the overall procedure was conducted in ~3 min. A phase analysis was conducted using a D8 Advance A25 (Brucker) x-ray diffractometer (XRD). The hardness test was carried out using an HX-1000 Vickers microhardness tester with a load of 0.98 N and a holding time of 10 s.

3. Results and discussion

3.1. The microstructure of the base material

Figure 2 shows the microstructure of the 7075 aluminium alloy BM after ECAP deformation. Figures 2(a) and (b) show that after four-pass ECAP deformation, the BM grains are equiaxed, fine and uniform in shape with a size of ~4.5 μm. This was because the extrusion and shear deformation occurred between the two channels of the die during ECAP deformation, which made the coarse grain (~90μm) transformed into fine grained base metal (~4.5 μm) after solution and natural aging. As shown in figures 2(c) and (d), the proportion of large-angle grain boundaries after four-pass ECAP deformation is ~41.8%, mainly between 45° and 60°. Figure 2(e) shows the BM pole figure after ECAP deformation. Combined with figures 2(a) and (e), it is shown that the texture on the (111) plane shows the strongest anisotropy, while the anisotropy on the (100) plane is the weakest. The grain orientation (C) was inclined in the ED and ND directions. This was because ECAP provided ideal shear deformation, and the texture orientation was rotated by 45° relative to the ECAP system.

3.2. The microstructure and properties of friction stir-welded joints

Figure 3 shows the thermal cycle curves under different cooling conditions in the welding process, and it can be seen that the temperature first increases and then decreases with time. The highest temperature at room temperature was 673 K, and 568 K with the assistance of CO₂, which were lower than the melting point of the base metal, indicating that the welding experiment was solid connection. The difference of peak temperature between the two welding environments is due to the rapid cooling effect of CO₂, which makes a large number of heat input loss. In the temperature drop stage, the cooling rate of the two welding temperatures is not the same (the slope of the curve when the temperature drops). It can be seen that the cooling rate under the assistance of CO₂ is greater than that at room temperature, because the temperature difference between liquid CO₂ and the joint is greater than that between room temperature and the joint, which is the main reason for the large cooling rate of CO₂. The greater the cooling rate, the faster the heat of the joint can be taken away.

The macroscopic morphology of a joint that had undergone four-pass ECAP under room temperature and CO₂-assisted FSW is shown in figure 4. The fly side on the advancing side (figure 4 AS arrow direction) was significantly more serious than on the retreating side (figure 4 RS arrow direction) because the plastic flow of the metals on both sides differed. Once plasticisation occurred, the metal moved from the forward to the retreating side, because the accumulation of heat on the retreating side led, the high dislocation density decreased, the fine grain coarsening and the plastic flow ability of the base metal increased after ECAP deformation. When macroscopically compared with an FSW joint, the surface of the latter was smoother; with the assistance of CO₂, the surface of the FSW joint was smoother and brighter, the flash was significantly reduced, and the shoulder scar was also reduced. This may have been because the rapid cooling effect of CO₂ could better remove heat during welding and thus reduce the heat input, thereby limiting the plastic flow of the metal, making the surface of the CO₂-assisted FSW joint brighter and smoother and creating an overall better welding effect [27].

The microstructures in different regions of FSW joints at room temperature and under CO₂-assisted conditions are shown in figure 5. The plastic flow of the metal was obvious under both conditions. Figures 5(a) and (d) show that the grains in the SZ region under the two welding conditions are equiaxed in a uniform distribution. The grain size in the SZ region under the CO₂-assisted condition was finer, the result of dynamic recrystallisation in the SZ region under mechanical stirring and thermal cycling of the stirring pin during FSW.
Figure 2. Microstructure of the base material after equal-channel angular pressing: (a) grain orientation, (b) distribution of grain size, (c) grain boundary distribution map, (d) angle grain boundaries and (e) pole figure.

Figure 3. The welding thermal cycle under different cooling conditions.
The rapid cooling effect of CO$_2$ inhibited the development of heat input and reduced the driving force of grain growth so that the grain size in the SZ region was finer compared with the conditions at room temperature. As shown in figures 5(b) and (e), the grains in the thermo-mechanically affected zone (TMAZ) show a different flow pattern from that in the SZ region, which is due to the deformation of the grains under the action of the pin and its shoulder. The orientation direction is related to the plastic flow direction. Due to an insufficient deformation strain, local dynamic recrystallisation occurred in the TMAZ region, and the grain size changes were not obvious. Figures 5(d) and (f) show the heat-affected zone (HAZ) areas under two welding conditions. The figure clearly shows that with the assistance of CO$_2$, the grain size remains slightly smaller and decreases gradually from the top to the bottom. This was because the friction between the shoulder and the upper surface of the BM provided heat input, but plastic deformation did not occur, which affected the grain growth behaviour within the upper surface. However, with a decrease in heat from the top to the bottom, the grain growth trend gradually weakened; this resulted in the inclined arrangement of the grain size [28].

The grain orientation in the SZ region of the FSW joint at room temperature and under the CO$_2$-assisted condition is shown in figure 6, where (a), (b), (e) and (f) show that the grain size in the SZ region of the FSW joint at room temperature is significantly finer than at room temperature. This was due to the rapid cooling effect of CO$_2$, which was consistent with front of this article. Dynamic recrystallisation was observed in the SZ region of the FSW joint, and the grains were equiaxed. However, compared with the BM grains, the grain size in the SZ region increased at room temperature to $\sim$5.54 $\mu$m, while the grain size in the SZ region was $\sim$2.9 $\mu$m under the CO$_2$-assisted condition and observably refined compared with the BM [26]. As shown in figures 6(c), (g), (d) and
(h), the grain boundary orientation reflects a bimodal distribution both at room temperature and for the CO$_2$-assisted case, and the proportion of high-angle grain boundaries (HAGBs) is higher compared with the BM. At room temperature, the proportion of HAGBs in the SZ region was $\sim 43.2\%$; with the assistance of CO$_2$, the proportion of HAGBs in the SZ region was $\sim 63.8\%$. This was because, during the welding process, dislocations were arranged and plugged to form sub-grain boundaries; additionally, observable severe plastic deformation occurred in the SZ region due to the high-speed movement of the stirring pin [29]. During the deformation, the proportion of low-angle grain boundaries increased due to the continuous absorption of dislocations by the sub-grain boundaries, which were transformed into HAGBs through continuous dynamic recrystallisation until they were finally transformed into equiaxed fine grains. Through the rapid cooling of carbon dioxide, the cooling rate of the welded joint was greatly improved, and the decrease in dislocation density caused by the thermal cycle was reduced. The sub-grain boundary increased its orientation difference by continuously absorbing any dislocation and transforming into a large-angle grain boundary. Therefore, the rapidly cooling of carbon dioxide could increase the proportion of large-angle grain boundaries [30, 31].

The pole and inverse pole figures of the SZ region of the welded joint at room temperature and in the CO$_2$-assisted condition are shown in figure 7. Here, (a) and (b) show that the maximum pole density of the SZ region of the FSW joint under the two conditions is significantly lower compared with the BM, (11.2 to 3.73 and 2.71, respectively). Furthermore, the anisotropy is weakened due to the high temperature and severe plastic deformation of the SZ region under the action of the stirring head, resulting in the phase transformation and rotation of the grains, which, in turn, leads to a decrease in the maximum pole density [32, 33]. With the decrease in temperature, the maximum pole density of the inverse pole diagram also decreased from 4.21 to 1.56, the upper surface of the BM.

The KAM diagrams and local orientation distributions in the SZ region for the fine-grained base metal, room temperature and CO$_2$-assisted welded joints after ECAP are shown in figures 8 and 9. The distribution of the KAM values in the three states was relatively uniform and close to 0° because the degree of recrystallisation
was relatively high in all three states. The recrystallised grains were new strain-free grains that had been generated from the deformed matrix; as such, their KAM values were low. Figure 8 shows that the KAM value near the grain boundary is higher. The higher KAM value inferred the presence of a higher dislocation density and greater plastic deformation. During ECAP deformation, formation of high density dislocations near grain boundaries, which increased the dislocation density and led to a higher KAM value near the grain boundary. The combined observation of figures 2(a) and (b) with figure 8(a) shows that deformation twins appear following ECAP (as indicated by the arrow in figure 8), and the plastic deformation degree is high at the deformation twin. As shown in figures 9, 8(b) and (c), the dislocation density in the SZ region after FSW is lower than in the fine-grained base metal. This was due to the presence of a thermal cycle in the FSW process, which decreased

Figure 7. Stir zone region of the friction stir-welded joints at room temperature and in the carbon dioxide-assisted condition. Pole figure: (a) at room temperature and (b) the carbon dioxide-assisted condition. Inverse pole figure (c) at room temperature and (d) the carbon dioxide-assisted condition.

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dislocation density after ECAP. Since the heat input in the CO\textsubscript{2}-assisted condition was lower than at room temperature, the KAM value was the lowest. A Schmid factor diagram of the \{111\} \{110\} grain slip system in the SZ region of the joint at room temperature and the SZ region of the joint welded with CO\textsubscript{2} assistance is shown in figure 10. Sun et al\cite{34} showed that the Schmid factor of materials is closely related to the complexity of the sliding system. The larger the Schmid factor, the easier the sliding system will be initiated, while the opposite will be true in terms of the strength of the material. It was concluded that the lowest Schmid factor value in the SZ region of the joint at room temperature was 0.29, which was higher than the minimum value of the Schmid factor in the SZ region of the CO\textsubscript{2}-assisted joint. The proportion of soft orientation (a Schmid factor of >0.45) in the SZ region of the joint at room temperature and with CO\textsubscript{2} assistance was 62.63% and 55.16%, respectively.

The 7075 aluminium alloy is a type of heat-strengthening aluminium alloy. The strengthening effect of a precipitated phase that is dispersed in the matrix was obvious\cite{35}. The XRD results of a fine-grained base metal welded at room temperature and under a CO\textsubscript{2}-assisted condition are shown in figure 11. The distribution of the precipitated phases of a fine-grained BM and an FSW joint has clearly changed and involves mainly the

Figure 8. The KAM of each region: (a) the fine-grained base metal, (b) the stir zone (SZ) region of the joint at room temperature and (c) the carbon dioxide-assisted joint SZ region.

Figure 9. Local orientation distribution of the fine-grained base metal, the stir zone (SZ) region of the joint at room temperature and the SZ region of the carbon dioxide-assisted joint.
Al$_2$CuMg, MgZn$_2$ and MgCu$_2$ phases. Among them, the Al$_2$CuMg phase with fine distribution had the best strengthening effect, followed by the long rod-like MgZn$_2$ phase [36]. The types of precipitates in the FSW joints at room temperature and under the CO$_2$-assisted condition were almost the same, and the diffraction intensity at room temperature was slightly higher than under the CO$_2$-assisted condition.

Figure 12 shows a hardness nephogram of the BM and the hardness curves of the FSW joints under room temperature and with CO$_2$ assistance. After four-pass ECAP deformation, the hardness of the BM increased from 100 HV to $\sim$140 HV, a noticeable increase of 40%. This was because when ECAP deformation was carried out, pure shear deformation occurred at the corner of the two channels of the specimen, which increased the dislocation density and broke up the large grains. The hardness of the specimen increased under the combined action of fine-grain strengthening and work hardening [37]. As shown in figure 12(b), the hardness of the FSW joints at room temperature and under the CO$_2$-assisted condition has a w-shaped distribution, and the hardness of the HAZ was the lowest ($\sim$95 HV at room temperature and 103 HV under the CO$_2$-assisted condition), all below base metal hardness (average 147 HV). No plastic deformation occurred in the HAZ region during FSW, but the heat input was high in the $\eta$-phase (MgZn$_2$) precipitation range; as a result, the fine precipitate ($\eta'$-Al$_2$CuMg) phase coarsened and transformed into a stable phase. Additionally, during the welding process, the internal stress after ECAP deformation decreased, the dislocation density decreased, and the heated grains and phases coarsened. The grain size was $\sim$11.23 $\mu$m at room temperature and $\sim$11.08 $\mu$m under the CO$_2$-assisted condition, which was observably coarser than in the SZ region [38]. The hardness of the SZ region was relatively stable, but compared with the BM, the hardness at room temperature was lower ($\sim$130 HV); this represented the softening phenomenon of the joint during welding. The grain size was finer than that of the BM under the CO$_2$-assisted condition, although the hardness was equal to that of the BM, which was slightly higher than at room temperature. This did not follow the Hall–Petch fine-grain strengthening theory, which posits that
fine-grain strengthening alone cannot improve the strength of a joint. $\eta'$ phase precipitation temperature of 7075 aluminium alloy is $663 \sim 703$ K, $\eta$ phase precipitation temperature is $573$ K [39]. During FSW, a large number of precipitates dissolved in the SZ region due to the high temperature, and the number of $\eta'$ was lower compared with the fine BM. According to the Orowan dispersion strengthening mechanism, the dispersion strengthening effect of a fine BM is better than that of FSW joints [40].

4. Conclusion

(1) Peak temperature is 673 K at room temperature and 568 K with CO$_2$ assistance, cooling efficiency can be improved by CO$_2$ assisted FSW. FSW joints of a 7075 aluminium alloy (BM) with an average grain size of 4.5 $\mu$m following ECAP were deformed at room temperature and under a CO$_2$-assisted condition, respectively. The surface of the FSW joints in the CO$_2$-assisted condition was more smoother.

(2) For the 7075 aluminium alloy (BM) FSW joint after ECAP, the grains in the SZ region were equiaxed. Due to the auxiliary effect of CO$_2$, the heat input in the SZ region was inhibited, and the grains were finer than those in the base metal ($\sim 2.9 \mu$m). At room temperature, the grain size in the SZ region was slightly larger than in the base metal ($\sim 5.54 \mu$m).

(3) Under the two conditions, the HAGB ratio of the FSW joint in the SZ area was higher than in the BM, and the HAGB ratio was the highest under the CO$_2$-assisted condition ($\sim 63.8\%$). After completing the FSW, due to the high temperature and severe plastic deformation in the SZ region, the maximum pole density decreased significantly. The KAM value decreased following the FSW, and the proportion of soft orientation in the SZ region was lower than at room temperature due to the forced cooling by CO$_2$.

(4) The types of precipitates after completing FSW at room temperature and under the CO$_2$-assisted condition were essentially the same and included mainly Al$_2$CuMg, MgZn$_2$ and MgCu$_2$. Compared with the fine-grained BM, the welded joint softened at room temperature, and the hardness of the SZ region was equal to that of the base metal under the CO$_2$-assisted condition.

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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.
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