Influence of tool rotational speed on the mechanical and microstructural properties of AISI 316 Austenitic stainless steel friction stir welded joints

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Abstract
Fabrication of steel joints using fusion welding technique will result in defects like hot cracking, and heavy residual stresses along with coarse microstructural features in the weldments. To overcome these problems, friction stir welding (FSW) technique was employed to fabricate steel joints. Tungsten based alloy tool is used in this investigation to withstand high dynamic frictional forces at elevated temperature generated during welding. Tool shoulder diameter of 25 mm, pin diameter of 3 mm and pin length 2.9 mm was employed to carry out the friction stir welding on SS316 steel plate of 3 mm. Sound joints produced at a constant welding speed of 40 mm min^{-1}, axial load of 15 kN at different tool rotational speed of 600 rpm and 700 rpm. In accordance with ASTM standards, Tensile, Compression, Micro hardness and impact tests were carried out to assess the mechanical properties of weld joints and the base metal. Mechanical testing results showed that tensile and hardness of the welded material was improved in compared with base metal. However, the impact strength of the welded metal is reduced. Furthermore, the base metal and welded metal were subjected to study microstructural analysis using optical and scanning electron microscopy. EDS and XRD analysis were also carried out on welded zone to understand about the formation of new phases in weld zone and it is surroundings.

1. Introduction

Austenitic stainless steel materials are the most common types of stainless steel. Particularly, grade AISI 316 has gained attentions of several engineering applications ranging from cryogenic temperatures to red-hot temperatures. Owing to its unique properties such as extremely formable, weldable and good mechanical strength, it is used in petroleum refining equipment, heat exchanger parts, exhaust manifold, chemical storage tanks, food preparation equipment and marine applications particularly coastal hand rails. A typical AISI 316 corresponds to the composition of UNS S31600 (ASTM A240/A240M), BS316S31, and X6CrNiMo17-12-2 [1]. Such stainless steel grades are manufactured by careful balancing of alloying elements namely C, Mo, Ni, and Cr. The role of each element is different in diverse applications. For example, in boiler tubes that operate at high temperatures, the percentage of carbon provides high strength, whereas in chloride-rich environments, molybdenum increases the resistance to corrosion, especially to localized attacks like pitting and crevice corrosion. As Nickel is used as a stabilizer of austenitic structure in room temperature, Chromium also forms oxide passive layer with anticorrosive property [2].

In aerospace, automobile, shipbuilding and power plants, metal joining is of paramount importance [3] The conventional welding process, the fusion welding involves many parameters like amperage, voltage, shielding gas, arc gap, wire feed, travel speed and electrode composition. The improper selection of welding parameters
leads to weld defects like poor fusion weldability, shrinkage, solidification cracking, and reduced porosity. In fusion welding, high heat input may be the source for distortion, increased hardness and reduced ductility. It can also induce cold cracking, or Heat Affected Zone (HAZ) cracking. Navid Moslemi et al [4] found that increasing heat input is the main cause for formation of sigma phase in welding, and this is the reason for reduction in notch toughness and impact resistance. Shankar et al [5] studies reveal that hot cracking or solidification cracking is formed in the center of the weld, which is considered as a acute issue in the fusion welding of Austenitic stainless steel.

To produce a good joint efficiency, Friction stir welding (FSW) is an attractive substitute to the fusion welding. It is worthy to mention that FSW is an eco-friendly process that involves only few significant parameters like rotational speed, transverse speed and axial load. Some of the metals / alloys often joined using Al, Cu, Ti alloys and alloy steel. The major problem in welding of harder materials such as steels and Ti alloys is a tool wear. The development of tool material is considered to withstand high stresses, elevated temperature, less wear rate and durable. Rai et al [6] reviewed the commonly used tool material in FSW process; their studies reported Polycrystalline cubic boron nitride (PCBN) is the best opted tool material for FSW of any harder base materials. However, tool cost of PCBN is too high, which acts as a barrier for welding high melting temperature materials. Meshram et al [7] is the pioneer to weld 316 materials by using PCBN material and found the optimizing parameters such as rotational speed of 1100 rpm and less welding speed of 8 mm min⁻¹, this experiment only reveals tensile strength of the weld which is higher than the base material

Mehmet Burak Bilgin et al [8] used the FSW Process to weld 3 mm thickness Ferritic stainless steel. WC-Co hard metal is used as tool material, and it showed notch impact energy stepped down as a result of increasing rotational speed. Further, it is also observed that, notch impact energy was improved by raising the traverse speed. In FSW process, tool design is a critical process. Lakshman Rao et al [9] studied the tool geometry in FSW applications. It was mainly focused study of pin tool geometry and tool design. Accordingly, three different base materials Al, Ti, and steel, weld tool geometry are examined and noticed that geometry plays a vital role in stirring action. Raghunathan et al [10] examined the degradation of tool material in FSW, and found that among the Tungsten alloy tool, 99% of W and 1% of La₂O₃ hold good property at high temperature with low degradation of the tool debris along with the base material. The tensile strength, impact toughness, hardness and bend test property are carried out for the friction stir welded HSLA steel. Hence the selection of tool material and tool geometry is prepared based on this work.

Solid state welding coalescence is produced below the melting point of base metal and without addition of filler metal. This is a group of welding process, works on same principle but the method of applying pressure and heat is different in all these processes. Based on the application of energy, this welding process can be classified into forge, friction, explosive, ultrasonic, diffusion and friction stir welding. Imran et al [11] studied the feasibility of joining AISI 316 L stainless steel by linear friction welding which resulted variation in welding parameters and has a major impact on the microstructure of welds. This was evident by the variation in fraction of delta ferrite at TMA zone of the welds, but this process mainly suitable for cylindrical jobs. Yeh et al [12] demonstrates SAE 316 stainless steel joints produced by diffusion bonding process. It shows a result of better bond strength obtained inserting super plastic interlayer. The constraint of this process is more time consuming to achieve better bonding joints.

The key benefits of FSW process the bond between the two pieces which made only by the base material and giving it similar tensile, compressive strength, fatigue characteristics of parent metal. FSW does not emit smoke, fumes, or gases. Whereas, it is a combination of frictional heating and mechanical loading, and transforms the solid state metal into a plastic state. Materials coalescence is achieved by stirring of tool in between the base metal under the action of load. In this process the good quality of weld is achieved by controlling parameters.

Generally there are two modes of metal flow phenomena in FSW. The Shoulder contributes first mode of metal transfer, when a small amount of metal is moved from front side to rear side that offers compactness to the weld. When shoulder pin influences second mode of metal transfer, the remaining metal transfers from front side to rear side. In this process, the base metals are securely clamped before welding. Abbasi et al [13] studied that the primary FSW process parameters are tool rotational speed, axial force and travel speed. Rotational speed influence frictional heat stirring, oxide layer breaking and mixing of metal. The axial force has effects on the generation of heat and maintaining contact condition, travel speed and control the appearance of weld. From the literature survey it is learnt that though many works were reported on AISI 316 Stainless steel related to fusion welding, but only limited works were reported in solid state welding. Thus, the present study is pertaining to investigate the effect of tool rotation speed on mechanical and microstructural properties of AISI 316 ASS friction stir welded joint.
2. Experimental work

Frictions stir welding process setup and tool material profiles are shown in figure 1. AISI 316 Stainless steel used as a base material for current study and its thickness is 3 mm cold rolled and annealed grade sheet.

Spark emission spectrometer (spectro MAX) has been employed to know about the chemical composition of base material. The spectrum analysis is done at three different locations to estimate the alloying composition and values are listed in table 1 and the mechanical properties of the base metal presented in table 2.

The cold worked annealed base metal has coarse equiaxed grain structure with annealed twins, evident of optical micrographs and SEM image of the base metal shown in figure 2. The average grain size of base metal is found to be $25 \pm 4 \mu m$.

A semi-automatic friction stir welding machine with the capacity of spindle speed 2000 rpm, Spindle tilt angle $+/-5$ deg, Z axial force 15kN and Rexroth controller with modified tool holder machine were used. The FSW tool material was made by 99% tungsten and 1% of $La_2O_3$, used for the experiment, that provide better wear resistant during the welding. In this process conical cylindrical tool with shoulder diameter of 25 mm, probe diameter of 12 mm, and pin length of 2.9 mm are used as tool geometry. To increase the bending stress of the tool material, conical sections are used. Thus the FSW joints are trialed at different tool rotational speeds of 400, 500, 600, 700 and 800 rpm at constant welding speed of 40 mm min$^{-1}$ and axial load of 15 KN. Welding conditions and parameters are shown in table 3.

Visual inspection is prerequisite for the application of other test method. Friction stir welded plates are inspected according to the standard EN 970 [14] Visual testing involves illumination of the welding surface of
the specimen using light. Direct visual examination is conducted for face and root sides of the weld surfaces. X-ray radiographic testing is conducted for welded specimen to identify the internal defect of the weld metal according to the ASTM E1032-19 [15] specifications. Hence the Radiographic testing machine model name (HIRAT) Capacity 250kv/500 A, Source to object distance 1000 mm, Exposure Time 30 s, Density 2–3 and Sensitivity 2X.

In metallographic examination, the welded specimen is cut to appropriate size and the specimen is polished with different grade sizes of emery paper for mirror finish according to the ASTM E3–11 specifications. The specimen is etched with 10% oxalic acid solution for 30 s and dried in air, now the polished specimen is examined under optical microscope and the images are captured at various magnifications 100X, 200X and 500X in base metal, weld metal and TMAZ regions. The microstructure of the weld metal is studied using Computerized Optical microscope (VERTIMET-CP) and high resolution Field emission scanning electron microscope (FESEM) model Number RA-ZE1-001 make by CARL ZEISS microscopy is attached with Energy dispersive x-ray spectrometer (EDS) model number QUANTAX 200 made by BRUKER, Germany, that studied fractography analysis. X-ray diffraction (XRD) analysis was performed to examine the various phases present in the material.

Tensile specimen were prepared in accordance to ASTM E8M-04 and experimental investigation was carried in a computer controlled universal tensile testing machine (M-30 model) to determine Tensile strength, Yield strength, and percentage of elongation are evaluated for both base material and welded samples in transverse directions. Charpy impact specimens were prepared as per ASTM E23-06 specifications. The specimens were tested by impact testing machine (AIT-300-EN model) 300 J capacity machine, where testing is performed at room temperature for the base material and weld center to check the impact toughness. Three points bend test the specimens were prepared according to ASTM E190-03 specification and tested in compression testing machine model no (TUE-CN-400) load capacity of 400 kN. The testing was done for both base material and welded specimen to check any crack present in it or not at 180°. The micro hardness evaluations were performed in a Vickers micro hardness testing machine, Model no: Qness Q10A + at base metal, weld metal and TMAZ at an applied load of 500 gf and dwell time of 10 s.

Table 3. FSW Process Parameters & Welding Condition.

| Process parameters | Values |
|--------------------|--------|
| Rotational speed of Tool used (rpm) | 400, 500, 600, 700, 800 |
| Welding speed used (mm/min) | 40 |
| Axial force (kN) | 15 |
| Diameter thickness (D/T) ratio of tool | 5 |
| Diameter of the Tool shoulder (mm) | 25 |
| Diameter of the Pin (mm) | 3 |
| Length of Pin (mm) | 2.9 |
| Tool inclined angle (°) | 0 |

Figure 2. Microstructure of Base metal (a) Typical OM Micrograph (b) Typical SEM micrograph.
3. Results and discussions

3.1. Macrostructure

Friction stir welded joints are done at different tool rotational speed of 400, 500, 600, 700 and 800 rpm.

The joints fabricated using 400, 500 and 800 rpm were end-up with volumetric defects due to lack of optimistic heat input at stir zone. Volumetric defects along the weld line must be avoided for better mechanical and metallurgical behavior of the joints. Whereas 600 and 700 rpm yielded defect free joints. Figure 3 shows transverse macrographs of Friction stir welded joints produced at different tool rotational speeds. Commonly FSW contains three different zones Base metal (BM) Weld stir zone (SZ), Thermo mechanical affected zone (TMAZ). It is interestingly noted that 'U' shaped shear bands are noticed in stir zones when the metal is joined at tool rotation speed of 600 and 700 rpm. Hence, those two joints were taken for further testing and other characterization.

In the figure 3, it can be observed that the tool rotational speed of 400 rpm tunnel defect was observed in the stir zone surface, because of the insufficient heat input and flow of metal. The weld produced at 500 rpm cavity formation is perceived due to low frictional temperature in the stir zone. Then, defect free weld is obtained at a tool rotation speed of 600 and 700 rpm and the 'U' shaped shear bands are observed in stir zone and near to TMAZ zone. Normally, the material flow occurs from that advancing side to the retreating side owing to subsequent heat generation in layer by layer, because of which the optimum plastic flow of material is obtained. Excessive flash formation is witnessed due to high tool shoulder pressure and extreme frictional heat generation that thermally softens the metal at a tool rotational speed of 800 rpm. From this observation, it can be understood that the shear band pattern in the weld zone was caused by the heat formation. Kumar et al 2016 [16] experimented FSW of 316 L ASS joints, then analyze mechanical and micro structural properties, which resulted in similar behavior of shear bands at tool rotation speed of 600 rpm.

3.2. Visual inspection

Visual inspection test reveals much information about the surface quality of welding. During FSW process, common issues like kissing bonds, cracks, voids, lack of bonding, cavity and sub-surface tunnel defects are observed. Figure 4 clearly indicates that the specimen welded at 600 rpm and 700 rpm were found to be free from volumetric defects. Further, it can be understood from the same figure that specimen welded at 600 rpm exhibit uniform onion rings in the face side when compared to the specimen welded at 700 rpm. Also, it can be noted that the beads in the root side of the weldments were occurred in 600 rpm.

Testing the weld quality by visual inspection is an important step followed in the welding process and it also ensures the weld zone free from macro defects. The surface appearance of weld metal is regular circular ripples and it is produced by the final sweep movement of shoulder towards the transverse direction. The rotation speed and transverse speed of the tool decides pitch between the ripples whereas, the selection of welding parameters determines smooth face of the weld metal. Hence uniform circular ripples throughout the joint ensure uniform metal flow and perfect dynamic recrystallization during welding.
3.3. X-ray radiography

Single wall technique is used for FSW butt joints in which the radiation passes through only one direction. X-ray film radiography is done according to the ASTM E1032-95 standards. After welding, the weld metal at 600 rpm and 700 rpm joints is subjected to x-ray radiography. In figure 5, it is observed that there is no internal defect at welding speed of 600 rpm and 700 rpm. Due to excess heat input and compressive load effect, the shoulder impinged into weld metal surface at 700 rpm. It indicates that the defect free weld joint is obtained due to sufficient heat energy produced at these parameters.

X-ray radiography is widely used as a non-destructive testing to ensure weldments quality [15]. Conventional X-ray single wall technique welding plate is placed in the middle of radiation source and photo film. Silver boride photographic film is used for recoding the internal defects of weldments. From these films, it is observed that there is no internal micro defects in weld metal produced at 600 rpm and 700 rpm. It specifies the quality and integrity of the weld joints.

3.4. Mechanical properties of material

3.4.1. Tensile and impact strength

Tensile fractured specimens and impact fractured specimens are shown in the Figures 6(a) and 6(b) respectively. Experimental results showed that 316 ASS joined using FSW process exhibit 12.68% improvement in yield strength whereas ultimate tensile strength remains unchanged compare to base metal when the tool rotates at a speed of 600 rpm. As tool rotational speed increases to 700 rpm both yield strength and tensile strength are
slumped in comparison to the joint made up of 600 rpm tool, on the other hand, ultimate tensile strength and yield strength remains the same as similar to base metal.

The impact strength test performed in Charpy impact testing machine, result showed that the base metal exhibit impact toughness of 50 J. As metal ASS welded under the tool rotational speed of 600 rpm, impact strength is reduced to 46 J. Further when tool rotational speed increases to 700 rpm, the impact strength decrease to 36 J. Hence, that is almost 28% decrease is found in impact toughness when compared to base metal. It is interesting to note that 8% reduction in impact strength was observed for 600 rpm in compare to 700 rpm. Mechanical properties of the base metal and FSW joints obtained are listed in Table 4.

From the figure 7(a) it can be observed that the yield strength of the FSW joint is quite greater than the base metal due to the effect of grain refinement and strain hardening in the weld zone. The ultimate tensile strength of the weld metal is lesser than base metal because of the severe compressive stress induced on the specimen, which blocks the grain boundary dislocation and reduce the percentage of elongation. It is seen in the figure 7(b) where the impact toughness of the welded specimen is reduced in comparison with base metal. According to A.K.
3.4.2. Bend test

Three point bend test is carried out for welded metal at face side and root side and it is observed that, there is no presence of surface cracks in welded metal as shown in figure 8(b). While comparing the compression strength of base metal and welded metal, the base metal was found to be 34 N mm\(^{-2}\), however the weld specimen exhibited 49 N mm\(^{-2}\) for the welding speed of 600 rpm. From the figure 8(b), it is clearly seen that the pin influenced region during friction stir welded with presence of cracks. Figures 8(a) and (b) depicts the bend Test Setup and Specimen photographs.

The faces bend and root bend three point test is carried out along the welded joint which is traverse to the longitudinal axis of the specimen. From the figure 9, it is noticed that the joints which are produced at 600 rpm and 700 rpm attain a compressive stress of 49 N mm\(^{-2}\) and 41 N mm\(^{-2}\) respectively, which is higher when compared to the base metal 34 N mm\(^{-2}\). It implies that all the joints which are produced by FSW are free from defects and have successfully passed the root and face bend test without any breakage.

3.4.3. Micro hardness test

Micro hardness examination has been carried out at various places over the transverse weld at different tool rotational speed. The Vickers micro hardness test shows the hardness of the base metal is 184 ± 5HV. The stir zone micro hardness was observed 231 ± 5HV and 217 ± 5HV at tool rotation speed of 600 rpm and 700 rpm. The results indicate stir zone hardness value is higher than the base metal. Figure 10, illustrates the transverse Micro hardness survey of FSW joints at 600 rpm and 700 rpm.

Hardness is measured along the transverse direction in the weld. In the stir zone, increase in hardness of about 25.54%, 17.94% is attained at 600 rpm and 700 rpm respectively, when compared to the base metal. It is mainly due to grain refinement and the effect of strain hardening in the weld zone. In TMAZ Zone, slight refinement of grains and shoulder impart plastic deformation which tends to improve hardness when compared to the base metal. The hardness value of the stir zone and TMAZ varies from 236 HV—212 HV.
the weld metal is increased because of equiaxed grains in the weld SZ. In TMAZ zone, there is an increase of hardness due to partial grain refinement and severe plastic deformation induced by tool shoulder at a rotational speed of 600 rpm. As per the study of Kim et al 2016 [18], FSW welds substantially have better hardness values compared to the base metal. This phenomenon is due to dynamic recrystallization resulted homogeneous grain refinement.

3.5. Microstructure analysis
3.5.1. Optical microscopy
The base metal is obtained as rolled 316 ASS sheet consists of equiaxed austenite grains containing several annealing twins. From the microstructure figures 11(a)–(c), it is observed that no twin boundaries are present in the nugget zone. During FSW Process, grains in the nugget zone are completely distorted by the stirring action of tool. Fine equiaxed grains are observed in stir zone. This can be attributed by the simultaneous effect of thermal and mechanical loads experienced by this weld metal. Microstructure representing the shoulder influenced region, shoulder-pin interface region and pin influenced regions were shown in figures 11(a) to (c) respectively. The formation of relatively low angle grain boundaries and similar orientation in the interface of the shoulder influenced region, shoulder-pin interface region and pin influenced region in stir zone have strong influence on mechanical properties of the joints. Also, these microstructures are evident for the significance of optimistic level of the process parameters as well as effect of tool rotational speed on formation of stir zone. Figure 11(d) and (e) represent the TMAZ retreating Side and advancing side of shoulder influencing region. The process of TMAZ distorted structure with grains oriented to the transverse direction is observed and found that this region grain size is slightly larger than stir zone. This is because the peak temperatures affect the dynamic recovery of the grains. Figures 11(f) and (g) indicates the river flow pattern in base metal and TMAZ interface zone.

3.5.2. Scanning electron microscopy
Figure 12 depicts the typical SEM fractographs of uniaxial tensile images of base metal and optimized weld metal. Figures 12(a) and (b) show the size and dimples distribution on the surface of broken image specimen, which indicate the characteristics of ductile fracture. The displayed tensile fractography consist of fine dimples in base
metal and weld metal as well. When compared to base metal, few non-uniform cleavage facets are found in weld metal which leads to reduce the ductility of the weld joint. This fractography analysis shows that same level of strength yielded by the weld joint whereas loss in ductility. Figures 12(c) and (d) illustrates typical SEM fractographs of impact images of base metal and optimized weld metal. Figure 12(c) shows the distribution of elongated dimples on the surface of broken image specimen and figure 12(d) shows the wavy like surface formation on the surface of broken image specimen, which indicates characteristics of shear failure. By comparing these two fractographs, it is noted that the weld metals having elongated and finer dimple structure is a clear indication of high energy mode of fracture, which is provides enhancement of toughness to the weld metal.

3.5.3. EDS analysis
Tensile and impact test EDS results of base metal and weld metal has been shown in the figures 13(a)–(d). There is no evidence of tungsten debris present in the weld metal which may have more possibilities in reduction of toughness. Also, this provides additional information such that no tool wear occurred during FSW process. It indicates adequate heat generation and optimal material strain developed by the rotation speed of 600 rpm in the weld zone.

3.6. XRD analysis
An XRD pattern of 316 stainless steel welded sample is shown in figures 14(a) and (b). The results showed a strong \(\{111\}\) \(\{100\}\) \(\{110\}\) crystallographic plane texture at the weld center. This is a typical shear texture observed in FCC materials. There are numerous sharp peaks corresponds to XRD pattern of austenite, also other week peaks that indicates secondary phases is due to the condition of annealing process.
Gerbson de Queiroz Caetano et al 2018 [19] studied that the process parameters strongly influence the heat generation, which later affect the heating and cooling cycle and subsequently the microstructure. Optimum tool rotation speed experienced higher material strain and reduced grain size in stir zone. Ramachandran et al 2016 [20] concluded that the tool rotation speed directly influence the mechanical properties, besides causing heat generation. McAuliffe et al. (2014) [21] investigated shear band formation and dynamic fracture in metals in different stages. Firstly, plastic strain occurs in a homogeneous phase following thermal softening which leads to strain hardening and shear collapses, a major phenomenon caused by severe softening of metal. Zumelzu et al 1999 [22] reported that fusion welding produces variations in composition, which influence the metallurgical and mechanical properties of the joint, which constrains the designer to control weld metal composition in order to obtain the desired properties. Friction stir welding process offers solution for many fusions welding process.

4. Conclusions

From the experimental study the following conclusion are drawn

- AISI 316 Austenitic stainless steel welded joints are successfully accomplished by friction stir welding process at an axial load of 15KN, constant welding speed of 40 mm min⁻¹ and tool rotation speed of 600 rpm and 700 rpm.
- Qualitative inspection such as visual and x-ray radiography ensure the weld quality and sound defect free FSW joints at (600 rpm and 700 rpm).
- Studies showed that Mechanical properties were directly influenced by process parameters of welding, maximum tensile strength of 602 MPa and compressive strength 51 N mm⁻² obtained at welding speed of 600 rpm.
- Micro hardness obtained for base metal is 184 ± 5HV. The stir zone micro hardness was observed maximum 231 ± 5HV at tool rotation speed of 600 rpm. This is due to the microstructural refinement weld metal attained by dynamic recrystallization, caused by stirring action of the tool at elevated temperature.
The impact toughness of base material is found to be 50 J, whereas for tool rotation speed of 600 rpm, welded specimen exhibited 46 J due to the reduction in impact toughness attributed to entrapment of tool debris in the stir zone.

XRD Results show a texture of 〈111〉〈100〉〈110〉 at the centre of the weld, typically it was nothing but a shear texture of face centre cubic materials and also ensure no secondary phase formation in weld zone.

SEM result shows the size and dimples distribution of tensile welded specimen, which indicates characteristics of ductile fracture.

Figure 13. SEM EDS tensile test- (a) base metal (b) weld metal impact test - (c) base metal (d) weld metal.

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Figure 14. X-ray diffraction pattern of SS 316 (a) Base metal (b) Welded sample.

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