

# Friction Stir Processing

## Characterization and analysis of processed aluminium alloys

AA5083-O and AA7022-T6

### Abstract

Friction Stir Processing (FSP) is based on the basic principles of Friction Stir Welding (FSW) in the metallurgical modification of the materials in the thermomechanical processed zone. FSP modifies the original microstructure through a grain size refinement on the materials processed zone and elimination of superficial and internal defects, e.g., porosities and cracks. The processed materials usually improve their mechanical properties, as well, their corrosion and wear resistance and fatigue behaviour. It is possible to reach superplasticity behaviours in some materials using the FSP.

In this work, it was made a literature survey on the issues involved which lead to the development of various tools for FSP, with different geometries, from where we selected two different tool geometries for the superficial FSP and two different tool geometries for the volume FSP. After this phase, it was performed an experimental analysis of tool geometry and the most important FSP parameters in the superficial and in volume FSP for the cast aluminium alloys AA5083-O and AA7022-T6 with 10mm in thickness.

It was performed several tests with different number of passes and different overlap ratios. The obtained results were analysed by metallurgical means using optical and electronic microscopy, hardness testing and tested in terms of bending resistance and superficial wear testing.

### 1 – Introduction

Friction Stir Processing (FSP) is a new solid-state technique, which uses the principles of friction stir welding to process materials leading to new improved metallurgical conditions. In FSP a tool containing a shoulder and a pin provides mechanical mixing and heating resulting from interfacial friction and visco-plastic deformation, in the area processed by the tool.

The FSP has four distinct regions; the nugget which is the region that is thermo-mechanically processed zone where the grain size is refined and homogenised, the thermo-mechanically affected zone (TMAZ) where the grain is elongated like it was mechanically deformed, the heat affected zone (HAZ) that has the same grain structure of the base material and the base material (BM) is the region that was unaffected by the process.

The improvement of properties is related to the refinement of grain size in the nugget due to the large processing strain. FSP has been very successful in modification of various properties such as formability, hardness, yield strength, fatigue and corrosion resistance. It is becoming very effective in the production of metal matrix composites. FSP has also been very successful in the production of materials with superplastic behaviour.

Santella et al. [1] confirmed that the hardness profile in the processed zone is uniform and that in some alloys the yield strength increases when compared to the base material. In another study, Kwon et al [2] studied an aluminium alloy AA1050 and verified that the yield strength and hardness decreases with the increase of the tool rotation. Chang et al. [3] studied

the effects of FSP in an Mg-Al-Zn alloy, and were able to produce a processed zone with a grain size smaller than 500nm with this were able to increase the hardness from 50HV to 120HV.

It is thought that the defects from the casting process that are usually associated to the initiation of cracks from where the material will fracture. So the elimination of these defects will increase the materials fatigue resistance. In studies made on the fatigue resistance [4, 5] it was verified that the FSP is able to eliminate or minimize these defects and studies have shown an increase in the fatigue resistance, in some cases the fatigue resistance doubled when compared to the base material fatigue resistance.

Since the FSP eliminates microstructures defects e.g. porosities and cracks, and with the refinement and homogenization of grain size makes the processed material very tolerant to damage and can be applied in formability operations. In the studies [6, 7] made it has been observed that the processed material greatly improve their ductility and in bending tests it has been possible to bend alloys 20mm thick without cracking.

The FSP is very suitable for the production of metal matrix composites because it produces a plastic deformed zone that promotes the mixture and refinement of the several material constituent phases and the high temperature helps in the formation of an intermetallic phase and suffers an hot consolidation which produces a very dense solid and also the joint particles will be homogeneously distributed making the material properties homogenous. In the studies [8, 9], it was proven that it is possible to produce metal matrix composites using Al-Ti powders and that the produced composites have great improvements in the yield strength and hardness. Morisada et al. [10] were successful in the production of metal matrix composites with the addition of carbon nanotubes, which improved the tensional properties and helped in the microstructure refinement.

Some materials after being friction stir processed can become superplastic, these materials gain this designation when they can be strained over 200% of their initial size before cracking. Materials with superplasticity at low temperatures are attractive for commercial formability since it reduces energetic costs, increases the lifetime of formable coatings and improves the superficial quality of a formable component. Charit et al [11] obtained superplasticity behaviours in an Al-Zn-Mg-Sn alloy and were obtained elongation of 1125% at 310°C and elongation of 525% at 220°C. In other related studies [12, 13], it was verified that the smaller the grain size the better was it superplastic behaviour, and it was also proven that there is an optimal point from which the material exhibits superplastic behaviour that is different for each material.

One of the characteristics of the FSP is the multipass, which is the use of multiple passes to process a material in full extent. Studies [14, 15] made have shown that the properties of the materials remain the same along the all the passes made on the material, although the material as shown a better superplastic behaviour in the AA7075 alloy when it was processed with only one pass than with multiple passes.

This shows that this technique is very promising as a superficial treatment and that it needs further evaluation, which was the determining reason for this study.

## 2 – Experimental procedure

One of the objectives of this work was to develop and test the applicability of new tool geometries that could friction stir process superficially and in volume. These tools should process more area than the conventional tools that are usually applied in friction stir welding. Therefore scrolled shoulders with slight variations between them and a concave shoulder with three semispherical non-concentric pins were specially designed for superficial FSP.

The aluminium alloys AA7022-T6 and AA5083-O used in this study, were obtained as a 210x300mm plate with 10mm in thickness. Two tool geometries were selected for the superficial FSP and two tools geometries were used for the in volume FSP (Fig. 1). For the in volume FSP, one of the tools had a Triflute pin with 8mm and 6mm in maximum diameter and length, respectively; the shoulder had a concave profile and was 19mm in diameter. The other tool had a shoulder with a concave profile and was 15mm in diameter, and the pin was threaded cylindrical with 5mm and 4.7mm in diameter and length, respectively. In the superficial treatment, one of the tools had a scrolled profile with four discontinuous streaks and the shoulder was 17mm in diameter, the other tool had a shoulder with a concave profile with 3 non-concentric semispherical pins and the shoulder had 18mm in diameter. The passes by FSP were made using a tool rotation rate of 710rpm and a tool transverse speed of 224mm/min using a tool tilt angle of 2°.

For the in volume FSP, the effect of the tool geometry and different overlap ratios was investigated, while on the superficial FSP it was studied the effect of tool geometry and processing parameters.



**Fig. 1** – Tools used in the FSP of the aluminium alloys

For the study on the in volume FSP, it was developed a constant that characterizes the overlap between each pass by FSP, this constant was denominated of overlap ratio (OR) and it is defined in equation 1.

$$OR = 1 - \left[ \frac{l}{d_{pin}} \right] \quad (1)$$

Where  $l$  is distance between centres of each pass and  $d_{pin}$  is the maximum diameter of the pin. From this equation it is defined that passes made in the same spot will have an OR=1 and that the OR will decrease with the increase of the distance between passes.

To study the effect of the overlap ratio it was made several passes with different overlap ratios, as described below:

- four passes with OR=1 from both sides of the plate;
- four passes with OR=1 from one side of the plate;
- three passes with OR=1/2 from one side of the plate;

- three passes with OR=0 from one side of the plate;
- and two passes with OR=-1 from one side of the plate.

This was made for both in volume FSP tools, after samples were taken from each condition and the various effects of the OR and the effect of the tool used were studied.

For the superficial we tested different conditions on the 5083-O alloy, where we tested different parameters starting with a tool transverse speed of 224mm/min and a tool rotation rate of 710rpm with a tilt angle of  $2^\circ$ , if this did not work it was tried other conditions like testing the opposite parameters like high tool rotation rate versus low tool transverse speed and low tool rotation rate versus high advancing speed. After this, samples were taken to study the obtained results.

The obtained samples from the FSP as well as the samples of the base material were polished and etched with Keller reagent to highlight the grain boundaries, precipitates and show the differences in composition or with Poulton modified reagent, which is better to highlight the grain boundaries and differentiates the recrystallized grains. After this the samples were characterized by optical microscopy on an optical microscope Olympus CK40M to observe the samples microstructure and study the effects of the processing variables, the measurement of the average grain size was determined using the mean linear intercept technique.

From the results of these observations, some samples were chosen to be characterized by electronic microscopy (SEM) and the hardness was also tested in several samples in a Struers Duramin microdurometer. The hardness testing on the base metal was made to obtain a value of the samples true hardness and to verify if there was any significant change on the hardness values on the sample. In processed samples, it was studied the evolution of the hardness at 1, 3 and 5mm from the surface in the samples processed in volume and at a distance less than 1mm in the superficially processed samples.

After determining the best FSP processing variables for the in volume FSP and superficial FSP, it was produced test samples for bending and wear testing.

The samples for the bending tests were 20mm wide and at least 190mm long and their thickness was 10mm, and were produced at a constant traverse speed of 224mm/min and a tool rotation rate of 710rpm and a tilt angle of  $2^\circ$  with an OR=1/2 for the in volume samples and an OR $\approx$ 1 for the superficial samples; the bending tests were made on an Instron 5566 equipment. It was made two test samples of each condition for these tests. The bending tests were made at a constant deformation speed of 5mm/min. The distance between support pins had to be wider for the tests because the equipment had a limit of a maximum applied force of 10kN, so the distance between support pins was increased so that the test would proceed in normal conditions. The adopted distance between support pins was of 100mm for the 5083-O samples and of 120mm for the 7022-T6 samples.

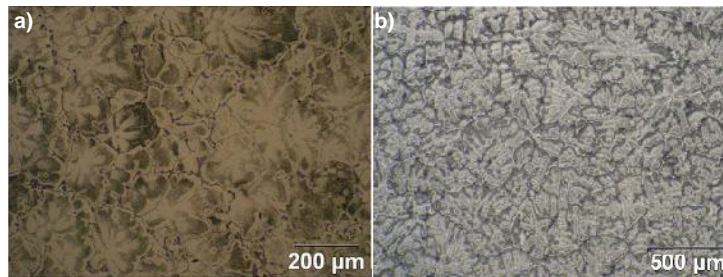
The wear testing was made from a qualitative point of view because only one sample of each condition was made and to have a proper study this analysis should be complemented with another characterization method. These samples were produced in the same conditions of the bending test samples. The samples made for wear testing were square with 45mm wide and

10mm thick. These tests were made on a pin-on-disc method assisted by a PC with LABView software to measure the friction coefficients between the samples and a tool steel pin. There were several parameters for this test like the applied force of 44.145N, the sliding speed of 4m/min, the pin contact area of 3.67mm<sup>2</sup>, the travelled distance of 360m and the test duration of 1.5h.

For each sample, its weight was measured before and after the test to know what was the lost of weight and from there it was calculated the wear constant and loss of volume per distance travelled.

### 3 – Discussion of results

The characterization of the base material was made through the data sheets available at Gleich GmbH Metallplatten-Service company and also metallographic characterization in which some samples were polished and etched and also hardness tested. It was observed that the grains in both alloys had a petal-like microstructure due to inoculants added to refine the grain size. These inoculants adopted a star shape and from each branch it grew a grain in which we obtained the following microstructures represented in Fig. 2.



**Fig. 2** – Base material samples polished and etched with Poulton modified reagent:

a) AA5083-O e b) 7022-T6

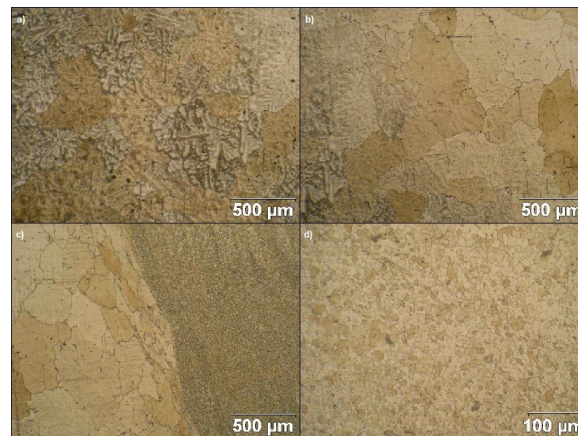
It was also measured the grain size and hardness for each alloy and obtained for the AA5083-O alloy an average grain size of 106.87μm and an average hardness of 76.9HV0.5, for the AA7022-T6 alloy it was measured an average grain size of 166.8μm and an average hardness of 132HV0.5.

After observing the several samples, it was concluded that the in volume FSP is best when Triflute pin was used because the threaded cylindrical pin tool creates defects in the processed area. The defects are due to the pins shape that causes its inability to drag the material and the most common defect that appears with this tool is the void defect. This kind of defects was not observed in the microstructure of the materials processed with the Triflute pin implying that this is the best tool for the job; since it can process the all thickness of the material used and the materials do not have any defects in the microstructure due to processing conditions. The other condition studied was the overlap ratio from which we concluded that the best overlap ratio was ½. This overlap for the Triflute tool guarantees that the all area is processed. The samples processed with an overlap ratio of 0, which meant that the passes were made with a distance from the previous pass equal to the maximum diameter of the pin, had unprocessed areas between nuggets because this pin was conic. It was also observed that

the AA5083-O alloy needed at least three passes in the place so that it would have a homogeneous processed area, unlike the AA7022-T6 alloy that only needs one pass.

In the superficial treatment it was proved that the superficial treatment with the scrolled shoulder with discontinuous streaks was the best because we could not process the material with the other tool and the material did not show any defects from the processing treatment.

In the AA7022-T6 alloy, it can distinguish the four different zones of the friction stir processing (Fig. 3). In the nugget we have a very small, homogeneous and equiaxed grains. In the TMAZ it can observe the elongated grains from the plastic deformation that this zone was submitted due to the flow of material caused by the rotating pin and it can also be seen that this area is depleted of precipitates due to the high temperatures of the process and that in some samples that were processed from both sides that this area is very large. The transition between the TMAZ and HAZ can be observed since the microstructure in the HAZ is very similar to the base material and therefore the final transition between HAZ-MB can not be distinguished.



**Fig. 3** – The different zones of the FSP in the AA7022-T6 alloy:

a) base material, b) HAZ, c) TMAZ, d) nugget

It can also observe an increase in the zone depleted of precipitates in the samples processed from both sides that must be due to a longer exposition to high temperatures when it was processed. It was measured inside the nugget a grain size of  $7.1\mu\text{m}$  that did not change independently of the number of steps and overlap ratios tested in this study.

In the AA5083-O alloy, it does not exist any zone with depletion of precipitates, and it is complicated to distinguish the HAZ from the base material. The TMAZ has the elongated grains but it is quite smaller when compared to the TMAZ of AA7022-T6 alloy, the nugget of the AA5083-O alloys is not homogeneous with just one pass and there are many differences of the grain size being bigger near the shoulder and smaller at the base of the nugget this fact is related to the capacity of removing the heat which is faster at the base of the nugget than near the shoulder. If the heat is not removed fast enough the grain will join together and become bigger. Inside the nugget it was measured an average grain size  $8.1\mu\text{m}$  near the surface and  $3.1\mu\text{m}$  near the base of the nugget for the sample processed only one time. For the sample processed four times with an overlap ratio of 1 we obtained a homogeneous processed area,

with a homogeneous grain size of 5.9 $\mu\text{m}$ . For the samples with overlap ratios of  $\frac{1}{2}$  and 0 it was observed that the grain size increased on the first passes with the subsequent ones because the generated heat from the subsequent processing is high enough to coalesce the grains.

In the superficial FSP since the deformation of the material was higher, it was obtained a smaller grain size, the measured grain size was of 1.6 $\mu\text{m}$ . The shape of the nugget in this treatment was semicircular therefore the processed thickness is not always the same, and to ensure that is thick enough and the most homogeneous possible it is necessary to use more force.

In the processed area it was observed the existence of some small particles and so with the help of scanning electronic microscope and EDS we were able to analyse and quantify the composition of these particles. The origin of these particles is in the inoculants used to refine the grain size of the base material and formed intergranular eutectics. In all measurements it is always present at least a small amount of silicon, which means that this was the inoculant used.

The particles in the nugget assume a spherical form with the increase of magnesium in the composition and a more elongated form with the increase of silicon.

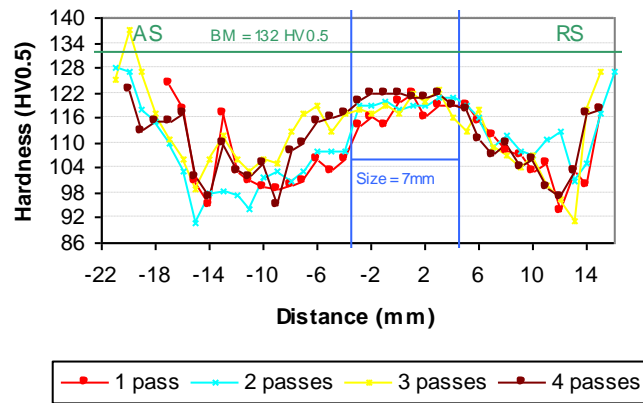
In the hardness testing for the AA7022-T6 alloys it was observed that the all area affected by the FSP, had a hardness lower than the hardness in the base material it was also confirmed that the nugget had the highest and most constant hardness inside the processed area around 120HV0.5, in the other zones the hardness decreases from the end of the nugget until a specific point and then it increases to the hardness of the base material (Fig. 4).

It was also observed an increase in hardness from the overlapping passes with OR=1 and this effect was more visible near the surface; this proves that overlap of various passes continues to homogenise the microstructure and its properties. The lowest hardness measured in the samples with an OR=1, was at about 14mm from the centre of the processed zone on the advancing side because this area must have been submitted to temperatures near 150°C which made it artificially age faster than the other zones influenced by the FSP.

In the samples processed with an OR=1/2 or 0, it was observed that the hardness did not decrease which confirms the stability of the microstructure inside the nugget.

In the hardness testing of the AA5083-O alloys it was observed, like the previous alloy, stability in the hardness inside the nugget and outside the nugget it decreases to the hardness of the base material hardness because unlike the AA7022-T6 alloy the hardness is the nugget is superior to the hardness of the base material confirming the law of Hall-Petch, that says that the smaller the grain size the higher the hardness for the same material.

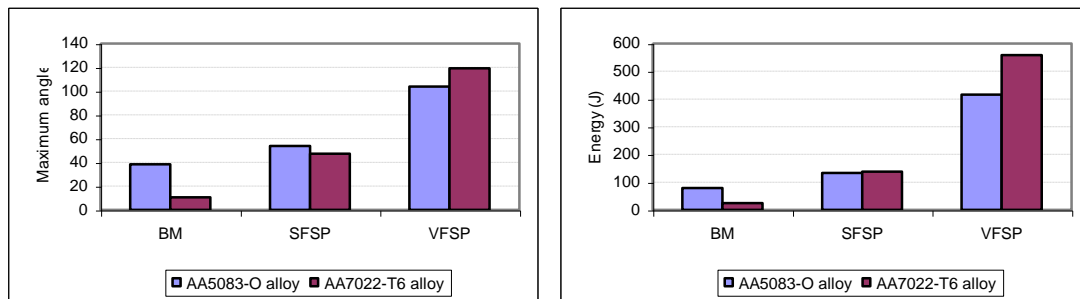
The hardness of the nugget stabilizes with the increase of the number of passes for an OR=1 which confirms what was seen in the microstructure that the grain became homogeneous with the number of steps. In the samples with OR=1/2 or 0 it is confirmed that the hardness of the previous processed zone decreases with the number of passes due to heat generated that coalesces the grain inside their nuggets.



**Fig. 4** – Hardness profile of a 7022-T6 alloy at a depth of 3mm

In the superficially processed samples it was observed that the hardness increased much more than in the in volume FSP sample of the same material, this once again proves the Hall-Petch effect since it was obtained a smaller grain size the hardness increased when compared to the in volume processed sample. The smaller grain size obtained was due to the higher deformation caused by the tool during the processing.

In the bending tests since the distance between supports was different for the two alloys, the only comparable results are the maximum angle before the crack initiation and the deformation energy necessary to initiate the crack because these will always be the same independently of the force made to bend.



**Fig. 5** – Bending tests results

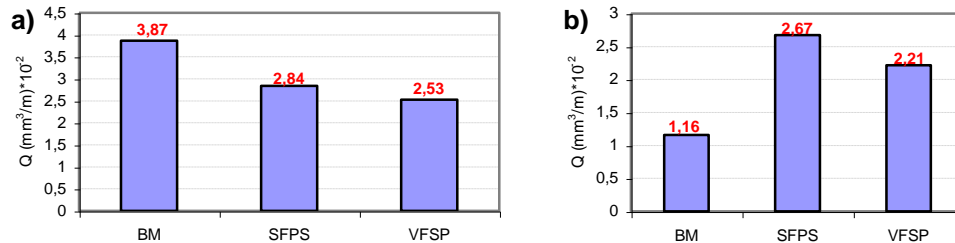
In both test results it can be observed that the base material in the AA7022-T6 alloy is much more brittle than the AA5083-O alloy, having a maximum angle of about 10° as opposed to the AA5083-O alloy that has a maximum angle of about 40°.

One of the conclusions that can be taken is that both the superficial FSP (SFSP) and in volume FSP (VFSP) improves the formability of both alloys, but the results for AA7022-T6 alloy are the most surprising since there was not any crack in the VFSP samples and the maximum angle and energy necessary for deformation were 12 and 25 times of the base material, respectively. In the samples that were superficially processed the maximum angle obtained is about the same for both alloys and that its important to have a constant processed thickness because if the processed thickness is smaller in one of the sides it will initiate the fracture in that side and it may propagate catastrophically depending if the base material is brittle or not.



The problem with the results of the samples superficially processed is that the nugget has a circular shape and because of this the processed thickness varies along the processed width so when choosing the overlap ratio so that it can be assured that the processed thickness is constant.

The wear testing was performed a qualitative analysis of the obtained results that would need to be at least confirmed with another characterization technique.



**Fig. 6** – Volume removed per distance travelled (Q) for the different processing conditions

a) AA7022-T6 alloy; b) AA5083-O alloy

It can be observed for the AA7022-T6 alloy that the FSP processing improved their wear behaviour of the processed samples. This is because the wear behaviour is related to several properties of the material such as hardness, ductility and microstructure, so difference in hardness in the processed samples for the base material was lower but there was a great improvement in ductility. So for the base material when the material cracks it will propagate catastrophically therefore will be a greater loss of material. In the processed samples since it has a greater ductility than the base material, it can deform without cracking and when there is a crack initiation it will have more difficulty to propagate therefore it results in a lower loss of volume in the processed samples.

For the AA5083-O alloys this behaviour should be similar or more effective in the processed samples since it has a greater hardness than in the base material, but this is not verified so there must be another reason for the decrease in the wear resistance in the processed samples. This reason lies in the microstructure in the particles that were observed inside the nugget, that must promote a new wear mechanism increasing the loss of material and therefore increase the volume removed per distance travelled.

#### 4 – Conclusions

The FSP is a process that shown to be very effective in the refinement of the grain size, reducing the grain size from 160 (AA7022-T6) and 106µm (AA5083-O) to an average grain size of about 6µm.

The best group of FSP parameters found under the technological conditions of the trials were constant transverse speed of 224mm/min, tool rotation rate of 710rpm and tool tilt angle of 2°. It was implemented an OR of ½ for the in volume FSP and an OR near 0 for the superficial FSP.

The hardness profile in the nugget processed zone is very homogeneous and similar to the hardness of the BM.

In the bending testing there was a great increase of the formability of the materials due to the increase of the materials ductility resulting from the refinement of the grain size.

It was concluded that the FSP improved the wear behaviour, but for the AA5083-O samples this behaviour worsened due to the emergence of a new wear mechanism, although it is necessary to make a deeper study of the wear behaviour using more samples and more characterization techniques.

## 5 – Bibliography

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