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Microstructural evolution and its effect on Hall-Petch relationship in friction stir welding of thixomolded Mg alloy AZ91D

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Microstructural evolution of a thixomolded magnesium (Mg) alloy AZ91D during friction stir welding was investigated. Friction stir welding resulted in a homogeneous microstructure consisting of fine recrystallised α-Mg grains in the thixomolded material. The microstructural homogenisation and refinement was attributed to dynamic recrystallisation accompanied by the dissolution of the eutectic structure during the welding. The grain refinement in the stir zone was effective in increasing the hardness, as predicted by the Hall-Petch equation. The effect of grain size on hardness was smaller than that in conventional and rapidly solidified AZ91. This phenomenon may be explained as being due to the microstructure of the stir zone which consisted of fine equiaxed grains with a high density of dislocations.

1. Introduction

Recently, Mg alloys have attracted increasing interest in transportation industries due to their low density and high specific strength. Active research has been directed to practical use of Mg structures, which are generally built using conventional fusion welding processes. However, fusion welding of Mg alloys often results in defects, such as porosities and hot cracks, which decrease the mechanical properties [1, 2]. The achievement of defect-free welds necessitates complete elimination of the surface oxide layer prior to welding and the selection of suitable welding parameters [3].

Friction stir welding (FSW) is a novel solid-state joining process that is more cost-efficient than fusion welding due to elimination of some defects, filler materials, shielding gases and weld preparation. FSW potentially offers considerable improvements in microstructure and mechanical properties of the welded joint of aluminium (Al) structures and has already been used in the production of Al structures [4–17]. On the other hand, FSW has not yet been applied to Mg alloys in practical use. Some previous studies [18–24] have shown that FSW can provide relatively good weldability of Mg alloys as well as Al alloys and have examined the mechanical properties and microstructures in friction stir welds of wrought and cast Mg alloys. Although these studies have shown that FSW results in relatively higher mechanical properties than fusion welding, microstructural evolution of Mg alloys during FSW is not fully understood. In the present study, the microstructural evolution of a thixomolded Mg alloy AZ91D during FSW and the effect of grain size on hardness in the stir zone of the weld were examined.

2. Experimental procedures

The material used in this study was a thixomolded Mg alloy AZ91D, nominally 2 mm thick. Two plates were friction stir welded using various welding parameters. The weld produced at a rotation speed of N rpm and a traveling speed of R mm/s is termed "*the $N \times R$-weld*" throughout the present paper. Following the FSW, the Vickers hardness profile of the weld was measured on the cross section perpendicular to the welding direction. The microstructure in the weld was observed by optical microscopy (OM). The surface for OM was etched with an acetic-glycol (60 mL ethylene glycol + 20 mL acetic acid + 20 mL water + 1 mL nitric acid) solution. Details of the fine microstructure in the stir zones were observed by transmission electron microscopy (TEM). Thin disks for TEM, 3 mm in diameter, were electrolytically polished in a 400 mL methanol + 25 mL butoxyethanol + 6 mL perchloric acid solution. TEM observations were carried out using a JEOL 2000EXII at 200 kV.

3. Results and discussion

3.1. Microstructural evolution during FSW

A low-magnification overview of *the 1220 × 1.5-weld* is shown in Fig. 1. Retreating and advancing sides of the weld correspond to the left- and right-hand sides of the weld centre, respectively. The weld has a typical nugget-shaped stir zone with an onion ring that has been reported in friction stir welds of some Al alloys [4–6].

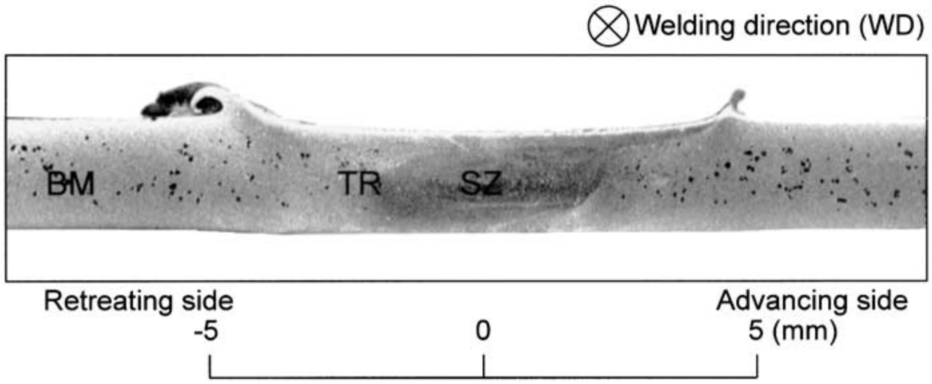

Figure 1 Transverse cross section of friction stir welded Mg alloy AZ91D.

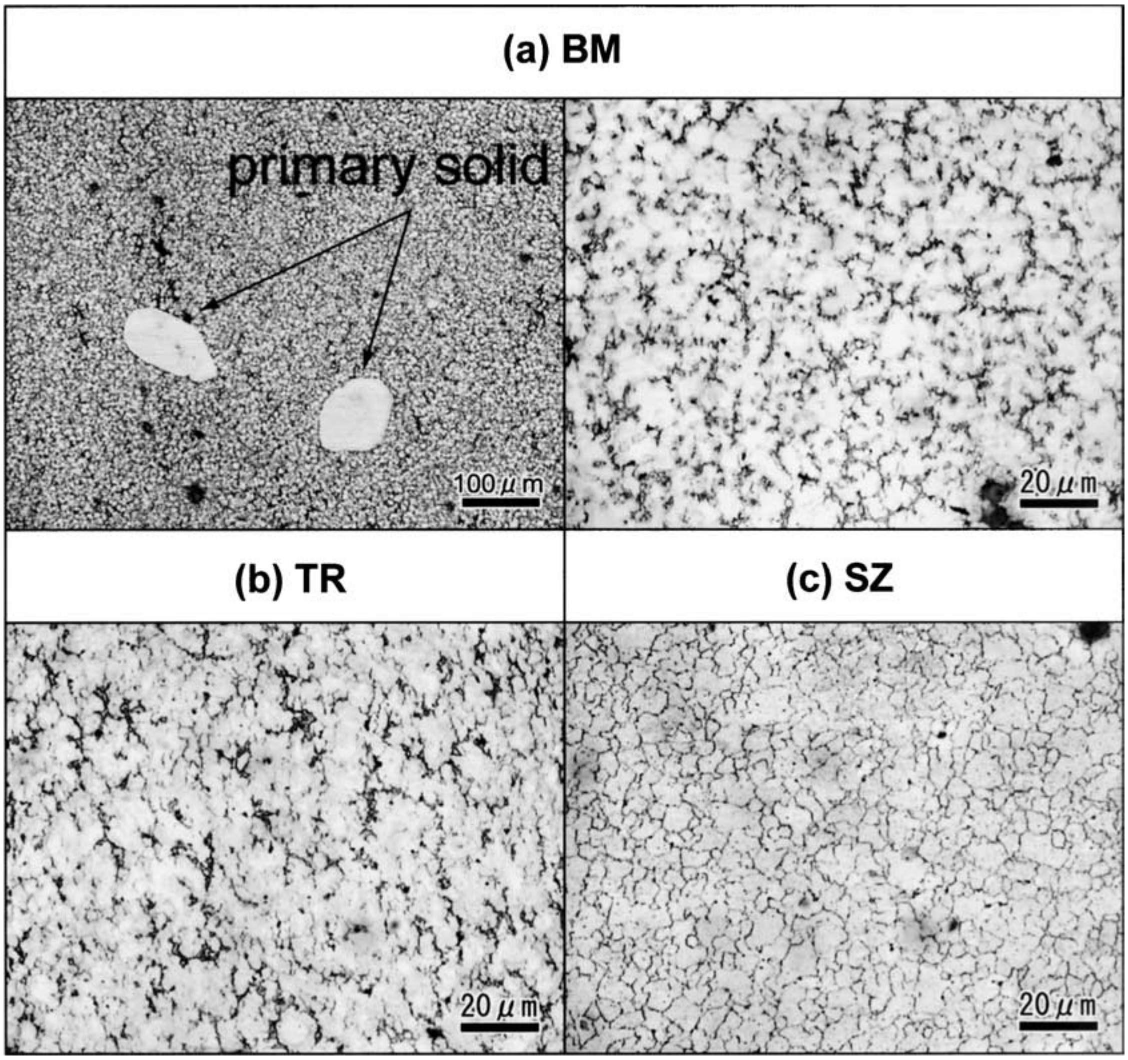

Figure 2 Optical micrographs at locations termed "BM," "TR," and "SZ," shown in Fig. 1.

No defects, such as cracks and porosity, were observed in the stir zone.

Optical micrographs of the welds are shown in Fig. 2. The base material region, transition region and stir zone correspond to the locations shown as "BM," "TR" and "SZ" in Fig. 1. The transition region is located just outside the stir zone. The base material region has both a cast microstructure and spherical coarse particles 100 μm in diametre. The spherical coarse particles are α-Mg solid phase existing in liquid phase during thixomolding, and they are expressed as primary solids in this figure [25–27]. The right-hand micrograph of the base material region indicates that the cast microstructure consists of an α-Mg phase and eutectic microstructure. The eutectic microstructure has been shown to consist of an α-Mg phase and $Al_{12}Mg_{17}$ phase in the thixomolded Mg alloy AZ91D [25]. The stir zone has equiaxed grains without either a eutectic structure or primary solid particles. The transition region exhibits a microstructure similar to that of the base material,

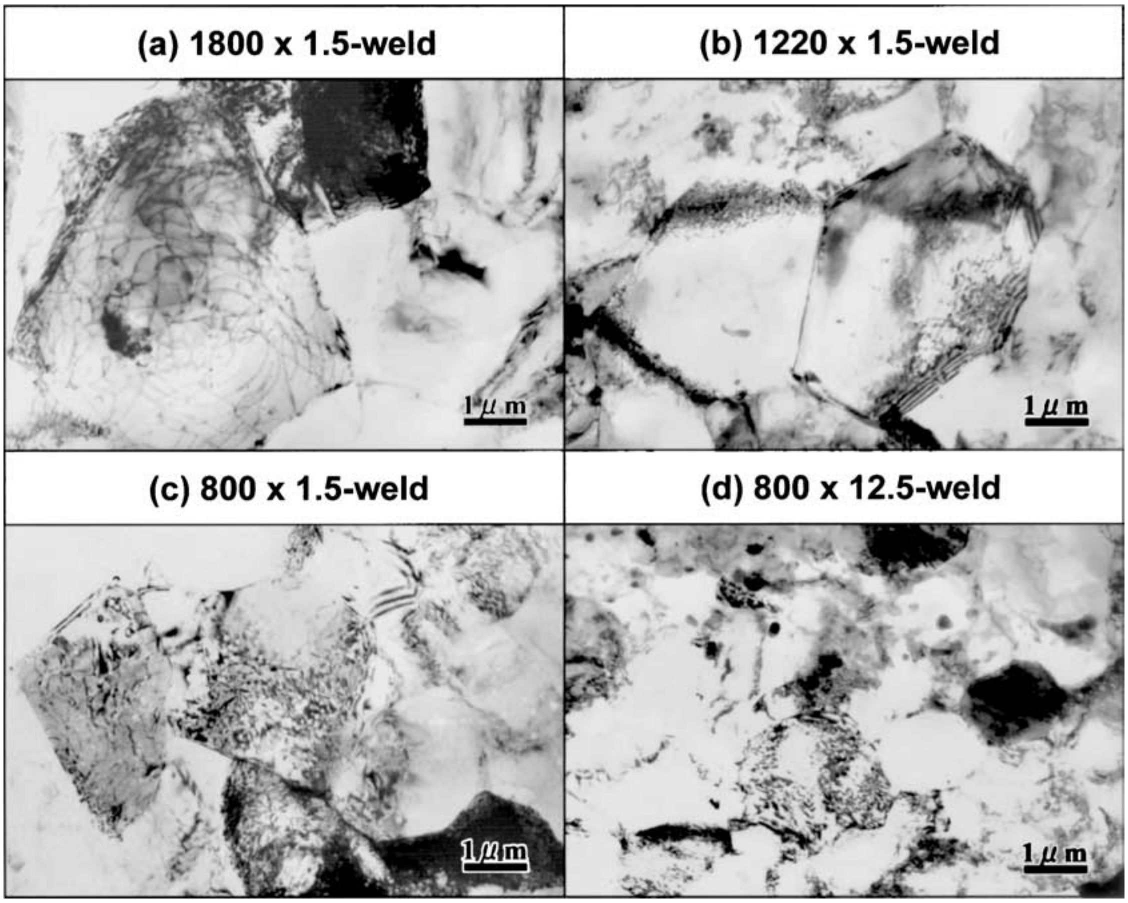

Figure 3 TEM images of the stir zone of (a) 1800 × 1.5-, (b) 1220 × 1.5-, (c) 800 × 1.5-, and (d) 800 × 12.5-welds.

although it contains a lower density of the eutectic structure than the base material.

TEM images of the stir zones of *the 1800 × 1.5-, 1220 × 1.5-, 800 × 1.5-* and *800 × 12.5-welds* are shown in Fig. 3. The TEM images confirm that all the stir zones consist of only equiaxed α-Mg phase with a high density of dislocations. The average grain sizes were about 5.36 μm, 2.48 μm, 1.82 μm and 0.93 μm in *the 1800 × 1.5-, 1220 × 1.5-, 800 × 1.5-* and *800 × 12.5-welds*, respectively. The temperature during FSW generally increases with increasing rotation speed or decreasing traveling speed. Some previous studies [14, 28, 29] have suggested that dynamically recrystallised grains are nucleated during the stirring of FSW and then grow statically during the cooling portion of the thermal cycle. Therefore, the stir zone should show a smaller grain size when the temperature rise during FSW is lower. This trend can explain the relationship between grain size and welding parameters in the stir zone of the present welds.

OM and TEM observations showed that in FSW, the cast microstructure of the thixomolded material was replaced with a fine homogeneous α-Mg grain structure, i.e., neither a eutectic structure nor primary solid particles were observed in the stir zone. Assuming that Mg alloy AZ91D is roughly regarded as Mg-9 wt% Al binary alloy, the eutectic structure would be completely dissolved into the α-Mg matrix at temperatures higher than about 643 K according to the Al-Mg binary phase diagram [30]. Additionally, the α-Mg matrix would also be dissolved when the temperature of FSW exceeds the solidus temperature of this alloy (about 773 K), resulting in an as-cast microstructure in this material during the cooling cycle. As shown in Figs 2 and 3, an as-cast microstructure is not observed in any of the stir zones, so that the stir zones are heated at temperatures between 643 K and 773 K during FSW. Exposure to this temperature range results in a supersaturated solid solution of the α-Mg phase in the stir zone. Besides frictional heating, plastic strain is also simultaneously introduced into the material during FSW, i.e., the stir zone experiences intense plastic deformation by the rotating tool. Both frictional heating and plastic strain lead to the formation of dynamically recrystallised grains in stir zones of the friction stir welded Al alloys [7, 14, 28, 29]. Mg alloys may experience dynamic recrystallisation more easily than Al alloys, because the recrystallisation temperature of Mg alloys is about 523 K, which is lower than that of most Al alloys [31]. Since the peak temperature of the stir zone is higher than the recrystallisation temperature, dynamic recrystallisation should occur in the stir zone during FSW stirring, accompanying the dissolution of the eutectic structure. This is a reason why the stir zone consists of the fine homogeneous α-Mg grains without a eutectic structure.

The transition region had a eutectic microstructure similar to that of the base material region, although the volume fraction of eutectic in the transition region was less than that in the base material. During FSW, the transition region was heated to a temperature lower than that of the stir zone, so that it would undergo only partial dissolution of the eutectic microstructure.

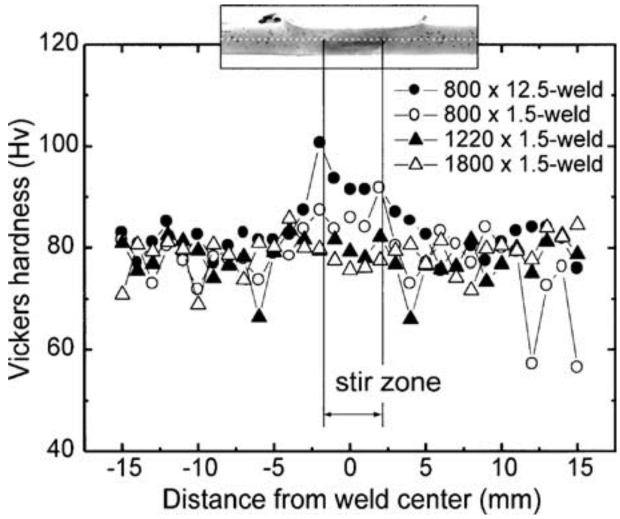

Figure 4 Hardness distributions of friction stir welded Mg alloy AZ91D.

3.2. Relationship between grain size and hardness in the stir zone

Vickers hardness measured along the dotted line of the cross section is shown in Fig. 4. The hardness of the base material ranged from 60 Hv to 85 Hv. *The 1220 × 1.5-* and *1800 × 1.5-welds* had relatively uniform hardness profiles. When the welding parameters with the lower rotation speed or the higher welding speed were used, the stir zone had a hardness value higher than that of the base material region. Hardness values of the weld centre were 76 Hv, 79 Hv, 86 Hv and 91 Hv in *the 1800 × 1.5-, 1200 × 1.5-, 800 × 1.5-* and *800 × 12.5-welds*, respectively.

The stir zones had homogeneous equiaxed grain structures, and the hardness values increased with decreasing temperature. The lower temperature resulted in smaller grain size, so that the hardness values were mainly related to the grain size in the stir zone. Fig. 5 shows the relationship between the hardness (Hv) and the reciprocal of the square root of grain size (d) for the stir zone. The Hv is proportional to the reciprocal of the square root of d. The Hall-Petch equation for the stir zone of the present weld is expressed as

$$\mathrm{Hv} = 64 + 27d^{1/2} \quad (\mathrm{kgf/mm^2}) \tag{1}$$

Nussbaum *et al.* [32] have reported the Hall-Petch equation for yield stress of rapid solidified Mg alloy AZ91 containing few dislocations as

$$\sigma_y = 130 + 210d^{1/2} \quad (\mathrm{MPa}) \tag{2}$$

Assuming that Hv is proportional to yield strength σ_y through the expression $\mathrm{Hv} = 3\sigma_y$ [33], the Hall-Petch Equation 2 can be converted to the following equation:

$$\mathrm{Hv} = 40 + 64d^{1/2} \quad (\mathrm{kgf/mm^2}) \tag{3}$$

In the conversion from Equation 2 to Equation 3, unit of strength, MPa, was converted to unit of hardness, $\mathrm{kgf/mm^2}$, using the expression 1 $\mathrm{kgf/mm^2} \approx 9.8$ MPa. Hall-Petch Equation 3 is also shown by a dotted line in Fig. 5. This figure shows that the effect of grain size on hardness is smaller in Hall-Petch Equation 1 than in Equation 3. This phenomenon may be explained as being due to the microstructure of the stir zone of Mg alloys. In the Hall-Petch relationship only high-angle grain boundaries are considered as obstacles to the dislocation movement. If a high density of dislocations, second phase particles and precipitates exist in the material, they would affect the Hall-Petch equation because these microstructural factors also work to obstruct the dislocation movement. Especially when the microstructural factors pin the mobile dislocations at smaller intervals than the grain size, they may weaken the effect of grain size on hardness. Some previous studies on FSW of Mg alloys [18, 22] showed that the stir zone had a recrystallised grain structure with a high density of dislocations. The TEM images shown in Fig. 3 indicate that the present stir zones also have fine equiaxed grains with a high density of dislocations. A high density of dislocations pins the mobile dislocations at smaller intervals than the grain size, which may have led to the lower slope of the Hall-Petch equation for the stir zone in the friction stir weld of AZ91D than in the study by Nussbaum *et al.* [32].

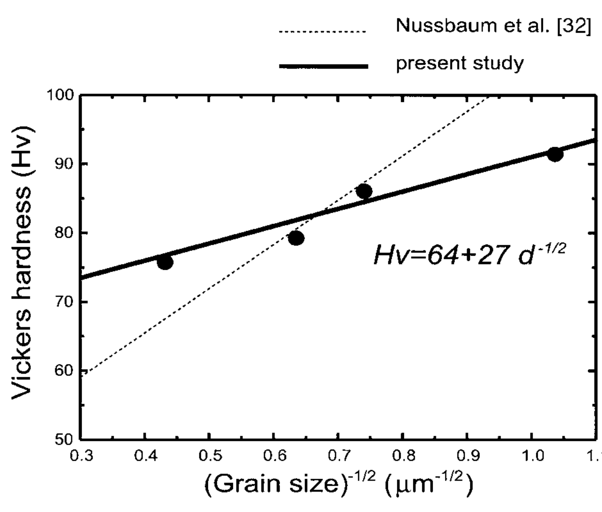

Figure 5 Hall-Petch relationship in the stir zone of thixomolded Mg alloy AZ91D with various welding parameters.

4. Summary

Microstructural evolution of a thixomolded Mg alloy AZ91D during FSW was investigated. The initial as-cast microstructure with primary solid particles changed into a homogeneous microstructure consisting of fine equiaxed grains of the α-Mg phase during FSW. The change in microstructure was explained as being due to the dissolution of the eutectic structure and dynamic recrystallisation during FSW. The hardness in the stir zone increased with decreasing grain size, in agreement with the Hall-Petch equation.

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