Tailoring the Microstructure of an AA5754 Aluminum Alloy by Tuning the Combination of Heat Treatment, Friction Stir Welding, and Cold Rolling

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Abstract: Friction stir welding (FSW) has now reached a technological impact and diffusion that makes it a common joining practice for several classes of metallic materials. These include light alloys (aluminum, titanium, magnesium), steels, and other metallic alloys. In addition, the combination of FSW with pre- or post-welding heat treatments or plastic deformation, such as cold rolling (CR), can favor minimal necessary plate thicknesses and induce effective alloy strengthening mechanisms that make the FSW joint lines as plate reinforcing zones. Process parameters, such as pin rotation and transverse speed, can be tuned to optimize the mechanical properties of the resulting joint. This work presents a microstructural study of the mechanical response of different sequences of heat treatment, FSW, and CR in a non-age hardened Al-Mg AA5754 alloy. By using polarized optical microscopy and microhardness tests, two FSW conditions were used to fabricate a joint; and were than subjected to different sequences of heat treatment and cold rolling. The results suggest that FSW conditions have a limited effect on the microstructure, while microhardness profiles show a higher variability of the different datasets related to the low welding speed investigated.

Keywords: heat treatments; friction stir welding; cold rolling; optical microscopy; microhardness

1. Introduction
Friction stir welding is a very useful process to produce highly performing joints whose application is rapidly taking advantage in some sections of the welding market [1] as a green and environmentally-friendly alternative compared to the classic and more widespread welding technology; since it does not involve consumables, such as shielding gases or electrodes, such as in the case of gas metal arc welding (GMAW) or laser beam welding [2]. These allow the joining of dissimilar metals and alloys, a topic which is still of very large interest also in the case of friction stir welding [3–8].

This particular welding technique is a solid-state joining process based on the friction exerted by a rotating non-consumable tool shoulde r with a pin [1,9]. A mechanical pressure is applied to the pin which is inserted on top of the surface of the metal plates; the plates need to be joined and then move horizontally to mechanically mix the base material, which is softened by the action of heat generated through the friction of the overall tool. The final FSWed joint is obtained by sweeping (by plastic deformation) the softened metallic material at first from the so-called advancing side (AS), and then from the retreating side (RS); directions are defined by the direction of the tool and the related clockwise or anti-clockwise rotation direction of the tool [10–12].

Friction stir welding can be applied to a variety of metallic materials, ranging from magnesium and titanium, to steel, copper and aluminum alloys. In the latter case, the study of friction stir welding applied to the 5XXX alloy series is attracting increasing interest from the scientific community. This is because the results show a strong connection between the mechanical properties of the welds and the grain size; and the dislocation density...
generated by plastic deformation and recrystallisation, which take place during the friction stir welding process [13].

Different combinations of values for the rotational speed of the pin, welding speed, and tool sinking have been reported in literature; they have been linked to the formation of particularly unfavorable microstructural features and to defects inside the welded joint [3–16].

However, the combination of friction stir welding, heat treatments, and other plastic deformation-based techniques such as cold rolling, which can minimize the welding sheet thickness reduction in the welded joint and have a strengthening effect that improves the overall efficiency of the final joint, is worth further investigation [17–21]. Indeed, currently the thinning of the weld is addressed with special tools or welding additive equipment [22]. It is worth mentioning that the combination with surface treatments, such as burnishing and bull-burnishing, is also very relevant in influencing the welding joint and its mechanical behavior; in particular, a local hardness increase [23,24].

FSW is especially used in aeronautics and the automotive industry; in addition, it is typically applied to light alloys and namely, aluminum AA5000, 6000, and 7000 series alloys [25–27]. In this respect, Al-Mg AA5754 is among the most used AA5000 series alloy in the aeronautical and automotive industries to make structural panels thanks to its excellent corrosion resistance properties; in particular, sea water and industrially polluted atmospheres, good mechanical strength, weldability, and low cost.

The aim of the present paper is to characterize the effect of the sequence of heat treatment, friction stir welding, and cold rolling on the overall microstructure and micro-hardness of the AA5457 alloy.

2. Experimental Procedures and Methods

AA5754 aluminum sheets 180 mm in length, 80 mm in width, and 1.5 mm thick in the H114 metallurgical state were joined in pairs by performing FSW with a machining center. The chemical composition of the alloy is shown in Table 1.

<table>
<thead>
<tr>
<th>Zn</th>
<th>Cu</th>
<th>Mn</th>
<th>Si</th>
<th>Fe</th>
<th>Mg</th>
<th>Cr</th>
<th>Ti</th>
<th>Ni</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.2</td>
<td>0.1</td>
<td>0.5</td>
<td>0.4</td>
<td>0.4</td>
<td>2.6–3.6</td>
<td>0.3</td>
<td>0.15</td>
<td>-</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

To analyze the role of plastic deformation in cold welding processes (Friction Stir Welding, FSW) in an Al-Mg alloy and how it is influenced by annealing treatment, four types of samples were studied. Figure 1 shows the sequence of operations performed to prepare each sample: sample 1 was subjected to FSW (FSW); sample 2 was subjected to FSW and subsequently to CR (FSW + CR); sample 3 was first subjected to FSW, then annealed (heating treatment, HT), and finally processed with CR (FSW + HT + CR); and sample 4 was annealed before being subjected to FSW and CR (HT + FSW + CR).
Figure 1. Schematics of the combinations of thermo-mechanical processes used in the present paper. Butt joints were obtained by FSW experiments carried out using the COMEC™ M50 (DRIVER M50 2T, Italy), a computerized numerical control machining center; the FSW process is schematized in Figure 2, which also shows the geometric characteristics of the pin tool (Table 2) and the welding parameters that have been set. The parameters used were selected on the basis of a previous study in which the weldability of the alloy AA5754-H114 was investigated.

Figure 2. Details of the FSW process: (a) schematic representation of the FSW process; geometric features of the conical pin tool in the H13 tool steel used in the FSW experiments and welding process parameters; and (b) picture of one of the welded parts, representative of all the processing conditions used.

Table 2. Features of the FSW system reported in Figure 1.

<table>
<thead>
<tr>
<th>Feature</th>
<th>Value</th>
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<tbody>
<tr>
<td>Shoulder diameter (mm)</td>
<td>15</td>
</tr>
<tr>
<td>Cone base diameter (mm)</td>
<td>3.9</td>
</tr>
<tr>
<td>Pin height (mm)</td>
<td>2.3</td>
</tr>
<tr>
<td>Pin angle (°)</td>
<td>30</td>
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</tbody>
</table>

Experiments were carried out using a tool sinking of 1.3 mm and a plunging angle of 2°; while the rotation speed and welding speed used were divided in two different parameters, labelled A and B summarized in Table 3; and were fixed during the overall welding process.
Experiments were carried out using a tool sinking of 1.3 mm and a plunging angle of 8°. The welding process could be considered stationary. The length side of the strips was parallel to the welded line. The strips were obtained in the central area of the weld, in such a way that the FSWed joints; so that the length side of the strip was parallel to the welded line. The strips were obtained in the central area of the weld, in such a way that the FSWed joints were fixed during the overall cooling was carried out by keeping the blanks in a furnace for 3 h, at a constant temperature of 415 °C; and then, cooling was carried out by keeping them in a turned-off furnace.

Since the main focus of the paper is to analyze the outcomes of the investigated thermo-mechanical processing sequences, only two conditions for the FSW process were considered. As per Table 3, condition A refers to a faster welding operation compared to condition B; however, it is also characterized by a lower rotational speed of the pin compared to B.

Metallographic inspections were performed by means of optical microscopy (OM) Leica, DMI-8 (Leica, Wetzlar, Germany) on surfaces polished mechanically and electrochemically etched with a 4% HBF4 solution at 15 V for a few tens of seconds. Polarized OM (POM) was used to properly highlight the grained structures of each sample.

### Table 3. Different parameters used for the friction stir welding.

<table>
<thead>
<tr>
<th>Parameter Set</th>
<th>Rotational Speed (rpm)</th>
<th>Welding Speed (mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1800</td>
<td>75</td>
</tr>
<tr>
<td>B</td>
<td>2400</td>
<td>50</td>
</tr>
</tbody>
</table>

The FSWed workpieces were cold rolled after joining and annealing (that is, FSW + CR, for sample 2; FSW + HT + CR, for sample 3; and HT + FSW + CR, for sample 4) to make the blank thickness uniform and increase its mechanical response. The cold rolling process was performed on strips, 65 mm in width and 110 mm in length, obtained by mechanical cutting of the FSWed joints; so that the length side of the strip was parallel to the welded line. The strips were obtained in the central area of the weld, in such a way that the FSW process could be considered stationary. The length side of the strips was parallel to the rolling direction of AA5754 sheet in the as-received condition.

Several rolling passes were performed in order to obtain a thickness reduction of about 20%; in samples 2 and 3, the desired deformation was reached with 4 rolling steps without noticing any sign of macroscopic damage to the material; while for sample 4, the cold rolling was interrupted after the second step with an overall reduction of 10% as macroscopic cracks appeared in the area affected by the welded joint, as highlighted in Figure 3.

![Figure 3. Panoramic POM of sample 4 after the 10% overall reduction. The onset shows a magnification of the welded joint with a large crack.](image-url)
Microhardness was performed along the middle-height thickness of the FSWed section. A Remet® HX-1000 tester (Remet, Casalecchio di Reno (Bo), Italy) was used; the measurements were spaced 0.5 mm apart.

3. Results and Discussion

By analyzing the cross section of an FSW welded joint, four zones with different metallurgical characteristics can be commonly identified (Figure 4):

- **i NZ (nugget zone):** this region undergoes the greatest plastic deformation and is typically characterized by an equiaxed and recrystallized grain structure;
- **ii TMAZ (thermo-mechanical affected zone):** a zone in which the material has been subjected to both plastic deformation and heating;
- **iii HAZ (heat-affected zone):** a zone in which the welding heat cycle caused alteration of the microstructure and mechanical properties of the material; however, there was no plastic deformation; and
- **iv PM (parent material):** an area that has undergone a weak heat cycle, if none; it is not plastically deformed, and does not show alterations to either the microstructure or mechanical properties.

![Figure 4. Comparison of panoramic micrographs of the welding joints processed with condition A.](image)

The polarized optical micrograph overviews reported in Figures 4 and 5 show the typical above-mentioned friction stir welding zones. It is worth to note that in both cases, the FSW + HT + CR results in a remarkable recrystallization of the grain structure promoting an uncontrolled growth of the grains due to the local high temperature reached in the welded joint area (from the nugget through the TMAS and HAZ). In addition, it should be mentioned that near the shoulder, the grains remain small as the deformation is compensated by the pressure exerted by the cold rolling equipment. This pressure-limited effect confines the largest amount of recrystallization to the nugget area; and allows somehow a smoother transition in terms of grain sizes between the parent metal and the heat affected zones.
Figure 6 shows NZ characterized by uniform and equiaxed grains in the alloy, not heat treated prior to FSW and prior to FSW + CR. By performing the heat treatment between the latter two processes, a strong growth of the grain dimensions is observed for both parameter sets used. This is in contrast to the weld joint characterized in the paper by Cabibbo et al. [18], where AA5754 sheets were characterized by a higher thickness (2.5 mm instead of 1.5 mm of the present paper). In addition, a key difference is represented by the friction stir welding parameters. These were as follows: a rather lower rotation speed (1200 rpm), a higher welding speed (100 mm/min), and a much lower value of the tool sinking (0.08 mm); a set of parameters that is similar to others reported in literature for 5xxx alloys [28–30]. The results outlined by the panoramic POM micrographs are confirmed by the comparison of the polarized optical microscopy images taken in the nugget zone for the four different processing conditions, and the two sets of FSW parameters A and B (Figure 6).

To correlate the grain size variation, whether induced by FSW, pre- or post-annealing, or by CR, the microhardness profiles were measured along the weld joint sections; in FSW, FSW + CR, FSW + HT + CR, and HT + FSW + CR experimental conditions. The corresponding side-by-side profile (advancing to retreating side) is reported in the multiple plots of Figure 7. The hardness profiles across the FSW joint show a certain degree of uniformity between the different characteristic zones, and the softening effect of the temperature in the thermally altered zones is recognized. It is noteworthy to outline that throughout the retreating side, the thermally altered areas (R-TMAZ) and (R-HAZ), the FSW-CR, and FSW-HT-CR showed a general hardness profile lower than the respective parts in the advancing side. The average hardness in the NZ of FSW + CR was the highest; while the two average values of FSW and FSW + HT + CR were quite similar. The higher average hardness found in the sample nugget subjected to FSW + CR agrees with the lower average grain size, compared to other conditions, as obtained with the metallographic inspection.
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Figure 6. Comparison of POM taken in the nugget zone.

Figure 7. Microhardness profile of the welded joint, for the FSW processing conditions A.

Figure 8. Microhardness profile of the welded joint, for the FSW processing conditions B.
Figure 8 shows that the microhardness profile outside and inside the welded joint. The processing parameter set B seem to result in a different mean value of the welded joint (−2/+2 mm) for the HT + FSW + CR condition. The reported values corresponding to the other experimental conditions at the NZ were quite similar to those obtained by using the parameter set A (see Table 2); that is, by performing the heat treatment before the welding and plastic deformation. Yet, in this latter case, an overall lower hardness was observed. Furthermore, from the microhardness point of view, an overall homogenization was reached by combining parameter sets and processing condition sequences. As the heat treatment to which the alloy was subjected in the two FSW conditions A and B (see Table 2) is the same, the strain hardening induced by CR seemed to be minimized by the effect of the FSW used parameters that induced the formation of microstructures characterized by lower residual stresses throughout the welding seam.

Thus, by using the sequences reported here of heat treatment FSW and CR, and by choosing the FSW parameter that is in Table 3, the advancing and retreating sides experienced a lower difference in terms of resulting microhardness.

Thence, it resulted that the profile in the AA5754 alloy, performing the heat treatment before and, in turn, friction stir welding and cold rolling, is able to provide FSW joints with a uniform hardness profile.

It is worth to mention that with both friction stir welding processing conditions A and B, the large recrystallized grains obtained after the FSW + HT + CR sequence did not lead to a significant change of microhardness profiles; although, according to what was reported in [31,32], the resulting mechanical response is not likely to be considered at its best.

The HT + FSW + CR joint showed a microstructure characterized by small grains and a highly homogeneous hardness profile throughout the FSW characteristic zones (from PM to the NZ). Indeed, the present results indicate that the proper heat treatment, FSW, and cold rolling sequence can be followed whenever a uniform hardness profile is foreseen and/or needed for a particular application in the case of Al-Mg FSW plates.

4. Conclusions

The present paper addressed the friction stir welding (FSW) of the AA5754 aluminum alloy, performed with two different conditions of rotation and welding speeds; coupled with different sequences of heat treatment (HT: annealing), FSW, and cold rolling (CR). The welded seam was characterized microstructurally (by polarized optical microscopy) and
mechanically (by micro-hardness measurements); and the main conclusions can be listed as follows:

1. The application of annealing and cold rolling before and/or after the FSW with condition A parameters (high advancing speed) showed neither remarkable variations nor trends in the microhardness profile of the joining, from the nugget to the parent metal;
2. For the samples welded with condition B FSW parameters, the application of the annealing heat treatment before the FSW process rather than after FSW and cold rolling results in an overall softening of the alloy; this is more pronounced on the advancing side of the welded sheet, where the other experimental conditions (FSW, FSW + CR, and FSW + CR + HT) show the highest overall microhardness mean values;
3. From a microstructural point of view, the sequence FSW + HT + CR resulted in a significant grain growth throughout the welding joint of both FSW conditions (A and B).

The main results showed that by tuning not only the FSW process parameters, but also a sequence of thermos-mechanical processes, it is possible to influence the resulting microstructure and hardness profiles throughout the thermally affected zones of the FSW seam. Therefore, these results cast a light on the viability of applying specific sequences of HT, FSW, and CR to 5XXX series alloys to induce the formation of an homogeneous hardened weld seam.

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References
5. Casalino, G. Advances in Welding Metal Alloys, Dissimilar Metals and Additively Manufactured Parts. Metals 2017, 7, 32. [CrossRef]