Original Article

Microstructure, texture and mechanical properties of a nickel-free high nitrogen duplex stainless steel processed through friction stir spot welding

S. Yousefian, A. Zarei-Hanzaki, A. Barabi, H.R. Abedi, M. Moallemi, P. Karjalainen

Abstract

A nickel-free high nitrogen duplex stainless steel holding high Mn-content was friction stir spot welded under the various rotational speeds. The microstructure/texture evolutions of the joints and the correlated mechanical properties were investigated in detail. The ferrite and austenite constituent massive phases were refined down to the mean grain size of 0.9 μm and 1.1 μm, respectively. The microstructure evolutions revealed the activation of continuous dynamic recrystallization as the main restoration mechanism. This was further verified through the formation of B- and A-fiber shear texture in the specimens processed under the various processing condition. Besides the activation of different restoration micro-mechanisms in both ferrite and austenite, dynamic phase transformation of ferrite to austenite was also contributed in grain refinement. The transformation was facilitated by providing more diffusional paths through substructure development and grain refinement. The local and bulk mechanical properties of the joints were also assessed. The outstanding tensile-shear peak load of 12.8 KN obtained for the sample welded at 400 rpm was discussed relying on the developed ultrafine-grained microstructure and the maximum peak extension of 1.7 mm achieved at 600 rpm was attributed to the role of ferrite phase in strain accommodation.

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Duplex stainless steels (DSS) consist of ferrite (\(\alpha\)) and austenite (\(\gamma\)) phases. The study of grain refinement in austenite during high temperature deformation and its implications on properties is crucial. The lean duplex stainless steels containing lower nickel and molybdenum content with corrosion resistance on a par with standard austenitic grades are being developed to reduce the production cost [2–5]. The conventional fusion welding of DSS meets frequently problematic issues such as hydrogen embrittlement, formation of intermetallic brittle phases and micro-segregation of alloying elements. These problems would also degrade both the corrosion resistance and mechanical properties of the welded joints [6–8]. To avoid such imperfections, solid state welding such as Friction Stir Welding (FSW) and Friction Stir Spot Welding (FSSW) has been employed effectively as a promising fabrication method. However, the influence of welding parameters of such novel methods on providing the optimal refined microstructure holding acceptable mechanical properties is crucial and needs to be assessed in details.

In this respect, Jeon et al. [9] have studied the grain refinement mechanism during FSSW of a single crystal austenitic stainless steel. It has been found that at a lower plunge depth, continuous dynamic recrystallization operates as the main restoration mechanism in both the thermomechanically affected zone (TMAZ) and stir zone (SZ). By contacting the shoulder to the work-piece, the amount of heat input and severity of deformation increase leading to activation of discontinuous recrystallization which is static in nature and occurred during cooling cycle. Similarly, Sato et al. [10] has reported the 304L stainless steel not only experience dynamic recrystallization in the course of FSW, but also static recrystallization may occur in the weld zone containing a high density of dislocation during post-process heat treatment. Liu and Nelson [11] investigated the grain refinement of a 304L austenitic stainless steel during FSW by ‘Stop-Action’ technique and denoted the occurrence of discontinuous dynamic recrystallization as the main restoration process in the SZ. Regarding the response of DSS to the severe high strain rate deformation imposed by FSW, Esmailzadeh et al. [12] reported that austenite has higher tendency to grain refinement than the ferrite phase has. In agreement, Sato et al. [13] found more grain refinement in austenite obtaining the mean equiaxed grain size of about 2.2 \(\mu\)m at the stir zone. A comprehensive study regarding the high temperature deformation behavior of DSS can be found in [14]. Apart from the grain refinement, the orientation of the grains within the SZ would also affect the subsequent mechanical properties. The Shear texture components are one of the common feature which are often observed in the SZ [15]. The shear component of B/\(\gamma\)(112) < 110> has been observed near the tool by Liu and Nelson [11]. This has been attributed to severe deformation conditions and evolved to C/C(001) < 110 > component when the tool passes through the material and the deformation severity is reduced. The tensile−shear properties of a friction stir spot welded (FSSWed) twinning induced plasticity steel has been correlated with the shear textures formed in the weld nugget [16]. It has been illustrated that the \(\alpha\)-fiber shear texture would suppress the cross slip and as a consequence twinning is promoted yielding to a significant peak load and extension.

The strain/thermal induced phase transformations are likely to happen in the course of FSW and affecting the capability of grain refinement. For instance, the formation of delta ferrite during FSW in a 304 stainless steel has been reported by Park et al. [17] owing to high amount of imposed strain. In the case of DSS, the phase transformation is common phenomenon which can occur at elevated temperatures. Ramirez et al. [18] have investigated the transformation of ferrite to the austenite phase during welding of different commercial DSSs. It has been revealed that supersaturated ferrite being enriched by the austenite stabilizing elements which leads to the precipitation of chromium nitride and secondary austenite. This phenomenon would retard the grain growth and consequently enhances the grain refinement influencing the room temperature mechanical properties of the joints.

In line to previous efforts, the present work for the first time focuses on friction stir spot welding of a nickel-free high nitrogen duplex stainless steel. The high content of nitrogen in such affordable chemical composition would definitely influence the austenite thermal/mechanical stability, therefore,

### Table 1 – The chemical composition (wt.%) of the studied lean DSS.

<table>
<thead>
<tr>
<th>Grade</th>
<th>C</th>
<th>Mn</th>
<th>N</th>
<th>Ni</th>
<th>Cr</th>
<th>Si</th>
<th>S</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>DSS</td>
<td>0.07</td>
<td>12.61</td>
<td>0.33</td>
<td>0.02</td>
<td>20.55</td>
<td>0.66</td>
<td>0.02</td>
<td>0.01</td>
</tr>
</tbody>
</table>

![Fig. 1](image_url) – Schematic of (a) Friction Stir Spot Welding (FSSW) process, (b) Tensile-shear specimen geometry.
the microstructure and texture evolutions would be more complex compared to austenitic stainless steel or conventional DSS. The activation of restoration mechanisms in both ferrite and austenite at various processing condition, the correlated texture evolution and probable thermal/strain induced transformations are the concepts which will be discussed in details. To explore the microstructure-processing-properties relationship, the local and bulk mechanical properties of the joints will be assessed through tensile-shear test and micro-hardness.

2. Material and methods

The high nitrogen nickel-free stainless steel was prepared using an induction furnace under N₂ atmosphere, followed by
Table 2 – Comparison of austenite grain refinement and phase transformation during FSW and FSSW of some steels.

<table>
<thead>
<tr>
<th>Material</th>
<th>Process Type/Rotation speed (rpm)</th>
<th>SZ grain size (µm)</th>
<th>Austenite Phase fraction</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>TWIP Steel</td>
<td>FSSW/1000</td>
<td>3.6</td>
<td>Roughly remain unchanged (~1% to ferrite)</td>
<td>[37]</td>
</tr>
<tr>
<td>Fe–20Cr–20Mn-0.95N</td>
<td>FSW/400</td>
<td>6.3</td>
<td>Roughly remain unchanged (~1% to ferrite)</td>
<td>[38]</td>
</tr>
<tr>
<td>304 L</td>
<td>FSSW/400</td>
<td>1.2</td>
<td>Roughly remain unchanged (~1% to ferrite)</td>
<td>[39]</td>
</tr>
<tr>
<td>DSS S31803</td>
<td>FSW/1500</td>
<td>6.5</td>
<td>No significant change (~4% to ferrite)</td>
<td>[40]</td>
</tr>
<tr>
<td>SAF 2507</td>
<td>FSW/550</td>
<td>4.3</td>
<td>Roughly remain unchanged (~1%)</td>
<td>[41]</td>
</tr>
<tr>
<td>SAF 2205</td>
<td>FSW/300</td>
<td>3.8</td>
<td>Roughly remain unchanged (~1%)</td>
<td>[42]</td>
</tr>
<tr>
<td>304L</td>
<td>FSW/300</td>
<td>13</td>
<td>Roughly remain unchanged (~1%)</td>
<td>[43]</td>
</tr>
<tr>
<td>SAF 2205</td>
<td>FSP/47, 57</td>
<td>10–30</td>
<td>~10% austenite to ε phase</td>
<td>[44]</td>
</tr>
<tr>
<td>Ni-free high N DSS</td>
<td></td>
<td>1.1</td>
<td>~20% increment in austenite fraction</td>
<td>Present work</td>
</tr>
</tbody>
</table>

Fig. 4 – TMAZ of the specimens FSSWed at (a, b) 400 rpm (c, d) 500 rpm (e, f) 600 rpm (EBSD boundary map of (a, c, e) austenite phase, (b, d, f) ferrite phase (HAGB are represented by blue, LAGB by red and green lines).
the electro-slag re-melting process. The chemical composition of the experimented material is given in Table 1. The casting ingots were homogenized at 1100 °C for 120 min, and hot rolled in the temperature range of 1100–1200 °C to 20 mm and finally solution treated at 1100 °C for 30 min.

In order to prepare samples for FSSW, the solution-treated material was cut and machined using an electro-discharge machine (EDM) to dimensions 30×100 mm and 2 mm thickness. Work pieces were clamped with a well-designed fixture (Fig. 1) and welded in the lap joint condition at three rotational speeds (400, 500 and 600 rpm), plunge depth of 2.5 mm, and 2 s dwell time by means of an 8 mm diameter cylindrical tungsten carbide (WC) pin with a shoulder diameter of 20 mm. For macro- and microstructural examinations, the FSSWed workpieces were cut by the EDM from the centre of the welds, as shown in Fig. 1. Specimens were cold mounted and ground with SiC papers up to 2000 grit and electro-polished by a solution containing HClO and C6H8O7 at 2.3 V for 2 min. Microstructure was characterized by a high-resolution scanning electron microscopy (HR-SEM, Hitachi SU6600) equipped with EDAX-TSL EBSD system with a 20 kV operating condition in order to study the phase transformation, restoration mechanisms and texture evolution. The scanning step size of 0.3–0.5 μm was selected depending on the overall grain sizes of each processed specimen. In order to investigate the presence of precipitates, the microstructures were characterized at 200 kV by a transmission electron microscope (TEM), JEOL JEM 2100. TEM specimens were prepared by grinding and polishing a thin foil down to 70 μm thick, followed by jet polishing at room temperature with a solution of 10% perchloric acid in acetic acid.

In order to trace the effects of microstructural evolution on the local mechanical properties of the processed material, the variation of micro-hardness was determined across the whole weld section under the load of 300g and dwell time of 10s. For each rotational speed, microhardness tests were performed five times. In order to evaluate the strength of lap joints, tensile specimens are shown in Fig. 1. And for each rotational speed, the tests were repeated three times.

3. Results and discussion

3.1. Macrostructure of the welded joints

The EBSD image quality and phase maps of the initial microstructure are depicted in Fig. 2. The microstructure consists of austenite (blue colour, ~75%) and ferrite (red colour), as is shown in Fig. 2b. The average grain size determined for austenite and ferrite was about 26 ± 5 μm and 27 ± 5 μm, respectively. In Fig. 3(a, e and i), the macrographs of the joints at various tool rotational speeds are illustrated. The bonding width in the samples welded at 400 rpm and 500 rpm is approximately equal (Fig. 3a and e) but decreases significantly by increasing the welding rotational speed to 600 rpm (Fig. 3i). This is attributed to the reduction of material’s flow and welding torque and has been thoroughly discussed elsewhere [16].

The different regions of the joints, heat affected zone (HAZ), thermomechanically affected zone (TMAZ) and stir zone (SZ), exhibit different grain size, grain morphology and texture evolution. The scanning step size of 0.3–0.5 μm was selected depending on the overall grain sizes of each processed specimen. In order to investigate the presence of precipitates, the microstructures were characterized at 200 kV by a transmission electron microscope (TEM), JEOL JEM 2100. TEM specimens were prepared by grinding and polishing a thin foil down to 70 μm thick, followed by jet polishing at room temperature with a solution of 10% perchloric acid in acetic acid.

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The different regions of the joints, heat affected zone (HAZ), thermomechanically affected zone (TMAZ) and stir zone (SZ), exhibit different grain size, grain morphology and phase fraction [20]. In Fig. 3d, h and l, the typical appearance of the HAZ is demonstrated, the microstructure of which is similar to that of base metal in respect of the grains and phase fractions, indicating that this region was just affected by heat generated during welding [9]. In TMAZ (Fig. 3c, g and k), which is the transition zone between HAZ and SZ, the grains have been elongated due to an upward flow of material near the SZ both during plunging and stirring steps. The similar features have been reported by several researchers, e.g. [21]. The phase fractions are the same as in the base metal apart from elongated shapes. In respect of the SZ, the extent of the heat input and strain rate are completely different owing to the different tool rotational speed. The mean austenite grain size (Fig. 3b, f and j) of the SZ under the rotational speeds of 400, 500, 600 rpm are 1.1, 1.4 and 1.4 μm, respectively, whereas the grain sizes for ferrite are 0.9, 1.1, 1.2 μm, respectively. Such
outstanding grain refinements in the both ferrite and austenite grains have not been observed in previous works. In this respect, the obtained results have been compared with those obtained by FSW or FSSW of austenitic stainless steel and DSSs (Table 2). As it is provided in Table 2, the extreme grain refinement to 1.2 μm, was achieved by Jeon et al. [6], FSSW of single crystal austenitic stainless steels, but this has been limited to few microns in the case of polycrystalline austenitic steels. The significant grain refinement achieved in the present work is attributed to the exclusive design of the chemical composition (Ni-free, high alloyed by Mn and N) and the severe deformation condition in respect of applied strain rate and temperature affecting the operation of restoration mechanisms in both ferrite and austenite. This will be discussed in details in the following sections.

3.2. Microstructure of the welded joints

3.2.1. Microstructural evolution in TMAZ

Considering the heat exposure and the applied strain, the activation of restoration mechanism and occurrence of phase transformation within the TMAZ are both probable. In this respect, the microstructures at the TMAZ of the specimens FSSWed at various rotational speeds are illustrated in Fig. 4. The phase fractions have been remained mostly unchanged. The recorded misorientation-angle distributions indicate that...
Fig. 7 – The phase equilibrium of DSS calculated by JMat Pro 7.0 software.

Fig. 8 – Misorientation angle distribution within the austenite phase at (a) 400 rpm, (b) 500 rpm, and (c) 600 rpm; (d) austenite grain size in SZ and subgrain size in TMAZ based on HAGBs and LAGBs respectively at various tool rotational speeds.
the material response was roughly similar and independent from the tool rotational speed, therefore, only the misorientation-angle distribution of the sample FSSWed at 400 rpm (Fig. 5) is discussed. The substructure developed in the austenite consists of high percentage of low angle boundaries (LAGBs), which are shown with green ($\theta < 5^\circ$) and red ($5^\circ < \theta < 15^\circ$) lines in Fig. 4a, c and e. The high frequency of LAGB in austenite (67%) (Fig. 5a) suggesting activation of dynamic recovery as the predominant restoration mechanism. In addition, strain concentration at the ferrite–austenite interfaces cause formation of clustered regions with high angle grain boundaries (HAGBs) (shown by red dashed lines in Fig. 4a, c and e) and the occurrence of partially recrystallization. This is attributed to the various strain accommodation capability of ferrite and austenite and load transition between two massive phases. It is also worthy to note that in Fig. 4a, c and e small austenite islands exist inside the ferrite grains, as outlined by yellow circles and pointed out with yellow arrows. This represent starting of the ferrite to austenite transformation especially on the $\alpha/\gamma$ interfaces. These results are in accordance with our previous research [22] regarding the hot compression behavior of the present steel under the various strain rates ($0.001$–$0.1s^{-1}$) at elevated temperatures. By means of microstructural based model which is comparison of microstructure in hot compression and TMAZ, deformation condition at TMAZ is estimated to have strain rates like hot compression strain rates ($0.001$–$0.1s^{-1}$).

3.2.2. Microstructural evolution in SZ

The notable reduction in the ferrite phase fraction with significant grain size refinement in both ferrite and austenite have been observed within the SZ, as indicated in Fig. 6a, b and c. The grain size of the SZ (Fig. 6d) has been decreased down to values smaller than those reported in similar studies in the previous literature, as listed in Table 2. The applied severe strain and strain rate at elevated temperatures are the main driving forces for the occurrence of dynamic recrystallization [9,11] and may justified such outstanding refinement (97% reduction comparing with starting microstructure). In fact, besides the activation of different restoration micro-mechanisms in both ferrite and austenite, dynamic phase transformation also contributes in grain refinement in the present case. It has been reported that the decomposition of supersaturated ferrite may take place during cooling stage and this phenomenon would happen at much longer time (~1000 s) due to diffusional origin of the transformation [18,23,24]. However, in the present work, the transformation seems to be boosted by means of developing more diffusional

Fig. 9 – EBSD maps of SZ superimposed with grain boundary and CSL boundary maps in austenite phase at (a) 400 rpm (7.5% CSL), (b) 500 rpm (11.5% CSL), (c) 600 rpm (15% CSL). (CSL boundaries are represented in red, Ferrite phase is green).
paths through substructure development during deformation and by the increment of grain boundary area due to intense grain refinement. It well confirms the previous reported results regarding the stabilization of austenite through FSW [25,26]. According to Sato et al. [11] the phase transformation plays a key role in both consuming of input energy and changing the balance of the phase fractions.

According to the calculated phase diagram by Jmat Pro 7.0 software (Fig. 7) and the measured phase fraction by EBSD, the approximate temperature that the SZ of the specimens welded at 400 rpm, 500 rpm and 600 rpm were experienced can be estimated, according to the microstructure based model explained in section 3.2.1, which are about 870 °C, 915 °C and 950 °C, respectively. The phase transformation is happened significantly and it is feasible from thermodynamic point of view, but the kinetics is relatively high in contrast with our previous research through hot compression (800 °C-1000 °C) at low strain rates (0.001/s-0.1/s) of this material [25] that no phase transformation is observed. Therefore, during the FSSW process decomposition of super saturated ferrite which formed at elevated temperature would occur. It has been reported that decomposition takes place during cooling cycle and this phenomenon would happen at relatively longer time (1000s) due to diffusional origin of transformation [18]. However, in the present work the kinetics of ferrite to austenite is boosted by means of developing more diffusional paths through substructure development and the increment of the area fraction of the grain boundaries due to grain refinement as well it confirms the previous results of researchers about stabilization of austenite by friction stir welding [25,26]. The ferrite is supersaturated from austenite stabilizer elements during the process and as it has been proved before [18] the microstructure have the driving force for phase transformation during the cooling cycle. Meanwhile, grain size of two phases are not comparable, although both underwent DRX. Phase transformation plays a key role for grain refinement which in previous study has not been taken place.

Figure 8a and 8b and c show the misorientation-angle distribution within the austenite phase deformed at various rotational speeds. Alongside with the appreciable grain refinement, the fraction of LAGBs is still high and the substructure has been developed at all deformation condition. Interestingly, the mean subgrain size in the TMAZ is very close to the average grain size in the SZ (Fig. 8d), which clearly indicates the continuous dynamic recrystallization as the dominant restoration mechanism. This is in agreement with the previous reported results on FSSW of a high Mn TWIP steel [16]. As is observed, the fraction of the S3 and S9 grain boundaries, which are shown by red lines, are increased from 7.5% to 15% by increasing the tool rotational speed (Fig. 9). The formation of S3 and S9 boundaries in DSS is typically associated with grain boundary migration induced by grain growth [27]. Therefore, this observation may illustrate the occurrence of grain growth during cooling stage which is intensified by...
increase of the tool rotational speed and thus increment of heat input. Coming to the point, the dynamic recrystallization, static recrystallization (during cooling stage) and phase transformation contribute in such extensive grain refinement. The grain structure remains relatively stable during cooling stage and fresh grains could be defined as static recrystallized grain. However, a slight grain growth seems to be under the higher rotational speeds in the SZ of the samples welded at 500 rpm and 600 rpm.

In the following, in order to investigate the restoration mechanism in ferrite phase the Fig. 10 represents misorientation angle distribution for three samples welded at different rotation speeds. For sample welded at 400 rpm (Fig. 10a) the LABs fraction is dropped dramatically in comparison with the TMAZ and there is a peak at approximately 44°. According to the random distribution function of Mackenzie the presence of a peak at 45° in misorientation angle distribution charts in ferrite assumes that DDRX is the main restoration mechanism [28]. By increase in the rotation speed the misorientation angle distribution of samples welded at 500 rpm and 600 rpm in ferrite (Fig. 10b & c), correspond a shift in dynamic recrystallization mechanism from DDRX in 400 rpm rotation speed to continuous dynamic recrystallization (CDRX) in 500 rpm rotation speed with high fraction of LABs suggesting an extended recovery illustrating the occurrence of CDRX. This finding is in contrary to results reported by Haghdadi et al. [14] on duplex stainless steel that by increasing the strain rate to relatively high strain rate of 10 s⁻¹ the restoration mechanism is turning into DDRX, from which it was in lower strain rates, CDRX. This contraction is related to the applied strain rates in the present work which is enormously higher than the

Fig. 11 — TEM micrograph from SZ of specimen welded at 400 rpm representing the presence of Cr₂N precipitant.
Haghdadi et al. [14] research and increase of ferrite phase fraction as it eases deformation at this high strain rate at higher welding rotation speeds and consequently, elevation in heat input and as well temperature. The enhancement in temperature and strain rate accelerates diffusional phenomenon like climbing and rearranging of dislocations which promotes DRV in ferrite phase with high stacking fault energy and CDRX becomes the dominant restoration mechanism. As it is in [18] phase fraction and temperature are not constant and especially phase transformation plays a key role in both consuming of input energy in microstructure and changing balance of phase fraction which leads to shifting in DRX mechanism.

Although it is reported thermal conductivity of stainless steels are comparatively low and thus the cooling cycle may be relatively long [9,27], the significant grain refinement occurred during welding is remained stable during cooling cycle. Also, a slight grain growth occurred by increasing rotation speed and consequently the enhancement of temperature results in grain growth in the SZ of samples welded at

![Image of orientation distribution functions (ODF, $\varphi_2 = 0$–$90^\circ$ sections) in the specimens FSSWed at (a) 400 rpm, (b) 500 rpm, and (c) 600 rpm, respectively.](image-url)
500 rpm and 600 rpm. But as it mentioned above, grain size remains stable. This could be also explained by means of energy that consumed during the phase transformation that occurred in the SZ during cooling cycle. Also, it is speculated that the grain boundary pinning by precipitants formed during process plays an important role which is detected by TEM and would be thoroughly discussed in the following.

3.3. Precipitation behavior

Figure 11 depicts a 65 nm precipitate near austenite/ferrite phase and the SAD pattern of this particle illustrates that it is Cr2N as in literature suggested before [18,29,30]. This indicates that the phase transformation occurred during cooling cycle of welding process. Also, the morphology of the precipitate as Fig. 11 illustrated, is granular. This morphology is reported to be the early stage of precipitation [29,31], in limited aging time. Similarly, Feng et al. [29] have observed a change in morphology from granular to laminar for Cr2N by increasing aging time in stainless steel, suggested that transformation of austenite to secondary austenite and chrome nitride occurs. Consequently, the observed fine and disperse precipitates with granular morphology after the welding is mainly due in part to limited time for diffusion during the cooling cycle.

As it is evident, alongside with the phase transformation of ferrite to austenite during cooling cycle, a cooperative nucleation and growth of precipitants and secondary austenite would occur. It is known that nitrogen and other austenite stabilizer elements have sufficient diffusional path to form secondary austenite through diffusion from super saturated ferrite phase in elevated temperature [32,33]. Moreover, stored energy in structure is greater than driving force for both recrystallization and phase transformation due to high strain rate and extended recovery and substructure development that has been noticed in the TMAZ which illustrates a step before SZ. Extraordinary grain refinement of present study is a result of consuming the energy for transformation and precipitation which had been generated during the process. Therefore, growth is restricted meanwhile precipitants size formed in phase transformation of ferrite to secondary austenite are at low order of magnitude so that pinning of boundaries could be a possible reason restricting grain growth. Also, these microstructural changes would affect the texture evolution of the microstructure.

3.4. Texture evolution

In order to evaluate the texture evolution, the collected orientation data have been arranged as orientation distribution functions (ODF) in the orientation space of \(0_1 = 0–360\), \(0 = 0–90\) and \(0_2 = 0–90\) (Bunge notation, with the triclinic sample symmetry) for the austenite phase in Fig. 12. It is worth mentioning that the ferrite texture evolution has not been considered as the volume fraction of this phase was quite low. The preliminary examination indicates the formation of B-fiber shear and A-fiber shear texture in the specimens welded at 400 rpm and 500 rpm (Fig. 12a and b), respectively. The intensities of the texture components in the specimen welded at 400 rpm are very low, and increases by enhancement in the severity of the deformation as a result of increase in the welding rotational speed. The presence of the fiber texture well indicates that continuous recrystallization has been occurred during the processing, which is in agreement with the results reported in [16]. By increasing the rotational speed to 600 rpm, the shear texture C is appeared. The occurrence of the single component shear texture shows that the recrystallized microstructure inherits the deformation texture and confirms the contribution of discontinuous recrystallization in the microstructure evolution. The proofs for the contribution of discontinuous recrystallization in grain refinement requires the stop-action technique and in-situ assessment of the microstructure evolution, which is the aim of our future works. In the case of austenitic stainless steel, the shear texture C has been observed under the lower severity of deformation [11]. Comparatively, observation of shear texture under the higher rotational speed in the present case, can be related to the increase in the phase fraction of ferrite and its contribution in accommodation of the imposed strain in the course of processing.

3.5. Mechanical properties

The variation of Vickers micro-hardness vs. distance from the key hole of the samples welded under the different rotational speeds is depicted in Fig. 13. In the SZ and TMAZ, the hardness values are much higher than that of the base metal, which is well attributed to substructure development and grain refinement also precipitation of Cr2N in the course of FSSW. The hardness value decreases with increase in the tool rotational speed that can be explained by grain size escalation due

<table>
<thead>
<tr>
<th>Rotation speed</th>
<th>400 rpm</th>
<th>500 rpm</th>
<th>600 rpm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bonding width (µm)</td>
<td>735 ± 5</td>
<td>835 ± 5</td>
<td>289 ± 5</td>
</tr>
<tr>
<td>Austenite grain size (µm)</td>
<td>1.1 ± 0.20</td>
<td>1.4 ± 0.10</td>
<td>1.7 ± 0.15</td>
</tr>
<tr>
<td>Ferrite grain size (µm)</td>
<td>0.9 ± 0.12</td>
<td>0.9 ± 0.15</td>
<td>1.1 ± 0.10</td>
</tr>
</tbody>
</table>
to Hall–Petch relationship. By getting away from the keyhole a drop in microhardness can be seen in graph, which is related to TMAZ transition zone between SZ and HAZ. Also due to grain growth in HAZ the hardness is lower than the TMAZ.

The red solid line in Fig. 13 is the hardness of the base metal and its hardness is similar to the HAZ microhardness that is related to approximate grain size equality to base metal grain size coarsening (Table 3) and the increasing ferrite fraction, as the softer constituent phase.

The tensile-shear tests have been also performed to determine the bulk mechanical properties of FSSWed joints. As it is illustrated in Fig. 14, the highest peak load (12.8 kN) has been obtained for the specimen processed at the 400 rpm rotation speed, whereas the highest peak extension (1.7 mm) has been achieved at 600 rpm. In the instance of sound joints, there is no logical correlation between the bonding width and bulk mechanical properties, as obvious also from Table 3. Thus, the microstructural features such as the grain size and the ferrite content are controlling factors of the joint strength and ductility. In this respect, the higher strength of the sample welded at 400 rpm can be related to the finer grain size (Table 3) and presence of Cr2N precipitates. Precipitates and increment in grain boundaries in 400 rpm sample act as barriers for dislocation movement leading to increment of strength level. On the other side the higher ductility of the specimen welded at 600 rpm can be justified considering the increase in the ferrite content which can effectively accommodate the imposed strain during subsequent room temperature deformation. In addition, the presence of shear texture type C may provide a proper condition for activation of cross slip, increase the strain accommodation factor and thus room temperature formability of the joints processed under the higher rotational speed of 600 rpm.

Fig. 14 – Tensile-shear strength (KN) and extension of FSSWed specimens versus experimented rotational speeds.

Fig. 15 – (a) Top view schematic of nugget pull out failure of welded joints, cross section of welded joints showing the failure initiation and route for (b) 400 rpm, (c) 500 rpm, and (d) 600 rpm.
Failure for all three joints were nugget pull out, which is reported in the literature that it is the desired fracture mode for spot joints [34], as it is schematically illustrated in Fig. 15a. The crack initiation and propagation route for each sample is schematically shown on cross section where the failure took place in Fig. 15b–d. The crack initiated from interface of SZ and TMAZ and proceed the propagation through the shoulder for all three welds. The strength of 400 rpm sample is dependent to microstructure since it has the finest grain size and it is independent of banding width (735 μm) and the route length (0.3 mm, as shown in Fig. 15b) where the crack propagated. But the elongation of 400 rpm sample seems to be correlated to the route that crack propagate since it has the least route to propagate. For samples welded at 500 rpm and 600 rpm it seems that elongation benefits more from grain size rather than route length since there is not a straight relation between route length and elongation for these two welds. Therefore, 600 rpm sample has higher elongation to fracture due in part to coarser grain size formed in the SZ and route of propagation.

4. Summary

Microstructure, texture and mechanical properties of the nickel-free high nitrogen duplex stainless steel joints achieved by friction stir spot welding, were investigated and the following results were obtained:

1. The joint welded at 400 rpm was effectively refined at the stir zone by the average grain size of 1.1 μm and 0.9 μm for the austenite and ferrite phases, respectively. At the higher rotational speeds of 500 rpm and 600 rpm, a slight increase in the grain size was observed due to higher generated heat input. In contrast, the thermomechanically affected zone of all joints experienced partial recrystallization specially at the austenite–ferrite interfaces. The predominant restoration mechanism in the austenite phase was concluded to be continuous dynamic recrystallization.

2. The ultrafine structure achieved through welding remained stable during the cooling stage. Such thermal stability was attributed to the consumption of the welding energy in simultaneous occurrence of phase transformation and recrystallization. The transformation supersaturated ferrite was boosted by means of developing more diffusional paths through substructure development and grain refinement.

3. The formation of B-fiber and A-fiber shear texture components in samples welded at 400 rpm and 500 rpm confirmed the occurrence of continuous dynamic recrystallization. With increasing the deformation severity at 600 rpm, a single component shear texture type C appeared as the dominant orientation depicting that the recrystallized microstructure inherited the deformation texture.

4. The maximum tensile-shear peak load (12.8 kN) and maximum extension (1.7 mm) were achieved for the sample welded at 400 rpm and 600 rpm, respectively. These were well justified considering the occurrence of grain refinement, and phase transformation under the various processing condition.

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**Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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**References**


