Microstructure Evaluation of Friction Stir Welded AZ91 with CaO Mg Alloy

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The present study was carried out to evaluate the microstructural properties of a friction stir welded AZ91 with CaO Mg alloy. For this work, friction stir welding (FSW) was performed at a tool rotation speed of 1250 rpm and a traveling speed of 32 mm·min⁻¹. A network of intermetallic compounds (Al₂Ca) was observed in the base metal (BM). In the stir zone (SZ), fine grains and intermetallic compound particles were formed due to dynamic recrystallization and mechanical stirring. The hardness profile showed that the hardness of the SZ was higher than that of the BM, probably due to the presence of fine grains and thermally stable intermetallic compounds. [doi:10.2320/matertrans.M2010271]

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1. Introduction

In recent years, vehicle weight reduction has been an important issue in the automotive industry, due to the necessity to reduce fuel consumption and global warming.¹⁾ The demand for Mg alloys has increased, due to their low density and good specific mechanical properties. With their increasing application in industry, it is necessary to develop effective welding techniques for the joining of Mg alloys. However, the fusion welding of these materials is difficult, due to the formation of vast porosity.²⁾ Friction stir welding (FSW), a novel solid joining technology, is a potential candidate, because of its many advantages compared with the traditional fusion welding processes. It can produce pore-free joints where the pores caused by casting solidification are eliminated, and fine-grained structures are generated in the joint where dynamic recrystallization occurs.³⁾ In previous studies, the FSW of Mg alloy was mainly focused on die-cast and extruded AZ alloys and AM alloys.⁴⁻¹¹⁾ However, there has been little research into heat resistant Mg alloys,¹²⁾ whose creep resistance is superior due to the existence of intermetallic compounds at the grain boundaries. In the present work, AZ91 with CaO¹³⁾ was friction stir welded and the microstructural properties after FSW were extensively investigated.

2. Experimental Procedure

The butt-welding of AZ91 with CaO (9.1 mass%Al–0.64 mass%Zn–0.01 mass%Ca–0.3 mass% CaO, Mg Bal.) was performed by FSW. Mg alloy plates were machined to a size of 70 mm × 140 mm × 4 mm for welding. The tool rotation speed and travel speed were fixed at 1250 rpm and

32 mm·min⁻¹, respectively. After FSW, the cross-sectional specimens were cut perpendicular to the welding direction, mechanically ground with abrasive paper, and polished with 1 and 0.3 μm alumina powders. Optical microscopy (OM) and electron probe micro analysis (EPMA) observations were carried out for the weld. And X-ray diffraction was used to phase analysis. The Vickers hardness profile of the weld was measured on the cross-section perpendicular to the welding direction.

3. Result and Discussion

Figure 1(a) shows an overview of the cross-section of joint, and the microstructure in the base metal (BM), thermo-mechanically affected zone (TMAZ) and stir zone (SZ) are shown in Figs. 1(b)–(d), respectively. The BM exhibits a typical solidification microstructure formed in the casting process. The grains in the TMAZ were greatly deformed due to the friction heat and mechanical deformation, and the grains have an obvious orientation along the metal-flow direction induced by stirring. The SZ contains fine recrystallized grains instead of dendritic ones, and the intermetallic compound (dark phase in Fig. 1) seems to be uniformly dispersed in the SZ.

Figure 2 shows the result of the EPMA analysis of the BM, TMAZ, and SZ. The microstructure of the BM, which is shown in Fig. 2(a), is mainly composed of α-Mg grains and a network of intermetallic compounds along the grain boundaries. And it shows that the concentrations of Al and Ca in the network structure are much higher than those in the α-Mg grains. The TMAZ (Fig. 2(b)), where both thermal and plastic deformation effects were evident, is composed of Al–Ca intermetallic compounds and recrystallized grains, and the former have an obvious orientation along the metal-flow direction induced by stirring. In the SZ (Fig. 2(c)), the original cast structure of the BM changed to a fine recrystal-

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Fig. 1 Each microstructures in the joint produced by friction stir welding: (a) Macro cross-section of weld nugget, (b) BM, (c) TMAZ, (d) SZ.

lized grain structure, and fine Al-Ca intermetallic compound particles were homogeneously distributed in the α -Mg matrix due to plastic deformation and stirring. It is worth noting that the intermetallic compounds existed in all three zones, i.e., the BM, TMAZ and SZ, although their shape and size differed.

For the correctly phase analysis, XRD analysis was carried out and its result is shown in Fig. 3. In case of BM (Fig. 3(a)), the matrix consisted with primarily of α -Mg as major phase and Al_2Ca as minor phase. Also in the SZ, as shown in Fig. 3(b), SZ has also α -Mg and Al_2Ca phases. Considering these results, it is suggested that the main network compound is Al_2Ca and it is quite stable at this processing temperature.

However, CaO was not observed in these materials; the CaO particles that were initially added were gone and, instead, Al_2Ca intermetallic compounds were observed over the Mg matrix. At the casting temperature of 953 K, the standard free energy change, ΔG_0 of reaction of $\text{Mg(l)} + 2\text{Al(l)} + \text{CaO(s)} = \text{Al}_2\text{Ca(s)} + \text{MgO(s)}$ was -9 kJ , indicating that the forward reaction could occur spontaneously.¹⁴⁾

Matsumoto *et al.* studied the influence of heat treatment on the microstructure of the as-thixomoulded Mg-6 mass%Al-3 mass%Ca material. It was shown that although the partial dissolution of the intermetallic compounds took place, most of the network structure was retained after being heated at 623 K for 1 h.¹²⁾ And F. Islam reported that ($\text{L} \leftrightarrow \text{Al}_2\text{Ca} + \text{Mg}$) reaction temperatures were 773.88 K~815.00 K in Mg-Al-Ca ternary system.¹⁵⁾ Also, it was reported that critical temperature of AZ61 was 593 K during friction stir welding.³⁾ From these result, it suggested that dissolution and reprecipitation of Al_2Ca intermetallic compound could not be the main reasons for the change in the intermetallic compound morphology, because the thermal condition is not sufficient for this mechanism. Rather the size and shape of the intermetallic compounds were changed mainly through mechanical fracture. It has been reported by many researchers that the microstructure of Mg alloys can be refined through FSW, since dynamic recrystallization occurs in the SZ during welding. The size of the Mg grains in the SZ ranges from 2 to 25 μm , depending on the chemical composition and welding parameters.⁶⁻¹¹⁾ In this research, a relatively fine grain structure with an average size of 1~2 μm

was produced by FSW. This microstructure was somewhat similar to that of a metal matrix composite and different from the microstructure produced by the FSW of other Mg alloys whose base metals are composed of dual phases, viz. Mg grains and intermetallic compounds such as $\text{Al}_{12}\text{Mg}_{17}$. After FSW, the microstructures of the SZ, consisting almost entirely of dynamically recrystallized Mg grains and intermetallic compounds, tend to disappear, due to their dissolution at elevated temperature. As regards the present material, both the BM and SZ were composed of two phases, viz. Mg grains and intermetallic compounds such as Al_2Ca . The main reason for the formation of this microstructure is the thermal stability of second phases such as Al_2Ca . Considering the Mg-Al binary phase diagram, the $\text{Mg}_{17}\text{Al}_{12}$ intermetallic compounds in AZ91 completely dissolve into the Mg matrix when the temperature is higher than 643 K. Therefore, the peak temperature of the SZ in this study is high enough for the $\text{Mg}_{17}\text{Al}_{12}$ intermetallic compound in AZ91 to dissolve into the matrix. However, as mentioned above, the Al_2Ca intermetallic compound in this material is very stable at high temperature. Therefore, it seems that these particles hindered the grain growth after dynamic recrystallization due to the Zener pinning effect.¹²⁾

Considering these results, it is suggested that the microstructure of this material during FSW was different from that of the FSW joints of other Mg alloys, due to the existence of this heat stable Al_2Ca intermetallic compound. Figure 4 shows the hardness profiles measured along the mid-thickness line of the cross section. It shows that the hardness of the SZ was higher than that of the BM; the average hardnesses of the SZ and BM were 65 HV and 90 HV, respectively. We believe that the improvement in the hardness of the SZ is related to the grain size and presence of intermetallic compound particles. Firstly, the grains of the SZ were finer than those of the BM and it is well known that fine grains play an important role in strengthening the SZ. Secondly, the small intermetallic compound particles also contributed to increasing the hardness according to the dispersion strengthening mechanism. The hardness profile of the FSWed joint of the same AZ91 without CaO is also illustrated in Fig. 4. In this material, the average hardness of the SZ was slightly higher than that of the BM, but lower than that of the SZ in

Fig. 2 Result of EPMA analysis in each region: (a) BM, (b) TMAZ, (c) SZ.

Fig. 3 XRD pattern of each region: (a) BM, (b) SZ.

Fig. 4 Vickers hardness distributions of the welds of AZ91 and AZ91 with CaO.

AZ91 with CaO. We assumed that although the intermetallic compound ($\text{Al}_{12}\text{Mg}_{17}$) dissolved due to friction heating, the fine grains contributed to increasing the hardness in the SZ. However, since the intermetallic compound (Al_2Ca) in AZ91 with CaO is stable at high temperature and dispersed uniformly in the SZ, these dispersed small particles contributed to increasing the hardness in the SZ through the dispersion-strengthening mechanism.

4. Conclusion

The microstructure of AZ91 with CaO which was welded by friction stir welding, was investigated. Compared with the BM, very fine grains were present in the SZ, due to dynamic recrystallization. The network of intermetallic compounds (Al_2Ca) in the BM was changed to very small particles due

to mechanical stirring. This increase in the hardness of the SZ was attributed to this stable intermetallic compound.

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