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# Microstructure characterization of the stir zone of submerged friction stir processed aluminum alloy 2219



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#### ABSTRACT

Aluminum alloy 2219-T6 was friction stir processed using a novel submerged processing technique to facilitate cooling. Processing was conducted at a constant tool traverse speed of 200 mm/min and spindle rotation speeds in the range from 600 to 800 rpm. The microstructural characteristics of the base metal and processed zone, including grain structure and precipitation behavior, were studied using optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Microhardness maps were constructed on polished cross sections of as-processed samples. The effect of tool rotation speed on the microstructure and hardness of the stir zone was investigated. The average grain size of the stir zone was much smaller than that of the base metal, but the hardness was also lower due to the formation of equilibrium  $\theta$  precipitates from the base metal  $\theta'$  precipitates. Stir zone hardness was found to decrease with increasing rotation speed (heat input). The effect of processing conditions on strength (hardness) was rationalized based on the competition between grain refinement strengthening and softening due to precipitate overaging.

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#### 1. Introduction

Friction stir welding (FSW) has proven to be an efficient, reliable, and environment friendly joining method for aluminum alloys [1,2]. During FSW, a rotational tool travels along the length of the abutting plates to be welded, and produces a high plastically deformed region through the associated stirring action produced by the tool. Friction stir processing (FSP), developed based on FSW, is emerging as an effective thermal-mechanical process to refine grains, achieve superplasticity, modify microstructures, and synthesize in-situ composite and intermetallic compounds [3–12]. As opposed to FSW, FSP is conducted on a single workpiece and is not designed as a joining method. During FSW/FSP, localized heating is generated by friction at the tool/workpiece interface, as well as from the plastic deformation of the workpiece. The combination of severe plastic deformation and high temperature exposure, promotes dynamic recrystallization in the stir zone and resultant grain refinement. The grain size in the stir zone of FSW/FSP of aluminum alloys without external cooling has been reported in the range of 2–10  $\mu$ m [13–16].

Since fine-grained microstructures are generally beneficial with respect to mechanical properties, there have been many attempts to achieve even finer grain size. Several researchers have reported that the grain size can be further refined by applying in-process or external cooling during FSW/FSP. Benavides et al. [17] performed FSW trials of 2024 aluminum alloy using liquid nitrogen to lower the initial temperature of the samples to -30 °C. They reported that the size of the grains was reduced to less than 0.8 µm when the starting workpiece temperature was reduced to 173 K (-100 °C). Rhodes et al. [18] used a mixture of dry ice and isopropyl alcohol on the surface of the processed 7050 Al plate in an



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attempt to "freeze in" the newly recrystallized microstructures and found extremely fine grains (50–100 nm). Hofmann and Vecchio performed submerged FSP on 6061-T6 Al alloy and obtained grains smaller than 200 nm [19]. Su et al. [20] obtained fine grains in the range of 100–400 nm by using a mixture of water, methanol, and dry ice to quench the friction stir processed 7075 Al plates immediately behind the tool. Similar to Su et al., Liu et al. [21] used room temperature water to quench the processed 7075 Al and obtained grains with an average diameter of 800 nm.

Although fine grains are beneficial to the strength of the stir zone, a loss of strength is often reported in the stir zone and HAZ by FSW/P of precipitation strengthened aluminum alloys. This could be caused by precipitate dissolution and transformation to an equilibrium state and coarsening. Woo and Choo found that softening in the stir zone of FSW joints of 6061-T6 Al alloy was primarily caused by precipitate dissolution [22]. It was reported by Dong et al. that the softening in the stir zone of 6005-T6 Al alloy was caused by the dissolution of  $\beta$ " during welding and limited re-precipitation during cooling [23]. They also reported that the softening in the HAZ was a result of the coarsening of the  $\beta'$  and Q' precipitates. Different precipitate evolution mechanisms were related to the thermal cycles during FSW. Therefore, in order to control the precipitate evolution, fast cooling was applied to limit the thermal effect during FSW. Fratini et al. [24] conducted in-process water-cooling on the top surfaces of weld samples during FSW and found that material softening in the thermo-mechanically affected zone (TMAZ) was reduced by 10-40 HV. Zhang et al. [25] performed underwater FSW on 2219-T6 Al alloys and found that the tensile strength of the samples was higher than that of the joint cooled in air, confirming the feasibility of improving the joint properties by water cooling. Furthermore, it was also reported that low tool rotational speed which leads to lower heat input, will result in lower stir zone temperature during FSW/FSP [25]. This can reduce the rate of precipitate dissolution or coarsening.

However, the effect of the tool rotational speed and associated cooling rate on the microstructure and hardness in the stir zone of FSPed Al–Cu alloys has not been systematically investigated. In the present study, submerged friction stir processing was conducted on a precipitation strengthened Al–Cu alloy at a constant travel speed over a range of tool rotational speeds. Microstructure evolution in the stir zone, including grain structure and precipitate morphology, has been analyzed. Hardness of the stir zone was also measured to quantify the effect of tool rotational speed and heat input on softening kinetics.

#### 2. Experimental Procedures

The submerged friction stir processing setup and the tool are shown in Fig. 1. Fig. 1a defines the processing coordinates. The processed plate was fitted into a tank, which was filled with room temperature water (Fig. 1b). A backing plate made of steel was used. The tool was a standard tool steel, consisting of a shoulder with a diameter of 22 mm and no pin feature, as shown in Fig. 1c. The base material used was  $300 \times 300 \times 2.5$  mm 2219-T6 aluminum alloy. The chemical composition (wt.%) of the base metal was Al–6.48Cu–0.32Mn–0.23Fe–0.06Ti–0.08 V–0.04Zn–0.49Si–0.2Zr. The tool rotational speed ( $\omega$ ) was varied in the range of 600–1000 rpm at a constant processing speed (v) of 200 mm/min. Displacement control was used for tool plunge with a plunge depth of 0.5 mm. The tilt angle of the rotational tool with respect to the Z-axis of the FSW machine was 2.5°.

Microstructure characterization was performed in two orthogonal directions of the friction stir processed plates, transverse cross section and plan view. The cross-section (YZ plane in Fig. 1a) of the plates was analyzed using Optical Microscopy (OM). Polished samples were etched with Keller's reagent (a mixture of 2.5 ml nitric acid, 1.5 ml hydrochloric acid, 1 ml hydrofluoric acid and 95 ml water) for 20 s. Vickers microhardness was measured on the polished cross-section using an automated tester under a load of 50 g for a dwelling time of 10 s. The center-to-center distance between the adjacent indents was 120  $\mu m.$  The plan view (XY plane in Fig. 1a) of the samples was characterized using transmission electron microscopy (TEM). The processed surface was lightly ground to remove the periodic surface bands from tool rotation. Samples were then mechanically ground on the unprocessed side (back side) until the sample thickness was reduced to  $80 \,\mu\text{m}$ . Disks with a diameter of 3 mm were punched from the center position of the thin foils and subjected to twin-jet electropolishing in a solution of 30% nitric acid and methanol at -20 °C. The TEM samples were examined using the Philips CM200 microscope operating at 200 kV to analyze the grain structure and precipitate morphology and distribution.



Fig. 1 – Submerged friction stir processing and the tool: a) schematic drawing of FSP, b) set up of submerged friction stir processing, c) the tool.

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