Production of Ultra-Fine Grained Aluminum Alloy using Friction Stir Process*

Yong-Jai Kwon, Ichinori Shigematsu and Naobumi Saito

Institute for Structural and Engineering Materials, National Institute of Advanced Industrial Science and Technology (AIST), Nagoya 463-8560, Japan

1050 aluminum alloy with an ultra-fine grain size was produced through friction stir process (FSP). The influence of tool rotation speed on the temperature profile, microstructure and mechanical properties of the friction stir processed zone (FZ) was also experimentally investigated. FSP was carried out with only a single pass at tool rotation speeds ranging from 560 to 1840 min\(^{-1}\) under a constant tool traverse speed of 155 mm-min\(^{-1}\). The maximum temperature of the FZ was lower than the melting point of the workpiece material, although increasing linearly with the tool rotation speed. The cooling rate of the FZ also increased linearly from 341 to 1473 °C-min\(^{-1}\) with the tool rotation speed. The FZ had very low dislocation density and was composed of fine equiaxed grains. These fine grains would result from the growth inhibition of 155 mm\(^{-1}\) friction stir process, grain refinement, ultra-fine grained material, 1050 aluminum alloy, severe plastic deformation, dynamic recrystallization, Vickers microhardness test, tensile test

1. Introduction

Recently, in many industrial fields, much attention has been focused on ultra-fine grained (UFG) materials with nanocrystalline and/or submicrocrystalline structures. These materials exhibit a wide variety of unique properties that result from a large volume fraction of their grain and/or interphase boundaries.\(^1,2\) For example, as well described by the Hall-Petch relationship,\(^3\) grain refinement is very effective for the improvement of mechanical properties, such as hardness and strength. In addition, UFG materials exhibit the superplastic behavior available for forming components with complex shapes. This superplasticity generally occurs in polycrystalline materials with grain sizes less than 10 μm at above 0.5 Tm (Tm means the melting point of a material) and relatively low strain rate.\(^4\) However, smaller grains lead to the superplasticity at lower temperatures and/or higher strain rates.\(^5\)

These UFG materials have been produced by an expanding variety of processing techniques, such as mechanical alloying (MA),\(^6\) crystallization of amorphous precursors\(^7\) and severe plastic deformation (SPD).\(^8-12\) Especially, there has been increasing interest in the SPD processes, such as equal channel angular pressing (ECAP),\(^8-10\) high-pressure torsion (HPT),\(^11\) and accumulative roll bonding (ARB).\(^12\) In these processes, grain refinement occurs as a consequence of dynamical recovery and recrystallization through the rearrangement of dislocations which are generated in large quantities within materials by very large plastic strains.\(^5\)

On the other hand, friction stir welding (FSW) has been studied as a new solid state welding technique of metallic materials, especially for aluminum alloys,\(^13-19\) since it was invented at The Welding Institute (TWI) in 1991.\(^20\) Sanderson et al.\(^15\) have reported that this welding technique can be applied to a large number of aluminum alloys. In addition, recent studies have demonstrated that the friction stir welded zone was composed of fine recrystallized grains resulting from severe plastic deformation experienced during welding.\(^16,17,21\) Then, we focused our attention on this unique characteristic of FSW and have studied this process not as a welding technique but as a new grain refinement process.\(^22\) Figure 1 shows the schematic illustration of the basic principle of the friction stir process (FSP) in the present research. In this process, a headpin rotating at high speed is inserted into a workpiece and then traversed horizontally to the top surface of the workpiece. Frictional heat is generated by the contact between the rotating tool and the workpiece. This heat induces the decrease in the deformation resistance of the workpiece with the increase in its temperature. The softened workpiece is severely plastically deformed by the

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![Fig. 1 Schematic illustration of the basic principle of the friction stir process (FSP) in the present research. The friction stir processed zone is hatched and labeled FZ and the rolling, transverse and normal directions of the workpiece are labeled RD, TD and ND, respectively.](image-url)
mechanical stirring action of the rotating headpin. Then, grain refinement occurs in the workpiece material as a consequence of dynamic recrystallization resulting from this severe plastic deformation.

In the previous paper, it was reported that the microstructure of the workpiece material affects the microstructure and hardness of the friction stir processed zone (FZ). However, the influence of processing parameters on the characteristics of the FZ was not investigated. Hence, in the present research, 1050 aluminum alloy with an ultra-fine grain size was produced through FSP. In addition, the influence of tool rotation speed on the temperature profile, microstructure and mechanical properties of the FZ was experimentally investigated.

2. Experimental Procedure

Cold-rolled plates of 1050 aluminum alloy were used as workpiece materials. The dimensions of the workpieces were 45 mm × 100 mm × 5 mm (Fig. 1). Figure 2 shows the schematic illustration of the specially designed tool used in the present research and the temperature measurement method of the FZ during processing. The tool was made from hardened SKD 61 (JIS). A shoulder diameter, headpin height and maximum headpin diameter were 7, 3 and 3 mm, respectively. The tool was rotated in the clockwise direction at speeds ranging from 560 to 1840 min⁻¹ and the rotating headpin was inserted into the workpiece. Then, the rotating tool was traversed perpendicularly to the rolling direction (RD) of the workpiece at a constant speed of 155 mm/min (Fig. 1). Tool rotation axis was perpendicular to the top surface of the workpiece. All FSP experiments were carried out with only a single pass. Temperature profiles of the FZ were measured during processing by a K-type thermocouple with a diameter of 0.5 mm. The hot junction of the thermocouple was placed near the tip of the rotating and traversing headpin, i.e., at a distance of about 2 mm from the undersurface center of the workpiece.

For microstructural observations and Vickers microhardness tests, friction stir processed (FSPed) specimens were cut perpendicularly to the tool traverse direction. The microstructural observations were carried out with optical microscopy (OM) and transmission electron microscopy (TEM). The hardness tests were performed under a testing load of 0.98 N at an interval of 0.5 mm in a range of 20 mm which contained both the FZ and the unprocessed zone (UZ). For tensile tests, the FSPed specimens were cut perpendicularly to the tool traverse direction and machined into tensile test specimens, as shown in Fig. 3. The FZ was located at the center of the tensile test specimens. That is, the parallel portion of the tensile test specimens was composed of the FZ and the UZ. The tensile tests were carried out at room temperature under a constant crosshead speed of 1 mm/min.

3. Results and Discussion

3.1 Temperature profile

Figure 4 shows the temperature profiles of the FZ during processing at the tool rotation speeds of (a) 560, (b) 980, (c) 1350 and (d) 1840 min⁻¹. The solid circle represents the maximum temperature at each tool rotation speed. In all cases, the temperature went up simultaneously with the
contact between the rotating tool and the workpiece. When the rotating tool traveled above the thermocouple, it reached the maximum temperature. Then, the temperature decreased rapidly as the rotating tool left from the thermocouple. That is, the FZ was exposed only for an extremely short time near the maximum temperature. This means that only a small portion of the workpiece was heated during FSP by the frictional heat in contrast to ECAP and ARB where materials are generally heated in whole during processing.

As the tool rotation speed increased from 560 to 1840 min\(^{-1}\), the maximum temperature also increased linearly from about 190 to 310°C. Since there was no external heating, this temperature increase must have been caused by the increase in the frictional heat resulting from the increase in the tool rotation speed. This result indicates that the maximum temperature which the FZ reached during processing is strongly dependent on the tool rotation speed. Hence, this maximum temperature can be controlled with the tool rotation speed for a constant tool traverse speed. In addition, in all cases, the maximum temperature was less than 646°C, i.e. the melting point of 1050 aluminum alloy (workpiece material). This result confirms that all FSP experiments were carried out in the solid state.

Figure 5 shows the heating and cooling rates of the FZ during processing at each tool rotation speed. In the present research, the heating and cooling rates were defined by eq. (1).

\[
HR \text{ or } CR = \frac{T_{\text{max}} - 100^\circ C}{|t_{T_{\text{max}}} - t_{T_{\text{max}}-100^\circ C}|}
\]

where, \(HR\) and \(CR\) are the heating and cooling rates of the FZ (°C min\(^{-1}\)), respectively, \(T_{\text{max}}\) is the maximum temperature which the FZ reached during processing (°C) and \(t_{T_{\text{max}}}\) and \(t_{T_{\text{max}}-100^\circ C}\) are the times at the maximum temperature and “the maximum temperature-100°C” (min), respectively. Namely, \(HR\) and \(CR\) were calculated from temperature variations between the maximum temperature and “the maximum temperature-100°C” during the heating and cooling stages, respectively. This was because the temperature changed almost linearly in these ranges. The heating rate increased significantly from 1220 to 2273°C min\(^{-1}\) with the increase in the tool rotation speed from 560 to 980 min\(^{-1}\). However, it had a tendency to be saturated at the tool rotation speeds above 980 min\(^{-1}\). In contrast, the cooling rate increased linearly without saturation from 341 to 1473°C min\(^{-1}\) with the tool rotation speed. This high cooling rate would result from above-mentioned unique temperature profile, i.e. localized heating of the workpiece just around the rotating tool.

For the above-mentioned SPD processes, dynamically recrystallized grains may grow up during processing and/or cooling, since materials are generally exposed for a relatively long time at a high processing temperature. However, for FSP, the growth of these grains will be inhibited because of the high cooling rate and the extremely short high-temperature exposure time, as indicated by the temperature profiles of the FZ. In consequence, the fine recrystallized grains will remain in the FZ without significant coarsening.

3.2 Microstructure observation

Figure 6 shows the optical micrographs of the cross-section perpendicular to the tool traverse direction of the specimen produced at the rotation tool speed of 560 min\(^{-1}\). The FZ is observed more brightly than the UZ. The UZ was composed of large grains highly elongated along the rolling direction (RD) of the workpiece material. In contrast, the microstructure of the FZ was completely different from that of the UZ. That is, the FZ was composed of very fine grains formed by FSP. It is noteworthy that the overall morphology of the FZ reflected that of the tool, i.e. the shoulder and headpin used in the present research (Fig. 2).

A microstructural transition zone was observed between the UZ and the FZ. On the retreating side (RS), where the tool rotation direction and the tool traverse direction were opposite, the relatively wide microstructural transition zone was observed. Namely, the microstructure changed gradually from the UZ to the FZ without a distinct boundary. However, on the advancing side (AS) where the tool rotation direction and the tool traverse direction were the same, the boundary was more clearly defined than on the RS. In addition, the microstructure changed drastically across this boundary. In the present research, the cause of these microstructural differences was not investigated. However, several researchers\(^{8,23,24}\) have suggested that there is a difference in the metal flow behavior during FSP between the RS and the AS. Hence, it is likely that these microstructural differences resulted from the different metal flow behavior on both sides.

Figure 7 shows the TEM micrographs of (a) the UZ (as cold-rolled 1050 aluminum alloy) and the central region within the FZ of the specimens produced at the tool rotation speeds of (b) 560, (c) 980 and (d) 1840 min\(^{-1}\), respectively. The UZ had very high dislocation density and was composed of highly elongated large grains with subgrains. In contrast, in the FZ, the dislocation density was very low and very fine equiaxed grains were observed. Especially for 560 min\(^{-1}\), the grain size decreased to even the submicron level of about 0.5 μm in spite of only the single pass of FSP. These results confirm that the FSP technique is a highly effective grain refinement process, considering ECAP and ARB generally require several passes for producing submicron-size grains in materials.

As the tool rotation speed increased to 960 and
Fig. 6 Optical micrographs of the cross-section perpendicular to the tool traverse direction of the specimen produced at the tool rotation speed of 560 min\(^{-1}\); (a) overall morphology, (b) transition zone on the retreating side (RS), (c) central region of the friction stir processed zone and (d) transition zone on the advancing side (AS).

Fig. 7 Transmission electron micrographs of (a) the unprocessed zone (as cold-rolled 1050 aluminum alloy) and the central regions within the friction stir processed zone of the specimens produced at the tool rotation speeds of (b) 560, (c) 980 and (d) 1840 min\(^{-1}\).
1840 min\(^{-1}\), the grain size also increased to 1–2 and 3–4 μm, respectively. In general, dynamically recrystallized grain sizes decrease with the increase in strain and/or strain rate.\(^{25-27}\) However, in the present research, the grain size increased in spite of the increase in the tool rotation speed, which was expected to lead to the increase in the strain and strain rate. This result suggests that some additional static grain growth occurred in the FZ during the cooling stage because of the increase in the maximum temperature of the FZ associated with the tool rotation speed (Fig. 4). Hence, it is clear that the maximum temperature of the FZ is a very important parameter in controlling its microstructure together with the tool rotation speed.

3.3 Mechanical properties

3.3.1 Hardness properties

Figure 8 shows (a) the Vickers microhardness profiles of the specimens produced at the tool rotation speeds ranging from 560 to 1840 min\(^{-1}\) and (b) the average hardneses of the FZ, UZ and annealed 1050 aluminum alloy. The hardness profiles were measured along the centerline of the FZ on the cross-sections perpendicular to the tool traverse direction. Many previous researchers have reported that the hardness of the FZ was less than or similar to that of the UZ.\(^{18,19,21,28}\) In the present research, however, the FZ exhibited higher hardness than the UZ for 560, 960 and 1350 min\(^{-1}\). It is noteworthy that, for 560 min\(^{-1}\), the average hardness of the FZ increased significantly to about 37% as compared with that of the UZ. 1050 aluminum alloy contains very small quantities of second phase particles and is classified into a non-heat-treatable alloy without age-hardening. Accordingly, the hardness of this alloy is primarily dependent on the dislocation density and the grain size. In all cases, the FZ had the extremely low dislocation density (Fig. 7). That is, the hardness of the FZ is more dependent on the grain size than on the dislocation density. Hence, for 560, 960 and 1350 min\(^{-1}\), it is evident that the high hardness of the FZ resulted from the grain refinement through FSP. This result confirms that the hardness of 1050 aluminum alloy can be greatly enhanced by the significant grain refinement through FSP.

The hardness of the FZ decreased with the increase in the tool rotation speed resulting in the increase in the grain size (Fig. 7). Especially for 1840 min\(^{-1}\), the hardness profile exhibited a different tendency from those for 560, 960 and 1350 min\(^{-1}\). In this case, the FZ exhibited lower hardness than the UZ in spite of its small grain size (Fig. 7(d)). This result cannot be explained by only the above-mentioned difference in the grain size between them. To investigate this phenomenon, a workpiece material, i.e. cold-rolled 1050 aluminum alloy, was annealed at 420°C for 5 hours. This annealed specimen had very low dislocation density like the FZ and was composed of very large equiaxed recrystallized grains ranging from about 30 to 40 μm. In addition, its average hardness was about 25HV0.1 lower than that of the FZ for 1840 min\(^{-1}\). In both cases, the dislocations would hardly influence the hardness because of their very low densities. Hence, the annealed specimen with the larger grain size exhibited lower hardness than the FZ for 1840 min\(^{-1}\). It is concluded that, for 1840 min\(^{-1}\), the UZ exhibited higher hardness than the FZ because of its very high dislocation density in spite of the larger grain size.

For 560, 960 and 1350 min\(^{-1}\), the hardness between the FZ and the UZ changed more rapidly on the AS than on the RS. This result corresponds with the above-mentioned microstructural difference that the microstructure variation between the FZ and the UZ was more drastic on the AS than on the RS (Figs. 6(b) and (d)).

3.3.2 Tensile properties

Figure 9 shows the tensile strength and elongation of the specimens produced at the tool rotation speeds ranging from 560 to 1840 min\(^{-1}\). For 560, 980 and 1350 min\(^{-1}\), there was no remarkable difference in the tensile strength and elongation, which were about 103 MPa and 36%, respectively. However, for 1840 min\(^{-1}\), the tensile strength decreased slightly to about 98 MPa and the elongation increased somewhat to about 41%. Figure 10 shows the appearances of the tensile tested specimens. In all cases, the fracture occurred in places distant from the center of the parallel portion, i.e., the center of the FZ. To specify the fracture locations, microstructure observations were carried out for the tensile tested specimens. Figure 11 shows the optical micrographs of the cross-sections parallel to the length.
direction, i.e. perpendicular to the tool traverse direction of the tensile tested specimens. For 560, 980 and 1350 min$^{-1}$, only the cold-rolled microstructure was observed in the fracture region. This result indicates that the fracture occurred in the UZ with the relatively low tensile strength since that of the FZ increased with the hardness by the grain refinement through FSP. However, for 1840 min$^{-1}$, fine grains elongated along the tensile direction were observed with the cold-rolled microstructure. This result shows that the fracture occurred between the UZ and the FZ. This would be because the FZ had lower tensile strength than the UZ for the same reason as the above-mentioned hardness decrease for 1840 min$^{-1}$ (Fig. 8).

Figure 12 shows the area reduction of the FZ during the tensile test. For 560 and 980 min$^{-1}$, the area reduction of the FZ was less than 1%. This result indicates that plastic deformation hardly occurred in the FZ during the tensile test.

Fig. 9  Tensile strength and elongation of the specimens produced at each tool rotation speed.

Fig. 10  Appearances of the tensile tested specimens produced at the tool rotation speeds of (a) 560, (b) 980, (c) 1350 and (d) 1840 min$^{-1}$.

Fig. 11  Optical micrographs of the cross-sections of the tensile tested specimens produced at each tool rotation speed; (a) 560, (b) 980, (c) 1350, (d) 1840 min$^{-1}$ and (e) enlarged (d).
Hence, it is evident that the deformation resistance of the FZ increased more than that of the UZ together with the increase in the hardness and tensile strength by the grain refinement through FSP. However, as the tool rotation speed increased to 1350 and 1840 min\(^{-1}\), the area reduction of the FZ also increased to about 3 and 18%, respectively.

The tensile strength of the FZ could not be measured in the present research. However, it can be predicted from the measured Vickers hardness by using the following eq. (2), which is applicable to aluminum alloy plates.\(^{29}\)

\[
TS = 3.24HV
\]  

\(TS\) and \(HV\) are the tensile strength (MPa) and the Vickers hardness, respectively. Figure 13 shows the predicted tensile strength of the FZ, UZ and annealed 1050 aluminum alloy, which were calculated from the measured average Vickers hardnesses (Fig. 8(b)). For 560 and 980 min\(^{-1}\), the measured tensile strength of about 103 MPa (Fig. 9) can be regarded as that of the UZ, since the fracture occurred in the UZ (Figs. 11(a) and (b)) and the FZ was hardly deformed during the tensile test (Fig. 12). This tensile strength is nearly equal to the calculated tensile strength of the UZ (about 113 MP). This result indicates that the eq. (2) can be applied to the prediction of the tensile strength in the present research. The tensile strength of the FZ for 560 min\(^{-1}\), which exhibited the smallest grain size and the highest hardness in the present research, is predicted to increase to even about 155 MPa.

4. Conclusions

1050 aluminum alloy with an ultra-fine grain size was produced through friction stir process (FSP) at tool rotation speeds ranging from 560 to 1840 min\(^{-1}\). In addition, the influence of the tool rotation speed on the temperature profile, microstructure and mechanical properties of the friction stir processed zone (FZ) was experimentally investigated. The following results obtained:

1. The maximum temperature of the FZ increased linearly with the tool rotation speed. However, this temperature was less than the melting point of 1050 aluminum alloy, indicating that FSP was carried out in the solid state.

2. As the tool rotation speed increased, the heating rate of the FZ was saturated but its cooling rate increased linearly from 341 to 1473 \(^{\circ}\)C.min\(^{-1}\). This high cooling rate will prevent fine recrystallized grains in the FZ from coarsening during the cooling stage.

3. The FZ had very low dislocation density and was composed of very fine equiaxed grains. The grain size of the FZ increased with the tool rotation speed. However, it is noteworthy that, for the 560 min\(^{-1}\), it decreased to even the submicron level in spite of only the single pass of FSP. On the advancing side (AS), the boundary between the FZ and the unprocessed zone (UZ) was more clearly defined than on the retreating side (RS) and the microstructure also changed more drastically in a narrower region on the basis of this boundary.

4. The average hardness of the FZ decreased with the increase in the tool rotation speed. However, it is notable that, for the 560 min\(^{-1}\), it increased to even about 37% as compared with that of the UZ. The hardness between the FZ and the UZ changed more drastically on the AS than on the RS, corresponding with the microstructural difference between both sides.

5. For 560, 980 and 1350 min\(^{-1}\), the tensile test specimens were fractured in the UZ. In addition, for 560 and 980 min\(^{-1}\), the area reduction, i.e. the plastic deformation, hardly occurred in the FZ during the tensile test. These results indicate that the tensile strength and deformation resistance of the FZ increased by the grain refinement through FSP.

6. For FSP, the maximum temperature of the FZ was a very important parameter in controlling its microstructure and mechanical properties together with the tool rotation speed.

7. The FSP technique was highly effective in producing an ultra-fine grained material with excellent mechanical properties.
REFERENCES