Pre and post-welding annealing influence on the microstructure of a 7475-T7351 aluminum alloy joining by friction stir welding

Influência do recozimento pré e pós-soldagem na microestrutura de uma liga de alumínio 7475-T7351 que se une por fricção

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Carolina Alencar Caldeira de Souza
Graduação em Engenharia Metalúrgica
Instituição: Instituto Militar de Engenharia (IME)
E-mail: carolina.souza@ime.eb.br

Saulo Brinco Diniz
Doutorado em Ciência dos Materiais
Instituição: Centro Federal de Educação Tecnológica Celso Suckow da Fonseca (CEFET-RJ)
Endereço: R. do Areal, 522 - Parque Perequê, Angra dos Reis - RJ, CEP: 23953-030
E-mail: saulo.diniz@cefet-rj.br

Angelo Siqueira da Silva
Mestrado em Ciência dos Materiais
Instituição: Instituto Militar de Engenharia (IME)
E-mail: angelosqr@gmail.com

Andersan dos Santos Paula
Doutorado em Ciência dos Materiais
Instituição: Instituto Militar de Engenharia (IME)
E-mail: andersan@ime.eb.br

Fabiane Roberta Freitas da Silva
Doutorado em Engenharia Metalúrgica
Instituição: Universidade Federal Fluminense (UFF)
Endereço: Avenida dos Trabalhadores, 420 - Vila Santa Cecília, Volta Redonda, RJ
CEP: 27255-125
E-mail: fabianesilva@id.uff.br

Luiz Paulo Brandao
Graduação em Engenharia Metalúrgica e de Materiais
Instituição: Instituto Militar de Engenharia (IME)
E-mail: brandao@ime.eb.br
ABSTRACT
Friction Stir Welding (FSW) promotes the joining of sheets due to localized plastic deformation and heating by displacing a non-consumable tool. This work aimed to verify the influence of an annealing heat treatment performed at 300 and 500 ºC with 15 minutes of soaking times, before and after the FSW of a 7475-T7351 aluminum alloy. The macrographs of the welded joint (top and cross section) and micrographs of the cross section was obtained by a stereoscope and optical microscope were analyzed and the mechanical behavior was characterized by uniaxial tensile tests. It was concluded that the annealing heat treatments did not influence significantly the nugget grain size, but there are indications that the realization of these before the FSW, provide a homogeneity in the mechanical properties of the welded joint and the base material.

Keywords: friction stir welding, annealing, 7475-t7351 aluminium alloy.

RESUMO
A soldagem por fricção (FSW) promove a união de chapas devido à deformação plástica localizada e ao aquecimento através do deslocamento de uma ferramenta não consumível. Este trabalho teve como objetivo verificar a influência de um tratamento térmico de recozimento realizado a 300 e 500 ºC com 15 minutos de tempo de imersão, antes e depois do FSW de uma liga de alumínio 7475-T7351. As macrografias da junta soldada (topo e seção transversal) e micrografias da seção transversal foram obtidas por um estereoscópio e um microscópio ótico foram analisados e o comportamento mecânico foi caracterizado por testes de tração uniaxial. Concluiu-se que os tratamentos térmicos de recozimento não influenciaram significativamente o tamanho do grão da pepita, mas há indícios de que a realização destes antes do FSW, proporciona uma homogeneidade nas propriedades mecânicas da junta soldada e do material de base.

Palavras-chave: soldagem por fricção, recozimento, liga de alumínio 7475-t7351.

1 INTRODUCTION
Aluminum is one of the most used metals due to several advantages [1-3], in which among the aluminum alloys, the 7XXX series alloys stand out. These alloys have a good strength/weight ratio, for this reason 7XXX series alloys are widely used in aircraft manufacturing. The aeronautical application of these alloys requires the use of joining processes such as welding [4-6]. However, when it comes to fusion welding, aluminum does not produce good results [7], thus generating structures with loss of mechanical properties, mainly due to solidification cracks and porosity in the melting area [8]. In particular, in 7XXX series alloys the problems are associated with the formation of zinc oxide during welding, cracking by solidification and brittle fracture. Thus, fusion welding process in 7XXX series aluminum alloys is not feasible for aeronautical applications [9].

The Welding Institute (TWI) has developed an alternative for welding aluminum alloys to be carried out in the solid state, and thus eliminates the solidification problems
that hindered their welding. This welding technique is called Friction Stir Welding (FSW), that consists of using a non-consumable tool which rotates in relation to its axis itself and that is inserted in the welded joint, whose movement and the heat generated by the friction promote a great plastic deformation and the mixing of the materials resulting in the joining of the plates without the occurrence of fusion [7,8,10].

The FSW results depends on the appropriate choice of welding parameters to be used, such as: traversing speed of the tool (welding speed) [11], rotational speed [7,11,12], tool penetration depth [11], joint type [7], tool inclination angle [13], and one of the most important is tool geometry [7,11,14]. With the correct choice of welding parameters, it is possible to get a joint in the welding region with a greater mechanical resistance than the base material (BM). However, in some cases depending on the chosen parameters, the resistance of the welded joint may be less than the BM, even if the visual appearance of the welding does not show defects [15]. Therefore, heat treatments become a viable alternative in some alloys in order to adjust the obtained properties according to the required requirements.

Thus, the main objective of the present investigation is to evaluate the effects of annealing heat treatments before and after the FSW on the microstructure of a 7475-T7351 aluminum alloy. This study presents a great relevance, as few researches have been found in the literature that present results of FSW and heat treatments being conducted on the 7475-T7351 aluminum alloy.

2 DEVELOPMENT

2.1 MATERIAL

For the present study, the 7475-T7351 aluminum alloy with a chemical composition of 5.67%p Zn, 2.38%p Mg, 1.78%p Cu, 0.20%p Cr and 0.28%p Fe+Si (% by weight) was conventionally cold rolled to 3.1 mm thickness (condition called C).

2.2 METHODS

Figure 1 shows the flowchart of the welding conditions and heat treatments that gave rise to the nomenclatures used in this article.
Figure 1. Flowchart of heat treatments and welding conditions.

Annealing heat treatments were performed at 300 and 500 °C, with a soaking time of 15 minutes and subsequent air cooling. For this stage, 2 different furnaces were used: before to welding, 50 x 100 mm (width x length) plates were heat treated in an electrical resistance furnace (manufacturer FDG, model 3000 - called 3FX and 5FX conditions). For the heat treatment after welding samples with dimensions of 4 x 50 mm (width x length) an electric resistance furnace was used (manufacturer NOVA Instruments, model NI 1385 - called XF3 and XF5 conditions).

The following parameters were used to achieve the welds by FSW: tool rotational speed of 2500 rpm, welding speed of 50 mm/min at 90° in relation to the plate rolling direction, 0.1 mm shoulder penetration, anti-clockwise direction of rotation, 3° tool angle and a top joint without chamfer. All the samples of the different conditions (3FX, 5FX, XFX, XF3 e XF5) were performed with one equipment (Model GG-7, Manufacturer MTI). The tool used in the present study presents a geometry composed of a 12 mm diameter shoulder with undercuts with rings shape and a concentric pin to the shoulder in conical format with bases of 5 mm and 4 diameters, threaded [12]. After the FSW, the plates were conducted for visual analysis, where a mobile phone camera (13 Mpixels) was used to record the images that portray the aspects on the upper and lower faces of the welded plates.

Samples with dimensions of 4 x 50 mm (width x length) were cut and embedded with acrylic resin of cold curing in order to analyze the transverse direction face of the weld line. Later, they were carried out to metallographic preparation. Grinding was performed with silicon carbide sandpaper from 80 to 2000 mesh and mechanical polishing with a 1/4 µm diamond paste. The chemical etching was performed with a KELLER solution (2.5% HNO₃ + 1.5% HCl + 1.0% HF + 95% H₂O) during approximately 60 seconds, with subsequent immersion in a 3% Nital solution (3% HNO₃ + 97% C₂H₂OH) for approximately 20 seconds.

Macro analyses were performed using a stereoscope, with a magnification of 1.25x. The microstructural analyses were performed using an optical microscope.
(Olympus Manufacturer, Model BX51M) with a photo camera (Model OLYMPUS, Manufacturer SC30) and a Stream Basic2 micrograph acquisition software. The micrographs photos were taken at ½ of sample thicknesses with magnifications of 50, 200, 500 and 1000x in four different positions in order to get information of different regions: (i) BM; (ii) BM, Thermo-Mechanically Affected Zone (TMAZ) and Nugget; (iii) BM or Heat-Affected Zone (HAZ), TMAZ, Nugget and the Shoulder Passage Affected Zone (SPAZ) and (iv) Nugget.

For the grain size calculation, micrographs of the Nugget regions (in a central position 1.5 mm thick, and ½ width) and BM of each specimen were used. The Heyn Intercept method was carried out with 5 lines in the X direction and 5 lines in the Y direction.

With the aim of characterizing the influence of annealing heat treatment on the joint by FSW, uniaxial tensile tests were performed on a universal testing machine (EMIC manufacturer, model DL30000). Cell phone camera was used to record the exact moment of specimen rupture. One sample (according to ASTM E08-M - subsize specimen) was used for each condition for a test velocity of 1 mm/min. The welded region was located in the central region of the gage length of the specimen.

2.3 RESULTS AND DISCUSSION

In Figure 2 (a), (b) and (c) are presented the visual aspects of the upper and lower face of 7475-T7351 aluminum alloy plates welded by FSW for XFX, 3FX and 5FX conditions, respectively. As the XF3 and XF5 conditions were generated from the heat treatment of the XFX condition, both visual aspects area similar. It can be observed that the welds carried out did not give rise to defects in the surface of the welds such as cavity defects [17] or the expulsion of material of the weld line [18].

However, there is a small number of barbs, observed in the region of beginning, middle and end of welding, common in plates joined by FSW. It is also observed that there is no surface defect along the root surface, so the good results obtained can be attributed to a correct choice of welding parameters used and the geometry of the chosen tool. Rotational speed is also an important factor for good welded joint quality [12]. Silva (2018) [17] performed the FSW union of 7475-T7351 aluminum alloys and found a higher presence of cavity-type defects in welded samples with lower rotational speeds. These defects may be caused by the low deformation and mixing of material due to the low heat generated.
On the other hand, in 2019, Souza and collaborators [12] found that excessive rotational speeds can also generate superficial defects, such as large amounts of barbs. Thus, as the joints of the present investigation did not present cavity defects or large number of barbs, it can be concluded that the rotation speed used was adequate to generate the necessary heat for plastic deformation.

The tool speed also plays an important role in welding results [11]. Silva (2018) [17], found that high feed speeds cause a greater number of defects. The reason is that with higher weld speeds, less amount of friction and heat is generated, reducing plastic deformation and material mixing. In this way, it can be concluded that, since there is no presence of superficial defects, an adequate advancement speed was appropriated selected to the process.

One of the best parameters found by Silva (2018) [17] in the execution of the FSW in 7475-T7351 aluminum alloys were 2500 rpm and 50 mm/min - the same ones used in the present research, where, as can be observed in the samples presented in Figure 2, no superficial defects were found. However, although the superficial aspect did not present cavity type defects, these can appear in the cross section what can be detected through the macroscopic analysis via stereoscope. Thus, it is necessary to evaluate the cross section of the welds.

Figure 3 shows the macrostructural aspects of the cross sections of the conditions visualized by stereoscope for the sample (a) XFX, (b) XF3 and (c) XF5. The 3FX and 5FX specimens presented an aspect like the XFX sample. They are presented with large magnification (50x) in Figure 3 (d) and (e). There, some regions of interest are
highlighted: AGG (Abnormal Grain Growth - abnormal grain growth region), BM, HAZ, TMAZ and Nugget

Figure 3. Cross section macrostructure for conditions (a) XFX, (b) XF3 e (c) XF5, e microstructure (50x) of conditions (d) XF5 e (e) XF3.

Figures 3 (a), (b) and (c) show that the welded samples did not show defects that are common in FSW welded samples such as: worm holes [19]; lack of penetration into the root surface [20]; and Kissing bond size [20-23].

In condition XF5 (Figure 3 (d)) the presence of larger grains size is observed in the upper region of the Nugget, where selective grain growth is reported in the literature as AGG (Abnormal Grain Growth) [24].

Through the variation of the rotation and welding speeds, Attallah (2004) [24] observed changes in the AGG area, and in this way, it can be concluded that the welding parameters can also influence the formation of AGG. Since, in the present investigation, the welding parameters were kept fixed and only for the XF5 condition there was the formation of AGG, this fact can be attributed to the heat treatment performed after welding at 500°C. The same behavior was observed by Attallah (2004) [24] after a heat treatment at 510 ºC in a 2XXX series aluminum alloy welded by FSW.

In Figure 3 (e), 4 distinct regions are highlighted in a joint welded by FSW. There the BM is the region that has not been influenced by the weld process, that is, it is not affected by heat or deformation resulting from the process [25]. In this way, Figure 4 presents in greater detail the BM microstructures of the conditions (a) XFX, (b) XF3, (c) 3FX, (d) XF5 and (e) 5FX.
There was no significant change in the grain aspects, which are elongated in the rolling direction, for XF3, 3FX and 5FX conditions in relation to the XFX condition. This modification can be due to a recrystallization process of the BM, since it was in the hardening state before the heat treatment was carried out at 300 and 500 ºC. In the future, analyses with higher magnification will be performed by scanning electron microscopy to confirm this fact.

On the other hand, it can be observed in the microstructure presented in Figure 4 (d) that the heat treatment at 500 ºC, after welding (sample XF5), promoted a significant modification of the BM by recrystallization of the grains. Probably, the presence of recrystallized grains in the BM of the XF5 sample and the non-recrystallization of the BM of the 5FX sample is due to the use of two different furnaces for heat treatments. The furnaces have different the heating rates because of its characteristic and chamber sizes and, in addition, the different re-heating capacity of each furnace and its ability to re-establish the temperature after each opening and closing during the sample replacements.

Between the BM and the TMAZ, there is the HAZ which is not affected by the deformation process, however it still suffers heat influence [7]. The border between HAZ and BM microstructural area is uncertain (the green line being only an indication) since is not possible observe a microstructural modification by optical microscope.

TMAZ is an affected area by both heats generated in the process and deformation generated by the tool [7]. Thus, the influence of TMAZ deformation can be observed when compared with BM, where grains are aligned in the rolling direction while the TMAZ grains present a direction change, indicating that they were deformed during the welding process. In this way, it is possible to infer that the TMAZ zone begins to the right of the blue line (Figure 3 (e)), where it is observed the beginning of the deformation of the grains, and ends in the yellow dashed line, where the Nugget area begins.

In order to identify the possible influence of the heat treatments conducted at 300 and 500 ºC before and after the weld by FSW, in Figure 5 are presented the micrographs...
with 500x of the Thermo-Mechanically Affected Zone (TMAZ) for the samples (a) XFX, (b) XF3, (c) 3FX, (d) XF5 and (e) 5FX.

It is possible to note in Figure 5, the grain deformations (being these inclined in relation to the rolling direction) and the influence of temperature, evidenced by the existence of some recrystallized grains. The recrystallization requires relatively high temperature to occur, that could be arised either from heat treatments or from friction arising from a deformation process [26].

![Figure 5. Micrographs of TMAZ (500x) for samples (a) XFX, (b) XF3, (c) 3FX, (d) XF5 e (e) 5FX.](image)

During the FSW, there was simultaneous microstructural changes in the Nugget region such as hardening, recovery and crystallization due to the deformation caused by
the pin and the heat generated in the process. When these phenomena occur during deformation, they are called dynamic recovery and dynamic recrystallization, respectively [26]. In dynamic recrystallization the nucleation of the grains occurs by the mechanism known as "necklacing", which occurs preferentially in the grain boundary in form of successive "collars" causing an intense grain refining.

Table 1 and 2 show the average grain size values at the central region of the Nugget and at the BM regions of the different samples. For the average grain size of Nugget, it should be noted that close values were obtained for all samples, independent of temperature and heat treatment order. This is possibly because the recrystallized grain size is more sensitive to deformation and less sensitive to temperature [26]. As the plates were rolled under the same conditions and the welds were carried out with the same parameters, the same deformation resulting from the process was imposed on all samples, and thus there was little change in grain sizes.

| Direction | Samples  
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<tr>
<td></td>
<td>XFX</td>
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<tr>
<td>X</td>
<td>1.78 ± 0.17</td>
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<tr>
<td>Y</td>
<td>1.39 ± 0.16</td>
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Table 1. Nugget grains sizes in X and Y directions (µm)

| Direction | Samples  
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<tr>
<td></td>
<td>XFX</td>
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<tr>
<td>X</td>
<td>&gt; 300</td>
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<tr>
<td>Y</td>
<td>5.58 ± 0.78</td>
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Table 2. BM grains sizes in X and Y directions (µm)

For the XFX and 5FX samples, it was not possible to identify the beginning and end of the grains in the micrographs. Because of it, it was not possible to measure the grain size in X direction. Therefore, the value of 300 µm was assigned, which corresponds to the width of the micrograph corresponding to the magnification applied. For the XF5 sample, a mean value of 22.22 ± 5.50 µm was obtained for grain size, which is much lower than that attributed to the other samples.

On the other hand, for the average grain size in the Y direction, there was a variation in the values obtained, which may be justified by the limitation of the optical microscope used and by the difficulty of identifying the grain boundaries in this orientation (Figure 4) mainly for XFX and 5FX samples.

In general, the average grain sizes for all samples were significantly reduced from BM to Nugget regions, which can be justified due to the dynamic recrystallization
mechanism which is known for refining the microstructure. In addition to being very refined, the Nugget microstructure is also equiaxial, as can be verified in both Table 1 and Figure 6.

Figure 7 shows images of the samples welded used in the uniaxial tensile test at the exact moment just after the fracture (indicated by a yellow arrow).

Figure 7. Fracture site of specimens subjected to the tensile test for samples: (a) XFX, (b) XF3, (c) 3FX, (d) XF5 e (e) 5FX.

It can be observed from the images that the 3FX and 5FX conditions were the only ones that presented the rupture in the BM area, that is, outside the welded joint. This fact is indicative that, although there is no significant difference that could be observed on the microstructure by optical microscopy, probably the annealing heat treatment before or after the FSW modified the mechanical strength of the BM region.

In future studies, uniaxial tensile tests will be performed in 3 or more specimens for each condition, as well as for the BM, in such a way that one can better understand influence of the heat treatment before or after the FSW in the mechanical properties obtained by the tensile test and also avoid suspicion of a fracture due to problems in machining test specimens.

3 CONCLUSION

According to the results presented, it can be concluded:
- the welds did not show defects in the visual appearance of the surface of the joints by FSW;
- the cross-sectional appearance of the samples did not show welding defects for all samples;
- heat treatment at 300 °C before or after FSW did not provoke recrystallization in samples base material;
- heat treatment at 500 °C after the FSW promoted the recrystallization of the base material grains;
- there was no significant change in the grain size of the central Nugget region for the studied samples. However, there was a considerable reduction in the average grain size from the BM to Nugget for the XFX, 5XF and XF5 samples;
- heat treatments before FSW probable took more homogeneous mechanical properties between base material and welded region.

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