Original Article

Microstructure and mechanical properties of friction stir welded 8 mm pipe SAF 2507 super duplex stainless steel

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\begin{abstract}

The microstructure and mechanical properties of 8-mm-thick friction stir welded SAF 2507 super duplex stainless steel pipe was examined. High-quality welds were successfully produced using polycrystalline cubic boron nitride (PCBN) tool, force control mode of 38 kN, travel speed of 50 mm/min, rotational speed of 200 rpm. Hardness and tensile tests were performed, showing hardness increase in the stir zone and failure on the base metal. Thermal simulation showed maximum global temperatures around 1176 °C, and different thermal cycles along the joint thickness, providing microstructural and mechanical differences along the welded joint.

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\end{abstract}

1. Introduction

Super duplex stainless steel (SDSS) are biphasic ferrite-austenite alloys based on the Fe–Cr–Ni system \textsuperscript{[1].} The two-phase structure of ferrite and austenite combines the beneficial effects of the phases and allows a global high strength (ferrite) and toughness (austenite) even at low temperatures \textsuperscript{[1–4].} SDSS exhibit higher resistance to stress corrosion cracking and strength than austenitic stainless steels, widely used in many applications as structural materials in various industrial sectors including chemical, petrochemical, power generation, pulp and paper, and pollution control industries \textsuperscript{[1,3]}

SDSS family alloys have good weldability, however, special care is required to assure mechanical properties are unchanged after the process. The interference on properties depends on the welding process used. In the case of arc welding, there is a direct influence of the welding energy, the process employed (energy density effect) and the deposition technique. These parameters affect the phase balance in SDSS...
resulting in poor mechanical properties and corrosion resistance [1,5,6].

In the case of friction stir welding (FSW), a solid-state process, there is an important influence of the thermal and mechanical history to which the material is subjected. The above-mentioned influenced some researchers to deal with the microstructure and mechanical properties of friction stir welded DSS [7–10].

Recent investigations showed that FSW can produce defect-free welds, on SAF 2507 (e.g. [6]) and SAF 2205 (e.g. [7,8]). In these studies, FSW refined the two constituent phases in the stir zone (SZ), and hence mean hardness and tensile strength were improved in the weld zone. Sato et al. processing 4 mm thick SAF 2507 plates, found that FSW in 2507 SDSS significantly refined the ferrite and austenite phases through dynamic recrystallization.

Saeid et al. [7] processing SAF 2205 duplex stainless steel plates relate the degree of grain refinement to the welding speed applied in the process. The grain size of SZ welded depends on two factors: thermal cycle and distribution of deformation. A faster tool welding speed resulted in a higher strain rate and lower peak temperature.

Despite the advance in solid-state joining of SDSS, the development of thicker joints in pipeline welding has also been an important factor in welding technology. Recent studies provided by The Welding Institute (TWI) have reported significant cost savings when using FSW to join thicker material sections [11]. While FSW typically travels slower than traditional arc welding processes, it is capable of joining thicker sections in a single pass resulting in significant cost savings when building large steel structures such as oil and gas pipelines, offshore platforms, or heavy machinery and maintaining its mechanical and corrosion resistance to values equal or superior to the base metal [11]. Recent studies have produced plate joints in 1.5 mm (e.g. [8]), 2 mm (e.g. [7]), 4 mm (e.g. [6]) and 6 mm (e.g. [12]) duplex stainless steels with excellent surface finish and complete penetration. The literature does not report any FSW study in thicker plates nor thicker pipes.

The present work characterizes mechanically and microstructurally 8-mm-thick SDSS SAF 2507 circumferential joints produced by friction stir welding. Hardness and tensile tests were performed to verify the mechanical resistance of the joint and further microstructural characterization was used to understand the effect of the process thermal cycles in the material.

### 2. Materials and methods

Two annealed SDSS SAF 2507 130-mm-long pipes were used in the tests. The chemical composition of the alloy is given in Table 1.

<p>| Table 1 – Chemical composition (in wt%) of the base metal. |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|</p>
<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Mn</th>
<th>Ti</th>
<th>P</th>
<th>S</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.021</td>
<td>22.13</td>
<td>7.68</td>
<td>3.76</td>
<td>0.84</td>
<td>0.002</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

The internal and external diameter were respectively 168.3 mm and 184.3 mm, conferring a total of 8 mm in thickness. A schematic view of the pipe geometry is illustrated in Fig. 1(a). No additional bevel preparation was used before the joining procedure.

The tool was a 7.72-mm-long step-spiral conical pin with a plane threaded shoulder made of metallic matrix composite PCBN–WRe (25%Re–W alloy reinforced with 60%vol Polycrystalline Cubic Boron Nitride), illustrated in Fig. 1(b).

A force control mode was made with a total tool penetration of 38 kN in the axial direction with a travel speed of 50 mm/min, spindle speed of 200 rpm. These parameters were selected based on a previous work in 2205 plates [6,9] and in-house development. During welding, axial force, torque and tool temperature were obtained and analyzed. Microstructure was examined by optical microscopy (OM) and scanning electron microscopy (SEM) along the stir zone (SZ), in both advancing (AS) and retreating side (RS). These notations are used throughout the present paper. Samples for OM and SEM examination were electrolytically etched in a 40% nitric acid solution applying a potential of 2 V for 120 s. Vickers hardness test was conducted on the cross-section perpendicular to the welding direction, using a Vickers indenter with a 200 N load for 15 s, to examine the hardness distribution. Hardness map was produced using 18,450 indents along the sample, assigning a color scale for representation. Transverse tensile

![Fig. 1 – (a) Pipe dimensions and welding direction schematics. (b) Tool geometry dimensions.](image-url)
Table 2 – Heat transfer coefficients applied to the thermal model.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>Free convection</td>
<td>20 W/m²K</td>
</tr>
<tr>
<td>Surface radiation</td>
<td>0.3</td>
</tr>
<tr>
<td>Side tool convection</td>
<td>20 W/m²K</td>
</tr>
<tr>
<td>Top tool convection</td>
<td>500 W/m²K</td>
</tr>
<tr>
<td>Backing plate</td>
<td>2000 W/m²K</td>
</tr>
</tbody>
</table>

Specimens were removed perpendicular to the welding direction. Tensile tests were carried out at room temperature on a 250 kN MTS tensile testing machine at a crosshead speed of 3 mm/min.

Thermal simulation was performed in three-dimensional finite element (FE) model developed using commercial software COMSOL v5.2. Assuming steady-state conditions, the model calculated the thermal cycles during the process. Welding parameters for the simulations were constant, such as axial force (38 kN), spindle speed (200 rpm) and welding speed (50 mm/min). Tetragonal elements with smaller sizes (ranging from 2 to 20 mm) in the regions near the tool than the rest of the workpiece were used at the mesh.

Free convection boundary conditions were applied to the workpiece surfaces. Thermal radiation boundary condition was also applied to the top surface of the workpiece with an emissivity coefficient. Heat transfer through the backing plate and tool top were considered and convection boundary conditions were applied to the side and to the top of the tool. All coefficients listed in Table 2 were based on previous work and in-house development [9]. Due to the complexity associated with modeling the friction stir welding tool with a step-spiral pin, a conical shape with a smooth pin surface (without threads) was used.

The heat source considers two conditions: a surface heat generation and a volume heat source, based on Schmidt and Hatel work [10]. The volume flux is calculated from the average power input \( P_{avg} \) during welding expressed by Eq. (1), where \( M_{avg} \) is the average torque measured during welding and \( \omega \) is the tool rotation speed.

\[
P_{avg} = M_{avg} \omega
\]  

Using Eq. (2), the volume flux can be calculated analytically using Eq. (2)

\[
Q_{volume} = \frac{f_{pin} P_{avg}}{V_{pin}}
\]  

where \( V_{pin} \) is the tool pin volume and \( f_{pin} \) is the percentage of heat generation from the pin side relative to the total heat generation. For this work, a value of 20% for \( f_{pin} \) was used based on the work of Colegrove et al. [13].

The surface heat flux expression must account for the heat generated by friction and by plastic deformation, expressed by Eq. (3)

\[
Q_{surface} = (1 - f_{pin}) \left[ \delta_{yield}(T) + (1 - \delta) r_{friction} \right] \omega r
\]

where \( r \) is the radial distance from the tool center, \( r_{friction} \) is the frictional stress at the tool/workpiece interface, \( \delta_{yield}(T) \) is the material yield shear stress at the welding temperature \( T \) at this same interface and \( \delta \) is a contact state variable that balances the heat generated by friction and by plastic deformation.

To account for the non-symmetric heat generation in the welding process, Eq. (3) can be rewritten as Eq. (4)

\[
Q_{surface} = (1 - f_{pin}) (\omega r - U_{weld} \sin \theta) r_{yield}(T)
\]  

The model coefficients were calibrated using an inserted thermocouple into the tool head and attached to the FSW machine. This thermocouple positioned by the tool shoulder does not provide the exact tool temperature at the interface with the processing material within the SZ. However, due to the high thermal conductivity of PCBN, it provides a comparative value for various welding parameters [14].

3. Results and discussion

The appearance of the weld surface is shown in Fig. 2. It is observed good surface conditions, with little flash and no superficial defects.

The results of force, torque and temperature are seen in Fig. 3. Both axial force and torque presented variation throughout the process, with values varying between 37.5 and 38 kN for axial force and 225 and 260 Nm for torque. These variations suggest geometrical irregularities presented in the workpiece, resulting in variation of the tool response throughout the process. Both axial force and torque values are dependent on both size and geometry of the tool and the material to which it interacts, so they are often not reported in publications. However, for comparison purposes, Saied et al. [7] welding 2 mm duplex Plates 2205 with a 3.8 mm tool obtained maximum values of axial force of 14 kN. The smaller values are due the less material displacement promoted by the 3.8 mm tool compared to 7.8 mm tool used in this paper. Temperature measured reached max values of 805°C in 160° position. The values of temperature changed according to the torque and axial response of the machine, rising after torque pikes and axial down pikes.

3.1. Hardness and tensile test results

Fig. 4 shows the cross-section of the weld bead at low magnification, indicating the advancing side (AS) and retreating side (RS) of the joint. It is apparent that a sound joint was produced.
It is also noted an incomplete root joint, possibly due to lack of penetration of the tool during welding.

The transverse hardness profile of the FSW SAF 2507 super duplex stainless steel is indicated in Fig. 5. Maximum values reached 400 HV in the advancing side/root region. The overall stir zone reached values around 325 HV, slightly higher on the advancing side. Esmailzadeh et al. [8] processing lean duplex stainless steels reached hardness values around 350 HV for 800 rpm rotational speed and 150 mm/min transverse speed. The author relates the hardness increase to the smaller grain sizes found in the stir zone.

Tensile test specimens failed roughly 40 mm from the weld center, located in the base metal, as seen in Fig. 6. Comparing to the hardness results, the failure occurred on lower hardness sites, which is proportional to the strength of metallic materials.

To understand the hardness and failure results, microstructural analysis was taken in the weld cross section.

3.2. Microstructure

Fig. 7 shows the SAF 2507 base metal. It displays white austenite (γ) islands embedded in a gray matrix of ferrite (α). Both have elongated shapes with volume fractions of 0.52 and 0.48, respectively, measured using a ferristoscope Fischer FMP30. The measured average grain sizes were 23.2 μm for austenite and 17.3 μm for ferrite [15]. Similar results were obtained by Sato et al. [6]. The base material showed no deleterious phases.
remaining in the manufacturing process after solubilization heat treatment.

The welded cross section was analyzed, focusing on three main regions: advancing side, retreating side and root, seen in Fig. 8 by optical (MO) and electronic analysis (SEM).

The phase balance after the process remained around 50%, indicating in all three regions similar phase balance of the base metal.

On the welded condition, the thermomechanically affected zone (TMAZ) is noticeable on the advancing side, showing a sharp morphology, being limited to 50 µm distance from the stir zone. In addition, the material in the TMAZ region shows severe grain deformation, re-orientated perpendicular to the weld traverse direction of the base metal due to the stirring action of the rotating tool. In the retreating side, a smoother transition between stir zone and base metal was noted.

In the stir zone, the morphology of ferrite and austenite islands was much different from that of the base material. The grain size of austenite and ferrite in the stir zone were smaller than those in the base metal. During FSW, the material in the stir zone experiences large shear stresses along the pin tool surface and exposure to high temperatures [16]. These conditions combined suggest that dynamic recrystallization occurred both in the austenite and ferrite phases during FSW.

Comparing the two sides of the stir zone, the grain size of austenite and ferrite (1.3 µm and 1.2 µm respectively) in the AS (Fig. 8b-II) showed smaller values compared to grains in the retreating side (2.1 µm and 1.9 µm respectively) (Fig. 8c-II), according to ASTM-E112-12 [15]. A previous study investigating material flow of FSW using an in situ marker technique showed that material located on the advancing side of the stir zone is displaced around the entire circumference of the pin tool, leading to a severe deformation, which would tend to break up the austenite islands producing a smaller grain size [17].

In the root, an intense grain refinement was also observed, with some elongated ferrite dispersed in the region. The smaller grain sizes found in that region suggest that material was exposed to lower thermal cycles and/or faster cooling rates, preventing grain growth in the region. Indications of secondary deleterious phases were not observed by scanning electrochemical microscopy, according to Fig. 8b-II, c-II and d-II. Santos et al. [9] and Saied [8]; Sato et al. [6] working with soldering of steels S32205 and S32750 did not observe any
To joints 7 stir intermetallic phase. Amigó et al. [18] studying welded laser joints of AID UNS S32205 indicated the formation of chromium nitrides of the thermally affected zone (ZTA) of the joint. This occurs due to the severe cooling rate that allows the reformulation.

In that way, complementary thermal simulation was performed to verify the thermal cycles along the joint thickness.

4. Thermal simulation

To support the microstructural evolution analysis, temperatures during the process were calculated by numeric simulation. The model was calibrated using the temperature measurements from the thermocouple located in the collar of the friction stir tool. As seen in Fig. 3, the temperature in the tool reached values around 780–800 °C. Fig. 9(a) shows the simulation results. The model exhibits good agreement with the reference tool thermal cycle curve. In Fig. 9(a), the maximum temperature calculated in the model was 1176 °C.

Santos et al. [9] predicted a global maximum temperature equal to 1183 °C located near the tool pin and 0.145 mm below the tool shoulder in a 6-mm-thick plate. In this way, it can be noticed that regardless of the thickness similar temperatures are achieved near the shoulder, which is the major energy source of the process.

According to Fig. 9(b), simulation results show that volume material close the tool shoulder (2 mm depth) experimented longer periods at high temperatures (>1100 °C) than the lower regions of the cross-section (7 mm depth). Also, due to the proximity to the backing plate, materials in lower regions experience faster cooling rates than outer region materials. In this way, material located in lower regions suffer deformation but at lower temperatures (~1000 °C) and faster cooling rates \( T_{12/8} = 40 °C/s \), conferring to ferrite and austenite grains, as seen in microstructural analysis, less energy to undergo grain growth after dynamic recrystallization.

This phenomenon is directly caused by the dimension of the pin and its limitation to provide the same amount of energy along the entire pin length. As the length of the pin increases, the distance between materials deformed and the major heat source of the process (tool shoulder) also increase. This results in different thermal cycles applied to the material along the thickness processed by friction stir welding. Different thermal cycles provide microstructures that exhibit disparate mechanical behavior along the welded joint.

Also, the cooling rates observed prevented the formation of secondary phases, for example sigma phase \( \sigma \). The statement is supported by Elmer et al. [19] work which monitored real-time transformations on 2250 steels in isothermal tests at temperatures of 700, 750, 800 and 850 °C. According to the study, sigma phases appears first after 96 s at 800 °C. In the present study, for the lowest cooling rate (22 °C/s), the total cooling time was 50 s, which suggest, supported by Fig. 8 SEM images, that no second phase is formed during welding.

5. Conclusion

The present study examined the microstructure and mechanical properties of an 8-mm-thick friction stir welded SAF 2507 super duplex stainless steel pipe. The following conclusions were made:

- Force control mode produced small flash and no superficial defects high-quality welds.
- Hardness tests revealed maximum values of 400HV in the root region. The overall stir zone reached values around 325 HV, slightly higher on the advancing side.
- Tensile tests resulted in failure on lower hardness sites, located in the base metal.
- In the root, an intense grain refinement was also observed, with some elongated ferrite dispersed in the region. The smaller grain sizes found in that region suggest that material was exposed to lower thermal cycles, preventing grain growth.
- The simulation results show that volume material along the upper regions of the pipe experimented longer periods at high temperatures (>1100 °C) than the bottom region of the cross-section. Material located in the bottom region receives less energy, which hinders grain growth after dynamic recrystallization.
- As the length of the pin increases, the temperature gradient between the outer and inner surfaces of the pipe also
increases, because the major heat source of the process is the tool shoulder. This results in different thermal cycles applied to the material along the thickness processed by friction stir welding, which provides disparate microstructures and mechanical behavior.

**Conflicts of interest**

The authors declare no conflicts of interest.

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**References**