Study of stirred layers on 316L steel created by friction stir processing

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Abstract. Nanostructured materials are known to exhibit attractive properties, especially in the mechanical field where high hardness is of great interest. The friction stir process (FSP) is a recent surface engineering technique derived from the friction stir welding method (FSW). In this study, the FSP of an 316L austenitic stainless steel has been evaluated. The treated layers have been characterized in terms of hardness and microstructure and these results have been related to the FSP operational parameters. The process has been analysed using a Response Surface Method (RSM) to enable the stirred layer thickness prediction.

1. Introduction
Friction stir processing (FSP) is a recent surface engineering technique derived from successful joining method friction stir welding (FSW) nowadays widely applied to aerospace, automotive and naval industry.

Invented and patented two decades ago, FSW is a solid state welding process environmentally friendly, more economically efficient, safer and easier to control compared to other joining processes [1-4]. The operating principle of FSW is very simple: a rotating non-consumable cylindrical tool plunges between, then slides along the edges of parts to be welded. Tools are designed with a threaded or unthreaded pin. Welded joints are formed by a thermomechanical process. Material is brought to flowing state due to frictional heat from tool pin and shoulder, and then is stirred between parts to be welded due to rotation movement of the tool. Researchers investigated and modeled material flow during friction stir in order to find how it affects welded joints quality [5-7]. It was found that the weld properties were dominated by the thermal input rather than the mechanical deformation by the tool [8]. Furthermore, Kumar et al. demonstrated that the pin transfers material layer by layer and the tool shoulder transfers material by bulk [9]. Regardless this difference between shoulder-driven and pin-driven flows, experimental studies [10] confirmed that FSW produces stronger and less defect joints than fusion arc welding. Among other technical benefits of FSW joints are: higher tensile strength, superior microhardness, refined microstructure with equiaxed grains [2,11-15], better corrosion resistance [12, 16,17] and improved fatigue resistance [18-21].

Studies on various materials demonstrated that the quality of FS welded joints generally depends on:
- base material mechanical and thermal properties
- tool material, shape and dimensions [22]
- process parameters (rotational speed, traverse speed, axial force, tilt angle, cooling system) [1,9,10,15].

Recently, friction stir processes (FSP) have been used to create superficial nanostructured layers on different materials (aluminium alloys, titanium alloys or steels) [10]. Friction stir processing goal is to ameliorate surface properties either by creating surface layers with...
refined structure or by creating composite nanolayers by adding different elements in surface layers [23]. Numerous studies have been carried out for aluminum and other soft materials, FSP of hard materials still needs to be studied and optimized due mainly to tool wear and non-homogeneity of the superficial layer after processing. In that sense, concerning the FSW/FSP of stainless steels several investigations have mainly aimed at the evaluation of corrosion behavior of so treated materials [24, 25]. An example is the work conducted by Park et al.[31] regarding the FSW of 304L stainless steel. They reported for the first time the formation of the sigma phase along the grain boundaries and concluded that the presence of sigma phase will not only affect mechanical properties of the material but also reduce the corrosion resistance. More recently Chen et al. [26] suggest that low heat input contributes to restrain sigma phase precipitation.

The present investigation has been conducted in order to study furthermore the behavior of the stirred 316L, tested to relatively severe processing input conditions (rotational speed, normal force and traverse speed) and to evaluate the possibilities to increase the hardness of the material for mechanical applications. In particular the primarily performance in term of size of nanocristallites and depth change have been investigated. The process has been also analyzed using a Response Surface Method (RSM) design in order to predict the thickness of the stirred layer as a function of input parameters. This approach could be useful from industrial point of view since FSP enable rapid treatments of surfaces using traditional machine tools and furthermore and can be used as guide line. The results presented are a first part of the works carried out by IRTES-LERMPS laboratory on FSP on stainless steels. If the hardness of the 316L base material can clearly be improved using our FSP process, the corrosion resistance of the treated surface is under evaluation in order to check the remaining “stainless” character.

2. Experimental

2.1. Materials
The present investigation has been carried out using of 316L stainless steel samples. This material is commonly employed in the manufacture of different components that could undergo friction and wear during operation and an elevated hardness of superficial layers could be interesting to enhance the service life. The material was provided in the shape of rectangular plates of 5 x 40 x 120 mm which were milled in order to decrease the surface roughness to Ra≈3.2±0.015 which is representative of real industrial surfaces. The composition of 316L steel is given in Table 1 and the initial grain size is about 40±5 µm as shown in Figure 1. The samples present a Vickers hardness of 200 ±10 % HV before FSP.

| Material | C | Cr | Ni | Mn | Mo | Si | P | S | N | Fe |
|----------|---|----|----|----|----|----|---|---|---|----|
| 316L     | 0.03 | 16 | 10 | 2 | 2 | 0.75 | 0.04 | 0.05 | 1 | bal. |
2.2. Friction stir processing
Friction stir experiments presented here have been performed at UTBM (IMSI Department plateform) on a 4-axis numerically controlled milling center (Figure 2), model Gambin50C adapted adding Kistler force measuring and control devices. The tool is a 10 mm diameter PCBN cylinder, without any pin.

Each test runs following two steps: pressure was first applied, then FSP was carried out by rotating the tool at a constant speed.
At the end of each test, microstructural analysis was carried out using an optical microscope (OM) and a scanning electronic microscope (SEM). The thickness of the stirred layers was measured by image analysis and indentation tests were conducted employing a micro hardness tester with a Vickers indentor. The indentation tests were performed at the same maximum load of 50 mN at 15 µm from the surface.

2.3. Design of experiments
In order to establish the functional relationship between friction stir process parameters (normal force, rotation speed and traverse speed) and the thickness of the affected layer by friction stir processing it was used a Box-Behnken experimental design [27], a Response Surface Method(RSM) design was applied to analyze and model experimental data using few
test runs. The factor combinations are placed at the center and at the midpoints of edges of the process space as is shown in Figure 3 for three factors.

![Figure 3. Box-Behnken experimental design for three factors](image)

The levels of the three factors were established after preliminary tests that set their limits of variation (Table 2).

| Independent variables | Coded | Levels          |
|------------------------|-------|-----------------|
|                        |       | -1 (low) | 0 (center) | 1 (high) |
| Normal force F (N)     | x1    | 900      | 1350       | 1800      |
| Rotation speed n (rpm) | x2    | 500      | 700        | 1000      |
| Traverse speed f (mm/min) | x3  | 100      | 140        | 200       |

3. Results and discussion

Changes in the 316L microstructure due to the dynamic recrystallization were obtained depending on the FSP conditions. Figure 4a shows a typical SEM cross section of the stirred steel obtained during the present experiments under a normal load of 900 N, a rotational speed of 700 rpm and a traverse speed of 200 mm/min. From the base material (BM) up to the surface, a foliated structure - the thermomechanically affected zone (TMAZ) followed by a layer with very fine grains - the stirred zone (SZ) can be observed. These structures are similar to those obtained in case of fretting or impact conditions [29]. As shown in Figure 4b the grain size has become finer in SZ in comparison to the base material. The as received 316L had an average grain size of 40±5 µm while in the SZ the average grain size at 15 microns below the processed surface was found to be 110 ±10 nm (Table 2). Concerning the
The evolution of the hardness, when fine grains are formed, the resulting hardness lies within the range $440 \text{ HV}_{0.05} \pm 20$ without significant variations between various samples.

Figure 4. Stirred layer on 316L under a normal load of 900 N, a rotational speed of 700t/min and a traverse speed of 200 mm/min.

To study the influence of the rotational speed and pressure on the stirred layer thickness, a mathematical model for depth of the friction stir zone was obtained from experimental data.

For three factors factorial Box-Behnken experimental design, standard design includes three replicates in the central point, therefore $N=15$ runs as shown in Table 3, table that also includes processing conditions and results.

The proposed model for the stirred zone $SZ$ can be expressed as a power function

$$SZ = a_0 \cdot F^{a_1} \cdot n^{a_2} \cdot f^{a_3} \cdot \varepsilon'$$

(1)

where $SZ$ is the thickness of the stirred zone ($\mu$m), $F$ is the normal force (N), $n$ is the rotational speed (rpm), $f$ is the traverse speed (mm/min), $\varepsilon'$ is the experimental error and $a_0$, $a_1$, $a_2$, $a_3$ are the model parameters to be estimated.

The first step in parameter estimation is transforming the nonlinear model stipulated in Eq. 1 into a first order polynomial model as in Eq.2

$$\ln(SZ) = b_0 + b_1 \ln F + b_2 \ln n + b_3 \ln f + \ln(\varepsilon')$$

or

$$y = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 + \varepsilon$$

(2)

where $y$ is measured $SZ$ on a logarithmic scale, $x_1, x_2, x_3$ are coded values for $F$, $n$ and $f$ and $b_0, b_1, b_2, b_3$ are linear model parameters.
### Table 3 Friction stir processing conditions and results

| Trial no. | Normal force $F$ (N) | Rotation speed $n$ (rpm) | Traverse speed $f$ (mm/min) | Levels of variation X1 | Levels of variation X2 | Levels of variation X3 | Stir zone SZ (µm) | Grain size (nm) |
|-----------|----------------------|--------------------------|-----------------------------|------------------------|------------------------|---------------------|-----------------|----------------|
| 1         | 900                  | 500                      | 140                         | -1                     | -1                     | 0                   | 65              | 212            |
| 2         | 1800                 | 500                      | 140                         | +1                     | -1                     | 0                   | 109             | 124            |
| 3         | 900                  | 1000                     | 140                         | -1                     | +1                     | 0                   | 111             | 128            |
| 4         | 1800                 | 1000                     | 140                         | +1                     | +1                     | 0                   | 150             | 89             |
| 5         | 900                  | 700                      | 100                         | -1                     | 0                      | -1                  | 100             | 117            |
| 6         | 900                  | 700                      | 200                         | -1                     | 0                      | +1                  | 70              | 88             |
| 7         | 1800                 | 700                      | 100                         | +1                     | 0                      | -1                  | 116             | 83             |
| 8         | 1800                 | 700                      | 200                         | +1                     | 0                      | +1                  | 114             | 102            |
| 9         | 1350                 | 500                      | 100                         | 0                      | -1                     | -1                  | 83              | 128            |
| 10        | 1350                 | 1000                     | 100                         | 0                      | +1                     | -1                  | 151             | 117            |
| 11        | 1350                 | 500                      | 200                         | 0                      | -1                     | +1                  | 51              | 121            |
| 12        | 1350                 | 1000                     | 200                         | 0                      | +1                     | +1                  | 114             | 96             |
| 13        | 1350                 | 700                      | 140                         | 0                      | 0                      | 0                   | 94              | 110            |
| 14        | 1350                 | 700                      | 140                         | 0                      | 0                      | 0                   | 83              | 112            |
| 15        | 1350                 | 700                      | 140                         | 0                      | 0                      | 0                   | 93              | 115            |

The independent variables were coded on a natural logarithmic scale using the relations presented in Eq. 3.

\[
x_1 = \frac{\ln F - \ln(1350)}{\ln(1800) - \ln(1350)}
\]

\[
x_2 = \frac{\ln n - \ln(700)}{\ln(1000) - \ln(700)}
\]

\[
x_3 = \frac{\ln f - \ln(140)}{\ln(200) - \ln(140)}
\]

Using the notation $y_m$ for the modeled response on a natural logarithmic scale, Eq. 2 becomes

\[
y_m = y - \epsilon = b_0 + b_1x_1 + b_2x_2 + b_3x_3
\]  

(4)

If interactions between factors are taken into account, factors $x_1x_2$, $x_1x_3$ and $x_2x_3$ are added to Eq. 4 and the predictive model is a first order polynomial function:

\[
y_m = y - \epsilon = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3
\]  

(5)

For parameter $b_0$, $b_1$, $b_2$, $b_3$, $b_{12}$, $b_{13}$, $b_{23}$, estimation it was applied the method of least squares and the following predictive response model for coded factors was obtained (Eq.6).
Parameter calculus, statistical evaluation of the results and graphic representations were performed using Minitab and MathCAD.

\[ y_m = 4.45759 + 0.18338x_1 + 0.28264x_2 - 0.14250x_3 - 0.05411x_1x_2 + 0.8620x_1x_3 + 0.05066x_2x_3 \]  

(6)

All predictive models must be tested in order to be validated. ANOVA analysis was applied to the linear model obtained previously (Eq.6) to test the adequacy and estimate significance level of model variables. The adequacy test, also named lack-of-fit test assesses the rightness of the model showing if the model should be accepted because it is complete and no other variables must be taken into account. In Table 4 are compared the mean square (MS) of the lack-of-fit to the mean square of the pure error in order to use F distribution for adequacy test. Calculated ratio \( F_{\text{calc}} \) of those two means of squares is smaller than the tabulated F corresponding to a 95% confidence limit showing that the lack-of-fit is within reasonable limits and no other significant factors must be added to the model.

| Variance source | df | SS     | MS     | \( F_{\text{calc}} \) | \( F_{\text{tab}}^* \) | Remarks               |
|-----------------|----|--------|--------|-----------------------|-----------------------|-----------------------|
| Regression      | 6  | 1.12224| 0.187042|                       |                       |                       |
| Lack of fit     | 6  | 0.11022| 0.018370| 3.67                  | 19.33                 | Adequate              |
| Pure error      | 2  | 0.01002| 0.005008| (\( F_{\text{calc}} \) < \( F_{\text{tab}}^* \)) |                       |                       |
| Total           | 14 | 1.24248|         |                       |                       |                       |

\*\( F_{0.05} \) for a 95% confidence level

Significance test (over-fit test) identifies factors without significant influence on response variable (Table 5). The mean of squares for each factor is divided by mean of squares of residual error and F ratio obtained \( F_{\text{calc}} \) is compared to the tabulated ratio for 95% confidence level \( F_{\text{tab}} \). If \( F_{\text{calc}} > F_{\text{tab}} \) the factor is considered significant, otherwise the factor is not significant and can be removed from the model. Results presented in Table 5 show that all factors are significant, the most influent of them being the rotation speed, while none of the interactions are significant.

| Variance source                          | df | SS     | MS     | \( F_{\text{calc}} \) | \( F_{\text{tab}}^* \) | Remarks     |
|------------------------------------------|----|--------|--------|-----------------------|-----------------------|-------------|
| Force (x1)                               | 1  | 0.26903| 0.269029| 17.90                 | 5.32                  | Significant |
| Rotation speed (x2)                      | 1  | 0.63908| 0.63908 | 42.52                 | 5.32                  | Significant |
| Traverse speed (x3)                      | 1  | 0.16244| 0.16244 | 10.81                 | 5.32                  | Significant |
| Force*Rotation speed (x1x2)              | 1  | 0.01171| 0.01171 | 0.78                  | 5.32                  | Not significant |
| Force*Traverse speed (x1x3)              | 1  | 0.02972| 0.02972 | 1.98                  | 5.32                  | Not significant |
| Rotation speed*Traverse speed (x2x3)     | 1  | 0.01026| 0.01026 | 0.68                  | 5.32                  | Not significant |
After the removal of non-significant interactions from the model, the predictive response function on the natural logarithmic scale is:

\[ y_m = 4.45759 + 0.18338x_1 + 0.28264x_2 - 0.14250x_3 \]  \hspace{1cm} (7)

The values of coefficients of determination \( R^2 \) and \( R^2_{adj} \) of 86.16%, respectively 82.39%, indicate a good fit between experimental data and the new predictive linear model (Eq.7). Therefore, based on the linear model the different parameters \( a_i \) in Eq.1 were calculated and the predictive nonlinear model for the thickness of the stir zone \( (SZ_m) \) was found:

\[ SZ_m = 0.038 \cdot F^{-0.62} \cdot n^{0.82} \cdot f^{-0.41} \]  \hspace{1cm} (8)

The graphic representation of the influence of process variables on the thickness of the transformed layer is shown in Figure 5. It can be observed that setting higher values for normal force and rotation speed increases the thickness \( SZ \), while increased traverse speed leads to a reduction of \( SZ \).
Figure 5. Graphic representation of transformed layer thickness $SZ$ as a function of normal force and rotation speed (a), as a function of traverse speed and normal force (b) and as a function of traverse speed and rotation speed (c).

4. Conclusion
Friction Stir Process on 316L stainless steel can lead to the formation of a stirred layer of thin grains whose thickness depends on input FSP conditions. A predictive model was provided for the thickness of the stirred layer depending on the process parameters: normal force, rotation speed and traverse speed. The model is valid within the limits of studied domain: $F=900-1800$ N, $n=500-1000$ rpm and $f=100-200$ mm/min.

- In the case of 316L the most influent factor on the depth of the transformed layer via friction stir is the rotation speed of the tool.
- Secondly, the depth of stirred layer is affected by the normal force. This is in good agreement with recent results obtained on 1045 carbon steel. For example, studying the FSP effects on this material, Langlade and al. have shown that the SZ thickness depends on the normal load in the case of a constant rotational speed [28].
- It can be assumed that increasing rotation speed and or/normal force, the depth of stir zone is augmented probably due to more severe mechanical and thermal conditions. Negative exponent of traverse speed in the predictive model and graphic representation (Figure 2) show that higher values of traverse speed lead to a reduction of the thickness of transformed layer, probably due to a more rapid cooling of processed surface.
Good agreement was shown between the predictive model results and the experimental measurements

5. References

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