

**INFLUENCE OF FRICTION STIR PROCESSING ON MECHANICAL
PROPERTIES AND CORROSION RESISTANCE OF UNS S32750
SDSS**

BY

HAFIZ MUHAMMAD ABUBAKER

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This thesis, written by **Hafiz Muhammad Abubaker** under the direction of his thesis advisor and approved by his thesis committee, has been presented and accepted by the Dean of Graduate Studies, in partial fulfillment of the requirements for the degree of **MASTER OF SCIENCE IN MECHANICAL ENGINEERING**.

Zuhair
Gasem

Digitally signed by Zuhair Gasem
DN: cn=Zuhair Gasem, o=KFUPM,
ou=Chair, ME Dept.,
email=zuhair@kfupm.edu.sa,
c=SA
Date: 2020.05.21 16:27:00 +03'00'

Dr. Zuhair Mattoug Gasem
Department Chairman

Dr. Suliman Saleh Al-Homidan
Acting Dean of Graduate Studies



14-06-2020

Date

Dr. Neçar Merah
(Advisor)

Dr. Ahmad A. Sorour
(Member)

Dr. Jafar Albinmousa
(Member)

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I would like to dedicate my thesis to my teachers, family, and friends.

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LIST OF ABBREVIATIONS

DSS	:	Duplex stainless steel
SDSS	:	Super duplex stainless steel
FSP	:	Friction stir processing
FSPed	:	Friction stir processed
FSW	:	Friction stir welding
CT	:	Compact tension
CTOD	:	Critical tip opening displacement
AS	:	Advancing side
RS	:	Retreating side
SZ	:	Stir zone
BM	:	Base metal
HAZ	:	Heat affected zone
WC	:	Tungsten carbide
PCBN	:	Poly crystalline born nitride
W-Re	:	Tungsten rhenium
K _{IC}	:	Plain strain fracture toughness
HISC	:	Hydrogen induced stress cracking
SSC	:	Stress corrosion cracking
XRD	:	X-Ray Diffraction
CR	:	Corroded
LEFM	:	Linear elastic fracture mechanics
PDP	:	Potential-dynamic polarization
LPR	:	Linear polarization resistance

ABSTRACT

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Super duplex stainless steel (SDSS) has excellent mechanical properties such as high strength, high toughness, high fatigue life and high corrosion resistance. SDSS is being used in applications such as desalination equipment, pressure vessels, marine/offshore applications and in oil/gas plants. Highly corrosive environment affects the behavior of the material by reducing its mechanical properties such as strength, hardness, and fracture toughness. There are different techniques that can be used to improve these properties and friction stir processing (FSP) is one of them, which is used to refine the grain structure and enhance the mechanical and electrochemical properties of the material. In this thesis work, friction process optimization was first performed and then, optimized FSP parameters were used to refine the microstructure of UNS S32750 SDSS material in order to improve its mechanical properties and corrosion resistance. Microstructure analysis revealed grains were refined from a size of around 160 μm in the base material to 2-30 μm in the processed zone. Stir zone presented non-uniform grain structure and the grain size increased from 2 μm on the advancing side of the friction stir processed material to 30 μm on the retreating side. This grain size reduction resulted in higher strength, hardness, fracture toughness and corrosion resistance of the material by up to 14, 11, 12, and 80 %, respectively. However, the ductility of the processed region was reduced by about 29 % and the material showed brittle behavior.

ملخص الرسالة

الاسم الكامل: حافظ محمد أبو بكر

عنوان الرسالة: تأثير معالجة الاحتكاك على الخواص الميكانيكية ومقاومة التآكل في UNS S32750 SDSS

التخصص: هندسة ميكانيكية

تاريخ الدرجة العلمية: أبريل 2020

يتميز الفولاذ المتميز المقاوم للصدأ على الوجهين (SDSS) بخصائص ميكانيكية ممتازة مثل القوة العالية، والمتانة العالية، والعمر الطويل ضد الانكسار، والمقاومة العالية للتآكل، تجده مستخدماً في معدات تحلية المياه، وأوعية الضغط، والتطبيقات البحرية و البرية القريبة من البحر، وفي مصانع النفط و الغاز. تؤثر البيئة شديدة التآكل على سلوك المادة عن طريق التقليل من الخصائص الميكانيكية مثل القوة، والصلابة، و المتانة ضد الانكسار. هناك تقنيات مختلفة يمكن استخدامها لتحسين خصائص هذه المادة. واحدة من هذه التقنيات هي معالجة تحريك الاحتكاك (FSP) التي تستخدم لتحسين بنية الجزيئات في المنطقة المعالجة لتعزيز الخصائص الميكانيكية و الكهروكيميائية للمادة. في هذه الورقة، تم إجراء بحث عن أفضل عوامل لمعالجة الاحتكاك أولاً، وبعد ذلك، تم استخدام العوامل الأفضل من معالجة تحريك الاحتكاك لتنقية وتحسين البنية المجهرية للمادة UNS S32750 SDSS وذلك لتحسين خواصها الميكانيكية ومقاومة الصدأ. أظهرت دراسات البنية المجهرية أن الجزيئات صغر حجمها من حوالي 160 ميكرومتر في المواد الأساسية إلى 2-30 ميكرومتر في المنطقة المعالجة. أظهرت في منطقة التحريك أثناء المعالجة هيكل جزيئي غير منتظم، و ازداد حجم الجزيئات من 2 ميكرومتر على الجانب المتقدم إلى 30 ميكرومتر على جانب إعادة المعالجة، وهما جانباً منطقة المعالجة. أدى هذا الانخفاض في حجم الجزيئات إلى تحسين القوة، والصلابة، والمتانة ضد الكسر، ومقاومة التآكل للمواد بنسبة تصل إلى 14 و 11 و 12 و 80٪، على التوالي. ومع ذلك ، تم تقليل ليونة المنطقة المعالجة بحوالي 29٪، وأظهرت المادة سلوكاً هشاً.

CHAPTER 1

INTRODUCTION

1.1 Background

Duplex stainless steel (DSS) is composed of ferritic (BCC structure and α -phase) and austenitic (FCC structure and γ -phase) components. DSS has excellent mechanical properties like high toughness, strength, hardness, and corrosion resistance. There are four basic types of DSS: lean DSS, regular DSS, and super DSS and hyper DSS. All four types of DSS have their own significance and their use mainly depends upon the application [1]. Super duplex stainless steel (SDSS) is mostly used in severe service conditions which affects the behavior of the material. Therefore, surface modification techniques such as friction stir processing (FSP) can be employed to improve the properties of the material. Studies of FSP of SDSS are rare because it is a very hard material and friction stir processing is mostly used for soft materials.

FSP is a solid-state technique wherein a stir tool mechanically stirs the material to modify its microstructure. FSP refines the microstructure through dynamic recrystallization of material [2]. Ultrafine size grains or even nano size grains can be produced by this technique. By refining the grains, mechanical properties such as strength, hardness, fracture toughness and corrosion resistance etc. will change. FSP is a form of friction stir welding (FSW) which is used to modify the behavior of the material, whereas FSW is employed for joining of metals [3].

1.2 Motivation

Duplex stainless steel is being used in different applications for multiple industries mainly owing to its high strength, high fracture toughness, fatigue, and corrosion resistance. Super duplex stainless steel (SDSS) has even more strength, hardness and higher corrosion resistance owing to its higher content of chromium, molybdenum and nickel [1, 4]. SDSS is used for desalination equipment, pressure vessels, marine applications, paper, pulp, offshore applications and in oil/gas plants where highly corrosive environment is present [5]. Severe corrosive environmental conditions affect the behavior of SDSS, therefore, FSP can be used to enhance the properties of SDSS. FSP refines the grain structure of SDSS and improves its mechanical properties. To the best of the author's knowledge, research work on the influence of FSP on fracture toughness behavior of SDSS is inexistent in the open literature. The only work involving FSP of SDSS is that carried out by Mishra [6] on the corrosion resistance of the processed alloy. This motivated us to initiate this research to study how this strong material can be friction stir processed and how FSP affects its microstructure, mechanical behavior, and corrosion resistance. Therefore, this research work will not only fill this research gap in the literature but also it will help improve the current knowledge of the influence of FSP on the behavior of SDSS.

1.3 Objectives

The overall aim of this research work is to understand the influence of FSP on the mechanical properties and corrosion resistance of UNS S32750 SDSS.

The specific objectives are:

- To study the tensile, hardness and fracture toughness behavior of UNS S32750 SDSS.
- To optimize friction stir processing of UNS S32750 SDSS.

- To study the influence of optimized FSP on tensile, hardness, corrosion resistance and the fracture toughness behavior of UNS S32750 SDSS.
- To study the effect of seawater on the fracture toughness behavior of as received and friction stir processed (FSPed) UNS S32750 SDSS.

1.4 Research Methodology

FSP was performed using different parameters (advancing speed, rotational speed, and tilt angle) in order to optimize the process. Optimization of FSP was based on the surface quality (smoothness and presence of defects) of the processed zone. Once FSP was optimized, samples for microstructural characterization, tensile, hardness, corrosion and fracture toughness testing were prepared.

Testing procedure and equipment used in this research work are described below:

- Microstructural characterization was performed using advance optical microscope.
- Tensile testing was carried out on Instron Machine 8801 according to ASTM standard E8/E8M-16a.
- Vickers hardness method was employed for hardness measurements.
- Fracture toughness testing was executed on Instron Machine 8801 according to ASTM standard E1820-20.
- Corrosion testing was performed using 3.5 % NaCl (synthetic seawater) solution on Gamry testing system.

All testing and analyses were carried out for both base and FSPed material to study the effect of FSP on the above-mentioned properties. In the end, comparison of tensile, hardness, fracture toughness and corrosion resistance of base metal and FSPed material revealed the influence of FSP on the behavior of UNS S32750 SDSS before and after corrosion testing.

CHAPTER 2

LITERATURE REVIEW

2.1 DSS, Classifications and Properties

DSS is basically a family of stainless steel which has austenite and ferrite in its composition in almost equal amounts. Their properties are the combination of properties of both ferritic and austenitic phases. They have excellent properties like high strength, hardness, fatigue life, corrosion resistance, and fracture toughness, among all steels [7]. Lean DSS, standard DSS, super DSS, and hyper DSS are in the family of DSS. Lean DSS has lower nickel content. Standard DSS has higher nickel and molybdenum content. Super DSS has around 25 % chromium, higher nickel, and molybdenum content. Hyper DSS has an even higher content of chromium, nickel, and molybdenum than SDSS [1, 4]. Different types and grades of DSS have different properties depending upon the element composition. UNS S32750 SDSS has good mechanical properties and corrosion resistance because of its composition [1, 4].

2.2 Friction stir processing

FSP is a solid-state processing approach in which a tool's pin is inserted inside the material for processing as shown in Fig. 1. During processing, tool's pin goes inside the workpiece and shoulder touches the surface of the workpiece to make tight contact between tool and workpiece [2, 8]. Severe plastic deformation takes place during processing and the pin mechanically stirs the material to modify its microstructure and the refinement of the microstructure of the material occurs through dynamic recrystallization [2, 9]. The side of the workpiece, where processing direction and direction of rotational speed are the same, is the

advancing side (AS) and the other side, where processing direction and direction of rotational speed are opposite, is the retreating side (RS) as shown in Fig. 1 [8, 10]. FSP is used to refine the grains of the material to enhance its properties. The parameters of FSP have influence on the refinement of the microstructure of the material and these parameters are rotational speed, advancing/processing and tilt angle of stirring tool [11]. Therefore, surface quality and grain refinement of the processed material will be changed by changing the parameters of FSP.

2.3 Influence of FSP on microstructure and tensile behavior

A lot of research has been done with different advancing speeds and rotational speeds, to examine the impact of these parameters on the microstructure and tensile behavior of the material. As FSP refines the microstructure and increases the grain boundary density which in turn offer more resistance to dislocation movements so the material strength will be enhanced. Emami, et al. [12, 13] researched 2205 SDSS to study the microstructural behavior and change in the properties by FSP. 2205 SDSS was processed at a rotational speed of 400 rpm and an advancing speed of 50 mm/min using tungsten carbide tool. Microstructural analysis showed that the fine grains were produced in the stir zone (SZ) due to high frictional heat and dynamic recrystallization. Esmailzadeh, et al. [14] investigated lean DSS to find out the effect of various parameters of FSP on the microstructure of the material. FSP was carried out at a rotational speed 800 rpm and at three different advancing speeds of 50, 100 and 150 mm/min using tungsten carbide stir tool. Their findings disclosed that the higher the advancing speed the smaller the grain size, resulting in improvements in hardness and tensile strength of the specimen.

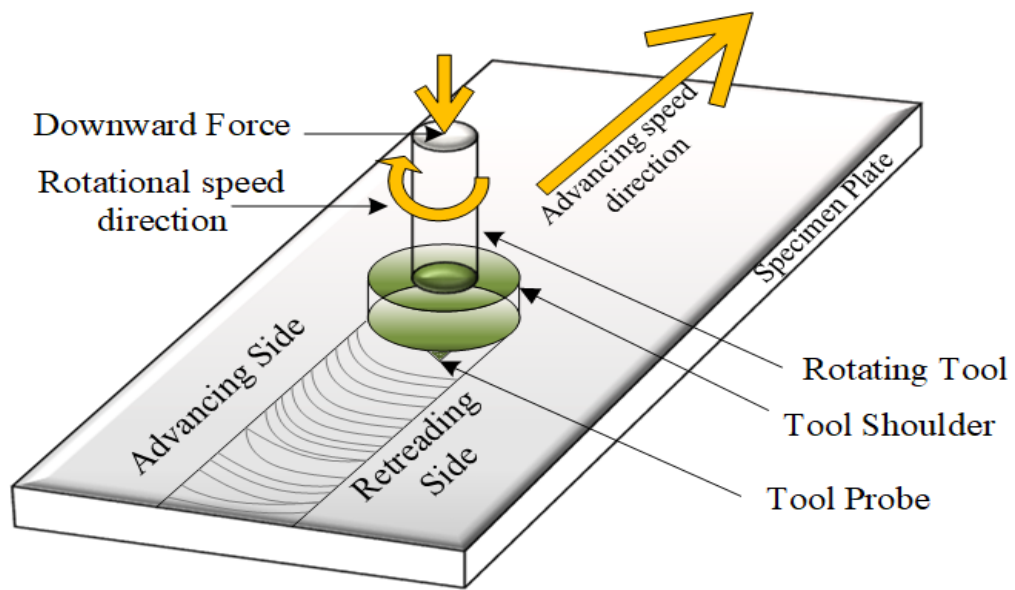


Figure 1 Schematic representation of Friction Stir Process [15].

Saeid et al. [16] investigated 2205 SDSS to examine the effect of different parameters of FSP on the microstructure and tensile behavior. FSP was executed at a rotational speed of 600 rpm and varying advancing speed of 50-250 mm/min using tungsten carbide stir tool. It was found that by increasing advancing speed from 50 mm/min to 200 mm/min, grain structure became more refined in the processed zone; however, groove like defect was produced at 250 mm/min. This was because, with the increase in advancing speed, heat input or peak temperature was decreased and at 250 mm/min, frictional heat was insufficient that created groove like defects. Also, it was discovered that with the reduction of grain size, hardness, and tensile strength of the SDSS increased. Other research on 22505 SDSS [17] reported that the grain sizes in the stir zone were not of same size and there was more grain refinement on the advancing side compared with the retreating side of the processed zone. Mishra et al. [18] studied 2507 SDSS to find out the effect of varying advancing speed (10-175 mm/min) on the microstructure of the material. It was observed that with the increase of advancing speed, grain size reduced until 100 mm/min then increased again. It was also found during research [19] that FSP produced sharp boundary on the advancing side of the processed zone and on the retreating side, gradual decrease in grain size was noticed from base material (BM) to the stir zone. Similar findings of increase in strength and hardness by grain refinement were reported [19] by analysis of friction stir processed 2507 SDSS. Santos et al. [20] studied the effect of FSP on four DSS UNS S32750, S32205, S32101, and S32760, with the processing speeds of 50-150 mm/min and the rotational speeds of 200-600 rpm using PCBN stir tool. It was noticed that the advancing side region experienced higher strains than retreating side, resulting in more grain refinement. It was also revealed that the stir zone had more tensile strength and hardness than BM. Table 1 summarizes the findings of different researchers on tensile behavior of DSS after FSP/FSW.

Table 1 Effects of FSP/FSW on tensile and hardness behavior.

Material	Processing Technique	Rotational speed	Advancing speed	Tool Material	Findings	Ref.
		rpm	mm/min			
lean DSS	FSW	800	50-150	WC based	3.5-11 % Increase in tensile strength. Increase in hardness.	[14]
2205 SDSS	FSW	600	50-250	WC based	Increase in tensile strength. Increase in hardness	[16]
2507 SDSS	FSP	800	10-175	Lanthanide-tungsten	7 % increase in tensile strength. 28 % decrease in ductility.	[18]
2507 SDSS	FSW	450	60	PCBN	Tensile strength was comparable. 40 % decrease in elongation. Increase in hardness.	[19]
lean DSS	FSP	300	100	W-Re	4 % decrease in tensile strength. 2 % decrease in elongation.	[21]

So, FSP refines the grains and because of grain refinement, the tensile and hardness behavior of the material will improve. Strength and hardness of material was improved because larger grain boundaries obstruct the dislocation movement; however, it reduced the elongation and ductility by making the processed material more brittle.

2.4 Effect of FSP on corrosion resistance

Corrosion has a negative effect on the behavior of the material. There are a lot of researches which revealed severe consequences of corrosion on properties of the material [22, 23]. Several researches are present in open literature to study the influence of FSP on the corrosion resistance of the metallic material. Sarlak et al. [24] examined the influence of FSW that was

performed at 800 rpm rotational speed and 50, 100 and 150 mm/min advancing speed on the corrosion behavior of lean DSS. The welded material was immersed in H_2SO_4 solution and corrosion resistance was evaluated by potentiodynamic polarization (PDP). Stir zone showed better corrosion resistance because of grain refinement and corrosion resistance was more enhanced by raising the advancing speed from 50 to 150 mm/min. Ajith et al. [25] investigated friction stir welded UNS S32205 DSS and found that welded specimen showed superior corrosion resistance than BM in 3.5 % NaCl solution. Similar research was presented on FSPed 2507 SDSS (FSP performed at 50 mm/min of advancing speed and 600 rpm of rotational speed by Mishra et al. [6] and corrosion resistance was assessed by PDP, open-circuit potential, and electrochemical impedance spectroscopy (EIS) that revealed that FSPed samples had better corrosion behavior than BM. Behnagh et al. [26] studied AA5083 aluminum alloy to find the influence of FSP on corrosion resistance. FSP was performed by using varying rotational speed (710-1400 rpm) and advancing speed (12.5-63 mm/min) and corrosion resistance was estimated by intergranular corrosion tests which showed that FSPed samples had better corrosion resistance than BM. Recent investigation on AISI 440C high carbon martensitic stainless steel [27] has done to examine the influence of FSP on corrosion resistance of the material. FSP was executed at rotational speed of 2000 rpm and processing speed of 150-300 mm/min using W-Re tool. PDP tests were performed in 3.5 % NaCl solution and results showed increase in corrosion resistance for FSPed samples than BM. Table 2 summarizes the findings of different researchers on corrosion resistance behavior of the material after FSP/FSW.

So, FSP refine the grains and because of grain refinement, corrosion resistance of material will enhance. Stable passive layer was developed on the surface of FSPed or welded material that enhanced the corrosion resistance.

Table 2 Influence of FSP/FSW on corrosion resistance.

Material	Processing Technique	Rotational speed	Advancing speed	Corrosive Environment	Findings	Ref.
		RPM	mm/min			
lean DSS	FSW	800	50-150	H ₂ SO ₄ solution	33-53 % increase in corrosion resistance.	[24]
S32205 DSS	FSW	-	-	3.5 % NaCl solution	Increase in corrosion resistance.	[25]
2507 SDSS	FSP	600	50	3.5 % NaCl solution	63-69 % increase in corrosion resistance.	[6]
AA5083	FSP	710-1400	12.5-63	5% NaOH and HNO ₃	Increase in corrosion resistance.	[26]

2.5 Effect of FSP on fracture toughness

Fracture toughness is the property of a material which describes how much energy will be absorbed by the material before fracture in the existence of an initial crack. The fracture toughness of any material must be known to understand the failure mechanism of the material because material having higher fracture toughness (more than K_{IC}) shows ductile failure whereas material having lower fracture toughness (less than K_{IC}) shows brittle failure [28]. As discussed earlier, FSP changes the microstructure so there will a change in the fracture toughness of the material. Researchers have examined the effect of FSP on the fracture toughness of the material. However, fracture toughness dependence on the FSP parameters (spindle speed and processing speed) is still not clear. Different studies have been performed on different materials to find out a proper relationship between fracture toughness and

processing parameters of FSP, but their findings were different. Some stated reduction in fracture toughness by FSP and some reported that fracture toughness of BM and friction stir processed material are comparable and some revealed increase in fracture toughness by FSP. But there is an agreement in literature that the change in fracture toughness by FSP depends on FSP parameters and material of tool, and specimen. Meinhardt et al. [29] investigated UNS S32760 SDSS to examine the effect of friction stir welding (FSW) on the fracture toughness of the material. It was revealed that fracture toughness of stir zone was reduced by about 19 % by FSW as compared with BM. Fairchild et al. [30] researched friction stir welded pipeline steel, grade X65 to X120. These welds were made at 300 rpm rotational speed and 51 mm/min advancing speed by PCBN tool. Their results showed that fracture toughness of weld material was significantly lower compared with BM. Similar work was performed by Santos et al. [31] on ISO 3183 X80M steel, to revealing that by using higher spindle speed (around 500 rpm), the fracture toughness of weld metal was reduced but for lower spindle speeds (around 300 rpm) fracture toughness of the weld metal was higher compared with BM. Similar results were stated by Tribe et al. [32] for API X80 steel who also found that fracture toughness was dependent on heat input and the rotational speed. They also showed that due to microstructure heterogeneity in the stir zone, the fracture toughness was different in the processed zone from weld center line to either direction. Avila et al [33] investigated on the pipeline steel API-5L-X80 to examine the behavior of fracture toughness of friction stir welded joints as compared with BM. The welding parameters used for FSW were 300 rpm and 100 mm/min of rotational and advancing speed, respectively. It was revealed that the stir zone produced by two passes of FSW had higher fracture toughness values than that of the stir zone of single pass weld samples which in turn was superior than that of the BM.

Table 3 Effects of FSP/FSW on fracture toughness.

Material	Processing Technique	Rotational speed	Advancing speed	Tool Material	Findings	Ref.
		RPM	mm/min			
S32760 SDSS	FSW	400	180	PCBN	19 % reduction in fracture toughness in welded area.	[29]
Pipeline Steel	FSW	300	51	PCBN	Significantly lower fracture toughness in welded area.	[30]
3183 X80M-Steel	FSW	300-500	100	PCBN	Fracture toughness depended on the rotational speed.	[31]
API X80 Steel	FSW	303-725	82-287	PCBN	Fracture toughness depended on the rotational and advancing speed.	[32]
API-5L-X80 Steel	FSW	300	100	PCBN-WRe	Significantly lower fracture toughness for single pass weld. Comparable fracture toughness for two pass weld.	[33]
A356	FSP	900	203.2	-	30 % increase in fracture toughness.	[34]

Sharma et al. [34] performed FSP on A356 aluminum alloy at a spindle speed of 900 rpm and advancing speed of 203.2 mm/min. They showed that fracture toughness of processed material increased by 30 % over that of BM.

It can be concluded that the fracture toughness depends on FSP parameters as well as on workpiece and tool materials. Although there is no specific trend available in literature on how

fracture toughness evolves with each of these variables. Table 3 summarizes the findings of different researchers on fracture toughness behavior of the material after FSP/FSW.

2.6 Influence of corrosion, secondary phases, and residual stresses on fracture toughness

Corrosive environments, secondary phases such as sigma phase and residual stresses affect the behavior of the material as discussed below.

2.6.1 Effect of corrosion on fracture toughness

SDSS have excellent corrosion resistance because of high Cr and Mo content in its composition. Despite its excellent corrosion resistance, in a severe corrosive environment and at higher operating temperature, fracture toughness behavior of the material is affected. Meinhardt et al. [29] investigated UNS S32760 to study the effect of synthetic seawater on the fracture toughness. They revealed that the fracture toughness was reduced owing to hydrogen induced stress cracking (HISC) and the stir zone was less sensitive to HISC than BM. Research by Lage et al. [35] on welded UNS S32750 reported similar finding that fracture toughness was reduced in the presence of hydrogen (corrosive environment) due to HISC. So, corrosive environment effects the fracture toughness of the material.

2.6.2 Effect of secondary phases on fracture toughness

Friction stir processing generates high temperature around the tool while processing the material. This high temperature could result in precipitation of secondary/intermetallic phases like sigma which will cause embrittlement, reducing the fracture toughness of the material [36]. A lot of researches have done to find out the effect of these phases on the material behavior. De Lacerda et al. [37] researched on UNS S31803 to investigate the impact of phase change

on material behavior. Samples were annealed at three different temperatures, 1060° C, 1200° C, and 1300° C. At these high temperatures, the change in α and γ proportions and undesirable phase precipitation (sigma phase and chromium nitrides) occurred. Research by Borvik et al. [38] on 2205 SDSS revealed that the fracture toughness reduced significantly by sigma phase and this decrease in fracture toughness was more than 90 % due to the high content (about 10 %) of sigma phase. Calliari et al. [39] researched on 2205 SDSS and reported that the amount/size of secondary phase decided the failure mode of the material whether it would be a ductile failure or a brittle failure. So, with the increase of the percentage of secondary phase, fracture toughness reduces, and material will behave in a brittle manner.

2.6.3 Effect of residual stresses on fracture toughness and corrosion resistance

Tensile residual stresses have a detrimental impact on material properties like strength, hardness, and fracture toughness. These stresses can be developed in the material by machining or by welding. FSP can also produce these residual stresses that can affect the mechanical behavior of the material. Moghadam et al. [40] investigated 2024-T351 aluminum alloy to examine the influence of FSP parameters on the residual stresses and the influence of these stresses on the fracture toughness. They revealed that residual stresses were developed in the material, resulting in reduction of fracture toughness by 18-49% than that of the BM. Cain et al. [41] examined Ti6AL4V which was produced by selective laser melting technique that caused residual stresses because of temperature fluctuations. They reported that there was a great effect of residual stresses on fracture toughness of the material. Moshayedi et al. [42] researched two A533B steel pipes, one was as received and other was welded by tungsten arc welding to study the effect of residual stresses on fracture toughness of the material. They stated that fracture toughness reduced in the welded pipe than as received pipe due to the residual stresses.

Residual stresses affect the corrosion resistance behavior of the material. Rajaguru et al. [43] studied SDSS to find out the impact of residual stresses on corrosion resistance of the material. Samples (as received and machined), exposed to a corrosive solution of MgCl_2 , revealed that as received samples had higher stress corrosion cracking (SCC) resistance and no cracks were observed on their surface. However, machined samples had low SCC resistance and cracks were observed on their surface due to the residual stresses induced during machining. Takakuwa et al. [44] examined the impact of residual stresses on corrosion resistance of the austenitic stainless steel 316L and reported that corrosion resistance was reduced by tensile residual stresses that developed in the material.

The effects of the tensile residual stresses can be reduced by several techniques, such as laser shock processing (LSP) which introduces compressive residual stresses in the material to compensate the influence of tensile residual stresses. An investigation on aluminum alloy 6061-T6 described that laser shock processing increased the fracture toughness of this alloy by 4-15 % using different pulse densities by eliminating the effect of tensile residual stresses [45]. A similar research on 2205 DSS reported that LSP increased the fracture toughness of DSS by compensating the effect of residual stresses developed by machining [46].

2.7 Conclusion

Based on the above literature review, it can be concluded that:

- UNS S32750 SDSS has good mechanical properties such as tensile strength, fatigue life, fracture toughness, and corrosion resistance.
- FSP refines the grain structure of material. So, FSP is expected to, in general, enhance the mechanical properties of material.
- Fracture toughness may be increased or decreased by FSP, depending on FSP parameters and tool geometry and material.

- FSP improves the corrosion resistance of the material.
- Corrosion reduces the fracture toughness of the material.
- FSPed material are less prone to reduction in fracture toughness than that of base metal when exposed to corrosive environment due to increases the corrosion resistance.
- FSP induces residual stresses in the material and these stresses can affect the behavior of material.
- FSP generates high temperature while processing, so at high temperature secondary/intermetallic phases such as sigma can produce, that can affect the behavior of the material.

CHAPTER 3

EXPERIMENTAL PROCEDURE

3.1 Material

The material used for this research is UNS S32750 super duplex stainless steel (grade 2507) with the chemical composition as illustrated in Table 4. This alloy is provided by ArcelorMittal in the form of 20 mm thick plate manufactured by hot rolling. The slab was sliced into plates of 6.5 mm thickness for sample manufacturing and processing.

Table 4 Chemical composition of UNS S32750 SDSS.

Cr	Ni	Mo	Mn	Si	N	Cu	P	C	S	Fe	PRE-N*
25.299	6.681	3.729	0.847	0.356	0.2758	0.114	0.027	0.026	0.0002	Bal.	42

* Pitting resistance equivalent number

3.2 Friction stir processing

FSP was performed using research base friction stir welder RM-1 (MTI-USA) which is equipped with necessary sensors and data acquisition system for measuring tool reaction loads and other parameters including but not limited to rotational and welding speeds and plunging depth. The machine is computer numerical controlled (CNC) and has the capability to perform FSW under position and force controls. In position control, plunging depth remains constant and force varies throughout the process while in force control, force remains constant and plunging depth varies. The machine forging capacity is limited to 65 kN, which is applicable for processing steel and stainless steel. A water-cooled tool holder, shown in Fig. 2, was used

to hold and drive the PCBN tool Q70 (Fig. 3), having 5.2 mm tapered pin length and 25 mm shoulder diameter. Both tool holder and tool are manufactured by Megastir (USA). Initially, tungsten rhenium (W-Re) tool, having 1.63 mm of taper pin length and shoulder diameter of 6 mm, was used for FSP. Five friction stir processes were performed with different processing parameters as listed in Table 5, but surface quality was not good. So, W-Re tool was not suitable for this research work and PCBN Q70 tool was used for FSP of UNS S32750 SDSS. PCBN tool was selected because it was mostly used by different researchers for processing SDSS.

There are various ways to optimize FSP, like surface quality (presence of defects and smoothness), grain refinement etc. In this research work, the optimization is based on surface quality. FSP was performed under five different parameters on UNS S32750 SDSS plate as listed in Table 6 using PCBN tool. Three friction stir processes were performed under position-control and two were performed under force-control. All FSPs were performed with argon shield (Fig. 2) and along the rolling direction of the plate. Rotational speed and tilt angle were also kept constant at 400 rpm and 2°, respectively. FSP-1 produced very rough surface and surface defects were observed in force control (FSP-4 and FSP-5) as shown in Table 6. Surface quality produced by FSP-2 was good among all friction stir processes. The parameters of FSP-2 that resulted in the best surface quality were selected for further processing.

Seven FSPs were performed on UNS S32750 SDSS plates as shown in Fig 4 using parameters of FSP-2 and infrared red camera was placed at 120 cm from the FSP tool to measure the peak temperature during the process. Three tensile, eight compact tension (CT) and a number of rectangular samples for corrosion and microstructural analyses were machined out of these plates.

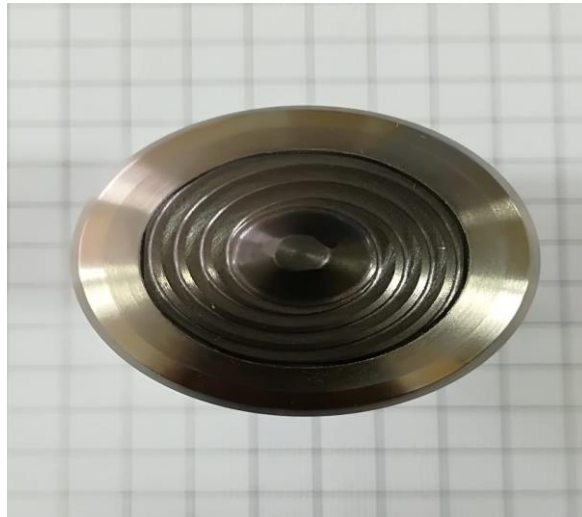


(a)

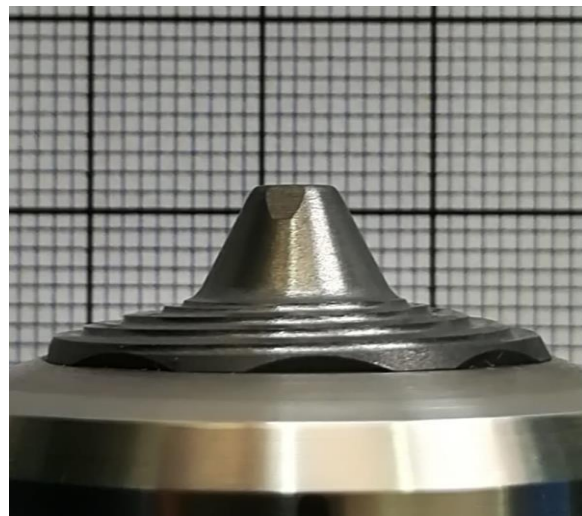


(b)

Figure 2 Friction Stir Processing setup: (a) RM-1 Friction Stir Welder, (b) Processing tool and platform.



(a)



(b)

Figure 3 PCBN tool profile: (a) Top view, (b) Front view.

After seven FSP operations, the tool and pin condition started to degrade, as shown in Fig 5. This is translated by a distorted pin and workpiece material build up on the tool shoulder. It can be seen in the Fig. 5 that pin and shoulder of FSP tool are severely damaged after processing because of the superior hardness of UNS S32750 SDSS.

Table 5 Processing parameters of FSP using W-Re tool.

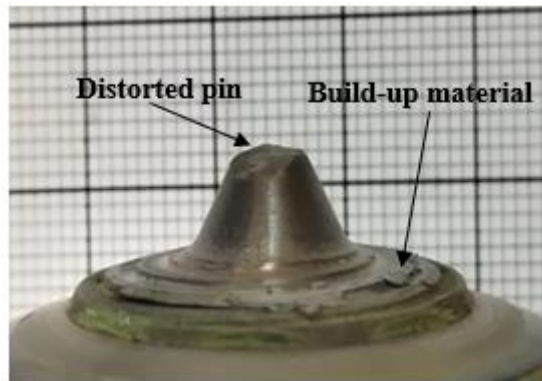
FSP No.	Advancing speed (mm/min)	Rotational speed (rpm)	Tilt angle	Control Method	Images
1	20	400	3°	Force	
2	20-75	800	3°	Force	
3	80	800	3°	Force	
4	75	800	3°	Position	

Table 6 FSP parameters for optimization of the process using PCBN tool.

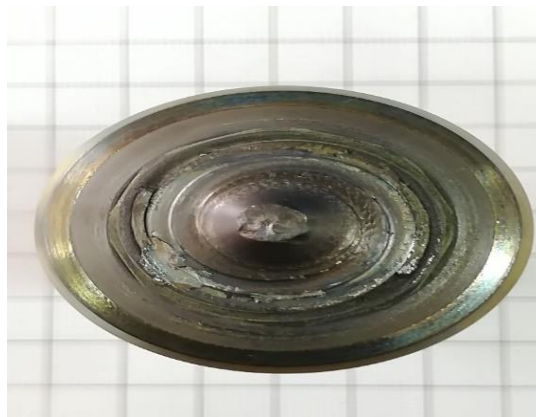
FSP No.	Advancing speed (mm/min)	Plunging depth (mm)	Control method	Images
1	50	5.2	Position	
2	100	5.8	Position	
3	150	6.2	Position	
4	150	6.0	Force (55 kN)	
5	150	6.0	Force (60 kN)	



Figure 4 UNS S32750 SDSS FSPed plates.



(a)



(b)

Figure 5 Tool profile after FSP: (a) Front view, (b) Top view.

3.3 Microstructural analysis

After FSP, plates were sectioned across the processing direction to examine the changes in the microstructure within the processed zone and neighboring areas. Standard metallographic sample preparations were performed which includes mounting, grinding, polishing, and etching. FSPed UNS S32750 SDSS plate was cut across the cross-section and then was grinded with silicon carbide grit papers (grit size of 240-800). After this, the surface of the specimen was polished using an alumina solution, before etching by a solution of 2g picric acid and 10 ml hydrochloric acid (HCL). Advanced optical microscope (DSX510 Upright Microscope, Olympus, Japan) was used to examine the etched samples and study the developed microstructure. Grain size was measured in the base metal and stir zone using line intercept method. X-Ray Diffraction (XRD) analyses of base metal and processed material were also carried out using Rigaku Miniflex to examine the formation of sigma phase during FSP. XRD was performed using Cu-K α radiation (30 kV and 15 mA) with a scan rate of 2°/min.

3.4 Mechanical Testing

Mechanical behavior of UNS S32750 SDSS was studied by tensile, hardness and fracture toughness testing of base and FSPed samples.

3.4.1 Tensile testing

Tensile testing of base metal and FSPed samples was carried out according to ASTM standard E8/E8M-16a [47], on Instron Material Testing Machine 8801 having a load capacity 100 kN. The sub size tensile sample dimensions are shown in Fig. 6a. Cutting was done along the processing direction, with the whole specimen in the processed region to exclude the influence of heat affected zone (HAZ) on processed material strength (Fig. 6b). Three tensile tests were

carried out on each of the base metal and the FSPed samples at a loading rate of 1 mm/min. Strain was determined using axial extensometer until a strain value of 20%.

3.4.2 Microhardness testing

Microhardness testing was performed to examine the effect of FSP on the hardness of the UNS S32750 SDSS. Vickers hardness measurements across the processed zone and base metal were performed at 500 gf. First, the samples were grinded by silicon carbide grit papers and then polished by alumina solution. For FSPed sample, hardness measurements were taken at nine different positions in the stir zone from advancing side to center of the processed material (C) to retreating side in the stir zone as illustrated in the Fig. 7. For base metal, hardness was determined at three different points across the rolling direction.

3.4.3 Fracture toughness testing

Fracture toughness testing of the BM and the FSPed samples was carried out using Instron 8801 machine, according to ASTM standard E1820-20 [48]. The effect of seawater on the fracture toughness of UNS S32750 SDSS was also studied. Fracture toughness testing was done on compact tension (CT) samples using the basic J-integral method described in ASTM E1820-20 [48]. The J- integral approach was chosen over the stress intensity factor (K_I) due to the presence of large plastic zone; making linear elastic fracture mechanics (LEFM) not applicable.

The geometry and dimensions of the CT sample are illustrated in Fig. 8a. CT samples, from the FSPed plate were cut such that the notch and crack tip are in the center of the stir zone and along the processing direction, as shown in Fig. 8b. Electrical Discharge Machining (EDM) was used to generate the groove and notch in the CT specimens. Testing setup for fracture toughness experiments is shown in Fig. 9. The CT samples were fatigue pre-cracked and a

high-resolution camera was employed to monitor the fatigue crack growth. The fatigue pre-cracking was done under load control using a stress ratio of 0.1 and a maximum load of 6 kN, for all samples. Fatigue pre-crack length was kept around 2 mm. After fatigue pre-cracking, fracture toughness testing was performed under position control at a loading rate of 1 mm/min and 60 loading/unloading sequences (Fig. 10a) for all samples. The load-crack opening displacement curves and Eqs. 1-5 [48] were used to determine the fracture toughness (J) using the basic method described in ASTM E1820-20 [48].

$$J = J_{el} + J_{pl} \quad (1)$$

$$J_{el} = \frac{K^2(1 - \nu^2)}{E} \quad (2)$$

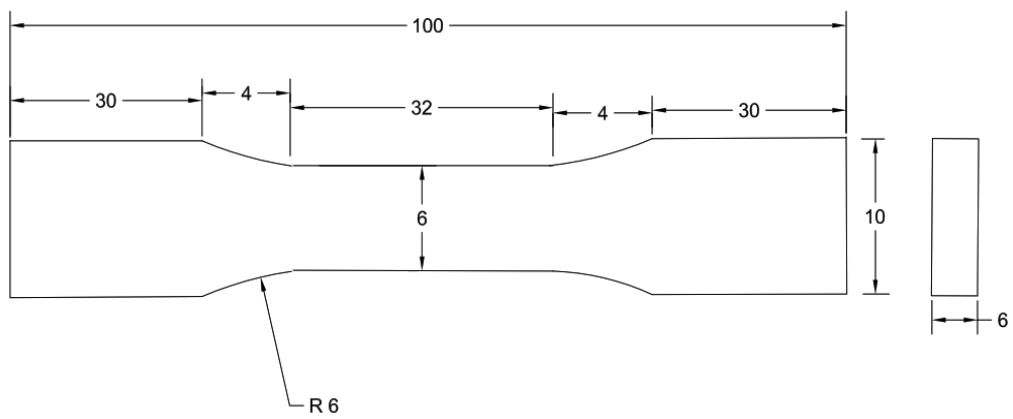
$$K = \frac{P_k}{(BB_N W)^{1/2}} f\left(\frac{a_0}{W}\right) \quad (3)$$

$$J_{pl} = \frac{\eta_{pl} A_{pl}}{B_N b_o} \quad (4)$$

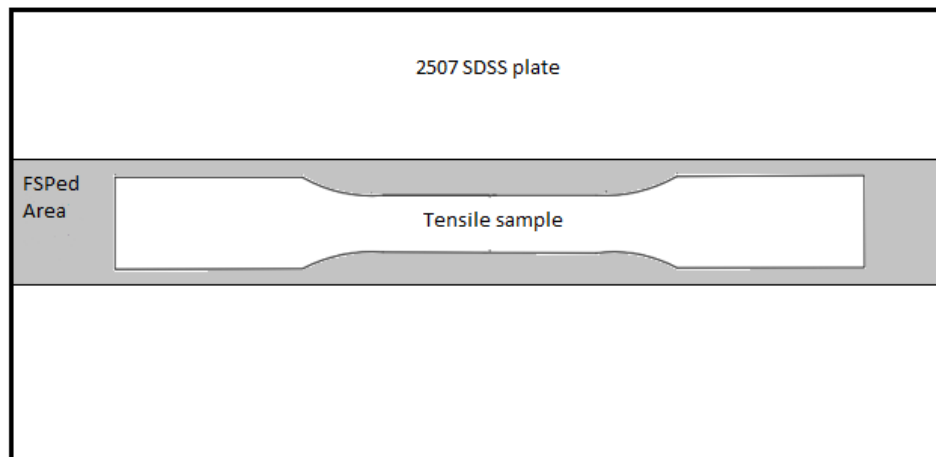
$$\eta_{pl} = 2 + 0.522 \frac{b_o}{W} \quad (5)$$

Whereas J_{pl} and J_{el} are respectively, the plastic and elastic components of fracture toughness (J). K , ν , E , P_k , B , a_0 , B_N and b_o are respectively, the stress intensity factor, poison's ratio, modulus of elasticity, peak load, thickness, initial crack length, net specimen thickness and uncracked ligament length. A_{pl} is the area under the load-displacement curve shown in Fig. 10b.

Fatigue pre-cracked, base and FSPed, CT samples were put in the beaker containing 3.5 % NaCl solution (Fig. 11) to examine the effect of FSP on fracture toughness in seawater. Voltage (-250 mV to +1000 mV) was applied to accelerate the corrosion rate. This is called potentiodynamic polarization (PDP) process. After that, fracture toughness testing was done to study the influence of corrosion on the fracture toughness of BM and FSPed UNS S32750 SDSS.



(a)



(b)

Figure 6 Tensile sample: (a) Geometry and dimensions in mm, (b) Sample position in processed area.

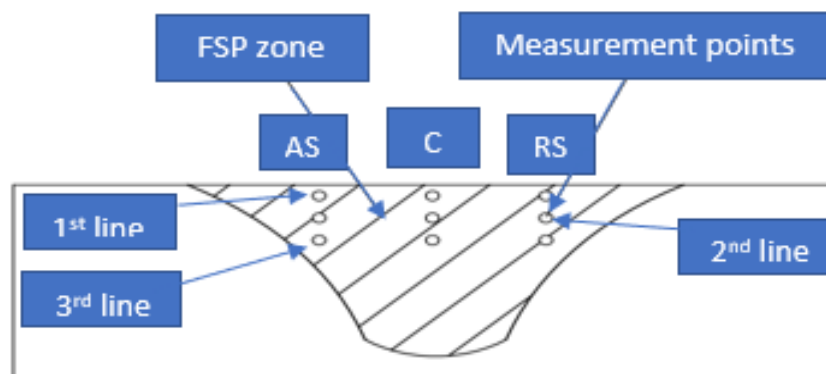


Figure 7 Hardness measurement spots in Stir Zone.

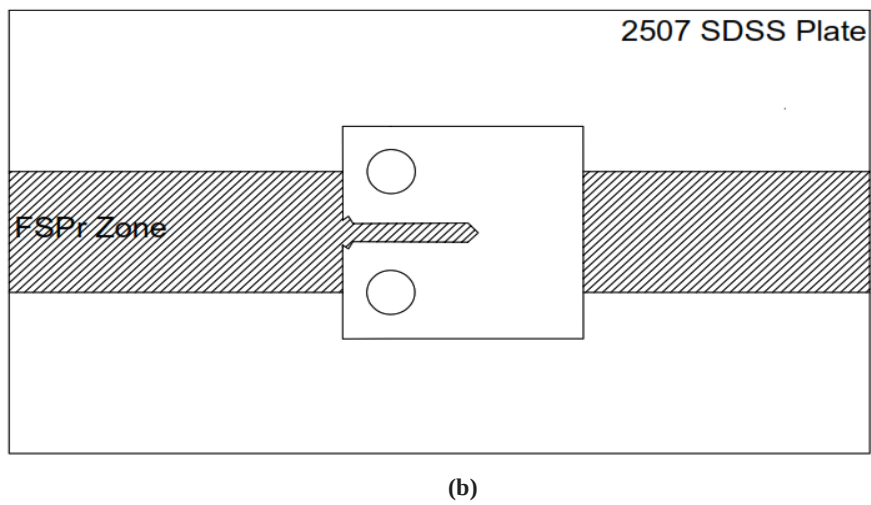
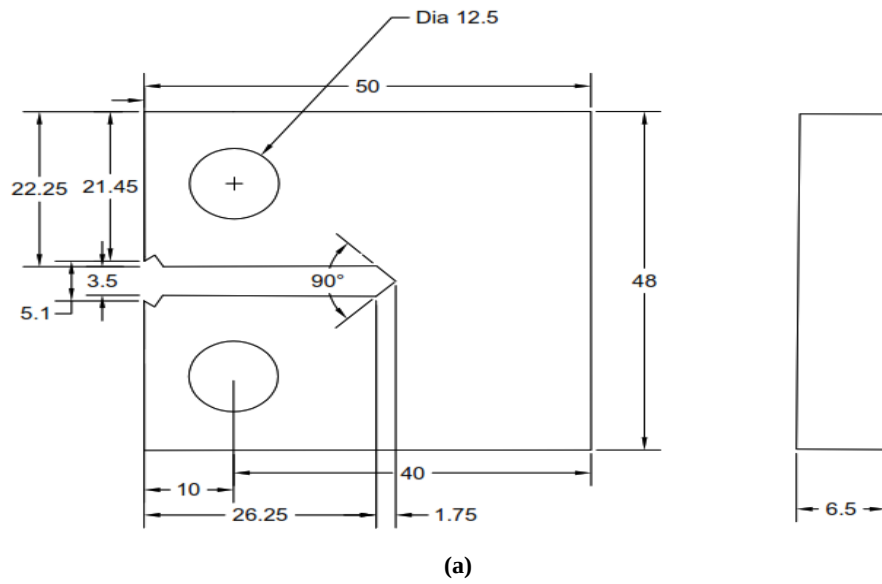


Figure 8 CT sample: (a) Dimensions in mm, (b) Position of CT sample in processed area.

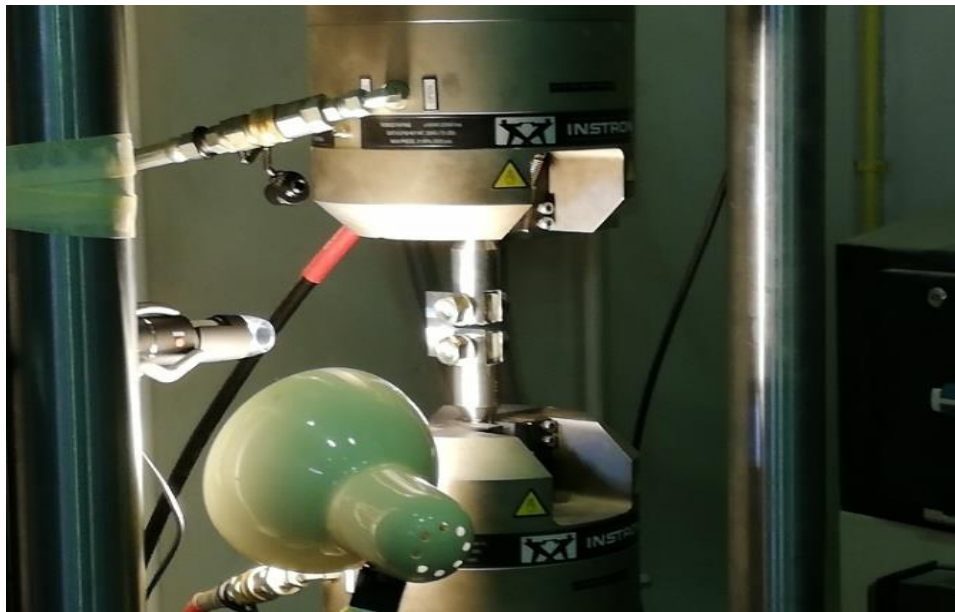


Figure 9 Fracture toughness testing setup of CT samples on Instron 8801.

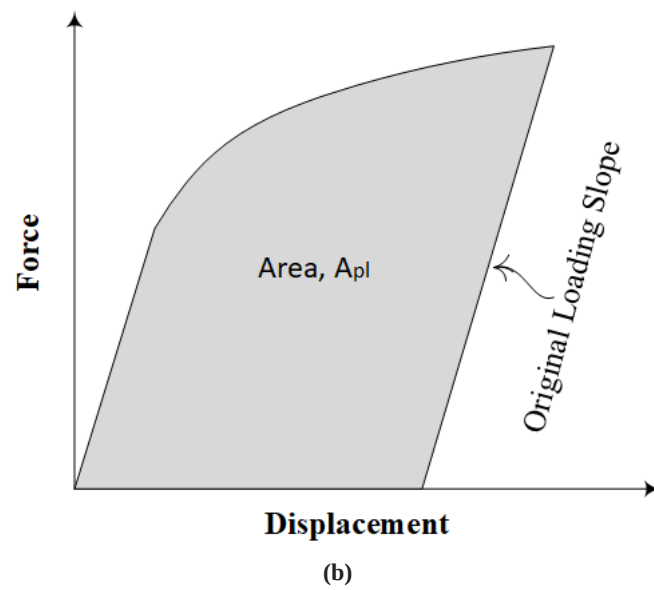
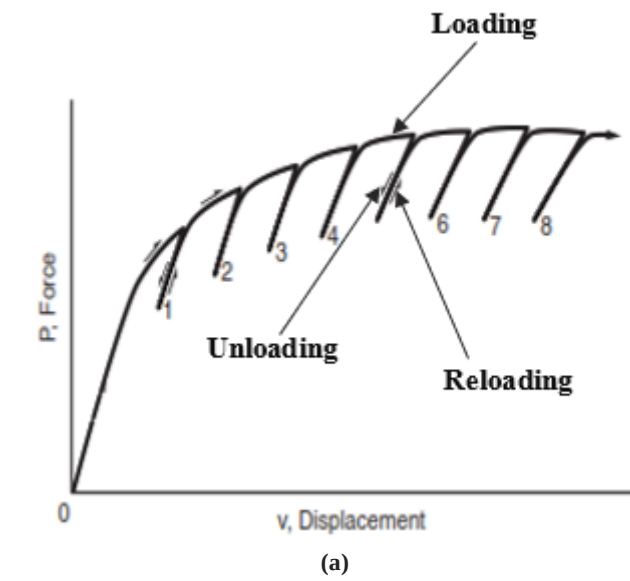


Figure 10 Definitions: (a) Loading/Unloading sequence [49], (b) A_{pl} area from Load-displacement curve.

3.5 Corrosion testing

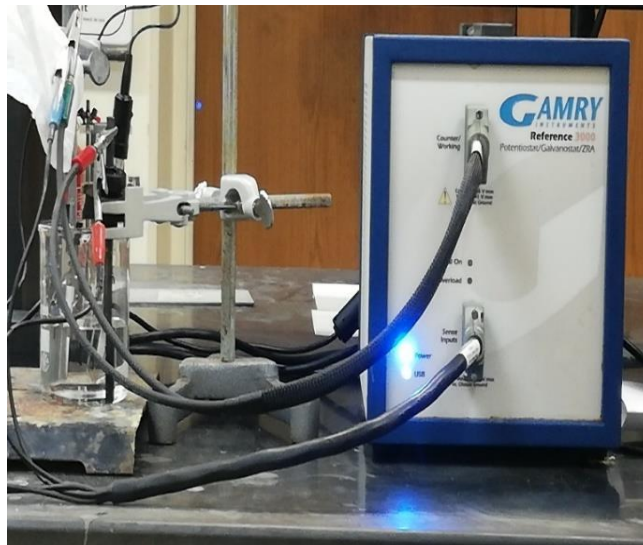
Electrochemical corrosion tests were carried out for the base and FSPed samples using Gamry Reference 3000 electrochemical system. A typical three-electrode cell assembly was selected to perform the corrosion tests, in which SDSS samples acted as working electrodes. Both the base and FSPed samples were ground by silicon carbide grit papers (240-1200) followed by polishing with alumina solution. Later, these samples were immersed in 3.5 % NaCl medium for 60 min (Fig. 12). to achieve the steady-state open circuit potential (OCP). During the test, about 16 mm² surface area of each sample was exposed to the corrosive environment. LPR tests were performed using -20 and +20 mV of electrode potential against the E_{corr} at the scanning rate of 0.125 mV/s. PDP test was performed in cathodic and anodic polarization regions by changing the electrode potential (- 250 mV to + 1000 mV) from OCP at a sweep rate of 0.197 mV s⁻¹. Corrosion rate of both samples were determined by using Eq. 6. All electrochemical corrosion tests were repeated three times to validate the results. EDS analysis of BM and FSPed metal was also carried out using SEM (JEOL JSM-6460) to examine the surface composition especially oxide layer.

$$Corrosion\ rate\ (mm\ per\ year) = 3.27 \times 10^3 \frac{i_{corr}}{density} Equivalent\ weight \quad (6)$$

In Chapter 4, the results of the microstructure characterization, tensile, hardness, fracture toughness and corrosion are discussed in detail and the effect of FSP, on the grain structure and mechanical behavior of UNS S32750 SDSS, is explained by the findings of these experiments.



(a)

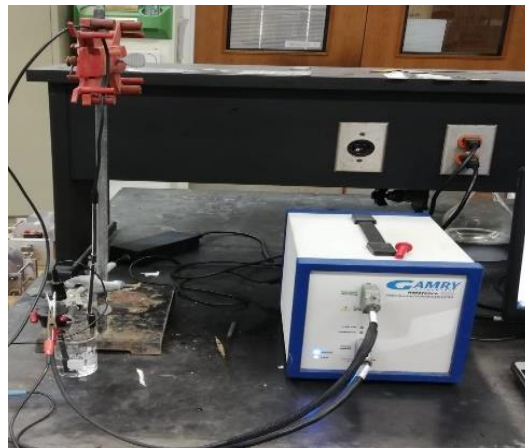


(b)

Figure 11 CT sample potentio-dynamic polarization: (a) CT sample immersed in 3.5 % NaCl solution, (b) Gamry Instrument for corrosion testing.



(a)



(b)

Figure 12 Experimental setup for corrosion testing: (a) Sample immersed in 3.5 % NaCl solution, (b) Gamry system for corrosion testing.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Effect of FSP on Microstructure of UNS S32750 SDSS

Figure 13 shows the microstructure of the processed region and Figure 14 presents images taken along the microstructure of processed region at different points (a, b, c, d) spanning from base metal through the stir zone.

The images shown in Fig. 14, were taken at the same magnification of 277x along the cross section of the processed region. It can be seen in Figs. 13 and 14b that there was a sharp boundary created by a sudden decrease in both austenite and ferrite grain sizes from base metal to stir zone on the advancing side, whereas on the retreating side the grain size reduced gradually from base metal to stir zone as shown in Fig. 14d. On the advancing side, rotational velocity vector and advancing velocity vector are in the same direction but on the retreating side, they are in opposite directions, as shown in Fig. 1 [50], resulting in higher net force on the advancing side. Thus, the grains on the advancing side experienced more plastic deformation compared to the grains on the retreating side. This caused a sharp boundary on the advancing side and the gradual increase in grain size from the advancing side to the retreating side in the stir zone. Additionally, peak temperature was different due to the difference in plastic strain in the stir zone [51]. The observed asymmetry in grain size, with respect to the processing centerline, was mainly due to this difference in plastic strains and temperatures from the advancing side to the retreating side. This observation is in agreement with the findings of Seidel and Reynolds [52]. Mishra et al. [18] concluded that the asymmetry of the stir zone can be attributed to the variation in temperature and plastic strain. The average grain size in base

metal is about 160 μm while it varies between 2 and 30 μm in the stir zone of FSPed UNS S32750 SDSS. As discussed earlier, the refinement of the grains in the stir zone is caused mainly by dynamic recrystallization due to plastic deformation and heat produced by friction. Grain size variation in the stir zone can also be observed in Figs. 13 and 14. These findings are similar to those reported by Mishra et al [18] and Santos et al [20] for the same type of material.

During FSP, peak temperature, measured by infrared camera, was around 1000° C and sigma (intermetallic) phase can occur as it is reported to form within the temperature range of 700°C to 900° C in DSS [53]. Sigma phase is a brittle and hard phase that influences the mechanical behavior of the material [54]. Thus, XRD was performed to examine the formation of sigma phase. It was clear that XRD pattern of FSPed sample (Fig. 15a) does not show any new peaks, thus sigma phase was not produced during FSP as sigma phase peaks occur in planes (0 0 2), (4 1 0), (2 1 2), (4 1 1), (3 3 1) and (2 2 2) [55]. Similar findings were stated by Li et al. [56] for FSPed hyper DSS. They explained that sigma phase was not formed because it needed more time to form at a temperature range of 700°C to 900° C. Sato et al. [57] revealed that around 52 minutes were required at a temperature of 827° C for sigma phase formation in FSW of DSS, while Elmer et al. [58] found that around 3 minutes were required at temperature 850° C for formation of around 1% sigma phase during FSW of 2205 SDSS. Cooling rate of FSPed samples are higher and temperature of the material reduces rapidly after FSP. Cui et al. [59] showed that the cooling rate was around 75° C/s for the carbon steels that were processed at a welding speed of 100 mm/min. It may be concluded that the absence of sigma phase in the present process was due to high cooling rate.

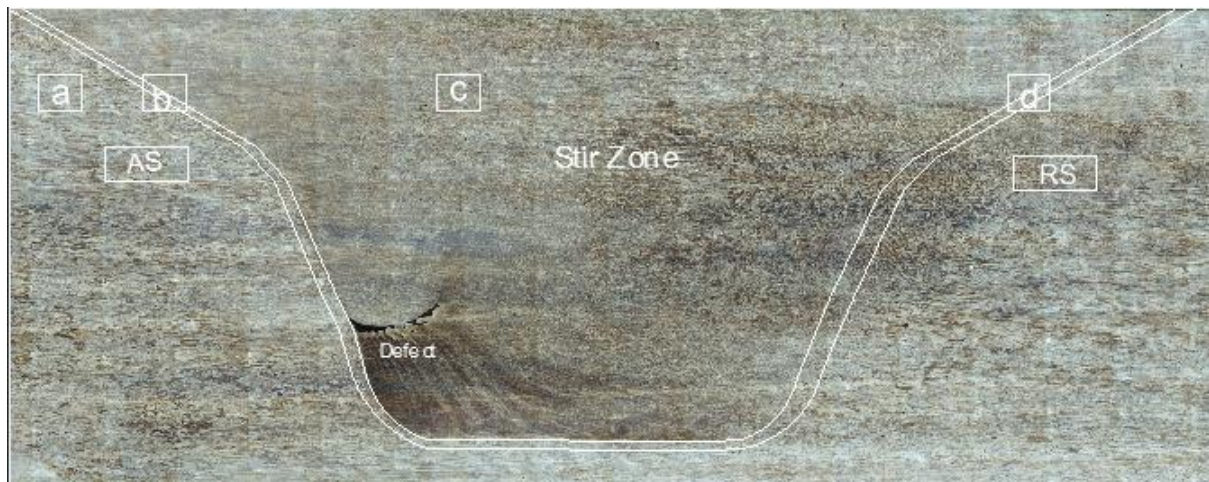


Figure 13 Microstructural Image of FSPed UNS S32750 SDSS. From Base metal (a) to thermo-mechanically affected zone (TMAZ) on advancing side (b) to Stir zone (c) to TMAZ on the retreating side (d).

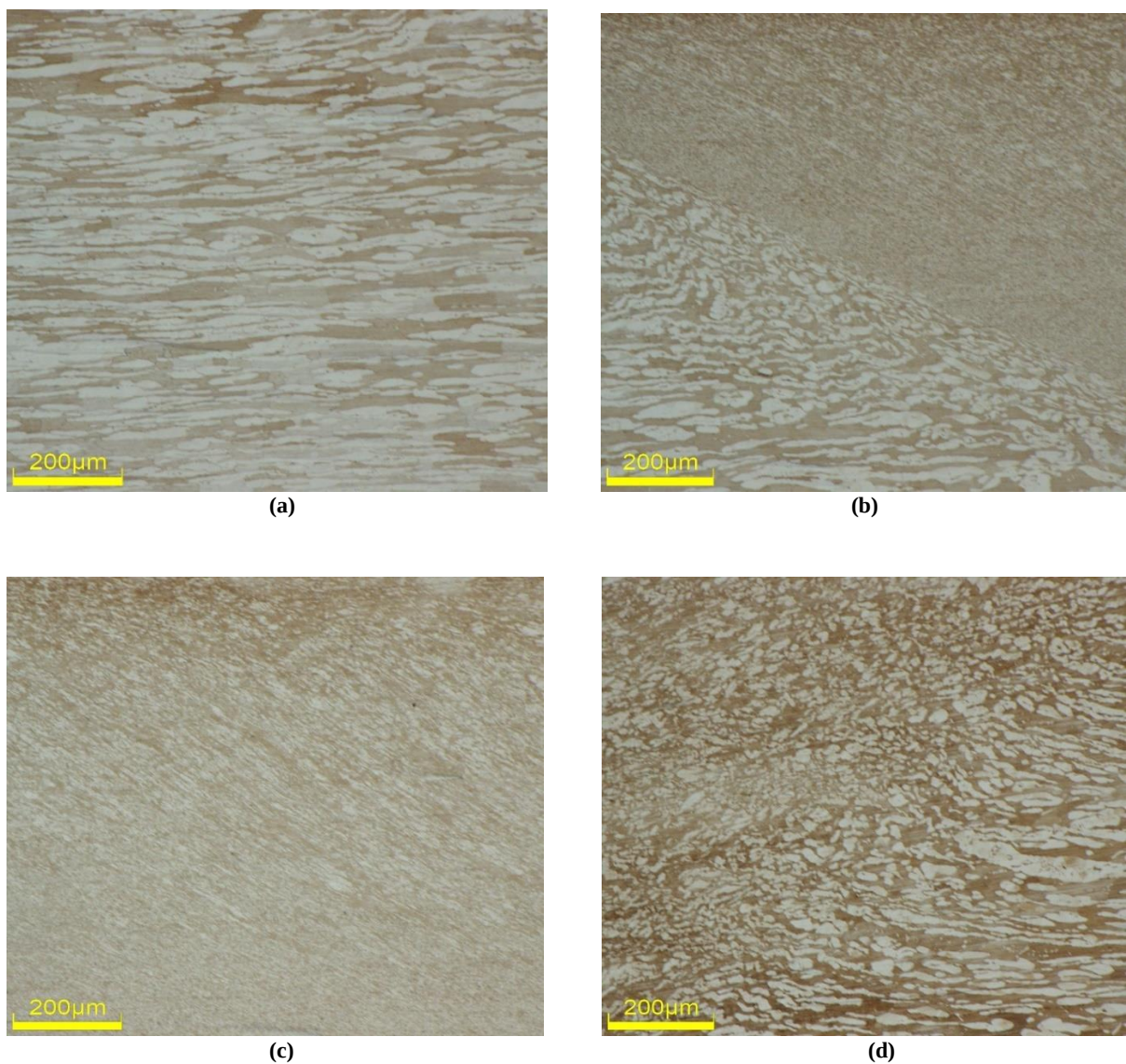
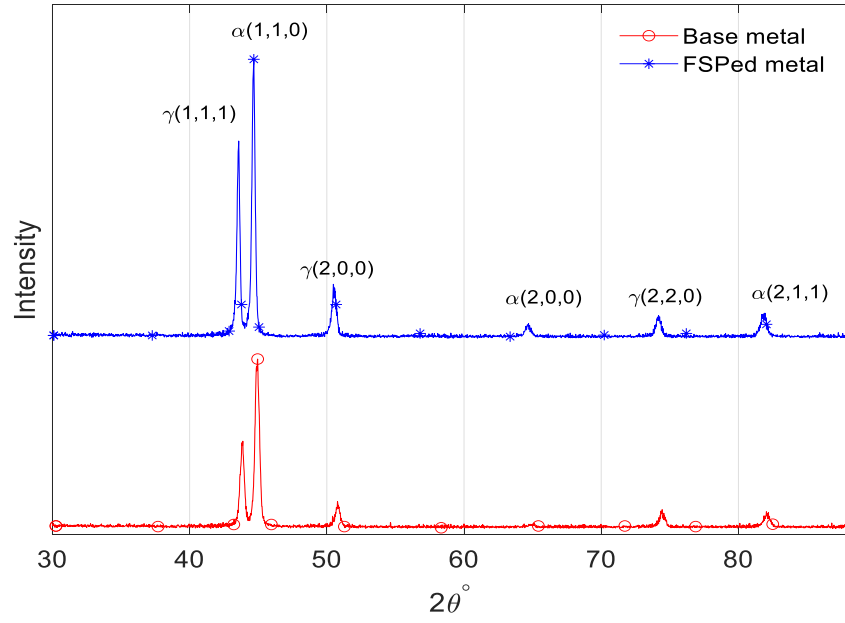
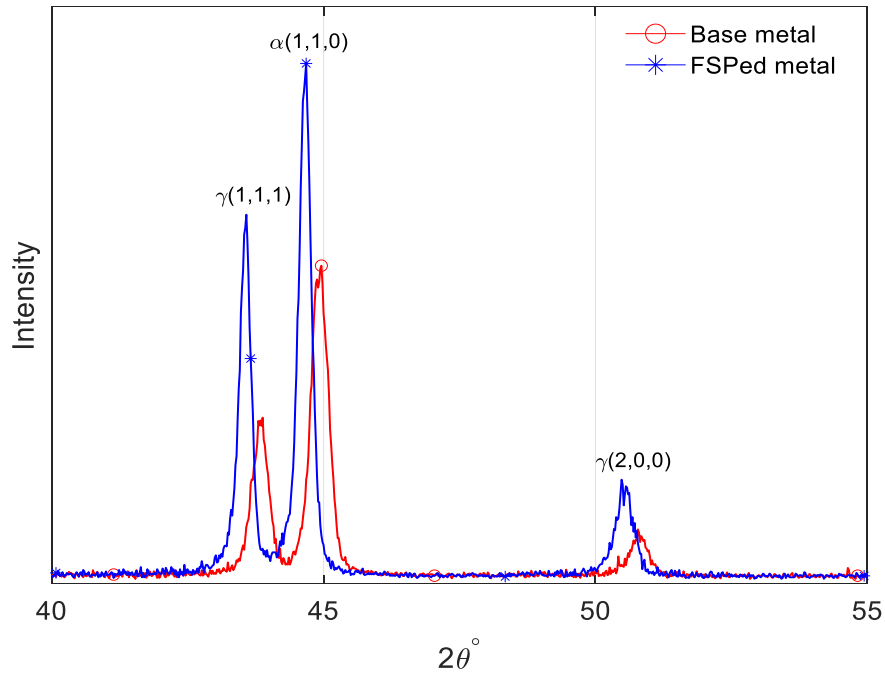


Figure 14 Microstructure of processed material: (a) Base metal, (b) Boundary of base metal and stir zone on advancing side, (c) Stir zone, (d) Boundary of base metal and stir zone on retreating side [15].



(a)



(b)

Figure 15 XRD of samples: (a) XRD of base and FSPed UNS S32750 SDSS, (b) Overlapped XRD of base and FSPed UNS S32750 SDSS [15].

The only difference between XRD patterns of FSPed and base metal was a little shift in the pattern to the left as shown in Fig. 15b. The shifting was uniform for the whole pattern and it was due to the strain developed in the material due to FSP [60]. It can also be noted that relative intensity in both patterns is the same for austenite and ferrite phases, so it was concluded that there was no change occurring in the concentration of both these phases after FSP.

4.2 Influence of FSP on Tensile and Microhardness properties of UNS S32750 SDSS

Figure 16 shows typical engineering stress-strain curves of tensile samples for BM and FSPed UNS S32750 SDSS. It is clear that the FSPed material displays higher strength and lower ductility when compared to base material. Table 7 illustrates the values of tensile and yield strengths for the six tensile specimens with the averages for base and processed samples. It reveals that the yield and tensile strengths of the processed material have improved by 14.3 % and 12.2 %, respectively. It is worth mentioning that while the material strength increased due to FSP its fracture strain decreased by an average of 29 %. This is expected because the improvement in strength is mainly due to grain refinement by FSP, the existence of more grain boundaries in the stir zone has diminished the dislocation motion and therefore the ductility of the FSPed material [61]. It can be seen, in Fig. 17, that larger necking occurred in the base material than FSPed material and that the FSPed sample showed less ductile fracture. This was due to the decrease in the ductility of material after FSP. The processing has increased the grain boundaries and these grain boundaries hinder the movement of dislocations resulting in dislocation pile-up and multiplication, causing semi-brittle fracture of the material [62-64].

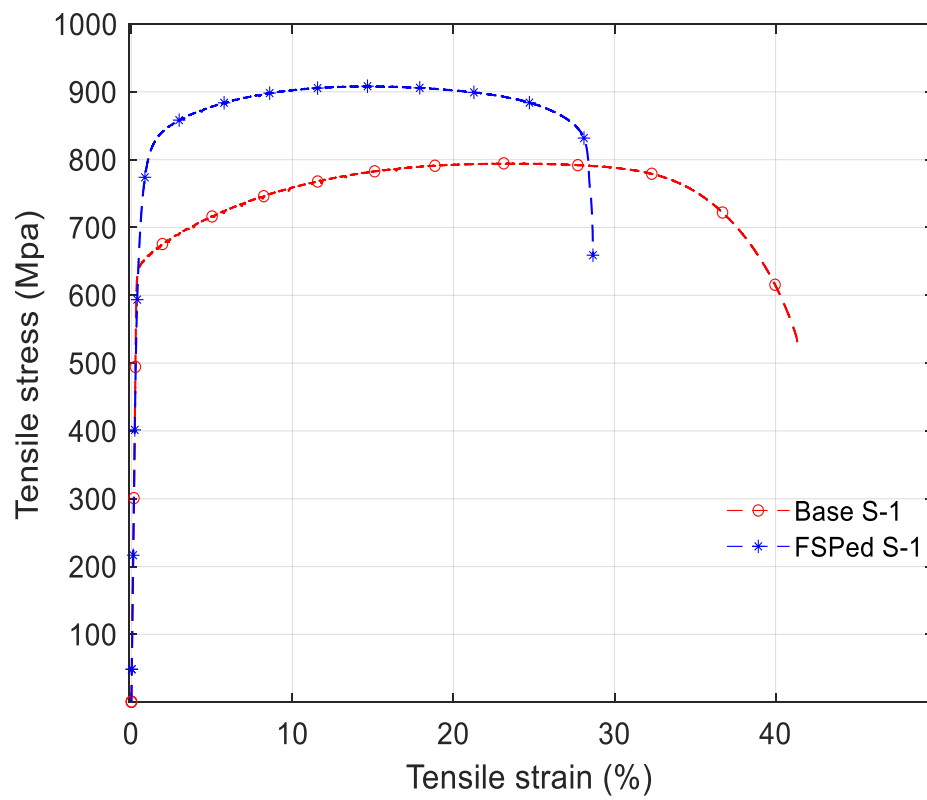


Figure 16 Tensile behavior of base and FSPed UNS S32750 SDSS [15].



(a)



(b)

Figure 17 Fractured tensile sample: (a) BM UNS S32750 SDSS, (b) FSPed UNS S32750 SDSS [15].

Table 7 Yield, tensile strength and fracture strain of base and FSPed UNS S32750 SDSS.

Sample No.	Yield Strength (MPa)	Tensile Strength (MPa)	Fracture strain (ϵ_f) (%)
Base S-1	651.75	794.43	41.37
Base S-2	665	784.3	-
Base S-3	650	802.2	-
Average	655.6	793.6	41.37
FSPed S-1	767.43	908.37	28.66
FSPed S-2	769.85	903.99	27.77
FSPed S-3	710.04	858.13	31.78
Average	749.11	890.16	29.40
% Increase/decrease w.r.t BM	↑ 14.94	↑ 12.05	↓ 28.93

The hardness measurement results are presented in Table 8 and the variation of the average values of the hardness is shown in Fig. 18. It can be noticed in Fig. 18 that the hardness increased in the stir zone from retreating side to advancing side because grain size decreased from retreating side to advancing side. The increase in the hardness in the stir zone was not uniform and this behavior was because of the heterogeneity of grain size in the stir zone. The average hardness improvement in retreating side is about 12% while that in advancing side is more than 14%.

It is known that the material having larger size grains has low number of grain boundaries and is weaker compared to material with lower size grains and significant number of grain boundaries. Because dislocation motion has to go through a lesser number of grain boundaries in larger size grain material [61, 65]. There are various models like Hall-Petch relation

available in the literature, which describe that grain size has inverse relation with the strength and hardness of the material and this increase in strength and hardness is compensated with decrease in ductility of the material [61, 66]. The findings of the present research concerning the tensile and hardness behavior of the UNS S32750 SDSS after FSP, are similar to those reported by a number of researchers [14, 16, 19, 20].

Table 8 Vickers Hardness of base and FSPed UNS S32750 SDSS.

Vickers hardness test (HV)					
	Sample No.	AS	C	RS	BM
1 st Measurement	1	327.6	310.4	326.9	276.0
	2	315.0	326.9	325.7	289.4
2 nd Measurement	1	313.3	313.3	309.3	279.0
	2	323.3	321.5	330.7	287.3
3 rd Measurement	1	305.4	319.1	287.7	271.8
	2	337.6	316.7	335.1	299.3
Average	1	315.4	314.3	308.0	275.6
	2	325.3	321.7	330.5	292.0
Overall average in stir zone	1	312.6			-
	2	325.8			-
% Increase w.r.t base metal	1	11.8			
	2	10.4			

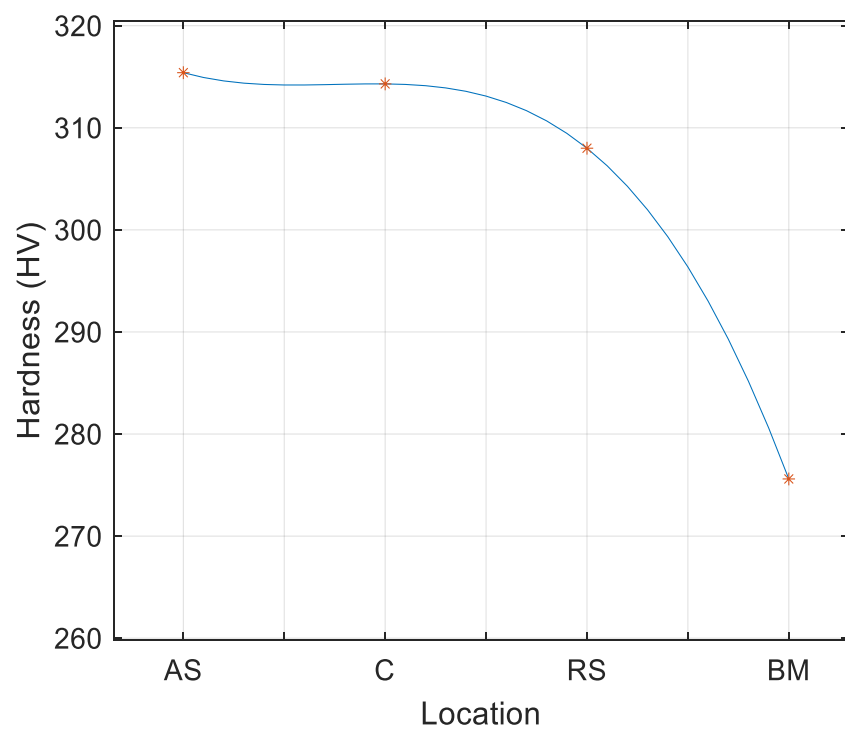


Figure 18 Vickers Hardness profile from BM through stir zone of FSPed UNS S32750 SDSS.

4.3 Effect of FSP on Fracture toughness of UNS S32750 SDSS

İriç and Ayhan [67] have shown that fracture toughness displays non-isotropic behavior because of change in microstructure in different directions. As, FSP modifies the microstructure of the material, it is expected that there will be a difference in the fracture toughness of base metal and FSPed material.

Eight CT samples are tested for fracture toughness measurement for the four conditions shown in Table 9. As mentioned earlier, the basic method described in ASTM E1820-20 was used to calculate the fracture toughness of samples and the results are shown in the Figs. 19 and 23-24 and Tables 11 and 15.

J-integral method for fracture toughness was used because linear elastic fracture mechanics (LEFM) was not applicable in this case. For LEFM, there are certain conditions that need to be satisfied as given in Eq. 7 [28]. As shown in Table 10, these conditions were not satisfied for this material and specimen dimensions.

$$t, a, (b - a), h \geq \frac{4}{\pi} \left(\frac{k}{\sigma_o} \right)^2 \quad (7)$$

Table 9 Conditions of samples for fracture toughness testing.

Condition	Samples	No. of samples tested
1 st	Base metal	2
2 nd	FSPed metal	2
3 rd	Base-corroded (CR) metal	2
4 th	FSP-corroded (CR) metal	2

Table 10 LEFM validity check.

Sample No.	a (mm)	$b - a$ (mm)	t (mm)	h (mm)	$\frac{4}{\pi} \left(\frac{K}{\sigma_o} \right)^2$	LEFM
Base S-1	10.54	29.46	6.5	24	38.25	Not applicable
Base S-2	10.43	29.57	6.5	24	42.14	Not applicable
FSP S-1	10.08	29.92	6.5	24	56.21	Not applicable
FSP S-2	9.1	30.9	6.5	24	55.80	Not applicable
Base-CR S-1	9.37	30.63	6.5	24	32.38	Not applicable
Base-CR S-2	9.58	30.42	6.5	24	28.51	Not applicable
FSP-CR S-1	10.46	29.54	6.5	24	59.10	Not applicable
FSP-CR S-2	10.53	29.47	6.5	24	46.78	Not applicable

Figure 19a, shows typical variation of load with crack opening displacement (COD) for base and FSPed materials. These load-displacement curves are used with Eqs. 1-5 to estimate the fracture toughness of base and FSPed UNS S32750 SDSS and the results are presented in Table 11. It can be noticed from Fig. 19a and the results of Table 11 that the average fracture toughness of FSPed samples has improved over that of base metal by about 12%. This is mainly due to the presence of a larger number of grain boundaries in the processed zone, which increased the resistance to crack growth. It can be seen in Figs. 19b and 19c that FSPed material has smaller plastic zone than the base metal. Plastic zone is the region where plastic deformation occurs ahead of the crack tip [68]. FSP has reduced the plasticity of the material because significant number of grain boundaries hinder the dislocation motion more, as discussed earlier. Tensile testing results have shown that base material had lower yield stress resulting in a larger plastic zone than the processed material.

Table 11 Fracture toughness of base and FSPed UNS S32750 SDSS.

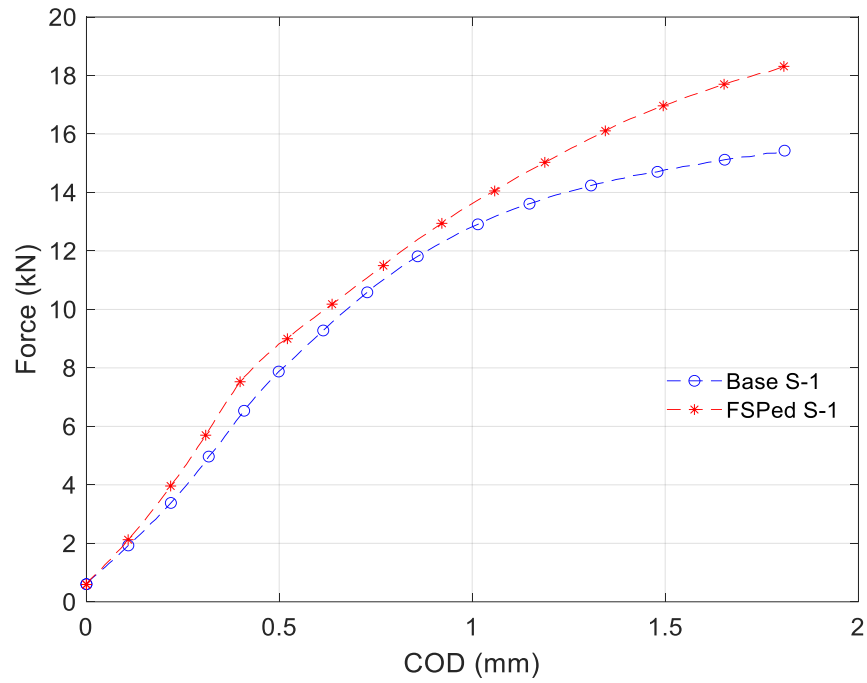
Sample No.	a_o (mm)	Load (N)	J_{el} KJ/m ²	J_{pl} KJ/m ²	J KJ/m ²	J_{Avg} KJ/m ²
Base S-1	20.43	15429.79	65.12	212.89	276.25	272.89
Base S-2	20.54	14573.48	59.11	214.16	269.54	
FSPed S-1	20.08	18312.90	86.86	220.19	306.86	304.66
FSPed S-2	19.10	19646.25	86.23	216.55	302.45	
Improvement in %						11.65

Plastic zone size was also calculated using Eq. 8 [28] which is based on Irwin's model and the result is shown in Table 12. It can be observed that the estimated plastic zone size of base metal is only 10 % larger than that of FSPed metal while Fig. 19 shows a much larger difference in plastic zone sizes. This is due to the fact that the estimated sizes of plastic zones are based on Irwin's model which assumes a circular plastic zone shape ahead of the crack tip while in our case plastic zone had the butterfly shape.

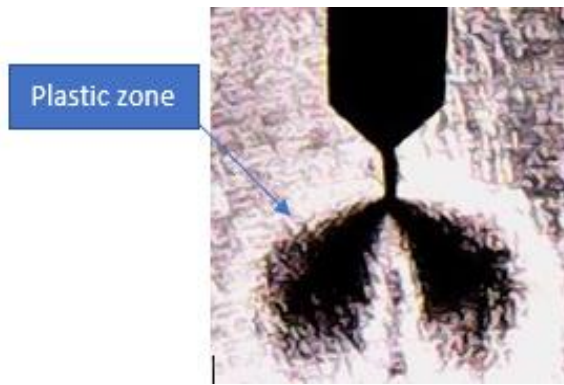
$$Plastic\ zone\ size = \frac{1}{\pi} \left(\frac{k}{\sigma_o} \right)^2 \quad (8)$$

Table 12 Plastic zone size of base and FSPed based on Irwin's model.

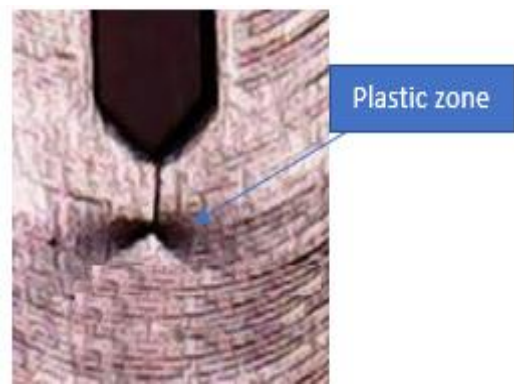
Sample	Load (kN)	F_p	K (Mpa√m)	Plastic zone size (mm)	Plastic zone size Avg. (mm)
Base S-1	14.57	10.07	112.94	9.45	9.94
Base S-2	15.43	9.99	118.54	10.42	
FSP S- 1	18.31	9.72	136.91	10.62	10.94
FSP S- 2	19.65	9.03	136.41	10.55	



(a)



(b)



(c)

Figure 19 Fracture toughness behavior of UNS S32750 SDSS: (a) Force vs COD curve of base and FSPed metal, (b) Plastic zone size of base metal, (c) Plastic zone size of FSPed metal [15].

4.4 Effect of FSP on Corrosion resistance of UNS S32750 SDSS

Corrosion assessment was carried out using PDP (potentio-dynamic polarization) and LPR (linear polarization resistance) tests in a 3.5% NaCl medium to evaluate the impact of FSP on the corrosion rate of UNS S32750 SDSS. Figure 20 displays the obtained PDP plots for base and FSPed samples while the extracted electrochemical parameters are summarized in Table 13. Observation of polarization curves demonstrates that the appearance of the anodic and cathode polarization branches remains identical for base and FSPed samples. However, the curves were notably shifted towards lower current density for FSPed material. From the observation of the obtained values in Table 13, it is indicated that the corrosion potential (E_{corr}) of FSPed sample was moved slightly to the nobler direction and its corrosion current density (i_{corr}) was pointedly reduced to about 80 % compared to base metal. In general, the value of i_{corr} is directly proportional to corrosion rate of the metallic material; thus, the lower i_{corr} value indicates reduced corrosion rate. Hence, the corrosion resistant behavior of processed metal was significantly increased, perhaps due to refinement of the microstructure with FSP.

As a non-destructive evaluation technique, the LPR test was also undertaken to estimate the corrosion rate of base and FSPed metal as shown in Fig. 21. The attained LPR values and calculated corrosion rates are summarized in Table 14. A rise in the value of LPR generally indicates high corrosion resistance of the investigated sample. LPR value of BM sample was found to be 243.70 k ohms, whereas for the FSPed sample it was found to be 785.57 k ohms, which further validated the improved corrosion resistance of FSPed sample in 3.5% NaCl medium. Tables 13 and 14 demonstrate that the i_{corr} was reduced for FSPed sample compared with base metal, and the corrosion rate was reduced by about 80-86 % for FSPed sample. Thus, the corrosion resistance of processed metal was increased due to refinement of the microstructure with FSP technique. EDS analysis of the base metal surface (Fig. 22a) shows a

little or no oxide component, most probably because of very thin oxide layer over the base metal. However, EDS analysis of the surface of FSPed sample (Fig. 22b) shows the existence of large amount of oxide and chromium component most likely because of the formation of thick Cr-enriched oxide layer. This oxide layer might be the reason of increase in corrosion resistance of the processed material. The microstructure of the material has a marked influence on the type and stability of passive film [69]. Ease of formation of passive (oxide) film is enhanced by the increase in grain boundary density [70]. Therefore, the formation of Cr-enriched oxide passive film on the processed material due to a decrease in its grain size [71] improved the corrosion resistance. As FSP decreases the grain size and increases the grain boundary density so stable and resistive passive film forms that increases the corrosion resistance. So, the improvement in corrosion resistance is attributed to the formation of Cr-enriched oxide film on the FSPed sample [6, 24].

Table 13 PDP test results of base and FSPed UNS S32750 SDSS.

Sample	E_{corr} (v)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	Corrosion Rate (mmpy)	Decrease in Corrosion rate w.r.t BM
Base metal	-0.0775	0.4308	4.537×10^{-3}	80.64 %
FSPed metal	-0.0712	0.0834	0.878×10^{-3}	

Table 14 LPR test results of base and FSPed UNS S32750 SDSS.

Sample	E_{corr} (v)	I_{corr} ($\mu\text{A}/\text{cm}^2$)	Corrosion Rate (mmpy)	Decrease in Corrosion rate w.r.t BM
Base metal	-0.0895	0.1852	1.951×10^{-3}	86.31 %
FSPed metal	-0.1284	0.0254	0.267×10^{-3}	

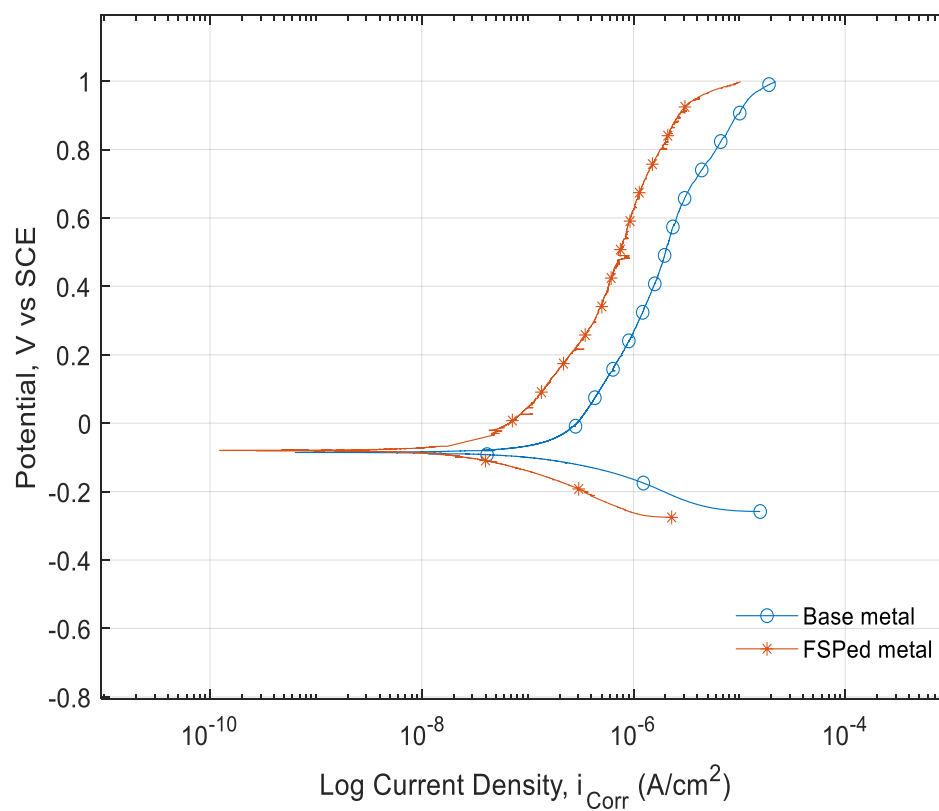


Figure 20 PDP result of base and FSPed UNS S32750 SDSS.

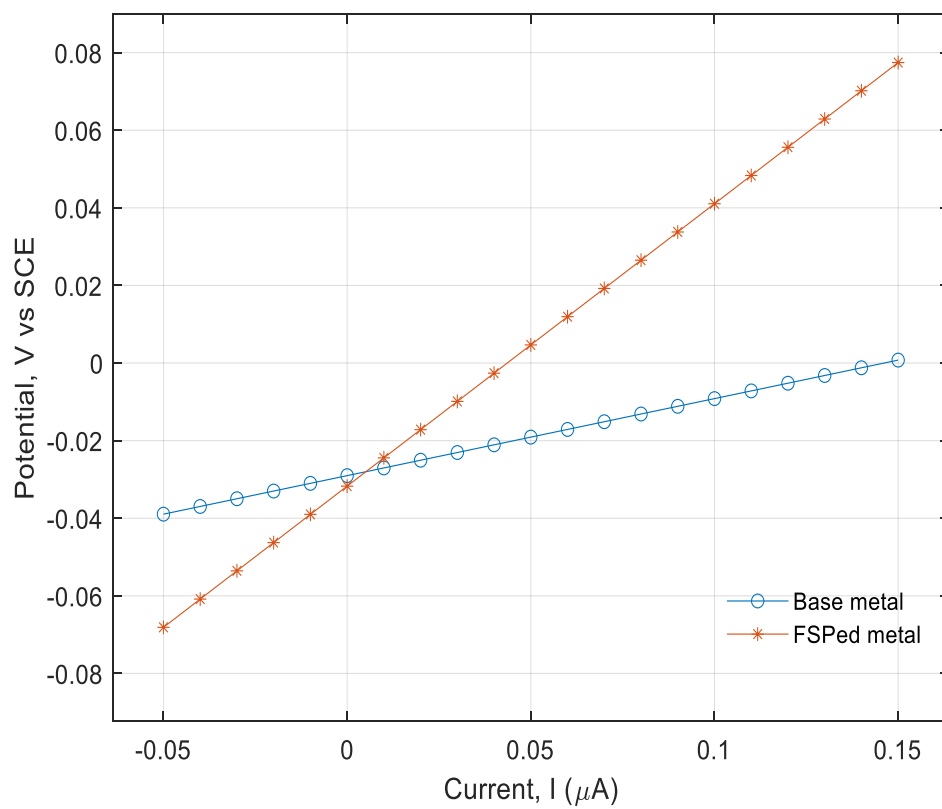
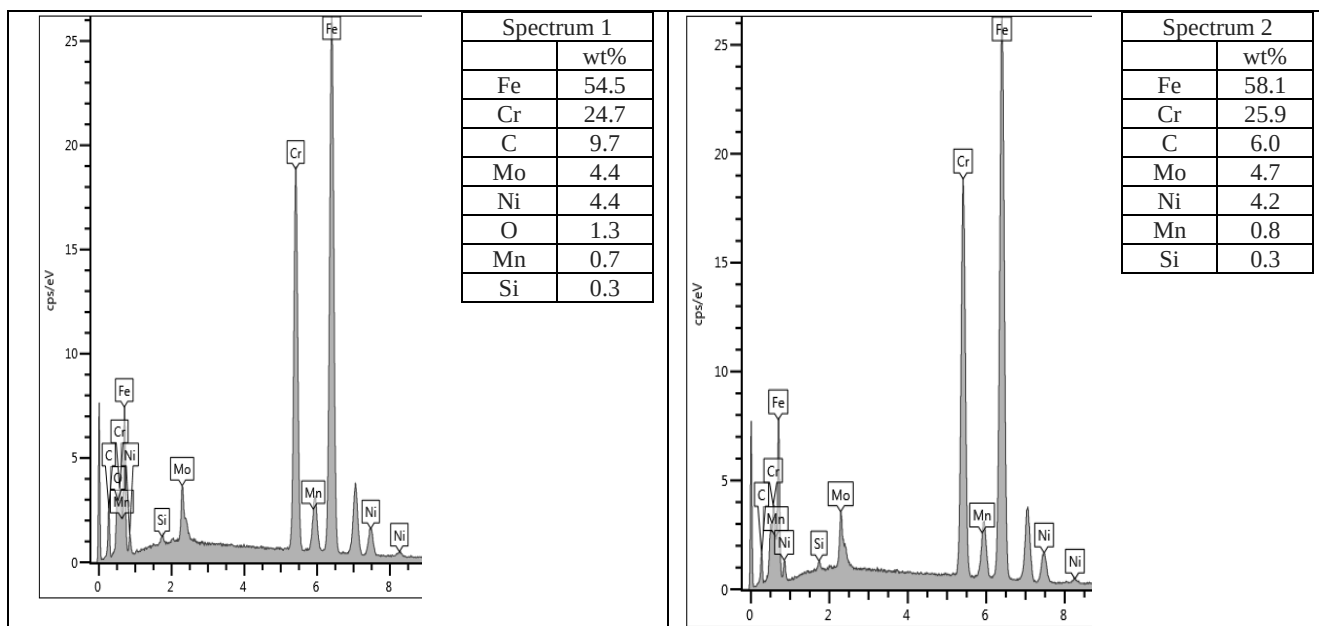
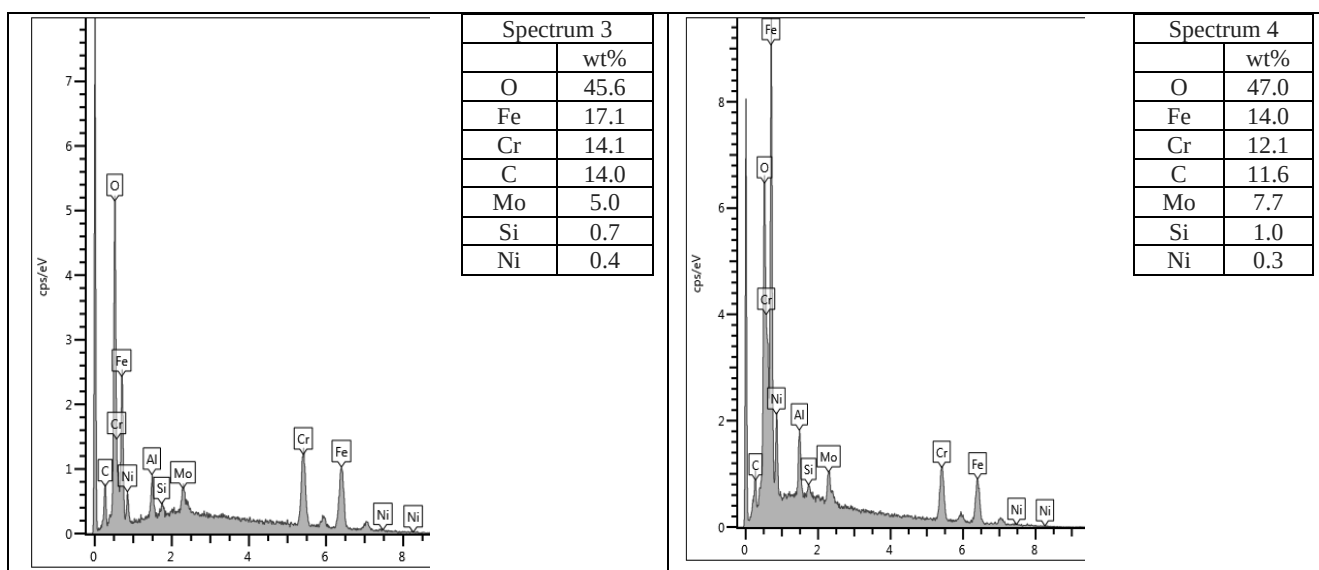


Figure 21 LPR result of base and FSPed UNS S32750 SDSS.



(a)



(b)

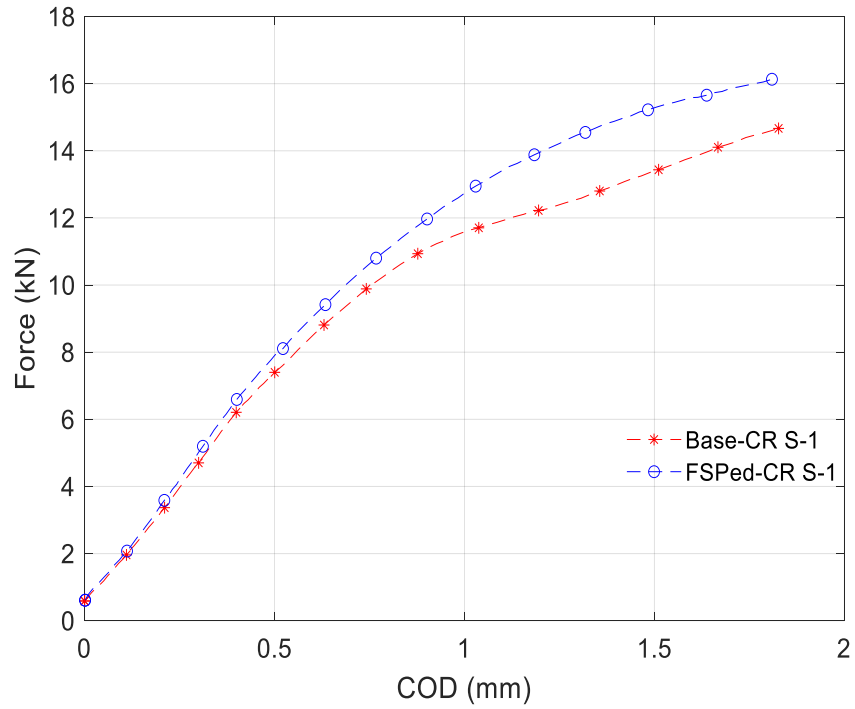
Figure 22 EDS analysis of samples: (a) Base metal, (b) FSPed metal.

4.5 Combined effects of FSP and Corrosion on Fracture toughness of UNS S32750 SDSS

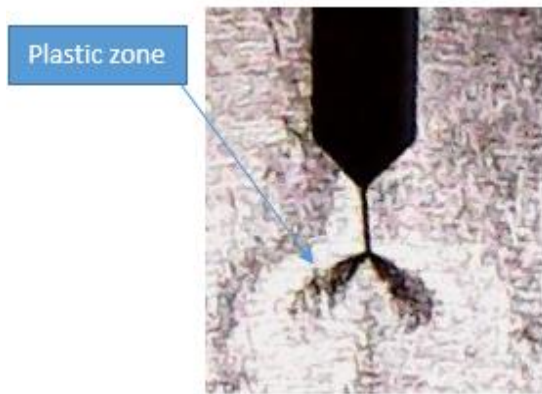
Figure 23 and Table 15 show that the corroded processed sample (FSPed-CR) showed higher fracture toughness than base-CR sample, which means that FSPed-CR sample showed more resistance to crack growth than base-CR sample. The fracture toughness of FSPed-CR samples was improved by about 26 % over that of base-CR sample as shown in Table 15. This improvement in fracture toughness was due to grain refinement and also the plastic zone size was reduced for FSPed-CR sample (Fig. 23c) compared with base metal (Fig. 23b) due to reduction in plasticity by FSP as discussed earlier. The improvement in fracture toughness of FSPed-CR sample was higher than the improvement of FSPed sample and this was most likely because of an increase in corrosion resistance (section 4.4) of the FSPed material. It is noted that, due to seawater corrosion, the fracture toughness of both corroded samples (base-CR and FSPed-CR) was lower than that of unexposed base and FSPed samples, as shown in Fig. 24. However, the influence of seawater corrosion on fracture toughness of base metal was more significant compared with FSPed metal. Seawater corrosion reduced the fracture toughness of base metal by 21.58 % but reduction in fracture toughness for FSPed metal was around 11 %.

Table 15 Fracture toughness of base-CR and FSPed-CR UNS S32750 SDSS.

Sample No.	a_o (mm)	Load (N)	J_{el} KJ/m ²	J_{pl} KJ/m ²	J KJ/m ²	J_{Avg} KJ/m ²
Base-CR S-1	19.37	14666.17	50.02	172.98	222.43	214.00
Base-CR S-2	19.58	13548.74	44.05	164.79	205.56	
FSPed-CR S-1	20.53	16131.00	72.31	272.16	270.88	269.01
FSPed-CR S-2	20.46	18229.70	91.33	267.62	267.13	
Improvement in %						25.7



(a)

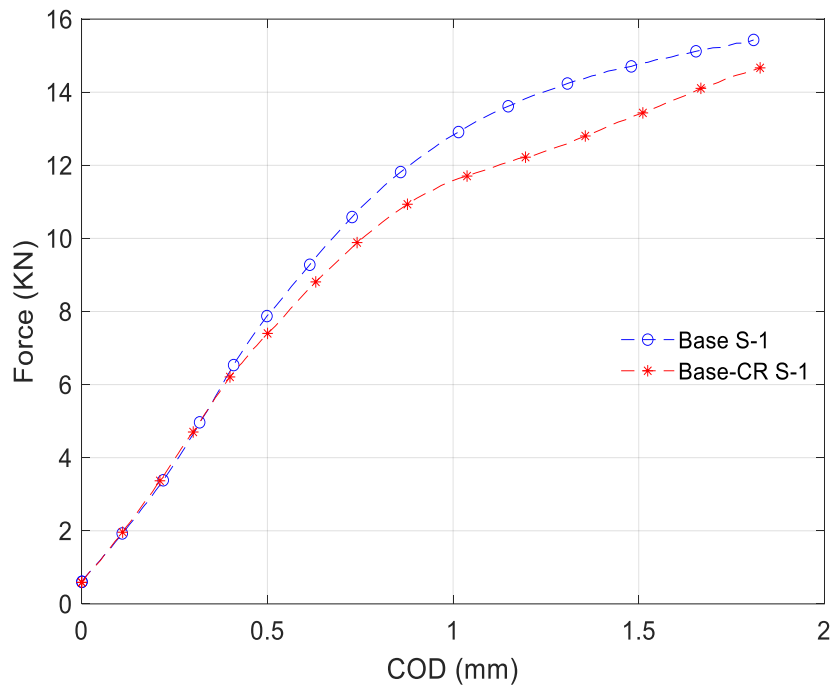


(b)

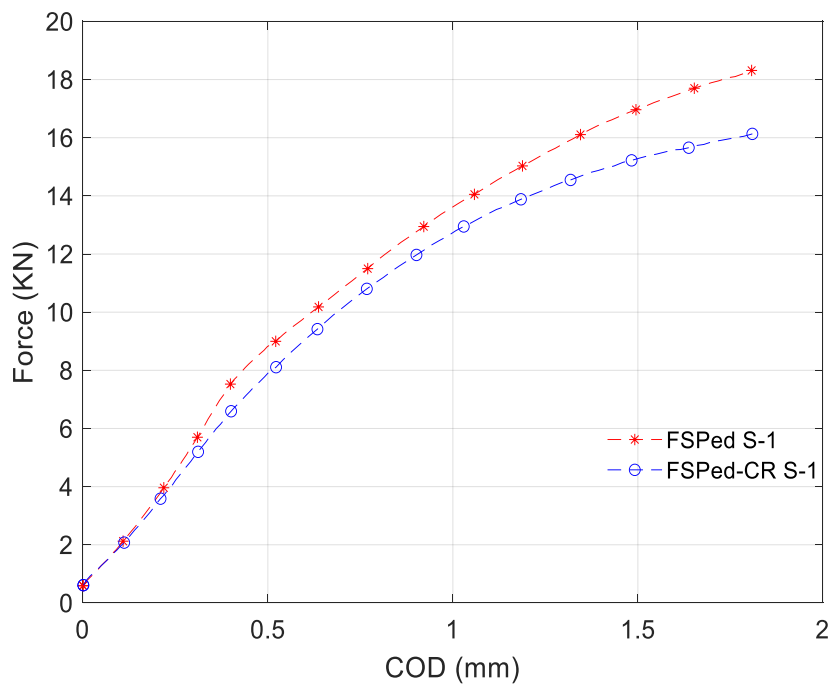


(c)

Figure 23 Fracture toughness behavior of UNS S32750 SDSS: (a) Force vs COD curve of base-corroded and FSPed-corroded metal, (b) Plastic zone size of base-corroded metal, (c) Plastic zone size of FSPed-corroded metal.



(a)



(b)

Figure 24 Fracture toughness behavior of UNS S32750 SDSS: (a) Base and base-corroded metal, (b) FSPed and FSPed-corroded metal.

CHAPTER 5

CONCLUSION AND FUTURE WORK

5.1 Conclusion

Friction stir processing (FSP) refines the grain structure of the material through dynamic recrystallization and this refinement depends on the FSP parameters, material of workpiece and tool, and geometry of tool. Properties of the material such as strength, hardness, fracture toughness and corrosion resistance depend on its microstructure and FSP enhances these properties by grain refinement.

Super duplex stainless steel (SDSS) is mostly used in corrosive environments because of its corrosion resistance. However, corrosive environments affect its behavior so FSP can be used to enhance behaviorist resistance to corrosion. The results of this study show that the mechanical properties like strength, hardness, corrosion resistance, and fracture toughness of UNS S32750 SDSS can be improved by an optimized friction stir process. Optimization of FSP was based on the surface quality of the processed plate. Several operations were performed by changing a number of processing parameters. Optimized parameters were found to be 400 rpm rotational speed, 100 mm/min advancing speed, and 2° tilt angle. Using these parameters, a smooth FSP surface was achieved with refined microstructure which lead to the improvement of most of the material properties.

The main conclusions of investigation of FSP on UNS S32750 SDSS are:

- FSP, with optimized parameters, refined the microstructure of UNS S32750 SDSS from grain sizes of about 160 μm to 2-30 μm .

- Microstructure refinement was asymmetric with respect to processed centerline and grains were finer on the advancing side (AS) in the stir zone because grains on the AS experienced more plastic deformation.
- Grain refinement increased the grain boundaries that hindered the dislocation motion, enhancing the strength and hardness and decreasing the ductility of UNS S32750 SDSS.
- FSP improved the yield strength, ultimate strength, and hardness of UNS S32750 SDSS by about 15 %, 12 %, and 11 %, respectively. However, its ductility was reduced by about 29 %.
- FSP increased the fracture toughness of UNS S32750 SDSS by about 12 %, because grain refinement increased the grain boundaries, enhancing the resistance to crack growth.
- FSP has improved the corrosion resistance of UNS S32750 SDSS by more than 80 % due to the formation of Cr enriched passive layer.
- Corrosion in seawater reduced the fracture toughness of the UNS S32750 SDSS by about 21 % for base metal and 11 % for FSPed metal.

5.2 Future work

The Influence of parameters of FSP on the properties of the material needs to be investigated thoroughly. FSP has different parameters which are rotational speed, advancing speed and tilt angle, as discussed above. These parameters have influence on refining the grain structure of the material. So, by changing these parameters, the microstructure and thus the behavior of the material will be affected. The effect of each parameter on the behavior of the material needs further studies. It is also recommended to investigate how the stir tool geometry and material influence the structure of the stir zone and thus the mechanical and electrochemical properties.

The thickness of the formed passive layer is an important parameter that can be used to study its effect on fracture toughness.

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Vitae

Name : Hafiz Muhammad Abubaker

Nationality : Pakistani

Date of Birth : 3/13/1993

Email : abubaker982@gmail.com

Address : House No. 14, Street No. 1, Tibba Baba Farid, District
Court Lahore, 54000, Pakistan

Academic Background : MS Mechanical Engineering, King Fahd University of
Petroleum & Minerals, Dhahran, 31261, Saudi Arabia.
BS. Mechanical Engineering, University of
Engineering and Technology (UET), Lahore,
Pakistan.

Publications : H. M. Abubaker, N. Merah, F. A. Al-Badour, J.
Albinmoussa, and A. A. Sorour, "Influence of Friction
Stir Processing on Mechanical Behavior of 2507
SDSS," *Metals*, vol. 10, no. 3, p. 369, 2020.
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