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Novel production for highly formable Mg alloy plate

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The principle and advantages of multi-pass friction stir processing (FSP) for the production of a highly formable Mg alloy, and some convincing experimental results are reported in this paper. FSP is a solid state processing technique which involves plunging and traversing a cylindrical rotating FSP tool through the material. FSP achieved grain refinement and homogenization of the as-cast microstructure in Mg alloy AZ91D. Multi-pass FSP produced a fine homogeneous microstructure having a grain size of 2.7 μm throughout the plate. The plate containing this FSPed microstructure exhibited fracture limit major strains six times larger than the diecast plate in the fracture limit diagram (FLD). The present study shows that multi-pass FSP is an efficient production method for a large-scale plate of a highly formable Mg alloy.

1. Introduction

Magnesium (Mg) and its alloys have a hexagonal close-packed (h.c.p.) crystallographic structure. At room temperature, only the basal slip system is active in h.c.p. structure of Mg, because the critical resolved shear stresses (CRSS) of the basal slip system is about one hundredth of those of non-basal slip systems on prismatic and pyramidal planes [1]. Since deformation of the polycrystal requires the activation of at least five slip systems, lack of active slip systems causes poor formability of Mg alloys at room temperature.

In 1960's, Chapman and Wilson [2] reported that the improvement of ductility could be achieved by refining its grain structure in Mg. Recently, some papers have also shown that high ductility is obtained in Mg alloys with grain size less than 20 μm [3–7]. Koike *et al.* [5, 8] demonstrated the reason for the improvement of ductility by the grain refinement in Mg alloys by transmission electron microscopy (TEM) for the tensile-deformed AZ31B Mg alloy. They concluded that the improvement of ductility was due to activity of non-basal slip systems and grain boundary sliding induced by plastic compatibility stress associated with grain boundaries, and dynamic recovery [5, 8]. These papers [2–8] suggest that grain refinement can raise formability in Mg alloys, but its formability, which is usually evaluated by forming limit diagram (FLD), has not been systematically examined yet.

ECA pressing [3–5], extrusion [6] and powder metallurgy [9] are widely used methods to produce fine grains in Mg alloys. However, it is extremely difficult to make large-scale samples of Mg alloy, consisting of fine grained microstructure, for structural applications with these methods. On the other hand, multi-pass friction stir processing (FSP) could produce a large-scale plate of Mg alloy covered by fine grained microstructure. The principle and advantages of multi-pass FSP for the production of the highly formable Mg alloy, and some convincing experimental results are reported in the present paper.

2. Multi-pass FSP as the production method of the highly formable Mg plate

FSP, a development based on friction stir welding (FSW), is a solid state processing technique used for microstructural modification, and involves plunging and traversing a cylindrical rotating tool through a material to produce intense plastic deformation [10]. This process is often utilized to refine the grain structure [10, 11] or to homogenize the heterogeneous microstructure [12]. A schematic illustration of multi-pass FSP is shown in Fig. 1. Besides the relative motion of the rotating tool to the workpiece (b), shifting the plunge position of the FSP tool transverse to the processing direction are also required in multi-pass FSP (d and f).

FSP and FSW can create fine recrystallized grains in the stir zone of Al, Mg alloys and steels by frictional heating and plastic deformation arising from the rotating tool. Grain sizes of the most recrystallized grains lie between 1 and 20 μm in the stir zone of Al and Mg alloys [13–19]. These values would be fine enough to provide high formability in Mg alloys.

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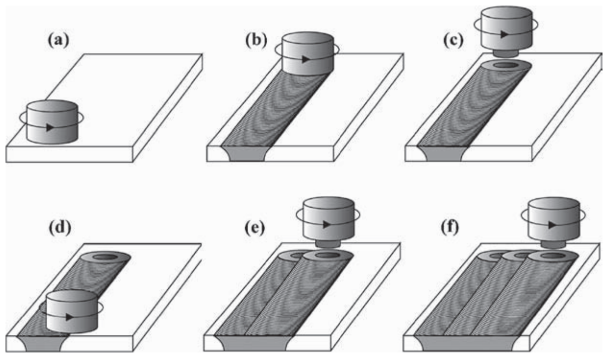

Figure 1 Schematic illustration of multi-pass friction stir processing (FSP).

Multi-pass FSP has superior advantages compared to the other processing methods for the grain refinement. The other methods, such as ECA pressing, extrusion and powder metallurgy, involve an immense amount of time and effort to make a small piece with fine grains. On the other hand, multi-pass FSP can produce fine grained microstructure in an entire plate of a Mg alloy by shifting the plunge position of the rotating tool and its relative motion to the workpiece, so that it would not be restricted by the shape and size of the plate. Additionally, the other processing methods are usually carried out under elevated temperatures [3–6, 8, 9]. Since Mg easily oxidizes and is sometimes flammable, the processing atmosphere and parameters must be strictly controlled during the other methods. FSP locally introduces the heat and plastic deformation arising from the rotating tool, so that control of the atmosphere is not required. It should be also noted that FSP can homogenize the heterogeneous microstructure consisting of α-Mg solid solution and intermetallic compound $Mg_{17}Al_{12}$, which adversely affects the room-temperature formability of the materials, in as-cast AZ91D Mg alloy by dissolution of the $Mg_{17}Al_{12}$ phase during the heating cycle [7, 20]. This suggests that FSP could completely omit any onerous heating processes, such as homogenization of the as-cast microstructure, before processing even when the base material has the as-cast heterogeneous microstructure. Moreover, ECA pressing and extrusion result in strong textures associated with the deformation nature in Mg alloys [4, 6]. Since mechanical properties of Mg alloys are significantly affected by texture [6, 18], presence of strong texture leads to strong anisotropy in Mg alloys, which is not often acceptable in the practical use. Friction stirring also produces a texture associated with the material flow [21–23], but in-plane anisotropy would be alleviated by texture control during FSP. The details of the texture control will be mentioned in the experimental session. Furthermore, if FSW is used to join the FSPed plates at the same parameters as the FSP, a semi-infinite large plate having the FSPed microstructure could be produced.

According to the above-mentioned background, multi-pass FSP would be a strong candidate for an efficiently method to make large-scale plate of highly formable Mg alloy. To confirm these advantages, the present study applied multi-pass FSP to a diecast AZ91D Mg alloy, and verified improvement of its formability.

3. Experimental procedures

The present study used plates of a diecast AZ91D Mg alloy (9 wt% Al and 1 wt% Zn), 2 mm in thickness. Multi-pass FSP was applied to this alloy using a general FSP tool. Dimension of the tool was determined on the basis of the following texture control during FSP. Some studies [21–23] suggest that the material flow during friction stirring arises from shear deformation along the pin column surface toward the rotating direction. In AZ61 Mg alloy, Park *et al.* [23] showed that material flow during FSW mainly activated the basal plane slip systems, which produced the strong basal plane textures along the pin column surface in the stir zone. On the other hand, Jin *et al.* [24] reported that shear deformation mainly occurred parallel to the surface of the tool shoulder during FSW of 2 mm-thick 5000-series Al alloys when the tool shoulder diameter was much larger than the plate thickness (They used a 15 mm shoulder diameter for 2 mm-thick plate). According to these results, the basal planes in most of the grains would be parallel to the shoulder surface if a much larger shoulder diameter is used. This suggests that in-plate anisotropy would be alleviated by texture control using a FSP tool with a large shoulder diameter. Considering these reports, the present study used a FSP tool having a 15 mm shoulder diameter, a 6 mm pin diameter and a 1.9 mm pin length.

During FSP, the tool-to-workpiece angle was 3 deg from the vertical axis in the FSPed alloy. The plunged position of the rotating tool was shifted 6 mm to advancing side of the FSP tool between FSPs, as shown in Fig. 1. In this study, multi-pass FSP was applied to this alloy at two FSP parameters: a travel speed of 12.0 mm/s and a rotational speed of 800 rpm, and a travel speed of 1.5 mm/s and a rotational speed of 1200 rpm. Heat input of friction stirring increases with increasing rotational speed and decreasing travel speed [25, 26]. Therefore, the FSPed alloys produced at these parameters ("a travel speed of 12.0 mm/s and a rotational speed of 800 rpm," and "a travel speed of 1.5 mm/s and a rotational speed of 1200 rpm") are expressed as "*the cold FSPed alloy*" and "*the hot FSPed alloy*" throughout this paper, respectively [27].

Microstructure in the FSPed alloy was etched in a 5 ml acetic acid + 5 g picric acid + 10 ml water + 100 ml methanol solution and then observed by optical microscopy. Grain size in the FSPed alloy was measured by mean linear intercept method for optical micrographs.

The formability was evaluated using a FLD. FLDs show the critical combination of ε_{major} and ε_{minor} in the sheet surface at the onset of necking. Higher ε_{major} in FLD means that the material has higher formability. To obtain an adequate FLD, uniaxial tensile tests, plane strain tensile tests, and hydraulic bulge tests were applied to the multi-pass FSPed alloys. Configurations of the test specimens are illustrated in Fig. 2. Uniaxial tensile test specimens were cut parallel and perpendicular to the FSP direction, while plane strain tensile test

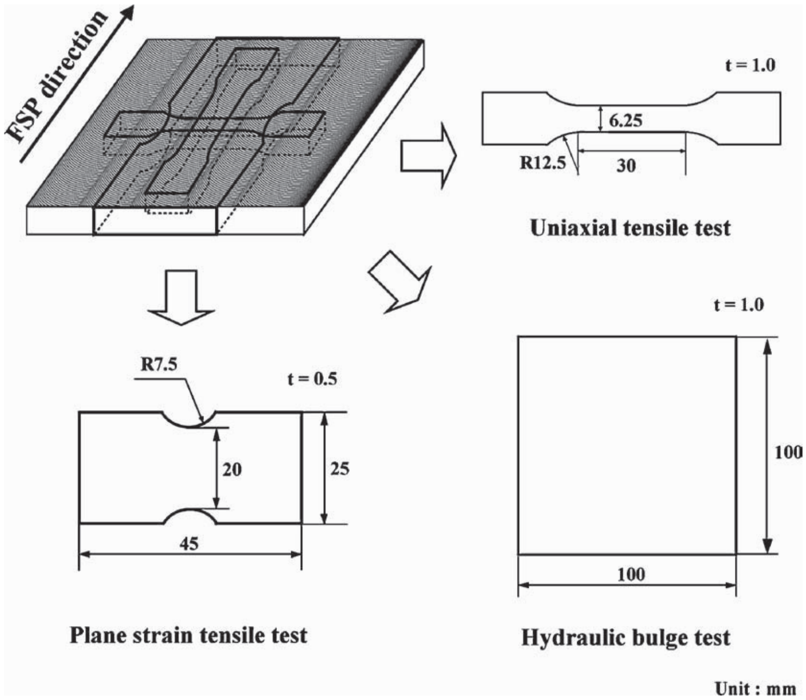

Figure 2 Configurations of the formability test specimens used in this study.

specimens were cut parallel to the FSP direction. In order to only examine the effect of microstructure on the strain in the FSPed alloy, the inequalities of the upper and lower surfaces were eliminated from the specimens. After the elimination of both the surfaces, many circles, 1.5 mm in diameter, were printed on the surface of the test samples. In the present study, we measured the $\varepsilon_{\mathrm{major}}$ and $\varepsilon_{\mathrm{minor}}$ using the following equation at the region closest to the fracture location after each test.

$$\varepsilon_{\mathrm{major}} = \ln(l_1/l_0)$$
$$\varepsilon_{\mathrm{minor}} = \ln(l_2/l_0)$$

where l_0 is circle diameter before test, and l_1 and l_2 are major and minor diameters of the ellipse closest to the fracture location. Tensile tests were carried out at room temperature at a cross-head speed of 1.67×10^{-2} mm/s.

4. Results and discussion

A cross section perpendicular to the FSP direction of the cold multi-pass FSP alloy is shown in Fig. 3a. Multi-pass FSP gives the FSPed microstructure to almost all of plate. The base material contained a lot of porosity which is usually inevitable in diecast alloys, while the FSPed zone does not have any porosity or defects. Each FSPed zone widens near the upper surface, and widths on the upper and lower surfaces of each FSPed zone were roughly equivalent to the diameter of the shoulder and pin, respectively.

Optical microstructures of the cold FSPed zone and the base material are shown in Fig. 3c. In Fig. 3c, regions "1," "1 + 2," and "1 + 2 + 3" correspond to locations "1," "1 + 2" and "1 + 2 + 3" shown in Fig. 3b, and they are affected by "only the first pass," "first and second passes," and "first, second and third passes" of FSP, respectively. The base material has an as-cast microstructure containing the α-Mg phase and an eutectic structure. The eutectic structure consists of the α-Mg phase and the intermetallic compound $Mg_{17}Al_{12}$. Grain boundaries were hardly detected in the base material region. On the other hand, all regions in the cold FSPed zone have fine homogeneous microstructures without any intermetallic compounds. The hot FSPed zone also had a fine homogeneous microstructure consisting of only the α-Mg phases as did the cold FSPed zone. Grain size distributions in the cold and hot multi-pass FSPed alloys are shown in Fig. 4. Data for the grain size were obtained in the regions shown by black points in Fig. 3a. The grain sizes lie between 2.4 and 2.8 μm in the cold FSPed alloy, while they range between 6.9 and 7.3 μm in the hot FSPed alloy. Average grain sizes were 2.7 and 7.0 μm in the cold and hot FSPed alloys, respectively. The difference in grain size between the cold and hot FSPed alloys can be explained by the heat input during FSP, because the cold FSPed alloy experienced a lower heat input than the hot FSPed alloy during FSP. It should be noted that the grain size profiles are roughly constant throughout the multi-pass FSPed alloys. This result suggests that the multi-pass effect on the grain size is negligible.

Formation of a homogeneous microstructure during FSP may be explained by dissolution of the $Mg_{17}Al_{12}$ phase during the heating cycle of FSP, because a phase diagram of an Al-Mg binary system [28] indicates that the $Mg_{17}Al_{12}$ phases completely dissolve in a Mg-9wt%Al alloy when the alloy is exposed to temperatures higher than about 643 K. Only the α-Mg phase is stable at temperature between 643 and 773 K in the phase diagram. Therefore, it would be deduced that the FSPed zones would be heated to this temperature range during stirring. This alloy simultaneously receives intense plastic strains by the rotating

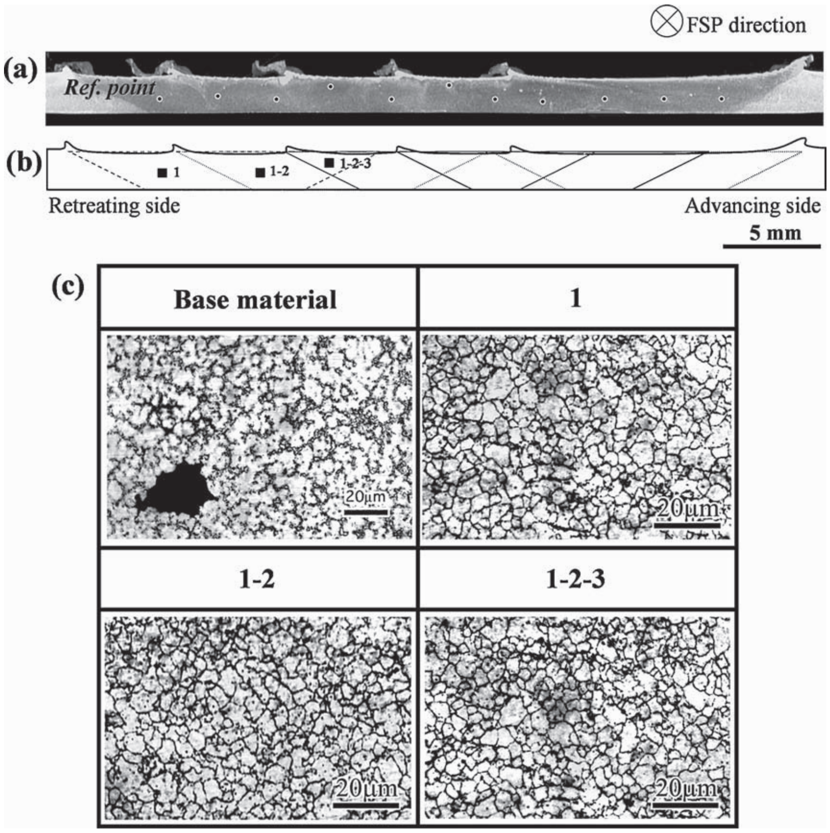

Figure 3 Cross section (a) and its schematic illustration (b) perpendicular to the processing direction of the multi-pass cold FSPed alloy. Optical microstructures of various regions shown in Fig. 3(b) on the multi-pass cold FSPed alloy are indicated in Fig. 3(c).

Figure 4 Grain size profiles in the multi-pass FSPed alloys.

tool during stirring. Mg alloys have the lower recrystallization temperature (about 523 K) than Al alloys [29], so that the dynamically recrystallized grains would be easily formed during FSP [18, 20]. This is a reason for the formation of fine grain structure in the FSPed zone.

There is a possibility that the FSPed microstructure is affected by the following passes of FSP, because it is exposed to temperatures high enough for grain growth of Mg alloy [4] during the following passes. However, distribution of grain size (Fig. 4) showed a negligible multi-pass effect on the grain size, which can be explained as follows. Some studies [30–32] suggest that the dynamically recrystallized grains produced by friction stirring drastically grow during post-processed heat treatment at high temperatures but they are stable during an exposure to temperatures lower than the peak temperature of friction stirring [32]. The peak temperature during friction stirring should be at a maximum in the vicinity of the rotating tool and decreases with increasing distance from the center of the friction stirring, because friction stirring produces frictional heat at the interface between the pin surface and the processed material. This situation suggests that the previous FSPed microstructure is unlikely to be exposed to temperatures higher than the peak temperature of the previous FSP during the following FSP in multi-pass FSP including the transverse shifts of the tool between FSPs. This is a reason why the multi-pass hardly affects the previous FSPed microstructure.

FLDs obtained from the diecast base material and the two FSPed alloys are presented in Fig. 5. This figure does not include data from the hydraulic bulge test for the base material, because the size of the base material plate was insufficient for the hydraulic bulge test. The FSPed alloys have larger fracture limit $\varepsilon_{\mathrm{major}}$ than the diecast base material. Especially, the cold FSPed alloy having grain size of about 2.7 μm shows fracture limit $\varepsilon_{\mathrm{major}}$ six times larger than the diecast base material under uniaxial and plane strain tensile tests. Since tensile data were roughly constant in both the FSPed alloys cut parallel and perpendicular to the FSP direction, in-plane anisotropy would be negligible in the multi-pass FSPed alloys. It is noted that the fracture limit $\varepsilon_{\mathrm{major}}$ increases with decreasing grain size in the

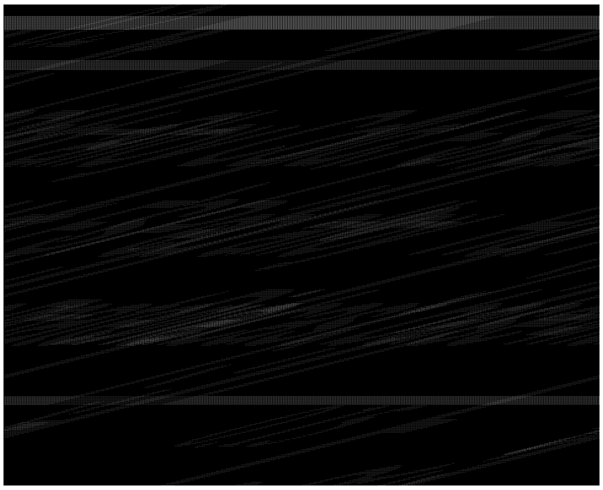

Figure 5 FLD obtained from the diecast alloy and multi-pass FSPed alloys.

FSPed alloy. Koike *et al.* [8] indicated that cross slipping from basal to non-basal planes occurred in grains with a grain size of 7 μm in AZ31B Mg alloy, while it was observed only near the grain boundaries in the grains with grain size of 50 μm. Additionally, it was also found that grain boundary sliding and dynamic recovery also occurred during room-temperature tensile deformation of AZ31B Mg alloy [8]. They concluded from these results that the improvement of ductility by grain refinement was attributed to activity of non-basal slip systems and grain boundary gliding induced by plastic compatibility stresses associated with grain boundaries, and dynamic recovery. In the present study, both the cold and hot FSPed alloys have an average grain size smaller than 7 μm, which is sufficient for the cross slipping in Mg alloys. Therefore, the grain boundary sliding would increase the fracture limit $\varepsilon_{\text{major}}$ with decreasing grain size in the FSPed alloys. Ductility significantly increases with decreasing grain size in Mg having grain size smaller than 5 μm [2], so that there would be a high possibility that further grain refinement would lead to a much higher fracture limit $\varepsilon_{\text{major}}$ in Mg alloys. The present study confirmed that multi-pass FSP was a strong candidate to produce the large-scale plate of highly formable Mg alloys.

5. Summary

The large-scale plate of the AZ91D Mg alloy with high formability was successfully produced via multi-pass FSP. FSP resulted in grain refinement and homogenization of the as-cast microstructure in diecast AZ91D Mg alloy simultaneously. The FSPed plate having grain size of 2.7 μm exhibited fracture limit major strains six times larger than the diecast plate. The present study shows that multi-pass FSP can be used to produce the large-scale plate of highly formable Mg alloy.

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