

# Evaluation of Grain Refinement and Mechanical Property on Friction Stir Welded Inconel 600

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To evaluate the dynamic recrystallization aspect and microstructures, electron back scattered diffraction (EBSD) analysis on friction stir welded Inconel 600 was employed. Friction stir welding (FSW) led to the dynamic recrystallization in all conditions as high angle grain boundaries were distributed more than 85% in fraction, with equiaxed grains, and the grain refinement was accelerated from average grain size of 19  $\mu\text{m}$  in the base material to 3.4  $\mu\text{m}$  in the stir zone by increasing the welding speed. Also, it increased microhardness by more than 20%, compared to base material microhardness, and the effect of grain size on the microhardness in the stir zones was satisfied with Hall-Petch relationship. [doi:10.2320/matertrans.MRA2008432]

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## 1. Introduction

Friction stir welding (FSW) was introduced for the first time by The Welding Institute (TWI), and performed on various materials with a low melting point, such as Al alloys, Mg alloys and Cu alloys, as well as high temperature materials such as steels, Ni alloy and Ti alloy.<sup>1)</sup> As a special feature of FSW, the grain refinement in materials can be controlled so that physical and chemical properties in stir zone increased significantly greater than that of the base material.<sup>2-5)</sup> However, FSW researches on Ni based superalloys with a high melting point which have been used in many parts of industry have rarely been reported.<sup>6,7)</sup> As these materials have high strength at high temperature, the welding speed is significantly slow than that of materials with a low melting point, less than 100 mm/min, which resulted in the limitation on the grain refinement of 12  $\mu\text{m}$  by Ye *et al.*<sup>6)</sup> and 14  $\mu\text{m}$  by Sato *et al.*<sup>7)</sup>

Ni base alloy is a material which has low stacking fault energy in F.C.C. metals. It is easy to occur the dynamic recrystallization at the condition which has high stored energy and enough heat input, compared to the materials with a high stacking fault energy, such as Al alloys.<sup>8)</sup> Since the FSW was introduced in 1991, the dynamic recrystallization aspect of materials with a high stacking fault energy has been reported at many researches,<sup>9-11)</sup> however, that of the materials with a low stacking fault energy in F.C.C. metals has not been reported mainly due to the difficulty of FSW on Ni alloy. Therefore, the present study was carried out to investigate the dynamic recrystallization aspect and the microstructural changes according to increasing the welding speed by EBSD technique and to evaluate relationship between the grain refinement and the mechanical property.

## 2. Experimental Procedures

The material used in this study was 75 mm  $\times$  150 mm  $\times$  2 mm plate of Inconel 600 (76 Ni, 15.5 Cr, 8.0 Fe, 0.25 Si, 0.50 Mn, 0.08 C, 0.008 S mass%—single phase type). FSW was performed at a tool rotation speed of 400 rpm and a

travelling speed of 150~250 mm/min, and a WC-Co (tungsten carbide-cobalt) tool with a shoulder of 15 mm in diameter and a probe of 6 mm in diameter and 1.8 mm in length was used. Also, in order to obtain the good welding property, the tool was tilted at 3° forward from the vertical, and argon shielding gas was utilized to prevent surface oxidation during the FSW. To observe the macrostructures and microstructures on friction stir welded materials, 97 ml HCl, 2 ml HNO<sub>3</sub> and 1 ml H<sub>2</sub>SO<sub>4</sub> solution was prepared, and samples were etched on surface.

In order to evaluate dynamic recrystallization aspect and grain boundary characteristic distribution (GBCD) on the stir zone, electron back scattered diffraction (EBSD) analysis was employed. For this work, samples were additionally polished using a Vibromet after mechanical polishing. EBSD analysis was performed using TSL-OIM™ incorporated with SEM for the base material and the stir zone. The Vickers hardness was evaluated to identify the mechanical property of the base material and the weld zone. Transverse hardness measurement was carried out on the cross section of friction stir welds with a load of 9.8 N, and a dwell time of 15 s was used.

## 3. Results and Discussion

The macrostructures of friction stir welded zone according to the change of welding speed are shown in Fig. 1. At the specimen made at 150 mm/min welding speed, it was perfectly welded from the surface to the bottom. However, band structure was observed in the center of stir zone, as shown in Fig. 1(a). It also has been reported by another research, similar to this study.<sup>12)</sup> The material made at 200 mm/min welding speed was welded from the surface to 1.8 mm in depth, and band structure was also observed in the stir zone similar to that at the 150 mm/min, as shown in Fig. 1(b). However, the specimen welded at 250 mm/min showed much shallower welded zone compared to 150 and 200 mm/min samples, and a groove like defect occurred at the advancing side, as shown in Fig. 1(c).

These differences of weld depth occurred by increasing the welding speed can be explained in terms of insufficient metallic plastic flow caused by the rotating probe. An

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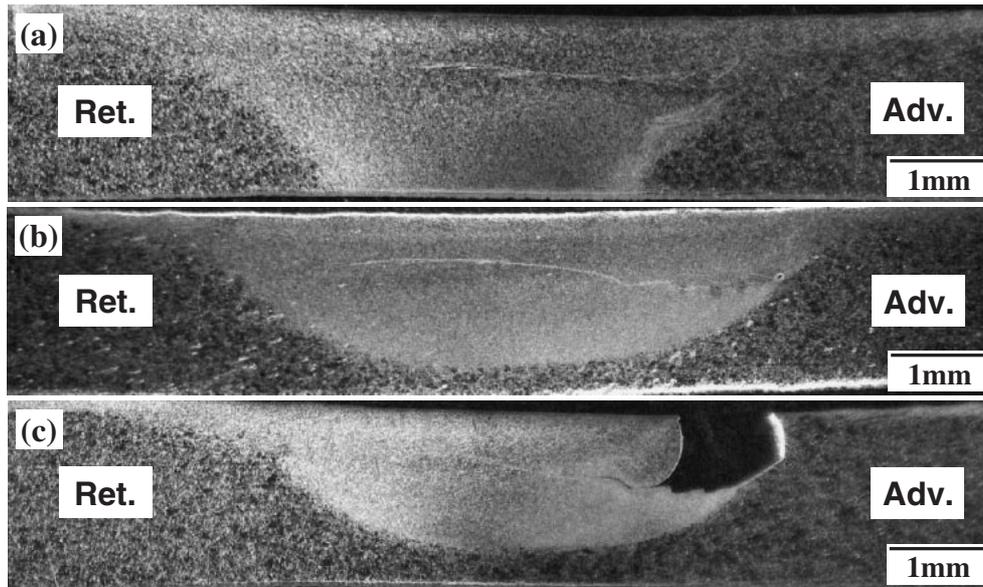


Fig. 1 The macrostructure changes of weld zone according to the increase of welding speed. (a) 150 mm/min, (b) 200 mm/min and (c) 250 mm/min in welding speed. Adv. and Ret. indicate advancing side and retreating side, respectively.

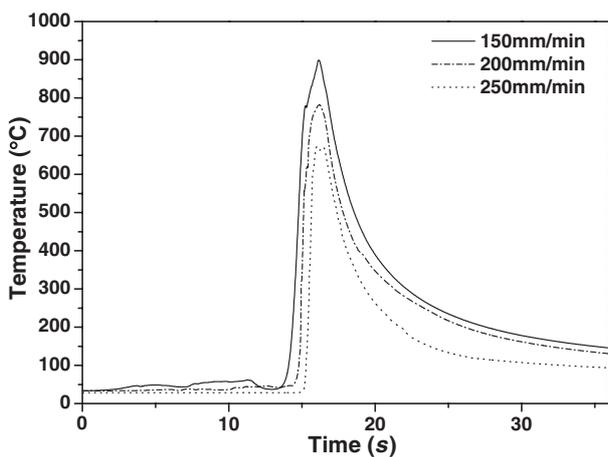


Fig. 2 The temperature hysteresis in the stir zone during the friction stir welding.

increase of welding speed led to a decrease in heat input into the weld material, and this results in insufficient metallic plastic flow, which can easily cause a groove like defect.<sup>1)</sup> In other words, the decrease of heat input can lead to the increase of the resistance of metallic plastic deformation, which results in the decrease in the depth of tool inserted into the base material at the constant force ( $2.3 \times 10^3$  kgf) during the FSW.

The temperature hysteresis of the stir zone according to the welding speed at the constant rotation rate is shown in Fig. 2. The temperature hysteresis was measured at the plate's backside on the center of the weld zone during the FSW. At 150 mm/min, the maximum temperature was 890°C in the stir zone, however, it was gradually decreased by an increase of the welding speed to 780°C at 200 mm/min and 670°C at 250 mm/min. Also, an increase of welding speed led to the increase of the relative cooling rate. Therefore, it can be explained that the heat input per unit length of the stir zone

decreases by the increase of the welding speed at the constant rotation speed.

To evaluate dynamic recrystallization aspect and microstructure in the stir zone, EBSD analysis was employed, and grain boundary maps for the analysis zones are shown in Fig. 3. In the initial state, base material consisted of grains of 5~45  $\mu\text{m}$ , with an average grain size of 19  $\mu\text{m}$ , as seen in Fig. 3(a). At the welding speed of 150 mm/min, the microstructure consisted of equiaxed grains ranging between 2  $\mu\text{m}$  and 20  $\mu\text{m}$  in size, with an average grain size of 5.6  $\mu\text{m}$ , which is significantly refined than that of the base material, as seen in Fig. 3(b). At the welding speed of 200 mm/min, grains in the stir zone were between 2  $\mu\text{m}$  and 15  $\mu\text{m}$  in size, with an average size of 4.4  $\mu\text{m}$ , as seen in Fig. 3(c). The increase of welding speed led to grain refinement more highly so that the average grain size at 250 mm/min was significantly decreased to 3.4  $\mu\text{m}$ , as seen in Fig. 3(d).

This grain refinement can be explained by high stored energy, low heat input and relatively high cooling rate according to the increase of welding speed, with the constant tool rotation speed. In other words, the recrystallization nuclei can be created more easily at the high speed welding because it has a more high stored energy and low heat input than that of the low welding speed. In addition, the high speed welding has a higher cooling rate than that of the low welding speed. Therefore, it can be obtained a more refined grain by the increase of welding speed.

Grain boundary characteristic distribution analyzed by EBSD is shown in Fig. 4. High angle boundaries, more than 95%, were distributed on the base material, as seen in Fig. 4(a). The 60 degrees distribution (22%) was identified by the annealing twin boundaries which were usually observed in materials with a low stacking fault energy in F.C.C. metals. In the stir zone welded at the welding speed of 150 mm/min, the high angle grain boundary was distributed over 95%, with 21% of annealing twin boundary, almost the same fraction as the base material, as shown in Fig. 4(b). In

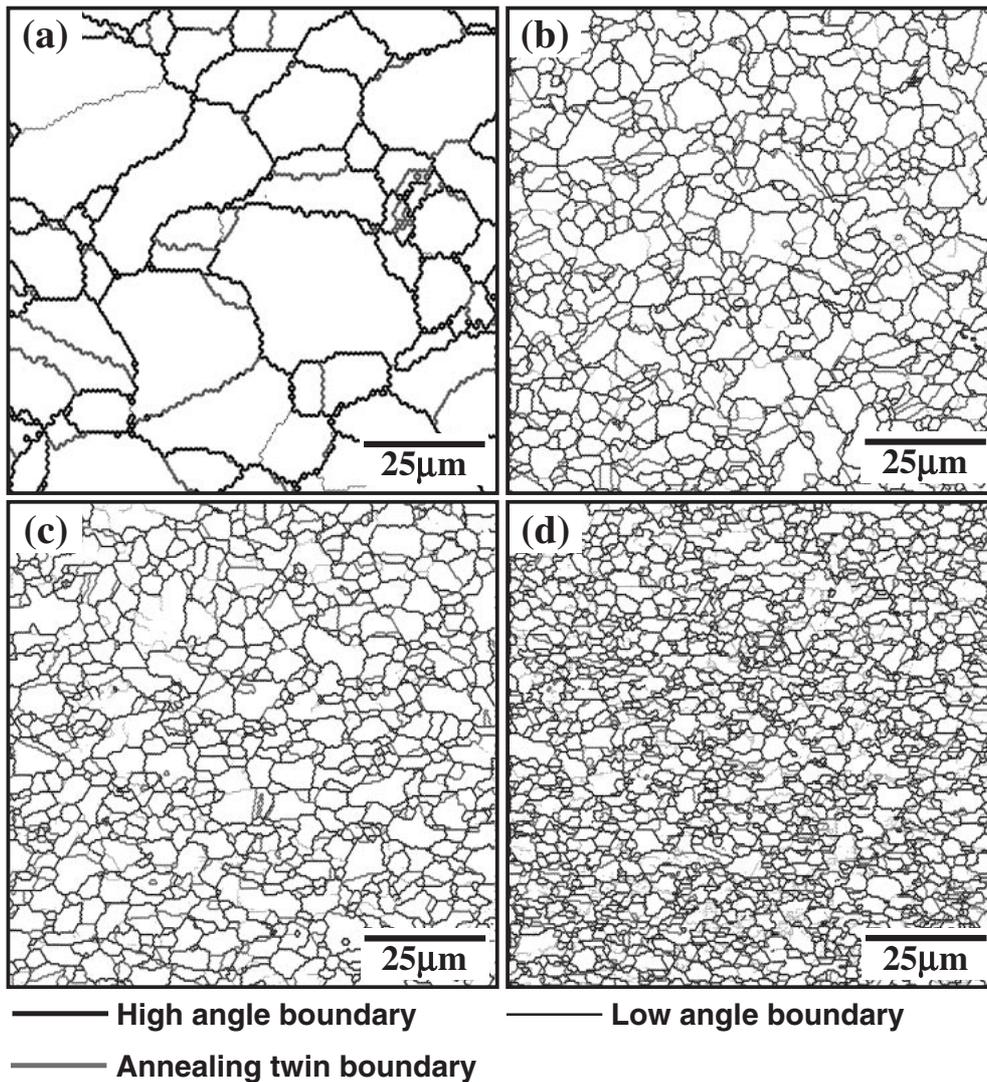


Fig. 3 Grain boundary maps of base material and stir zones. (a) base material, (b) 150 mm/min, (c) 200 mm/min and (d) 250 mm/min in welding speed.

case of 200 mm/min welding speed, high angle boundaries were distributed more than 90%, and annealing twin boundary was decreased to 16%, as seen in Fig. 4(c). The increase of welding speed led to the decrease of high angle boundary distribution. As a result, at the 250 mm/min welding speed, the high angle boundary and annealing twin boundary decreased to 85% and 12%, respectively, as seen in Fig. 4(d). The change of boundary fraction observed in this work is simply shown in Fig. 4(e).

The stacking fault energy in F.C.C metals plays an important role in determining the grain refinement, mechanical property and texture.<sup>13)</sup> The materials with a high stacking fault energy are easy to rearrange dislocation by elevated temperature from the severe plastic deformation state, resulted in dynamic recovery, so that fewer dislocations are available to create high angle grain boundaries, and also limited stored energy gives rise to easy grain growth.<sup>14)</sup> Also, this process can make a strong texture than that of materials with a low stacking fault energy.<sup>15)</sup> However, the materials with a low stacking fault energy are easy to create the recrystallization nuclei in the grain boundaries and grains

with a higher dislocation density during the elevated temperature, resulted in dynamic recrystallization.<sup>8)</sup> Also, the materials with a low stacking fault energy can be easily formed annealing twin during the recrystallization and grain growth, resulted in random texture due to twinning variants in the grains.<sup>13)</sup> This work showed the distribution of annealing twins, 60 degrees in boundary angle, at all conditions, as seen in Fig. 4. In case of 150 mm/min, annealing twins was formed more than 20%, similar to that of the base material. Also, these results can be led to random texture distribution by the twinning. Therefore, the dynamic recrystallization in F.C.C metals can be known to be affected by the difference of stacking fault energy.

The microhardness distributions according to the change of welding speed are shown in Fig. 5. The average microhardness of the base material was 163 Hv in Vickers hardness. At 150 mm/min, the microhardness of the stir zone was in the range from 180 to 193 Hv, indicating a higher hardness than that of the base material. In case of 200 mm/min welding speed, a more increased distribution was shown, ranging from 180 to 220 Hv. An increase of welding speed

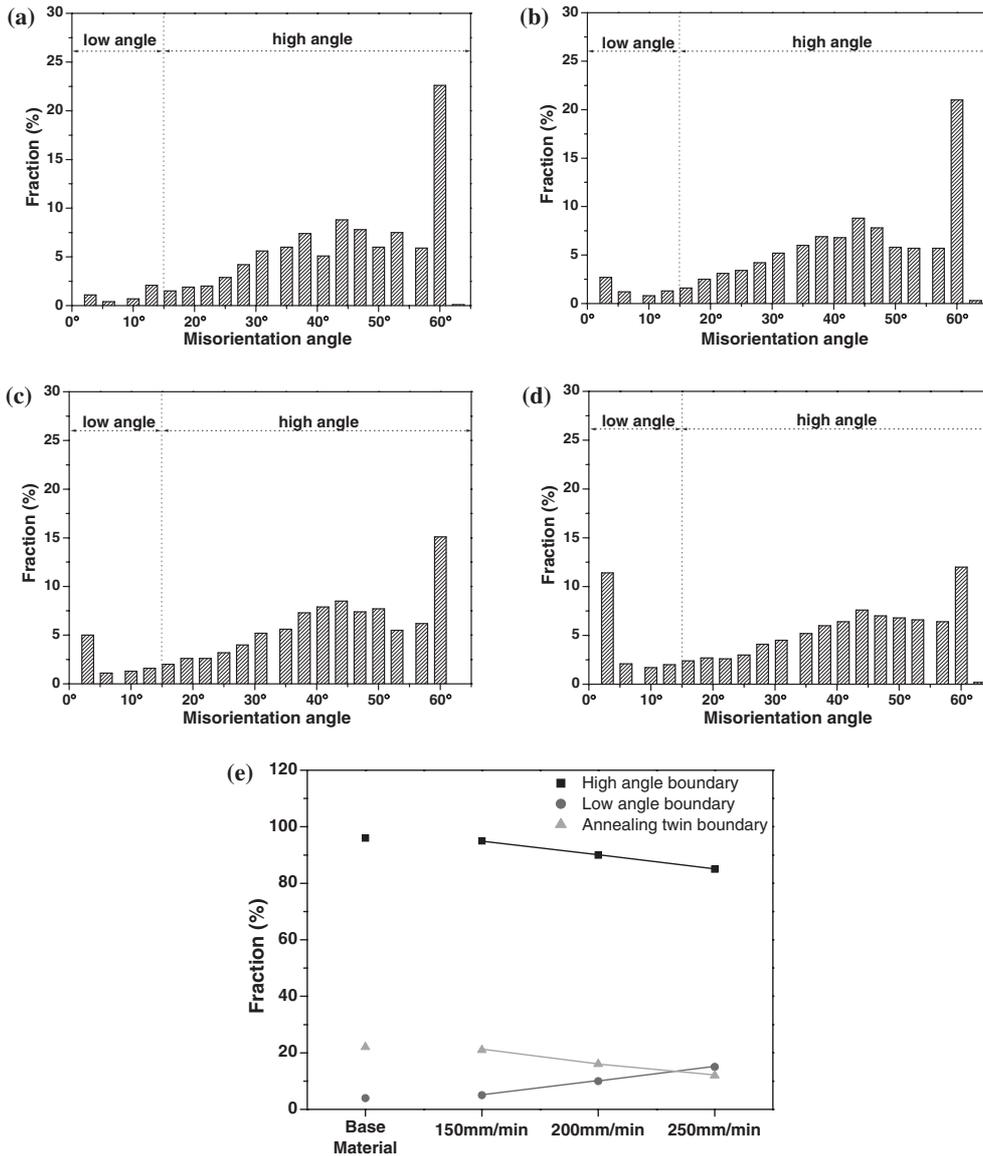


Fig. 4 Changes of grain boundary character distribution and boundary fraction by friction stir welding. (a) base material, (b) 150 mm/min, (c) 200 mm/min, (d) 250 mm/min in welding speed and (e) boundary fraction.

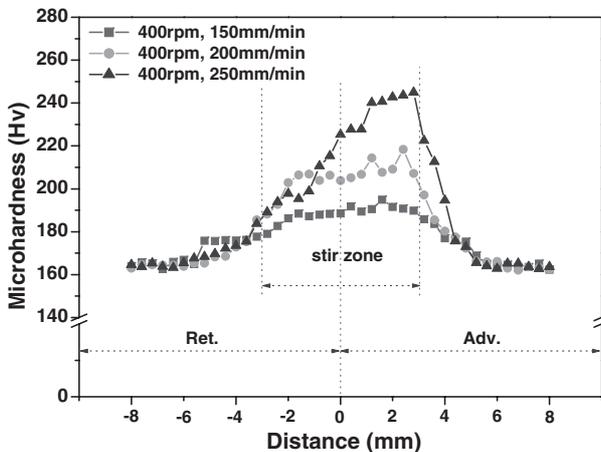


Fig. 5 Distribution of Vickers microhardness in friction stir welded material. Adv. and Ret. indicate advancing side and retreating side, respectively.

led to more increased microhardness, as a result, at 250 mm/min welding speed, microhardness in the stir zone was distributed from 180 to 245 Hv. This indicates a notable increase of the hardness in the stir zone, more than 20%, than that of the base material.

The relationship of grain size on the microhardness in the center of the stir zone is shown in Fig. 6. The microhardness versus grain size of stir zone satisfied the Hall-Petch relationship. The following relationship was derived,  $Hv = 53 + 325 d^{-1/2}$ . Also, it is easy to estimate that the effect of dislocation density on the microhardness is very low due to the absence of low angle boundary in the grains at all conditions, as seen in Fig. 3. Therefore, it can be known that the microhardness was gradually increased by the grain refinement during the FSW.

The grain refinement mechanism by FSW can be simply explained on the basis of plastic flow and dynamic recrystallization. By applying FSW, the high stored energy in the material was accompanied by severe deformation

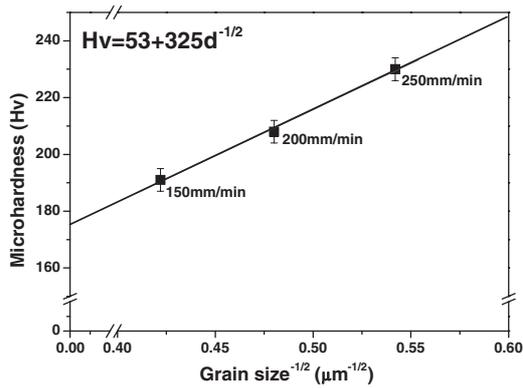


Fig. 6 The Hall Petch relationship between the grain size and the microhardness in the stir zone.

process. An increase in the stored energy can be explained by an increase in the dislocation density, and there is a higher dislocation distribution due to the plastic flow which is the foundation of FSW. Also, an important point for this process is that dynamic recrystallization is accompanied by the friction heat between the tool and material during the FSW.<sup>1)</sup> In addition, as the Inconel 600 alloy used in this study has a low stacking fault energy, it is difficult to rearrange the dislocations by dynamic recovery compared to the material with a high stacking fault energy, such as Al alloys, while dynamic recrystallization is easy to occur.<sup>8)</sup> Therefore, when enough heat input and stored energy are provided during the FSW, the formation of the recrystallization nucleus could occur coincidentally from the grain boundaries and grains with a high dislocation density. As a result, the microstructure with refined grains can be obtained.

#### 4. Conclusions

The FSW of Inconel 600 can be successfully performed at 150 mm/min and 200 m/min welding speeds without defect. Also, the dynamic recrystallization was observed at all conditions, as high angle boundary was distributed more than 85%, accompanied by annealing twin. The application of

FSW led to the notable grain refinement by the increase of welding speed. As a result, the average grain size of 19 μm in the base material was significantly refined to 3.4 μm in the stir zone by FSW. It affected the increase of mechanical properties so that the microhardness increased from 163 Hv in the base material to 220 Hv at 200 mm/min welding speed in the stir zone. Also, the effect of grain size on the microhardness in the stir zone satisfied the Hall Petch relationship. Therefore, the application of friction stir welding is effective to induce the dynamic recrystallization on the materials with a low stacking fault energy in F.C.C. metals and increase the grain refinement and the mechanical properties in the FS weld zone.

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