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Analysis of mechanical properties of an Aluminum + MWCNT compound manufactured by FSP

Análisis de propiedades mecánicas de un compuesto de Aluminio+NTCMC fabricado por PFA

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Abstract— The technique of Friction Stir Processing (FSP), can alter the microstructure of a metallic alloy generating a refinement of grain in the material by modifying it and closing porosity of previous processes, creating new crystallographic textures that may contribute to the magnetic performance material is different in comparison with the base material. This technique is considered appropriate for the introduction of agents that alter the mechanical and tribological properties, in this work was chosen as the reinforcing agent of Carbon Nanotubes Multilayer (MWCNT) that strengthen an aluminum alloy of an increment by the time in the industry. To analyze the change in the behavior of the material (Al+CNT) traction tests were carried out and scratched at room temperature.

Key Word — Agitation Friction Processing (FSP), Aluminum, Multi-layer carbon nanotubes (MWCNT), grating test, tensile test.

Resumen— La técnica de Procesamiento por Fricción Agitación PFA, puede alterar la microestructura de una aleación metálica generando un refinamiento de grano en el material modificándolo y cerrando porosidades de procesos previos, generando nuevas texturas cristalográficas que pueden contribuir a que el desempeño magnético del material sea distinto en comparación con el material base. Esta técnica se considera apropiada para la introducción de agentes reforzantes que alteran las propiedades mecánicas y tribológicas, en este trabajo se eligió como agente reforzante los Nanotubos de Carbono de Multicapas (NTCMC) que reforzarán una aleación de Aluminio de uso cada vez más frecuente en la industria. Para analizar el cambio del comportamiento del material (Al+CNT) se realizaron pruebas de tracción y rayado a temperatura ambiente.

Palabras clave— Procesamiento por Fricción Agitación (PFA), Aluminio, Nanotubos de carbono de múltiple capa (NTCMC), prueba de rallado, prueba de tracción.

I. INTRODUCTION

The Aluminum is highly used in automotive, aeronautics and structural industries, thanks to its good relation weight-resistance and resistance to oxidation. It has been the subject of multiple investigations to modify and improve its mechanical properties for use in increasingly demanding applications. To create compound materials on its surface, the technique of Friction Stir processing has been gaining the attention of researchers because of the advantages mentioned above. However, the lack of knowledge of how the process parameters: rotation speed and agitation, overlap, pin and shoulder geometry and their interactions affect the areas processed using the FSP technique in an aluminum alloy [1], keeps the technique still in the trial and error approximation stage, making it difficult to obtain adequate process windows and limiting its application in the industrial sector to companies that have the possibility of dedicating resources to the determination of the appropriate parameters. Therefore, in this study, we will analyze the methods of initial deposition for subsequent mixing using FSP, a quantity of reinforcing agents, and analysis of proper parameters of the technique to know how they influence the result of the mechanical and tribological properties to be studied.

II. CONTENT

In this research, we used Aluminum 1100, a material with 99% Al, 0.95% of Fe+S and Zn and Mn at 0.05%. This alloy is of high commercial and industrial use, since thanks to its high degree of purity in Al it allows to analyze with a clear vision that in other alloys the behavior of the material in different conditions of study, among them this one; how would be their mechanical behavior with and without any kind of treatment by means of FSP. This study will have three conditions for analysis: AA1100 base material, AA1100 subjected to FSP without nanotubes and AA1100 subjected to FSP with nanotubes. With this in mind, it is possible to analyze what changes FSP generates with respect to the base material, as well as how the introduction of nanoparticles alters the response of the alloy to the tensile test. For the execution of the process, two types of tools were used, one of them with concave shoulder, straight threaded cylindrical pin and the other with straight shoulder with rounded edge and square pin with threaded edges, the components of a standard tool in FSP are shown in Figure 1. The aluminum plates used were 300mm long, 80mm wide and 5mm thick. They were placed on the FEXAC industrial milling machine, from which 2 different types of rotation speeds were chosen, 4 different feed speeds and a 2° angle of inclination, Table 1. The technique was executed perpendicular to the direction of lamination of Al.

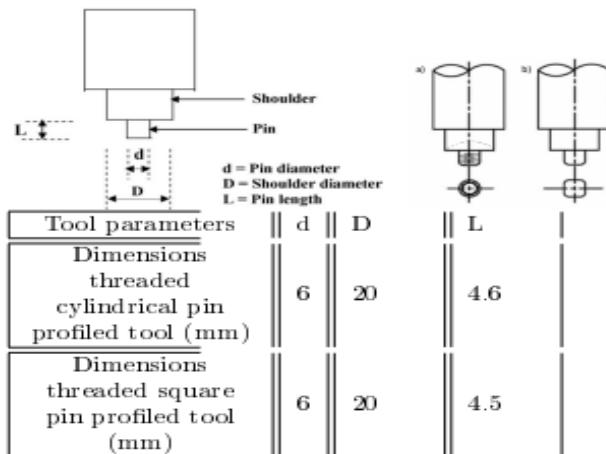


Figure 1: Tool parts, specifying Length and diameter of pin and shoulder. a) circular pin, concave shoulder, b) square pin with rounded corners and straight shoulder.

Tool parameters	Dimensions [mm]	Authors
d	5, 4, 4, 6.5	[1, 2, 3, 4]
D	16, 10, 10, 20,	[1, 2, 3, 4]
L	4, N.A, 2.2, 3	[1, 2, 3, 4]

Table 1: Tool parameters, respectively with each author.

Once the analysis of the different processed zones was carried out, it was concluded that the parameter that gave the best results, with good surface finish and without defects in the Processed Zone, was 1400 rpm and 45 mm/min, being selected as the set of parameters to be used in the plates to introduce the MWCNT, these parameters are also recommended by [2].

A. Particle deposition

Several authors suggest making a slot in the plate to deposit the particles and reinforcing agents [3, 4, 7, 8]. In the case of this project to deposit the MWCNT, a 2 mm deep and 2.5 mm wide slot was created along the length of the Aluminum plate, with the idea that the tool should pass through the middle of it, sealing the slot, distributing material and the nanoparticles inside it, Figure 2.

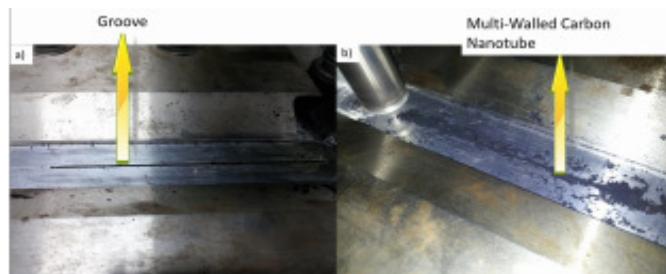


Figure 2: a) Plate with the slot for the deposition of nanotubes and b) plate with nanotubes deposited in it.

Once the material was processed, tensile tests were carried out using the Universal Ag-250 KNG Shimadzu machine with specimens that follow the E8 rules for this type of process. This being so when the samples were obtained with the MWCNT, the microstructural and mechanical characterization stage was passed. However, Figure 4 shows that the FSP process seals the slot, filling it with side metal and MWCNT, but leaving defects in the cross section and longitudinal section of the processed zone. This defect had a negative impact on the tensile tests, Figure 3, generating premature failures that were nucleated in the defect. In this way, another method had to be used for the deposition of carbon nanotubes; and although many references use a slot to confine the NTC, this method was discarded, according to what happened with the authors K. Elangovana et al. [2].

However, when it was observed that this method left defects inside the material (indicated by the red line), Figure 4, a different method of deposition of the MWCNT was searched for.

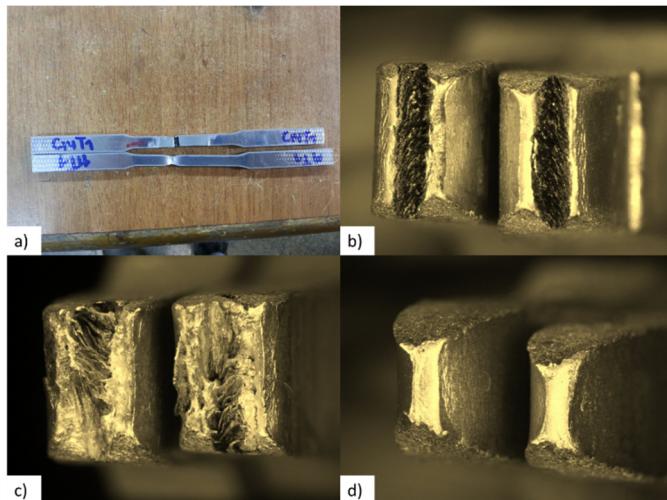


Figure 3: Tensile specimens with processed zone defects because of deposition slot. a) Breakage of processed material (top) and base material (bottom), b) Breakage caused by the slot defect, c) Breakage of the material in a brittle and ductile manner, d) Ductile breakage of base material.

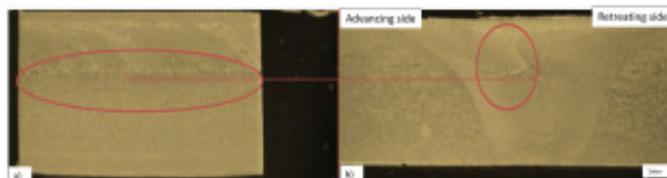


Figure 4: Processed zones with defects due to particle deposition slot, a) longitudinal view, b) transverse view.

The method designed to achieve controlled and quantifiable deposition consists in creating a zone where the mixture could be confined; the region had to be the same size as the shoulder, about 20 mm full and with a depth of 0.2 mm. As this deposition was superficial, there was no possibility of any defects inside the material. With this in mind, the next step to follow is to place the particles in the slot, but being this shallow and the MWCNT so light and volatile, that the passage of the tool would remove them as the shoulder creates an air vortex. To prevent the MWCNT from escaping was used Lauryl Ether (LE), a surfactant that combines with water to form a mixture with the MWCNT, depositing them without fear of escaping from the surface. Something very similar to what the authors Chi-Hoon Jeon et al. report in the article [7], with the difference that in this work it is a question of confining the mixture without external support as plates perpendicular to the plate to be processed, which restrict the dough, the parameters for

making this mixture are. The Lauryl Ether mass to be mixed with 100ml of water would be:

$$m_{LE0.5} = 20.175 \text{ g}$$

The mass of MWCNT to be mixed with the substance of water and surfactant would be:

$$m_{NTCMC0.5} = 0.12075 \text{ g}$$

The total mass of the mixture with MWCNT with water and the LE at 0.5 molar would be:

$$m_{m0.5} = 120.295 \text{ g} \approx 120.3 \text{ g}$$

Once the mixture is deposited on the surface, Figure 5, it must wait 6 hours for the mixture to solidify and be ready to perform the FSP technique.



Figure 5: Deposition of the mixture LE+MWCNT+H₂O in channel

B. Execution of fsp over the plate

Thanks to the high temperatures generated between the tool and the plate due to the friction of the two components, the mixture began to boil, evaporating the Lauryl Ether and the water, leaving the MWCNT exposed, but adhered to the plate. The two degrees (2°) of inclination of the tool contributes to the fact that the machine in its front part gives space to the Carbon Nanotubes to be submerged by the pin, Figure 6, avoiding burrs

that might have separated these nanoparticles from the process.



Figure 6: FSP on Aluminum plate with solidified mixture

III. RESULTS

The study of the cuts was made from the following Processed Zone, Figure 7, among the materials processed one was created with a concave circular shoulder pin, and the other was obtained with a straight shoulder square pin.

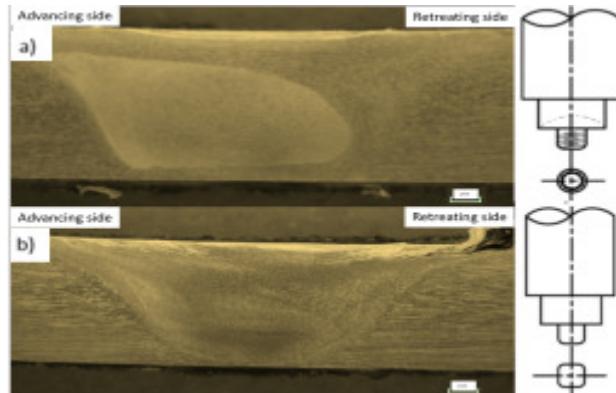


Figure 7: Processed zones that were obtained using two different types of pins. a) Straight Circular Concave Shoulder b) Straight Square of Straight Shoulder. Growth 0.75x.

It is important to note that although the pins of both cases had a similar length and the same active area [2], the Processed Areas have significant differences: The nugget of the processed area with a straight circular pin with a concave shoulder is more full at the bottom, the changes in grain size are notorious, between the base material and the processed area, but the transition between the ZTMA and ZTA zones is not clearly visible. On the other hand, in the nugget of the treated zone with straight shoulder square pin there is a more defined parabolic shape, and the nugget is smaller than in the previous case; also the changes in grain size between the base material and the processed zone are notorious; additionally, the transition between the ZTMA and ZAT zones is more noticeable than in the previous case.

Figure 8 provides a closer view of the microstructure of the processed area focusing on the characteristic regions:

1) Base material: in this section you can see the elongated

grains of the material as a result of the previous process of obtaining the veneer.

2) Transition between base material and processed material: In this part of the image we can differentiate between the base material, the ZTA and the ZTMA, created as a consequence of the severe plastic deformation and the temperature gradients caused by the friction between the tool and the Aluminum.

3) Processed Zone: is the area directly affected by the pin, the metal suffers from plastic deformation and refines its grain size, its shape and structure changes depending on the type of pin used.

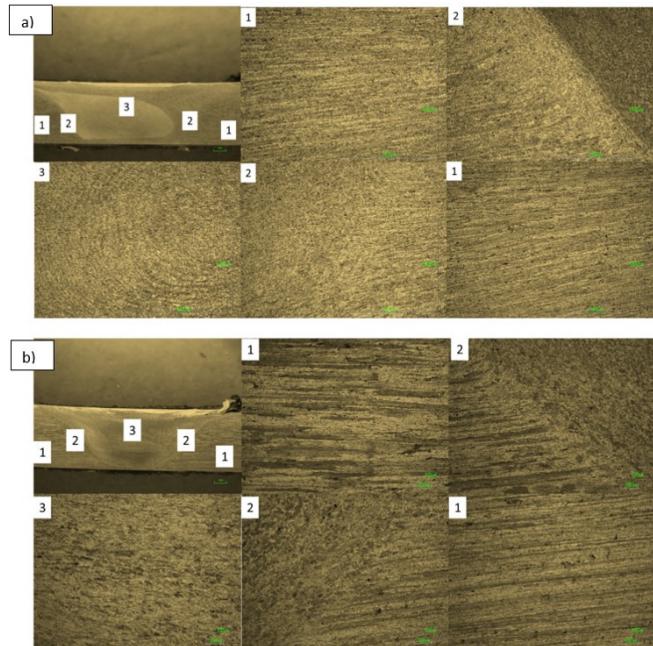


Figure 8: Transversal section of the two specimens subjected to FSP, a) circular pin, b) square pin. Magnifications 0.75x and 100x.

A. Specimens for traction tests

The traction tests were carried out in the same conditions as previously, using the Universal Ag-250 KNG Shimadzu machine, but on specimens that had no slot defect, this test was performed in two directions, one perpendicular to the processed material lace and the other parallel to the processed material, Figure 9. On this occasion, the breaks did not present a defined pattern as in previous cases, in which the material was broken on its retreating side according to the direction of rotation and feed of the tool. In total, ten different parameters were considered to perform this test.

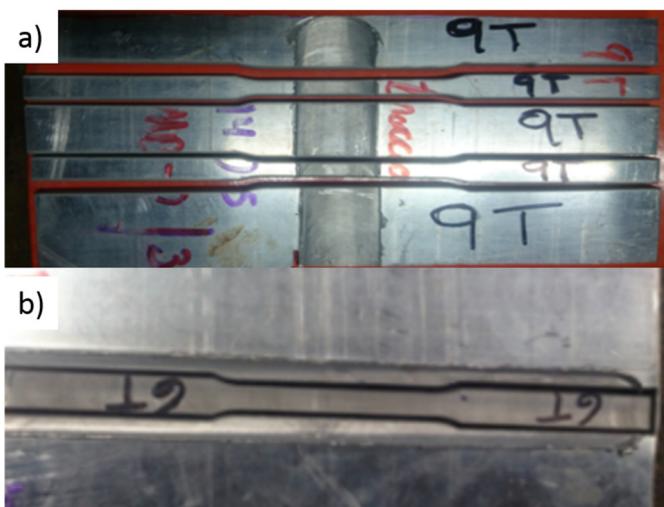


Figura 9: Tensile specimens, a) Specimens perpendicular to the processed material cord, b) Specimens parallel to the processed material cord.

In total, eleven different types of samples were considered for this test. It is important to note that each traction curve presented is representative of the three tests that were performed for each condition described:

1. Base material (BM).
2. Processed material with circular pin and concave shoulder, specimen perpendicular to the processed area and one pass (BM-P-O).
3. Processed material with square pin and straight shoulder, specimen perpendicular to the processed area and one pass (BM-P-Sq).
4. Processed material with circular pin, concave shoulder, longitudinal specimen to the processed area and one pass (BM-P-O | 1P | APM).
5. Processed material with square pin, straight shoulder, longitudinal specimen to the processed area and one pass (BM-P-Sq | 1P | APM).
6. Processed material with circular pin, 0.5 molar concave shoulder with double MWCNT deposition layer and one pass, specimen perpendicular to the processed area (0.5M-O-2C | 1P).
7. Processed material with square pin, 0.5 molar straight shoulder with double MWCNT deposition layer and one pass, specimen perpendicular to the processed area (0.5M-Sq-2C | 1P).
8. Processed material with circular pin, 0.5 molar concave shoulder with double MWCNT deposition layer and three passes, specimen perpendicular to the processed area (0.5M-O-2C | 3P).
9. Processed material with square pin, straight 0.5 molar shoulder with double MWCNT deposition layer and three passes, specimen perpendicular to the processed area (0.5M-Sq-2C | 3P).
10. Processed material with circular pin, 0.5 molar concave shoulder with double deposition layer, longitudinal specimen to the processed area and three passes. (0.5M-O-2C | 3P | APM).
11. Processed material with square pin, 0.5 molar straight shoulder with double deposition layer, longitudinal specimen to the processed area and three passes. (0.5M-Sq-2C | 3P | L.Z.P).

B. Analysis of results

Using AA1100 aluminum as a reference, we studied the effect of aspects such as the introduction of MWCNTs, the geometry of the tool and the number of passes. Figure 10 and Table 2 describe the stress vs. deformation curve and the values obtained in it of this alloy. It is observed that its level of deformation is close to 42%, has a tensile strength of 127.7 MPa and Young's moduli of 4487.8 MPa. Figure 11 shows the fracture after the tensile test and how it changes drastically in appearance from the slotted deposition method. This image allows visualizing how the fracture type has a characteristic appearance of ductile behavior and appreciable reduction of the transversal section area at the rupture site.

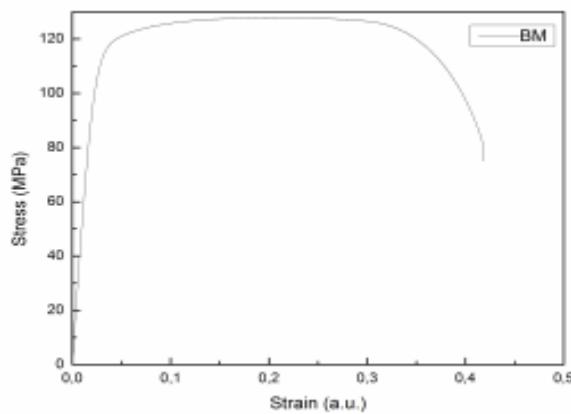


Figura 10: Curve Effort vs. Deformation of the base material.

Sample	MB
Elongation (%)	42
Cadencie effort (MPa)	118.5
Maximum effort (MPa)	127.7
Young's module (GPa)	4.4

Table 2: Mechanical properties of the Al 1100

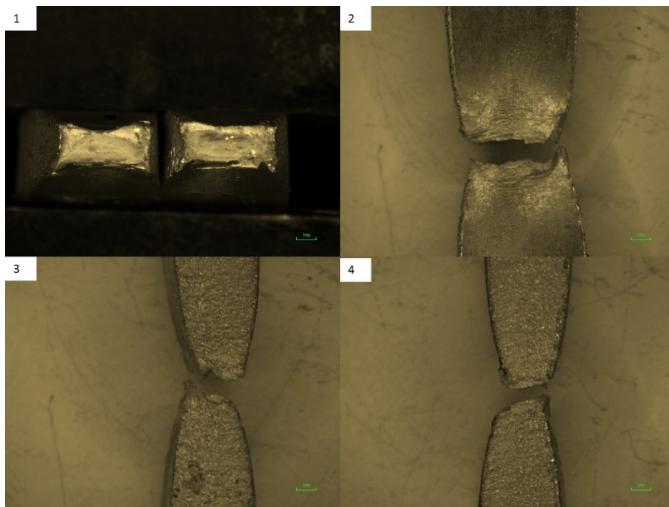


Figure 11: Base material (MB), 1) front view of the fault, 2) top view of the fracture, 3) left side view, 4) right side view. 0.75x.

Figure 12 shows a comparison of the traction curve between the base material processed with a circular pin (MB-P-O) and the base material processed with a square pin (MB-P-Cua). Table 3 presents the properties obtained for each material in this tensile test. Figure 13 shows photographs in which the fractures of the specimens can be seen.

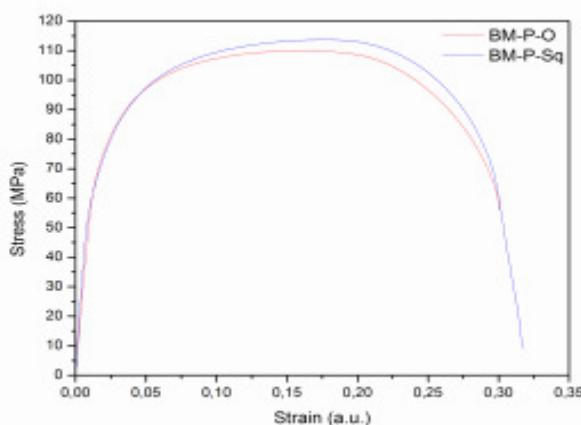


Figure 12: Effort vs. Deformation Graph a) processed material with a circular pin vs. b) Processed material with a square pin.

Sample	MB-P-O 1P	MB-P-Cua 1P	MB
Elongation (%)	30	31.6	42
Cadencie effort(MPa)	95	101.6	118.5
Maximum effort (MPa)	109.8	113.5	127.7
Young's module (GPa)	5	5.4	4.4

Table 3: Properties of the processed material with circular pin versus processed material with square pin.

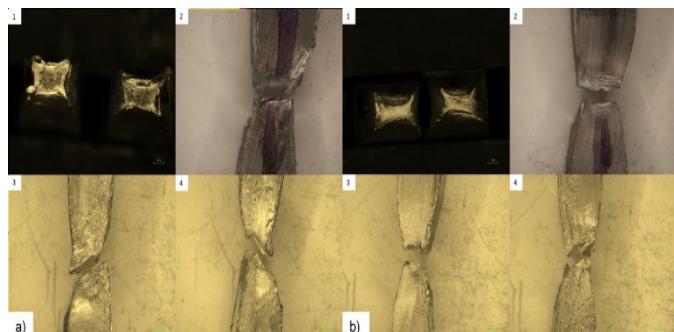


Figure 13: a) material processed with circular pin and concave shoulder and b) material processed with square pin 1) front view of the fault, 2) view of the top of the fracture. 3) left side view, 4) right side view. 0.75x.

This change in the fracture pattern may be associated with a different residual tension distribution caused by the pin geometry. However, this effect is not sufficient to generate appreciable changes in the mechanical properties. On the other hand, the tensile strength and elongation values are similar to each other.

Figure 14 is the comparison of the traction curve between the processed material longitudinal to the processed zone (L. Z. P) with a circular pin (MB-P-O L.Z.P.) against the treated material with a square pin longitudinal to the processed zone (MB-Cua-O L.Z.P.). In table 4 shows the values obtained for the properties of each material in this tensile test.

The base material, subjected to the longitudinal tensile test to the processed area, shows in Figure 14 how the level of deformation of the treated material increased considerably, regardless of the pin used, this is due to the fact that this material has a smaller grain size than the base material and also that recrystallization plays an essential role in the mechanical behavior of the alloy. Figure 15 shows fractures of both types of samples.

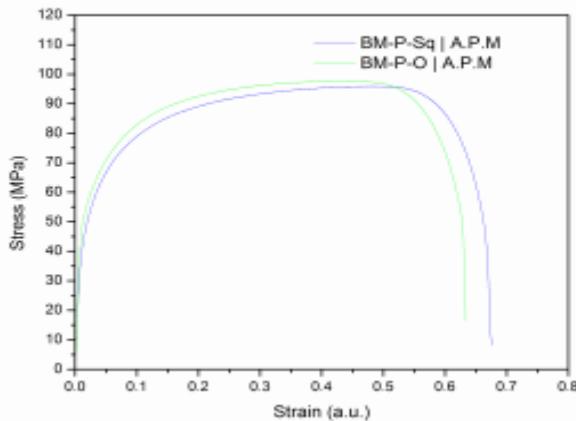


Figure 14: Effort vs. Deformation Chart a) Processed material, circular pin longitudinal zone processed versus b) Processed material, square pin longitudinal zone processed.

Sample	MB-P-O 1P L.Z.P	MB-P-Cua 1P L.Z.P	MB
Elongation(%)	63.3	67.5	42
Cadencie effort(MPa)	48.2	43.1	118.5
Maximum effort (MPa)	97.4	95.8	127.7
Young's module (GPa)	4.8	3.4	4.4

Table 4: Properties of the processed material with circular pin versus base material processed with square pin.

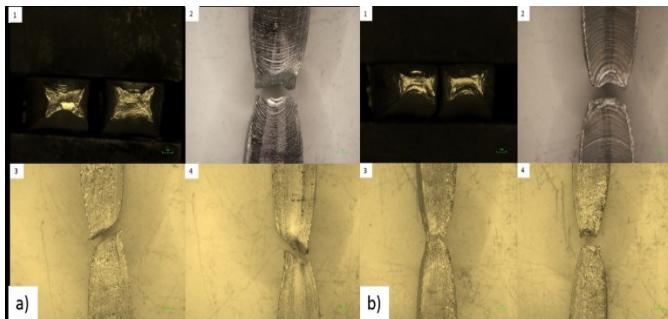


Figure 15: a) material processed with circular pin and concave shoulder longitudinal to the processed area and b) material processed with square pin longitudinal to the processed area 1) front view of the fault, 2) view of the top of the fracture, 3) left side view, 4) right side view. 0.75x.

Based on the results of the base material, it is studied the behavior of an aluminum compound AA1100+MWCNT obtained by FSP with two different pin geometries and with one pass or 3 of the tool. Figure 16 shows that none of the results obtained from the tensile test depends on the type of pin. Comparing these results, which are shown in Table 5, with those of the base material and the processed stuff, it is observed that they are generally lower. The traction resistance of the base material is 127.7MPa, that of the processed material is

113.5MPa, while that of the compound is 94.9MPa; in itself, the properties are below compared with that of the base material. Figure 17 shows a detachment in the top layer of the specimen made with a circular pin, the depth of this layer is approximately 0.5mm, this is because the shoulder of this tool is concave and has an inclination of 2°, giving the Aluminum the possibility to move a small layer of material from its forward side to the backward side, generating a coverage in the deposition of the MWCNT, instead the straight shoulder and square pin tool creates a different effect, because due to the geometry of this tool that the particles of MWCNT were pressed in the process and distributed in the processed area.

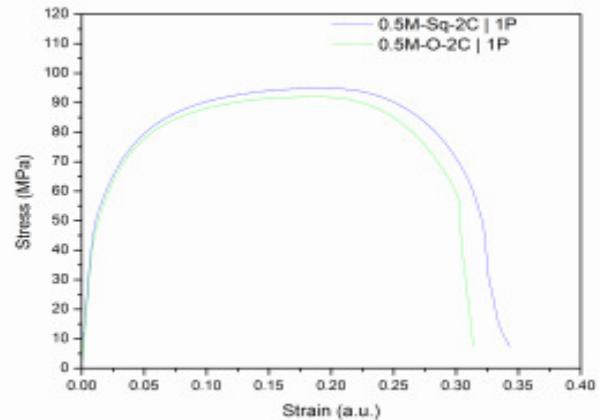


Figure 16: Effort vs. Deformation curve of compound AA1100+MWCNT, a) Material processed with a circular pin, concave shoulder at 0.5 Molar and one pass (0.5M-O-2C) b) Material processed with a square pin, straight shoulder at 0.5 Molar and with a tool pass (0.5M-Sq-2C).

Sample	0.5M-O-2C 1P	0.5M-Cua-2C 1P	MB
Elongation (%)	31.4	34.3	42
Cadencie effort (MPa)	40.9	51	118.5
Maximum effort (MPa)	91.9	94.9	127.7
Young's module (GPa)	4.8	4.5	4.4

Table 5: Properties of the compound Al+MWCNT processed circular pin versus the properties of the compound Al+MWCNT processed square pin.

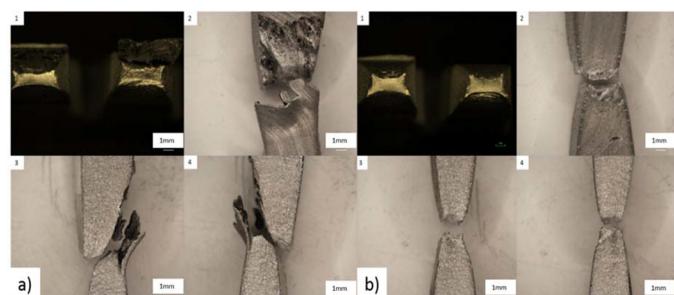


Figure 17: a) Processed material with circular pin, 0.5 molar concave shoulder with double deposition layer and one pass (0.5M-O-2C | 1P) b) Processed material with square pin, 0.5 molar shoulder with double deposition layer and one pass (0.5M-Sq-2C | 1P).

On the other hand, if the number of passes increases from one to three, the properties associated with the strength of the material decline; but its level of deformation increases considerably. Figure 18 shows how the deformation of the compound changes, having in the previous case deformations of up to 35%, something that with the three passes of the tool increased up to 45%. The contribution of the increase of the three passes of the tool over the same processed zone is intended to generate a homogeneous distribution of the Carbon Nanotubes over the entire processed zone and to refine the grain size even further. Now if we compare the data visualized in Table 6 it shows how the two samples of Al+MWCNT manufactured with the two types of pins and three passes of the tool present a similar response to the tensile test with each other, but as mentioned, the properties of the compound in comparison with the base material and the processed material are lower. The resistance to traction of the base material is 127.7MPa, and of the processed material is 113.5MPa. While the strength decreased, the ductility and Young's module increased compared to the material processed with Carbon Nanotubes and one pass; reaching approximately 0.53GPa and 7% respectively. In Figure 19 does not show the detachment of the top layer of the circular pin specimen, as seen in the samples of one pass, which can be attributed to the fact that the three passes of the tool distributed the nanoparticles in a more homogeneous way than when a single pass was used; additionally, the compound processed with a square pin maintains the behavior described above and did not show any detachment of the top layer of the material, in addition to that the transversal section of the specimen processed with a square pin had a reduction of the area higher than the one of the circular pin. The specimen on side b) generated a double neck in the process of the traction test.

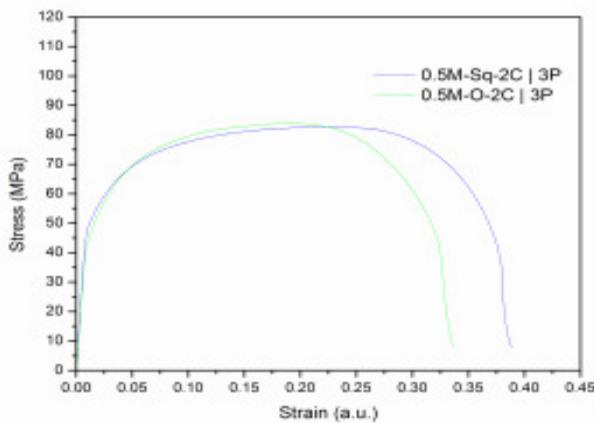


Figure 18: Effort vs. Deformation curve of the composite AA1100+MWCNT, a) material processed with circular pin, 0.5 molar concave shoulder with double

deposition layer and three passes (0.5M-O-2C | 3P) b) material processed with square pin, 0.5 molar shoulder with double deposition layer and three passes (0.5M-Sq-2C | 3P).

Sample	0.5M-O-2C 1P	0.5M-Cua-2C 1P	MB
Elongation(%)	31.4	34.3	42
Cadencie effort (MPa)	40.9	51	118.5
Maximum effort (MPa)	91.9	94.9	127.7
Young's module (GPa)	4.8	4.5	4.4

Table 6: Properties of the compound Al+MWCNT processed circular pin three passes. Against the properties of the compound Al+MWCNT processed square pin three passes.

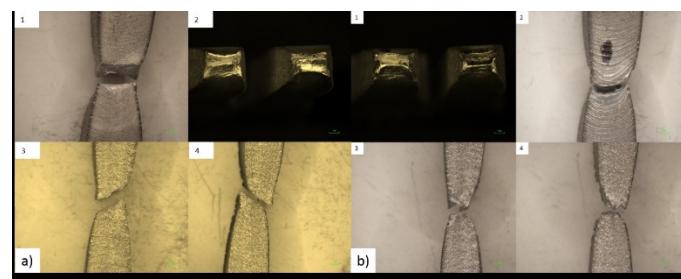


Figure 19: a) material processed with circular pin, concave shoulder at 0.5 Molar with three passes (0.5M-O-2C | 3P) b) material processed with square pin, straight shoulder at 0.5 Molar and three passes (0.5M-Cua-2C | 3P).

In all the cases, due to the recrystallization that the material suffers once the friction agitation processing is executed, the resistance to traction decreased concerning the base material, but the capacity of deformation increased in the samples obtained longitudinally to the processed zone by approximately double compared with the base material.

C. Fesem of traction tests

The images taken by FESEM JEOL are very similar to those taken in the study of the article [8], where the aspect of the material due to the multiple passes of the tool is different from the base material.

Figure 20 shows the typical behavior of a ductile material, with its respective dimples. Figure 21 shows the rupture of the material processed with carbon nanotubes with a single pass of the tool and circular pin, in it the different transitions that the material suffers can be appreciated, as they are in the surface the processed material affected by the shoulder, in the middle the content affected by the pin and in the low part the base material, by using FESEM the MWCNT can be observed coupling in the matrix of the AA1100.

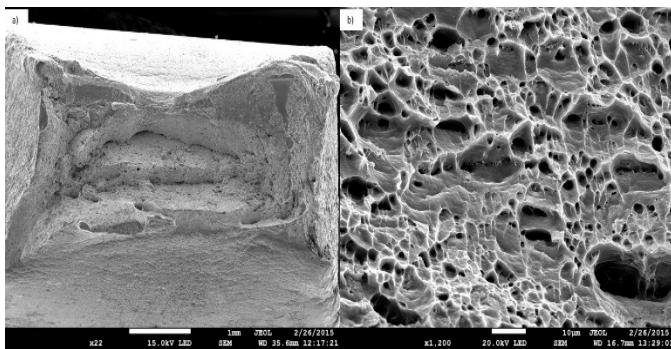


Figure 20: Base material (BM) a) 22X approach and b) 1200X approach.



Figure 21: Material processed with circular pin, concave shoulder at 0.5 molar with double deposition layer and 0.5M-O-2C 1P tool pass, a) 20X approach and b) 12000X approach.

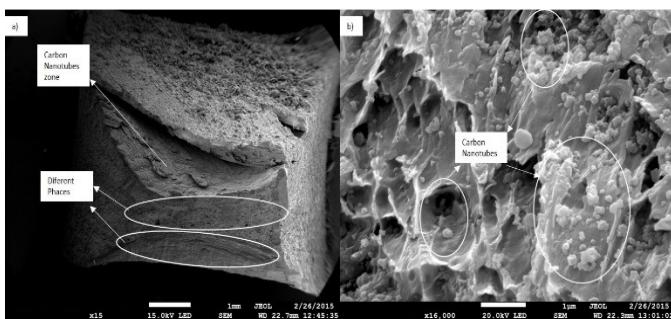


Figure 22: Material processed with circular pin, concave shoulder at 0.5 molar with double deposition layer and three passes (0.5M-O-2C | 3P), a) 20X approach and b) 16000X approach.

IV. CONCLUSIONS

The results of the effort vs. deformation tests of this study reveal that friction-agitation processing generates changes in the mechanical response of this material. This response will depend on the orientation of the specimen at the time of testing, will also depend on the number of tool passes and if FSP modified the material with or without the MWCNT.

Probably due to the recrystallization that the material suffers once the friction agitation processing is executed, the resistance to traction decreased with respect to the base material, but the deformation capacity increased in the samples obtained

longitudinally to the processed zone by approximately the double in comparison with the base material; with this in mind, if a traction test is executed transversal to the treated region, the levels of deformation and traction resistance of the base material will not be achieved, but if the traction test is performed longitudinal to the processed zone, the traction resistance does not equal that of the base material but the deformation increases.

The square pin with straight shoulder generates greater grain refinement and a homogeneous distribution of the particles without surface detachment of the processed zone layer, with higher flexibility and the formation of a double neck in the tractioned samples transversal to the treated zone.

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