Original Article

Microstructure and mechanical properties’ modification of low-temperature friction stir welded non-combustive Mg-9A1-1Zn-1Ca alloy joint

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ABSTRACT
Friction-stir-welded (FSW) Mg alloys usually exhibit an undesirable combination of strength and ductility compared to the base material, posing disadvantages to practical engineering applications. In this work, low-temperature FSW was applied on a non-combustive Mg-9A1-1Zn-1Ca alloy to modify the microstructure and mechanical properties of the welded joint. Results show that many dislocations, second-phase particles, and (10-12) twins were introduced into ultrafine grains of the welded joint, which could randomize the basal texture intensity and enhance strength throughout the joint, leading to fractures in the base material in the transverse tensile test. The appearance of coherent twin boundaries can efficiently accommodate dislocation, thereby elevating ductility and ultimate tensile strength.

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1. Introduction
Mg alloys have advantages of low density and high specific strength and are the most promising structural materials in the fields of aerospace and automobile manufacturing [1,2]. However, Mg alloys often exhibit poor plastic formability at room temperature due to their close-packed hexagonal (HCP) crystal structure [3,4]. Therefore, it is important to study the welding performance of Mg alloys to expand their applications in the manufacturing of large or complicated components. Friction-stir-welding (FSW) is a new solid-state welding technology, offering notable advantages in the joining of low-melting-point materials such as Al alloys [5,6]. For example, FSW can eliminate defects associated with fusion welding and produce ultrafine grains in the stir zone (SZ). In recent years, increasing research has focused on the FSW of Mg alloys, which have a similar melting point to Al alloys.

Unfortunately, a strong basal texture is often generated in the weld joint, leading to a drastic change in grain orientation from the SZ to the base material (BM) [7–13]. The (0001) crystal plane of grains in the weld are nearly parallel to the probe surface [7–10]. For this kind of grain orientation, the BM/SZ interface becomes a weakened region, and the fracture is preferentially located there during transverse...
tensile tests. A reduction in yield strength after FSW can be attributed to the texture-induced softening mechanism due to strong anisotropy of the weld. Therefore, post-welding deformation has been conducted to randomize the basal texture and improve mechanical properties of the FSW Mg alloy joint [14–16]. The dominant strengthening mechanism has been attributed to the (10-12) twins formed in the welded joint; these twins can simultaneously improve the strength and ductility of the FSW Mg joint. However, the relatively complex processing steps are not convenient in engineering applications. Therefore, a simple one-step processing method should be developed to modify the microstructure and mechanical properties of the FSW Mg joint, hence the main motivation of the current study. Recently, Sun et al. developed low-temperature friction stir spot welding technology to join Al alloy [17] and steel [18]. In this method, the welding temperature can be reduced to below 200 °C for a highly reduced tool rotation rate and increased axial force. The microstructure and mechanical properties of the joint can be enhanced significantly to improve the thermal circle. This technology should be further expanded to butt welding due to its various advantages. In this work, low-temperature FSW (LT-FSW) was designed to join non-combustive Mg-9Al-1Zn-1Ca (AZX911) alloy plates. This work aims to construct an FSW Mg joint with a randomized basal texture and well-matched strength and ductility.

2. Experiments

The as-received materials were non-combustive AZX911 Mg alloy plates (3 mm in thickness). A WC-Co based FSW tool with a right-threaded probe was used in this work. The welding tool and welding process are schematically shown in Fig. 1. To prevent the joint from oxidizing during welding, argon gas flowed around the tool at a flow rate of 20L/min. A K-type thermocouple was used to measure the temperature in the SZ, and a peak temperature of 168 °C was recorded during the welding process. After welding, the cross-sectional surface perpendicular to the welding direction (WD) was initially...
characterized using optical microscopy (OM). Microstructural features of the weld were further characterized by
electronic backscattering diffraction (EBSD) set in a field-emission
scanning electron microscope (FE-SEM). Solute elements in
the \(\alpha\)-Mg were determined using an energy dispersive X-ray
spectrometer (EDS), which was also set in the FE-SEM. Sub-
structures that could not be detected with EBSD in the SZ
were examined by transmission electron microscopy (TEM).
To prepare TEM samples, the samples were mechanically pol-
ished to 50 \(\mu\)m and then double-jet electropolished using a
solution of methanol:glycerin:nitric acid (6:3:1 in volume) at
20 V and \(-30\) °C. To evaluate the strength of the welded joint,
tensile samples covering the weld and the BM with a gauge
size of \(5 \times 12.5 \times 2.7\) mm\(^3\) were cut according to the JIS
Z2201(13B) standard. To investigate the strength of SZ, ten-
sile samples containing only the SZ were cut with a gauge
size of \(12 \times 3 \times 2\). All tensile tests were conducted at room
temperature at a crosshead speed of 1 mm/min.

3. Results and discussion

Fig. 2a shows the cross-sectional overview of the LT-FSW
AZX911 Mg alloy joint. Defects were not detected, indicating
that the as-received materials were successfully butt-welded
by LT-FSW. In addition, a basin-shaped SZ was clearly
observed at the joint center. The SZ area was relatively small
compared to the conventional FSW or double-sided FSW Mg
weld [19–21]; the volume of stirred material declined due
to the material’s reduced flow ability caused by the lower
welding temperature. The BM exhibited a homogeneous grain
structure, having an equiaxed \(\alpha\)-Mg matrix and precipitated
phases. Precipitated phases were identified as \(\text{Mg}_{17}\text{Al}_{12}\) and
\(\text{Al}_{2}\text{Ca}\) in the AZX911 magnesium alloy as reported in previous
studies [22,23]. The heat-affected zone (HAZ) exhibited a sim-
ilar grain structure as the BM due to a substantially improved
thermal cycle. Fig. 2b and Fig. 2c depict OM microstructural
features of two typical regions on the cross-sectional surface.
Grains in the thermo-mechanically affected zone (TMAZ)
had an orientation that aligned clearly with the material
flow direction caused by intense stirring; these grains were
also partially refined due to resulting plastic deformation
(Fig. 2b). The SZ exhibited an ultrafine grain structure, and
the precipitated phases were uniformly distributed in the SZ
(Fig. 2c). The Al content was relatively high for the AZX911 Mg
alloy, as the \(\beta\)-Mg\(_{2}\)Al\(_{12}\) phase was often generated based on
a eutectic reaction \((L \rightarrow \alpha\text{-Mg} + \beta\text{-Mg}_{2}\text{Al}_{12})\) [24]. The addition of
Ca caused the \(\text{Al}_{2}\text{Ca}\) phase to form in the SZ as well [25,26].
Therefore, the \(\beta\text{-Mg}_{2}\text{Al}_{12}\) and \(\text{Al}_{2}\text{Ca}\) phases each formed in
the as-received AZX911 Mg alloy. Interestingly, \(\beta\text{-Mg}_{2}\text{Al}_{12}\) and
\(\text{Al}_{2}\text{Ca}\) particles were both detected in the SZ as well. The
eutectic \(\beta\text{-Mg}_{2}\text{Al}_{12}\) phase will dissolve into the \(\alpha\)-Mg matrix at
temperatures higher than \(370\) °C due to poor thermal stability.
For conventional FSW, the welding temperature is usually higher than 370 °C, thus, the eutectic $\beta$-Mg$_{317}$Al$_{12}$ phase cannot be detected in the SZ. This phenomenon was also found in our previous study [27]. In this work, the $\beta$-Mg$_{317}$Al$_{12}$ phase remaining in the SZ suggests that the processing temperature was extremely low, consistent with the temperature measurement results. The Al$_2$Ca phase demonstrated better thermal stability than the $\beta$-Mg$_{317}$Al$_{12}$ phase, and the dissolution temperature was approximately 545 °C [26]; therefore, the precipitated phases were identified as Mg$_{317}$Al$_{12}$ and Al$_2$Ca in the SZ which similar to those of the BM.

EBSD examinations were conducted to characterize microstructural details of the BM and the SZ; findings are displayed in Figs. 3 and 4, respectively. The mean grain size and high-angle boundary (HAB) fraction of the BM were 8.4 μm and 92.3%, respectively. The misorientation angle distribution was similar as a random distribution of HCP crystallites as indicated by the black solid line in Fig. 3b; this a typical recrystallized grain structure because the as-received material was produced by hot rolling. Accordingly, the BM exhibited a rolling texture, wherein the (0001) plane of most grains was nearly perpendicular to the normal direction (ND). The SZ exhibited an equiaxed and uniform grain structure with a mean grain size of 1.2 μm. As displayed in Fig. 4b, two prominent peaks appeared for misorientation angle distribution. The peak at 2–5° indicated that massive dislocations were introduced into the SZ. Another peak around 86° occurred because HCP crystals contain few slip systems, which causes more frequent (10-12) twinning [28]. Such twinning behavior led the {0001} crystal plane in the twin to rotate about 86° around the <10-11> direction; therefore, the peak at 86° is attribute to (10-12) twinning behavior. The LT-FSW process apparently promotes formation of {10-12} twins.

Grains in the SZ often showed shear textures due to simple shear deformation caused by the rotating probe [29,30]. For conventional FSW reported in prior studies [15,19–21], the <0001> crystal direction of most grains in the SZ center was nearly parallel to WD and exhibited strong basal texture intensity. By contrast, in this study, the maximum texture intensity of the LT-FSW joint became smaller compared to the conventional FSW. The (0001) pole figure displayed two orientation components of <0001>||WD and <0001>||ND. With a decline in deformation temperature, (10-12) <10-11> twinning behavior tended to occur, resulting in the <0001> crystal direction rotating by about 86° back towards the ND. Compared to the BM, a lower-angle boundary (LAB) with a misorientation angle lower than 5° was generated in the SZ based on Kernel average misorientation (KAM) maps; see Fig. 5. Increasing the KAM corresponds to an increase in dislocation density [31–33]. Grains in the SZ showed relatively higher KAM than those in the BM. Given the relatively low temperature in the welding process, the annealing effect after welding was reduced. The
Fig. 5 – Kernel average misorientation map obtained by EBSD measurement of (a) BM and (b) SZ.

Table 1 – Number fraction of LAB, HAB, and TB of TMAZ-1 and TMAZ-2 in Fig. 6.

<table>
<thead>
<tr>
<th>Location</th>
<th>LAB (%)</th>
<th>HAB (%)</th>
<th>TB (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TMAZ-1</td>
<td>14.2</td>
<td>87.3</td>
<td>16.3</td>
</tr>
<tr>
<td>TMAZ-2</td>
<td>22.3</td>
<td>77.7</td>
<td>9.3</td>
</tr>
</tbody>
</table>

Fig. 6 – Grain boundary map of TMAZ on retreating side of FSW AZX911 Mg alloy joint. The low-angle boundary (LAB, 2° ≤ θ < 15°), high-angle boundary (HAB, 15° ≤ θ), and twin boundary (TB, θ = 86°) are indicated by green, black, and red lines, respectively.

Because the SZ of the LT-FSW joint exhibited different microstructural features compared with the BM and conventional FSW, a unique microstructural evolution followed. To understand grain structure transformation from the BM to the SZ, an EBSD examination was conducted on the TMAZ; results are shown in Fig. 6. The TMAZ was divided into two regions [i.e., TMAZ-1 and TMAZ-2] according to morphological differences. Table 1 summarizes the misorientation details of these regions. Grains near the BM in the TMAZ-1 region were significantly elongated. The LAB number fraction was higher than that of the BM because of applied plastic deformation. Conversely, the TMAZ-2 region was near the SZ and thus experienced more intense deformation, resulting in a mixed microstructure of ultrafine grains and elongated grains. Therefore, the number fractions of the HAB and twin boundary (TB) were higher than in the TMAZ-1 region due to the dynamic recrystallization (DRX) progress. The main mechanism of grain refinement is generally attributed to discontinuous DRX (DDRX) and continuous DRX (CDRX) [34]. The typical features of DDRX and CDRX are indicated by arrow 1.
Fig. 7 – Schematics of several DRX mechanisms during the LT-FSW of AZX911 Mg alloy joint. LABs and HABs are indicated by fine lines and thick lines, respectively.

and arrow 2, showing a neckless structure and transformation of LAB to HAB via dislocation recombination, respectively. Another important characteristic not reported previously was detected in this study: as indicated by arrow 3, the initial HAB and TB demonstrated partially serrated morphology. Due to the relatively large axial load during the FSW, fine grains can coalesce via migration of the bulging HAB and TB following grain refinement caused by geometric DRX (GDRX) [35]. Similarly, the three DRX mechanisms, i.e., CDRX, DDRX and GDRX, occurring in hot deformation of various magnesium alloys have also been studied [36–46]. In order to make the grain refinement process easy to understand, the DRX schematic diagrams are illustrated in Fig. 7.

To investigate tensile properties of the SZ, a micro tensile test was conducted with findings illustrated in Fig. 8. Compared to the BM and earlier studies, the SZ showed an enhanced yield strength of 195 MPa. Moreover, a good strain-hardening capacity was achieved, leading to high ultimate tensile strength and uniform elongation of 348 MPa and 24%, respectively. A relatively good combination of strength and ductility in the SZ was generated by LT-FSW technology. Since the mechanical properties of the whole welded joint were significantly improved, the fracture occurs in BM for the tensile sample covering both welded joint and BM.

The strengthening and toughness mechanisms of the SZ can be attributed to the following four factors:

(i) Grain boundary strengthening $\sigma_{HBP}$. For polycrystalline materials with grain sizes ranging from several microns to several hundred microns, the dependence of yield strength on average grain size can be described by the Hall-Petch relationship:

$$\sigma_{HBP} = \sigma_0 + kd^{-1/2}$$  \hspace{1cm} (1)

where $\sigma_{HBP}$ is yield strength, $\sigma_0$ and $k$ are constants, and $d$ is the average grain size. This relationship confirms that yield strength increases as grain size declines. Based on previous studies, the values of $\sigma_0$ and $k$ were 11 MPa and 164 MPa·m$^{1/2}$, respectively [47].

(ii) Orowan strengthening $\sigma_{Orowan}$. In this work, globular Mg$_7$Al$_2$ and Al$_2$Ca each precipitated in the SZ (Fig. 9a);
thus, increased yield strength caused by the strengthening of precipitated particles can be predicted using the following formula [48]:

$$
\sigma_{\text{Orowan}} = \frac{Gb}{2\pi\sqrt{1-\nu} \left( \frac{0.779}{\sqrt{f}} - 0.785 \right)} \ln \frac{0.785d_i}{b} \tag{2}
$$

where \( b \) is the Burgers vector \( (3.21 \times 10^{-10} \text{ m}) \); \( d_i \) and \( f \) are the precipitated particle diameter and volume fraction, respectively; and \( \nu \) \((0.35) \) is the Poisson’s ratio.

(ii) Solid-solution strengthening \( \sigma_s \). Compared to Zn and Ca in the AZX911 Mg alloy, Al is the main alloy element, and its content is relatively high (about 9 wt.\%). Zn and Ca are mainly involved in the formation of intermetallic compounds. In this work, only solid-solution strengthening of the Al element was considered. Given the EDS result in Fig. 10, contents of the Al solute in the \( \alpha- \text{Mg} \) was \( 3.89 \text{ at.\%} \).

The influence of the solid element on the yield strength is given by

$$
\sigma_s = \sigma_U + \frac{3.1\epsilon G C^{1/2}}{700} \tag{3}
$$

where \( \sigma_U \) is the yield strength of pure Mg \((39 \text{ MPa}) \), \( \epsilon \) is a constant of 0.74 MPa \([50]\), \( G \) is the shear modulus of pure Mg \((1.66 \times 10^4 \text{ MPa}) \), and \( C \) is the concentration of the Al element (at.\%).

(iv) Dislocation strengthening \( \sigma_{\text{GRD}} \). Dislocation is related to dislocations caused by severe plastic deformation during welding. Many dispersed particles were found in the SZ as revealed in Fig. 2c. Because of the incompatibility between particles and the matrix, massive dislocations were generated during welding, leading to an enhanced strain-hardening rate during the tensile test. The contribution of dislocation density is predicted as follows \([52]\):

$$
\sigma_{\text{GRD}} = \alpha Gb \left( \frac{f \sqrt{\nu}}{b d t} \right)^{1/2} \tag{4}
$$

where \( \alpha \) is a constant \((1.25) \), and \( \nu \) is the shear strain \((0.013) \). The dislocation density was relatively high (see Fig. 5b), thus enhancing the yield strength. The predicted yield strength value, including the abovementioned strengthening mechanisms in the SZ, is plotted in Fig. 8. The experimental yield strength value was close to the predicted value, indicating that these strengthening mechanisms can accurately predict yield strength in the SZ.

(v) Toughening mechanism. The improved uniform elongation may be attributed to the appearance of \( \{10-12\} \) twins,
as illustrated in Fig. 9b. Coherent TBs can accommodate dislocations efficiently, thereby elevating ductility and strain hardening during the tensile test [53–55].

4. Conclusion

Non-combustible AZX911 Mg alloy sheets measuring 3 mm thick were successfully joined by LT-FSW. The peak temperature declined substantially, allowing for successful removal of HAZ. Ultrafine grains with high dislocation density and {10-12} twins were produced in the SZ. CDRX, DDRX and GDRX contributed to grain refinement in the SZ. The welded joint fractured in the BM during the transverse tensile test due to the highly strengthened SZ caused by grain boundary strengthening, solid-solution strengthening, second-phase strengthening, and dislocation strengthening. Formation of {10-12} twins reduced the basal texture intensity as well as the strong dislocation accommodation belonging to the same slip system, leading to improved strain hardening and ductility.

Conflicts of interest

The authors declare no conflicts of interest.

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References


