Application of Friction Stir Processing as a Pretreatment to Fusion Welding

The pretreating technique resulted in an improved weld heat-affected zone and weld metal microstructure in several nickel-based alloys

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ABSTRACT

Friction stir processing (FSP) was applied as a pretreatment technique to modify the base metal microstructure of nickel-based alloys, including Inconel® Alloy 600 (IN600), Alloy 625, Alloy 718, and Hastelloy® X. Microstructural analysis of gas tungsten arc (GTA) welds placed on pretreated Ni alloys indicates simultaneous refinement of the heat-affected zone (HAZ) and weld metal (WM), which is otherwise not achievable using conventional techniques. The grain refinement resulting from FSP resulted in increased epitaxial growth sites for the WM as well as up to a threefold reduction in near-fusion boundary (FB) HAZ grain size for IN600. Augmented strain weldability testing of Alloy 625, Alloy 718, and Hastelloy X suggests HAZ liquation cracking susceptibility was reduced as a result of FSP pretreatment. Alloy 718 showed the highest degree of HAZ refinement (1.9 times untreated base material) and also showed the greatest reduction in maximum HAZ crack length and total crack length. Weld heat input was also shown to have an effect on the near-FB HAZ of pretreated alloys. It can be expected that fusion weld parameters will have an effect on the efficacy of FSP pretreatment.

Introduction

Friction stir processing (FSP) is mechanically similar to friction stir welding (FSW); however, no weld is created in the former. Friction stir processing utilizes the microstructural changes resulting from severe plastic deformation beneath a rotating tool to locally modify the properties and performance of metals. Some applications for FSP have included crack repair or microstructural modification of fusion welds. Several studies have investigated using FSP as a post-arc welding technique to reduce weld discontinuities and improve the weld mechanical properties of various Al alloys (Refs. 1, 2) and NiAl bronze (Ref. 3). However, very little attention has been focused on the prospect of using FSP to modify base material microstructure such that weldability issues such as hot cracking are reduced or eliminated for subsequent fusion welds. Friction stir processing pretreatment can be used as an alternative conditioning process for the rejuvenation of microstructures with deleterious features or phases, thus enabling subsequent fusion welding.

Naturally, welding in the solid state via FSW is a simpler process than the combination of both FSP and fusion welding. However, in some situations, using FSP exclusively is not entirely practical, especially for high Tm materials. By nature of the FSW process, joint geometries are limited to primarily butt and lap configurations. Other joint configurations require complex fixturing and/or complex machine control schemes (Ref. 4). Additionally, high process forces experienced during FSW of high-Tm materials, especially for thick sections, limits welding to only very robust FSW machines with large associated workspace footprints. Such practical limitations hinder FSW capabilities in the field. One can envision FSP pretreatment performed remotely to only modify the microstructure where necessary, e.g., a portion of the workpiece that is under high restraint, stress concentration, etc. The locally modified workpiece can then be transported to the field after FSP where final joining can be accomplished via conventional fusion welding techniques.

To date, only a single study has investigated the utility of using FSP as a pretreatment for microstructural modification of subsequent fusion welds. The unique work, by Mousavizade et al. (Ref. 5), focused on the effect of FSP pretreatment prior to laser welding of cast IN738. The top ~1.5 mm of 5-mm-thick IN738 plate was friction stir processed. Following FSP, the stir zone (SZ) region was laser surface remelted. Any cracking that occurred during laser surface remelting was the result of inherent mechanical restraint. The authors noted qualitative improvements in HAZ liquation cracking susceptibility due to increased material homogeneity and refinement of liquation-inducing microconstituents from FSP. The HAZ of non-FSP IN738 contained many cracks transversely oriented to the fusion boundary. In contrast, the FSP-treated material exhibited no cracks in the near-fusion boundary HAZ after laser surface remelting. The authors suggest that constitutional liquation of coarse primary γ and interdendritic γ′ eutectic in the cast base material was circumvented as a result of FSP from breakdown and refinement of...
Beneficial changes resulting from pretreatment are not limited to the HAZ and may also occur in the weld metal (WM) of pretreated material due to the refinement obtained from FSP. The WM grain structure during arc welding is primarily controlled by epitaxial and preferred growth from the fusion boundary with competitive growth dominating remote from the fusion boundary (Ref. 6). Like a casting, the WM grain structure can be directly influenced by altering the extent of growth (Ref. 7) — with increased growth site density leading to decreased grain size. Many studies have investigated the effects of increasing the heterogeneous growth site density to refine WM grains by using additions of inoculant particles. Studies have been performed in several alloy systems including ferritic steels (Ref. 8), Al-Zn-Mg alloys (Ref. 9), and Al-Li alloys (Ref. 10). However, a significant downside to the practice of introducing inoculant particles is the alloy-specific change in chemistry required for the base material or filler material. Additionally, the thermal stability of the particles makes the inoculation effect somewhat dependent on the welding parameters. For some applications where WM refinement is sought, a change in alloy composition is not feasible, e.g., autogenous welding. Noncompositional methods such as arc manipulation techniques can also be used to change the extent of growth in fusion welds via several mechanisms including decreased weld pool temperature (Ref. 7). Example methods include electromagnetic stirring (Refs. 11–13), mechanical vibration (Refs. 11, 14), and AC pulsed current (Refs. 15, 16). These techniques have demonstrated effectiveness in altering WM grain morphology. However, arc manipulation requires direct implementation of additional equipment, and adds complexity to the welding processes. Furthermore, additional parameters such as arc oscillation and current pulse frequency must be developed and controlled for different materials. Increasing growth site density by grain refinement using FSP prior to fusion welding is a promising straightforward technique that does not require compositional or fusion welding process parameter alternations.

The objective of this work is to evaluate the effect of FSP pretreatment on the microstructural evolution of subsequent fusion welds. The effects of FSP pretreatment on the HAZ liquation susceptibility for three HAZ liquation-susceptible Ni-based alloys was investigated. For alloys that included Alloy 625 (Ref. 17), Hastelloy® X (Ref. 18), and Inconel® 718 (Refs. 19, 20) prior studies have identified mechanisms and effects.
Friction stir processing parameters, including the spindle rotation rate and traverse rate, were adjusted such that defect-free process zones were able to be obtained. Table 2 shows the processing parameters used for FSP of the three HAZ liquation-susceptible alloys. For Alloy 600, two process parameters were chosen that represent the extremes of the processing window with respect to heat input, i.e., high and low heat input. Friction stir processing parameters for Alloy 600 are also listed in Table 2.

Following FSP, autogenous gas tungsten arc (GTA) welds were placed atop the FSP material. Prior to arc welding, the samples were cleaned with acetone. Arc welds were placed on the top plate surface entirely within the stir zone and made in the same direction as the FSP traverse. A programmable GTA welding machine (Jetline Engineering, Inc., Model TKM-72-M, Irvine, Calif.) was used with welding speed, current, and voltage of 1.57 mm/s (3.7 in./min), 110 A, 11 V, respectively.

The HAZ liquation susceptibility of FSP pretreated materials was evaluated using the spot Varestraint technique (Ref. 21). Prior to testing, pretreated plates were ground flat to facilitate crack detection after testing — Fig. 1. Figure 2 shows a schematic of the spot Varestraint test. The spot Varestraint test utilizes a GTA torch to create a spot weld on the test specimen (180 A, 20 s arc time) such that the circular spot weld was centered along the edge of the FSP SZ. Using this method, both base material and FSP pretreated material are tested simultaneously. After the spot weld pool is stabilized, the arc is extinguished and load is applied at a stroke rate of 6 in./s (152 mm/s). The applied load deforms the sample around the die block with a predetermined radius thus imposing a known level of strain. Strain levels ranging from 1 to 7% were evaluated. After Varestraint testing, any oxidation was removed using SiC grinding papers. Heat-affected zone cracks orthogonal to the fusion boundary were observed using a stereo microscope at 20X magnification. Both total crack length (TCL) and maximum crack length (MCL) values were measured using stereo optical microscopy at a 20X magnification. All crack lengths were measured perpendicular to the spot weld fusion boundary as shown in Fig. 3. Metallographic analysis was performed using a combination of techniques including light optical microscopy (LOM), scanning electron microscopy (SEM), and electron backscatter diffraction (EBSD).

Specimens examined using LOM were electrolytically etched using an aqueous solution of 10% oxalic acid with a current density of 0.86 A/cm² to reveal general microstructure. A SEM (FEI, Model Quanta 200, Hillsboro, USA) equipped with an EBSD camera was used for crystallographic orientation analyses. Electron backscatter diffraction was used to gather information regarding the distribution of crystallographic orientations within the FSP and arc weld regions. Samples for LOM and SEM/EBSD were mechanically polished with the final step consisting of vibratory polishing using colloidal silica. Maps generated from EBSD data were used for measurements of grain size and grain boundary length. The ASTM linear intercept method was utilized for LOM grain size measurements. EBSD grain size was determined using an equivalent area method that determines an equivalent grain diameter based on the measured area of the grain. A grain tolerance angle of 5 deg was used to define a grain.

Results and Discussion

Characterization of Friction Stir Processing Pretreatment of IN600

Extremes in the IN600 processing window were selected to represent the gamut
of grain sizes obtainable via FSP. The high and low-heat input parameter combinations of 150 rev/min; 2 in./min and 100 rev/min; and 4.5 in./min, respectively, were selected. All resulting stir zones were representative projections of the FSP tool having a truncated shape with SZ width of ~0.75 in. and depth of nearly 0.125 in., which correlates to the shoulder diameter and length of the pin. The resulting IN600 SZs for high- and low-heat-input FSP runs exhibited average SZ grain sizes of 15 and 9 μm, respectively. For comparison, the grain size of nonpretreated base material was significantly larger, 54 μm.

Following FSP, autogenous GTA welds created directly atop the FSP SZ such that welds were contained entirely within the SZ and in the same direction the FSP traverse. Figure 4 shows a transverse cross section of the autogenous GTA weld made atop the FSP region. The size of the GTA weld is such that the corresponding HAZ still is located within the former SZ. For both FSP heat inputs, coarsening of the prior SZ is readily apparent. Compared to the starting SZ grain size, the average grain size in the HAZ along the fusion boundary increased to the initial FSP grain size by a factor of 4 to 5, depending on the sample. The initial SZ grain differences resulting from the different parameter combinations did not have a significant effect on the resultant near-fusion boundary HAZ grain size. While prior SZ grains coarsened as a result of the autogenous GTAW pass, near-fusion-boundary grain size remained below 100 μm, unlike the untreated IN600 base material. Near-fusion-boundary grains for untreated base material are several hundred microns in diameter. Figure 5 shows EBSD inverse pole figure maps of transverse sections near the fusion boundary. The pole figure maps clearly illustrate the difference in near-fusion-boundary microstructure for FSP and nonprocessed material with respect to grain size. Despite coarsening of near-fusion-boundary grains from GTAW, the FSP-pretreated samples exhibit grains on average three times smaller than the GTAW HAZ grains of the non-FSP pre-treated BM. Clearly, FSP is a viable method for inducing HAZ grain refinement and thereby reduce the severity of grain coarsening of a base material before fusion welding.

The HAZ grain size reduction obtainable using FSP pretreatment has the potential to improve fusion weldability issues such as HAZ hot cracking. A number of weldability studies have observed a relationship between near-fusion-boundary HAZ, grain size and liquation cracking susceptibility. In a study by Thompson et al., the HAZ liquation cracking susceptibility of Inconel Alloy 718 was shown to be linearly dependent on grain size (Ref. 22). The benefit of reduced hot cracking susceptibility with finer grain size is attributed to the increased grain boundary area associated with smaller grains. Provided the liquid wets the grain boundary, the larger grain boundary area promotes the spreading of liquid (assuming a constant volume of liquid), thereby reducing the thickness of liquid films present on the boundary. The increased boundary area along with liquid spread across a larger boundary area reduces the strain concentration and crack susceptibility. Because IN600 is not as susceptible to HAZ liquation cracking as other solid solution-strengthened alloys with richer compositions, weldability testing of FSP pretreated IN600 was not performed.

**As-Friction Stir Processed Microstructure of Liquefaction Cracking-Susceptible Alloys**

Examination of stir zone material reveals considerable grain refinement relative to the base material for Hastelloy X, Alloy 625, and Alloy 718 — Fig. 6. Hastelloy X grain size was reduced from 88 μm in the base material to an average of 6 μm. Similar grain size refinement was also observed for both Alloy 625 and 718 — Fig. 6. Average base metal grain size for Alloy 625 and Alloy 718 was 26 and 44 μm, respectively. Compared to the bimodal grain size distributions observed in Alloy 625 and 718 base material, the distribution of grain size in the SZ was considerably more uniform. Lastly, NbC particle size appeared to be unchanged following FSP, suggesting such carbide constituents within the size range observed in the base material (approximately 2–8 μm) are simply translated during stirring and are not broken down mechanically.

**Varestraint Testing of FSP Pretreated Materials**

The susceptibility to HAZ liquation cracking was determined in terms of maximum crack length (MCL) and total crack length (TCL) within the HAZ of the spot weld created during Varestraint testing. The MCL and TCL as a function of strain for the three liquation-susceptible alloys...
are shown in Fig. 7A, B, respectively. Cracking behavior during spot Varestraint testing was found to exhibit a threshold behavior. The threshold strain to induce cracking was found to exist between 1 and 2% strain. For material tested in the as-received condition, the MCL and TCL measurements suggest that HAZ liquation cracking susceptibility is most severe for Alloy 718 followed by Alloy 625 and Hastelloy X. This observation is consistent with thermodynamic predictions of the solidification temperature range (STR). Thermocalc® was used to predict the STR using equilibrium as well as the Scheil-Gulliver model, which assumes complete mixing of solute in the liquid and no diffusion in the solid. Table 3 lists the predicted STR for the three tested alloys. As with the trends in observed magnitude of maximal crack length and frequency, thermodynamic predictions show the largest STR for Alloy 718 followed by Alloy 625 and, lastly, Hastelloy X.

For all three tested alloys at all tested levels of stain, FSP pretreatment decreased the MCL. The reduction in observed MCL is attributed to the reduction in near-FB HAZ grain size as a result of FSP pretreatment. The long (20s) dwell of the spot weld created during the Varestraint test results in coarsening of the original fine grains formed during FSP. However, despite the expected coarsening, near-fusion-boundary grains in FSP pretreated alloys remain 1.9, 1.6, and 1.5 times smaller than nonpretreated base material for Alloy 718, Alloy 625, and Hastelloy X, respectively. Interestingly, large differences exist in the susceptibilities as measured via spot Varestraint testing for the tested alloys in spite of similar near-fusion-boundary grain size after pretreatment. Such differences are related to the inherent differences in alloy composition that lead to varied inherent liquation susceptibility. The largest decrease in MCL was observed for Alloy 718. Pretreatment via FSP decreased the MCL by as much as 30%. The greatest overall improvement in MCL was observed for Alloy 718, which is inherently the most prone to HAZ liquation cracking. Reduction in MCL was also observed for Alloy 625; however, to a lesser degree than Alloy 718. Similar liquating constituents (NbC and Ni3Nb (Ref. 23)) present in Alloy 718 are also present in Alloy 625 although present in smaller quantities due to the leaner alloy composition of Alloy 625 with respect to Nb. Hastelloy X, which contains liquating constituent phases including (M23C6, M6C, Laves, and P Phase) (Refs. 18, 24, 25) shows the smallest reduction in MCL after FSP pretreatment. As with the measured MCL, values obtained for TCL as a function of strain decreased as a result of the FSP pretreatment. Like the value for MCL, the degree of improvement was largely dependent not only on the applied strain level, but also the alloy system. The greatest improvement was realized with Alloy 718. Total crack length for Alloy 718 was reduced by as much as 25%. For Alloy 625, which has liquation cracking susceptibility that lies between Alloy 718 and Hastelloy X, only demonstrated a maximum im-

![Fig. 7 — Spot Varestraint results. A — Maximum crack length; B — total crack length as a function of applied strain for Hastelloy X, Alloy 625, and Alloy 718.](image1)

![Fig. 8 — Arc weld HAZ grain size response to heat input. Solid lines represent FSP material and dashed lines represent base material.](image2)

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The high heat input of the 180-A GTA weld metal fusion boundary with fusion zone to the left of the line.

Fig. 9 — Inverse pole figure maps along the fusion boundary for the following: A — FSP pretreated; B — untreated Hastelloy X base material. Sample sectioned and examined along the ND-RD plane.

The effect of FSP processing parameters (i.e., the effect of starting grain size) was examined using EBSD orientation maps of WM plan sections — Fig. 10. The extent of grain refinement within the WM for the FSP pretreated base material is readily apparent compared to the as-received base material. Figure 11A shows the difference in measured WM grain size using EBSD for the low and high FSP heat input parameters compared to the as-received base material. Interestingly, despite the differences in starting grain size for the two different FSP heat input conditions prior to arc welding, the average WM grain size was quite similar for both conditions prior to arc welding, the average WM grain size was quite similar for both FSP pretreatment conditions. For applications such as repair welding of coarse-grained base materials, FSP pretreatment is a viable solution for local refinement of resulting fusion weld microstructures.

Weld Metal Refinement Via FSP Pretreatment

Also apparent in the orientation maps shown in Fig. 5, the weld metal (WM) grain size is reduced along with the HAZ grain size as a result of the FSP pretreatment. Because the growth of solidification grains in the WM occurs epitaxially (Refs. 26, 27), an increase in growth sites associated with finer grains along the fusion boundary will decrease the WM grain size. The effect of increased epitaxial growth resulting from finer grains is mechanistically analogous to other techniques used to increase heterogeneous growth in welds (Ref. 27).

The effect of FSP processing parameters was examined using EBSD orientation maps of WM plan sections — Fig. 10. The extent of grain refinement within the WM for the FSP pretreated base material is readily apparent compared to the as-received base material. Figure 11A shows the difference in measured WM grain size using EBSD for the low and high FSP heat input parameters compared to the as-received base material. Interestingly, despite the differences in starting grain size for the two different FSP heat input conditions prior to arc welding, the average WM grain size was quite similar for both FSP parameter combinations. Due to the relatively small EBSD scan area and the highly columnar nature of WM grains, measurements of total grain boundary length using EBSD more clearly demonstrate the extent of WM microstructural modification by FSP. Neglecting solidification subgrain (cell and dendrite) boundaries, WM microstructures examined were almost entirely comprised of high angle grain boundaries (> 10 deg misorientation). Total boundary length measurements for the three conditions are shown in Fig. 11B. The WM formed from the low-heat-input FSP condition demonstrated 455% higher total grain boundary length compared to non-FSP base material. Compared to the high-heat-input FSP condition, the low-heat-input FSP condi-
tion exhibited slightly higher total grain boundary length (along with average grain size) likely due to the difference in starting SZ grain size. This refinement of WM grains can be directly attributed to the greater extent of epitaxial growth sites and resulting competitive growth of WM grains from refined fusion boundary grains created by FSP pretreatment.

Face-centered cubic materials such as IN600 do not exhibit strong grain size dependence for Hall-Petch strengthening (Ref. 28). Hardness measurements of the low- and high-heat-input FSP conditions were 73 and 70 HRB, respectively. Without FSP prior to arc welding, the measured WM hardness was 69 HRB. Although large strength improvements are not expected from the extent of weld metal refinement obtained via FSP pretreatment, other mechanical properties such as ductility are expected to improve with increased weld metal grain refinement. As with the HAZ, refinement of WM microstructure has the potential to more effectively accommodate liquid present at grain boundaries as well as reduce grain boundary stress concentrations (Ref. 24). As a result, numerous weldability issues such as weld metal liquation (for multipass welds), ductility dip cracking, and strain age or reheat cracking (in multipass welds) can potentially be minimized (Refs. 24, 29) via reductions in WM grain size.

**FSP Pretreatment in Other Material Systems**

Fiction stir processing pretreatment for HAZ and WM refinement is not limited to Ni-alloy systems. Other systems that are prone to significant coarsening of HAZ and WM microstructure resulting from fusion welding are expected to have a similar benefit. Titanium alloys, for example, are especially susceptible to severe coarsening of weld metal grains (prior beta grain size). For applications requiring good resistance to fatigue crack initiation resistance, large grain sizes are detrimental to performance (Ref. 30). To reduce coarsening, solid-state joining techniques such as FSW have an advantage with respect to preventing severe grain growth. However, because FSW cannot be applied universally, FSP pretreatment before fusion welding may be advantageous. Figure 12 shows a plan-view section of FSP Ti-5111 (a near-α alloy) with a spot weld placed such that one half is contained within the fine-grained SZ (Ti-5111 SZ grain size 1–2 μm) and the other half is contained within the HAZ, which is comprised of very coarse prior β grains similar to the parent β-processed microstructure. On the FSP side of the spot weld, the near-fusion boundary prior β grains are significantly coarsened; however, the grains remain considerably smaller than those on the non-FSP side. The smaller grains on the FSP side resulted in increased epitaxial growth sites and an increase in refinement compared to the adjacent side of the spot weld. Weld metal grains solidified from the FSP-pretreated material were reduced in size by nearly an order of magnitude larger in average diameter.

**Conclusions**

Fiction stir processing as a viable method for the modification of fusion weld microstructures was successfully demonstrated. Refinement of both weld metal and HAZ grain size was achieved by friction stir processing of the base metal prior to fusion welding by autogenous GTAW. As a result of the HAZ thermal excursion from arc welding, fine grains of the FSP stir zone were coarsened by a factor as high as eight depending on location within the HAZ. However, HAZ grains near the fusion boundary for stir-processed material still remained smaller than the base material by a factor of three, depending on alloy system. Heat-affected-zone grain refinement can be expected to have several practical benefits, especially related to the weldability of hot-cracking-

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**Fig. 11** — A — Average WM grain size as measured by EBSD; B — average WM grain boundary length for untreated and FSP pretreated IN600.

**Fig. 12** — A — Optical micrograph of plan view section of GTA spot weld along FSP SZ/BM boundary in Ti-5111; B — higher magnification view near boundary between FSP pretreated and untreated base material. Microstructure revealed using Kroll’s etchant.
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