

Article



# A Microstructural Evaluation of Friction Stir Welded 7075 Aluminum Rolled Plate Heat Treated to the Semi-Solid State

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**Abstract:** Two rolled plates of 7075 aluminum alloy were used as starting material. The plates were welded using a simultaneous double-sided friction stir welding (FSW) process. One way of obtaining feedstock materials for Semi-solid processing or thixoforming is via deformation routes followed by partial melting in the semi-solid state. As both the base plate materials and the friction weld area have undergone extensive deformation specimens were subjected to a post welding heat-treatment in the semi-solid range at a temperature of 628 °C, for 3 min in order to observe the induced microstructural changes. A comparison between the microstructural evolution and mechanical properties of friction stir welded plates was performed before and after the heat-treatment in the Base Metal (BM), the Heat Affected Zone (HAZ), the Thermomechanically Affected Zone (TMAZ) and the Nugget Zone (NZ) using optical microscopy, Scanning Electron microscopy (SEM) and Vickers hardness tests. The results revealed that an extremely fine-grained structure, obtained in the NZ after FSW, resulted in a rise of hardness from the BM to the NZ. Furthermore, post welding heat-treatment in the semi-solid state gave rise to a consistent morphology throughout the material which was similar to microstructures obtained by the thixoforming process. Moreover, a drop of hardness was observed after heat treatment in all regions as compared to that in the welded microstructure.

**Keywords:** 7075 aluminum alloy; rolling; post-welding-heat treatment; friction stir welding (FSW); Semi-solid processing

## 1. Introduction

AA 7075 (Al-Zn-Mg-Cu) alloy is a heat treatable alloy possessing remarkable strength due to the precipitation of MgZn<sub>2</sub> phase when Zn to Mg ratio is 1:2 or 1:3. This class of aluminum alloys is widely used in the aerospace and automotive industries where parts must have particular properties to meet the required demands as well as being reliably weldable and machinable [1–3]. Among welding techniques, friction stir welding is well suited for joining of aluminum alloys, as the process leads to the grain refinement in the nugget zone, with potential to further improve the strength of aluminum alloys [4,5].

Friction Stir Welding, as a severe plastic deformation approach, has been considered the most important welding technology during the last twenty years. FSW is defined as a solid state joining process in which the workpieces being welded are not melted [6–8]. Continuous improvements to the performance of FSW joints during the process, has been achieved by variations of the process, as for instance, double-sided FSW that offers advantages over conventional FSW methods such as higher transverse speed and even distribution of heat input into the workpieces to be joined [9–11].

Over the last few decades, many studies have been conducted on FSW in order to make use of its advantages, and more recently researchers have focused on the effects of heat treatment of FSW joints

to investigate the stability of the very fine grain structure obtained by FSW at elevated temperatures. A common observation that has been reported after a post welding heat treatment is the abnormal grain growth in the nugget zone that might have negative effects on the mechanical properties of these welds [4,12].

The aim of the present study is to investigate the effects of FSW on the microstructure and mechanical properties of welded plates of AA 7075 along with the evaluation of a post welding heat treatment in the semi-solid region, at a temperature of 628 °C. Optical microscopy, Scanning Electron Microscopy and Vickers hardness tests of the welded and heat-treated specimens have been employed to achieve these goals.

#### 2. Materials and Methods

One way of obtaining feedstock materials for Semi-solid processing or thixoforming is via deformation routes followed by partial melting in the semi-solid state. It has been observed by many researchers that [13–15] materials that have undergone deformation can be manipulated by heat treatment in the semi-solid region to obtain a non-dendritic near spheroidal microstructure that is the key to Semi-solid processing or as it is more generally known, thixoforming. Such near spheroidal microstructures induce materials to behave thixotropically when placed under shear, therefore allowing them to be shaped into complex near net-shaped products. It was decided to subject both the base plate materials and the friction weld area to a post welding heat-treatment in the semi-solid range at a temperature of 628 °C, for 3 min in order to observe the induced microstructural changes, as they all have undergone extensive deformation during their processing. All specimens were heated treated at 628 °C, which is between the Liquidus and Solidus for this particular alloy, for three minutes before quenching in water as a lot of information exists on microstructures obtained under similar conditions [16–18].

In detail, two rolled plates of 7075 wrought aluminum alloy, 62 mm thick, were welded using FSW at TWI Rotherham, using a simultaneous double-sided technique with a coarse threaded TriFlat probe mounted in a 40 mm concave shoulder employed as a stirring tool. The transverse speed of 100 mm/min was applied, the lower tool lead the upper tool by 15 mm and the weld was force cooled by circulating ethylene glycol using copper bars in intimate contact with the workpiece immediately adjacent to the two weld lines. This technique was used to try and maintain the properties of the parent material as much as is possible. Welding thick material requires a high heat input to plasticise the metal and can potentially reduce the alloy's properties as elements close to the weld are taken back into solution and then re-precipitate. By using two tools to make a simultaneous double pass along the weld line the heat input was evened out to try and give a more even distribution of heating through the material thickness than would otherwise be the case if a full penetration single pass weld was made. Traversing the tools quickly limits the time for which the metal is heated locally, and force cooling close to the weld line extracts heat that is no longer performing a useful function (plasticisation).

Typically we see grain refinement in the thermomechanically affected zone. There is the potential for grain growth in the HAZ but high weld travel speeds and forced cooling minimize this.

The chemical composition of the alloy is shown in Table 1 [2].

Table 1. Chemical composition of wrought 7075 Al alloy (mass %) used in this study [2].

Alloy	Cu	Zn	Mg	Mn	Cr	Ti	Si	Fe	Zr	В	Al
7075	0.94	4.52	2.24	< 0.01	0.22	0.05	0.02	< 0.01	< 0.01	< 0.01	Bal.

The microstructure of the welded plates was investigated using standard metallography with specimens from both the weld line and parent material being prepared and ground with standard SiC grinding paper and polished with 6  $\mu$ m and 1  $\mu$ m monocrystalline diamond suspension and 0.05  $\mu$ m silica suspension. Specimens were etched using 10 g sodium hydroxide diluted with 100 mL water. Using Nikon Eclipse LV150 optical microscopy and TM3030Plus Tabletop Scanning

Electron Microscope, the microstructure of the specimens was evaluated in the BM, HAZ, TMAZ and NZ.

Image J software was used to measure the average grain size from the optical images using the linear intercept method. The Vickers hardness test was conducted using Zwick hardness tester with 10 kgf applied force for 10 s across the plate in various locations and mean values calculated. To investigate the effects of the post welding heat treatment at semi-solid temperature on the microstructure and mechanical properties of the welded plates, specimens were heated to 628 °C and kept at temperature for 3 min followed by immediate quenching in water.

# 3. Results and Discussion

## 3.1. Microstructural Evolution of the Friction Stir Welded Plates

Figure 1 shows the optical micrographs of the two plates that have been successfully friction stir welded. As can be seen from 1a, the microstructure contained the following zones: Nugget Zone (NZ), Thermomechanically Affected Zone (TMAZ) and the Heat Affected Zone (HAZ). In FSW, a tool is embedded on a rotating probe that moves along the length of the plates to be welded and a nugget zone is produced by the interference between the welding tool and the workpieces during the stirring stage. Moreover, the TMAZ is generated by the friction between the tool shoulder and the top plate surface as well as the contact between the adjacent material and the edges of the tool [19].



**Figure 1.** Optical micrographs of 7075 aluminum alloy after FSW showing (**a**) different zones formed after FSW; (**b**) weld area; (**c**) pancake-shaped grains in BM and (**d**) ultra-fine grains in the NZ.

In the TMAZ, the material experiences lesser amount of strain and strain rate as compared to the NZ and the pancake-shaped grains along the interference between the NZ and TMAZ are elongated and bent, so it does appear that FSW causes drastic distortion in the elongated grains [9,20]. In addition, the grain structure in the HAZ is closer to that in the base metal than other regions [21,22].

Figure 1b shows the weld area, where it can be observed that the weld line is free of common defects present in conventional welding processes including intergranular cracks. The base metal consists of pancake-shaped grains elongated in the rolling direction, as can be seen in Figure 1c, and the grain structure in the nugget zone, shown in Figure 1d, shows that the grains are extremely fine, so much so that the grain size could not be measured by conventional optical methods. However, in literature, an average 0.5–10 µm is reported for the grain dimension in this area after a single pass of FSW [21–24]. Generally, the grain size in the NZ is determined by two main thermomechanical factors; the heat input and the degree of plastic deformation. Therefore, during FSW the elongated grains of the parent material are mechanically turned into a fine grain structure as a result of the extensive plastic deformation that the material undergoes during the process as well as the heat generated during the welding process. Recovery and recrystallization in this zone which can be accompanied by an improvement in the mechanical properties [19,21,25]. Atul Kumar et al. [25] have conducted FSW of aluminum 7075 alloy with different transverse speeds, from 25 to 85 mm/min, and suggest that the recrystallized grain size is a function of transverse speed and that higher transverse speeds give rise to reductions in heat input per unit length and therefore prevent the recrystallized grains to coarsen. Thus, the formation of the extremely fine grains in the NZ of the present study can be attributed to the high transverse FW speed used (100 mm/min). In addition, there is no evidence of recrystallization in the TMAZ, due to the lower temperatures caused by friction stir welding process, although the material undergoes plastic deformation due to the relatively high amount of heat during the welding [20].

#### 3.2. Post Welding Heat Treatment

Optical microscopy images of heat-treated friction stir welded specimens can be seen in Figure 2. Specimens were prepared from along the length of the joint from the top to the bottom of the welding line in order to evaluate the influence of the heat treatment on the microstructure in the various zones: NZ, TMAZ, HAZ and BM.

Optical microscopy shows a considerable change in the grain structure and morphology of the specimens from the various zones throughout the microstructure. As was shown in Figure 1c, the base metal contained pancake-shaped grains that were elongated in the rolling direction, and the structure of grains in the HAZ was also the same as that in the BM. In addition, the TMAZ consisted of the pancake like grains that were bent due to the high plastic deformation during FSW, and a NZ with extremely fine grains. However, after heat treatment, the morphology of grains in the BM, TMAZ and HAZ is converted to a near spherical fine grain structure that is uniformly distributed throughout the material. Furthermore, the grain structure in the NZ is similar to that of the other regions, and the effects of the FSW cannot be observed on the microstructure in this region and in addition, substantial grain growth was observed in the NZ. Cerri et al. [4] also reported abnormal grain growth in the NZ after post weld heat treatment at different temperatures up to 300 °C for the double-lap FSW joint of Al 2024T3 and 7075T6 alloys.

From the optical micrographs presented in Figure 2a,b, it can be seen that heat treatment at 628 °C resulted in a microstructure which is very similar to that obtained by the thixoforming process, a process that shapes metals in the semi-solid state. In addition, SEM images shown in Figure 2c, demonstrate a consistency in the material microstructure after the post welding process. Grain size was also measured at different areas of the specimens showing a uniformity of the microstructure after heat treatment with an average grain size of around 43 µm.

In the thixoforming process, one way of generating the prerequisite non-dendritic microstructure that is the key to the process, is through a deformation route followed by a heat treatment in the semi-solid temperature range that allows for recrystallization and partial melting along high angle grain boundaries. Previous work has shown that around 628 °C from DSC data there is around 48% liquid formed and that the main phases present, in very small quantities are Mg<sub>2</sub>Si, Al<sub>13</sub>Fe<sub>4</sub> and Al<sub>6</sub>Mn [16,26]. The resulting near spheroidal non-dendritic microstructures exhibit thixotropic

properties, i.e., the material behaves like a solid if left undisturbed but flows like a liquid when under shear. It is this particular behaviour of the non-dendritic microstructures that has allowed such materials to be shaped in the semi-solid state into complex near net-shape products under low injection forces. Of course the deformation route is one of a number of possibilities that are available in delivering non-dendritic microstructures, one of the more interesting ones is that of spray forming, where molten metal is gas atomized and accelerated towards a substrate where it builds up into a solid billet. The shape of substrate depends on the particular application, i.e., billet, bar, tube etc. [27]. The microstructures of spray formed materials are homogeneous and quite fine and when re-heated to their semi-solid state become near-spherical microstructures typical for thixoforming processing.

Examples of similar studies on the microstructures of extruded and spray formed materials show typical non-dendritic near spheroidal microstructures following thixoforming as illustrated in Figure 3.



**Figure 2.** Optical microscopy images of heat-treated specimens illustrating the microstructure of the material (**a**) top, middle and bottom of the welding line; (**b**) higher magnification of same images of material in top, middle and bottom of the welding line (as described in Figure 4) and (**c**) SEM images of the specimens taken from similar regions.



**Figure 3.** Examples of thixoformed microstructures obtained from: (**a**) extruded and (**c**) sprayformed feedstock materials; (**b**,**d**) show the resultant microstructures after thixoforming [20].

### 3.3. Vickers Hardness Results

Figure 4 represents results of the Vickers hardness test performed for the FS welded plates of aluminum 7075 alloy. The hardness measurements were conducted on specimens selected from various parts along the weld joint as shown in Figure 4a. It can be observed that the graph follows an upward trend from the base metal to the nugget zone implying that the hardness in the NZ is higher than that of the TMAZ and a decrease in the BM. Broadly speaking, some factors such as grain size, morphology and interface features of the precipitates can determine the hardness profile for the heat treatable aluminum alloys. These factors are controlled by heat input, cooling rate of the material and resultant temperature generated during and after the FSW [25]. Therefore, as aluminum is a relatively soft material, lower amount of frictional forces are required before the material starts to plasticize. Hence, temperatures that the workpiece experiences during the welding are relatively low compared to those of hard materials, leading to grain refinement in the NZ that gives rise to the improvement of properties including hardness and strength in this region [21]. In this study the simultaneous double-sided welding technique is followed by rapid cooling along the weld line and the process is completed in a short time with heat distributed evenly, resulting to improved mechanical properties in the NZ. However, a drop of hardness in HAZ/TMAZ and NZ relative to the parent material has been reported by some researchers, where it has been explained that the hardness decrease in HAZ/TMAZ is associated with the dissolution or coarsening of the strengthening precipitates due to the relatively high temperatures that materials experience in these regions, and the high cooling rates preventing precipitation of phases leading to a lower amount of hardness in HAZ/TMAZ [24]. Moreover, hardness reduction in NZ has been described due to the same phenomena. However, the fine-grained structure in this area causes higher hardness as compared to that in the HAZ/TMAZ [24].



**Figure 4.** (a) Schematic illustration showing two plates of Al 7075 alloy welded by FSW; (b) hardness gradient for different points of the material; values are the average of three readings.

Figure 5 shows graphs of hardness for heat-treated samples of FS welded plates that were prepared along the length of the weld line. The hardness tests were performed from the edge over the length of specimens. As can be seen, unlike the results of the hardness obtained for FS welded specimens before the heat treatment that presented an upward trend from the BM to the NZ, hardness values are scattered and do not follow a particular trend. However, both graphs have a similarity related to hardness numbers for specimens analyzed from the middle of the weld zone showing higher hardness than both top and bottom regions before and after the heat treatment. This can be justified due to the friction stir welding process, as the impact of two stirring tools on the middle of the weld line is higher than the two sides and the material in this region experiences more shear forces.



VICKERS HARDNESS

**Figure 5.** Graph of hardness versus position from the weld centre for Al 7075 alloy. FS welded plates after heat treatment at 628 °C.

From the graph, it can be observed that mean hardness values presented for top and bottom of the weld are roughly the same and average hardness numbers for the middle of the weld are higher than both top and bottom regions. In addition, the maximum peak is 150 HV on the graph related to the middle of the weld, which is still lower than the maximum hardness that were obtained in the nugget zone of the specimen from the same region of the weld before heat treatment. The reason for this can be explained by significant grain size growth after heat treatment at 628 °C as compared to ultra-fine grains in the NZ of the specimen before the heat trial. Cerri et al. [4] conducted a post welding heat

treatment for the same alloy of aluminum at different temperatures and reported a hardness decrease in the NZ after heat treatment at 300  $^{\circ}$ C due to grain coarsening in this region.

It should be noted that low melting point constituents are melted during the heat treatment and some of the second phase comes into solution but that the bulk of the material remains solid. The incipient melting of the low melting point phase allows penetration around high angle grain boundaries to take place resulting in the 'spheroidisation' of the grain structure as seen after the heat treatment.

### 4. Conclusions

Plates of aluminum 7075 alloy have been welded by a simultaneous double-sided friction stir welding technique. Because these welds undergo extensive plastic deformation, a post welding heat treatment was conducted in the semi-solid region at a temperature of 628 °C, in order to investigate if the resultant microstructures develop in a similar way those encountered in semi-solid metal processing or thixoforming.

The following conclusions can be derived from this study:

- (a) The base metal consists of typical pancake-shaped grains elongated in the rolling direction but the grain size is significantly decreased in the nugget zone due to extensive plastic deformation that the materials undergo during FSW;
- (b) Grains start to grow throughout the material during heat treatment at 628 °C, resulting in uniform non-dendritic microstructures similar to those obtained by thixoforming;
- (c) Hardness tests show that hardness in the NZ is higher than that in TMAZ and BM due to the ultra-fine grain structure attained in this area.

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Conflicts of Interest: The authors declare no conflict of interest.

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