Paper No. PPU15



# Surface Modification to Enhance the Properties of SA 210 Grade A1 boiler Steel by Friction Stir Processing

#### **Supreet Singh**

Assistant Professor in Department of Automobile Engineering Chandigarh University supreetsingh2333@gmail.com

#### **Manpreet Kaur**

Associate Professor in Department of Mechanical Engineering, Baba Banda Singh Bahadur Engineering College, Fatehgarh Sahib, India

#### ABSTRACT

Friction stir processing (FSP) is used for localized modification and control of microstructure in nearsurface layers of processed metallic components for specific property enhancement. In the current investigation, FSP was developed on a boiler tube material namely SA 210 Grade A1 which is commonly used in high-temperature steam generating plants. The FSP was carried out at the rotation speed of 900 rpm with feed rate of 70 mm/min and threefold pass of 100 % overlap. The microstructure, mechanical properties were studied in detail. It was observed that after FSP the microstructure and the mechanical properties of the steel like microhardness, tensile strength, and yield strength improved. Thereafter, high-temperature corrosion behavior of the unprocessed and FSPed materials was investigated at 900°C for 50 cycles in Na<sub>2</sub>SO<sub>4</sub>-82%Fe<sub>2</sub>(SO4)<sub>3</sub> molten salt environments. Weight-change measurements after each cycle were made to establish the kinetics of corrosion. The FSPed specimen showed higher corrosion resistance than the unprocessed steel.

Keywords: Friction Stir Processing; medium carbon steel; microhardness; grain refinement; high temperature corrosion behavior.

NIGIS \* CORCON 2017 \* 17-20 September \* Mumbai, India Copyright 2017 by NIGIS. The material presented and the views expressed in this paper are solely those of the author(s) and do not necessarily by NIGIS.

#### INTRODUCTION

In high-temperature steam generating plants, the structural materials suffer from various types of surface degradations such as corrosion and wear. Ferritic steels such as medium carbon steels used in boilers and reheater tubes are subjected to high-temperature environments. These metallic materials possess special properties like easy availability, low cost and ease of fabrication, but they are not able to withstand high temperatures for long periods due to their poor corrosion resistance. The hot corrosion of a boiler steel/alloy usually occurs in the environments where molten salts such as sulfates (Na<sub>2</sub>SO<sub>4</sub>), chlorides (NaCl), or oxides (V<sub>2</sub>O<sub>5</sub>) are deposited onto the surfaces. The Na<sub>2</sub>SO<sub>4</sub>-82pct Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> environment is found usually in coal-fired boilers where the coal ash corrosion is induced by the deposition of complex iron-alkali sulfates, (Na,K)<sub>3</sub>Fe(SO<sub>4</sub>)<sub>3</sub>. [1] Therefore, the development of high-temperature oxidation protection systems in industrial boilers is an important topic from both engineering and an economic perspective.

In the current investigation, a boiler tube steel namely SA210 Grade A1 steel was selected. The steel is a well known candidate for fabricating boiler tubes in steam generating plants. However at elevated temperatures, the steel lack resistance to oxidizing/corroding environments. If there could be some means by which the microstructure of the steel is refined and the local mechanical properties are improved, the corrosion resistance can be enhanced. Nowadays a new surface modification technique Friction stir processing (FSP) is used for localized modification of mechanical properties and control of microstructure in near-surface layers of processed metallic components for specific property enhancements. The FSP technique is cost-effective, environment and user friendly technique [2].The present study is undertaken to study the effect of FSP on corrosion behavior of Grade A1 steel.

Materials processed via friction stir processing (FSP) have shown improvement in both hardness and toughness due to microstructural refinement [3-7].However, the performance of FSPed materials has not been evaluated yet under high-temperature corrosion environment. All the corrosion studies reported till date are room temperature corrosion studies on FSPed steels and various aluminium and magnesium alloys. To explore the benefits of FSP in the field of corrosion a novel attempt is made by the authors in the present study by investigating the high-temperature corrosion behavior of the unprocessed and FSPed material at 900°C for 50 cycles in Na<sub>2</sub>SO<sub>4</sub>-82%Fe<sub>2</sub>(SO4)<sub>3</sub> molten salt environments. The microstructure, mechanical properties, and corrosion resistance of the unprocessed and FSPed materials have been evaluated

#### EXPERIMENTAL PROCEDURE

#### SELECTION OF MATERIAL AND FRICTION STIR PROCESSING

The material used in this work is a medium carbon steel namely ASTM-SA210 grade A1 (GrA1) with chemical composition in wt. % as 0.27 C, 0.93 Mn, 0.035 P, 0.1 Si, 0.035 S and remaining 98.63 Fe. The steel is being used as a boiler tube material in various coal-fired thermal power plants in northern parts of India. It was procured from Guru Nanak Dev Thermal Power Plant located at Bathinda in Punjab. The Gr A1 steel plate (120 mm x100 mm x 4 mm in size) were friction stir processed, using high performance vertical CNC milling machine.

## NIGIS \* CORCON 2017 \* 17-20 September \* Mumbai, India

A specially designed fixture was fabricated from stainless steel grade EN32 to clamp the Gr A1 steel samples on machine for performing FSP. FSP was carried out in longitudinal direction using a tungsten carbide (WC) tool without pin. The various friction stir process parameters are shown in table 1. After process, the plate was cooled with dry ice.

Spindle speed	Transverse speed	Plunge Depth	Pass of tool	Diameter of		
(rpm)	(mm/min)	(mm)	on substrate	tool (mm)		
900	70	1	3	10		

#### Table 1: Friction stir process parameters

#### CHARACTERIZATION OF BASE STEEL AND FSPED STEEL

The characterization was done at Indian Institute of Technology Ropar, Roopnagar, India. Surface characterization of the FSPed specimens was studied on scanning electron microscope (SEM)(JEOL make, Model: JSM 6610LV), equipped with energy dispersive spectroscopy (EDS). The SEM micrographs along with the EDS spectrum were taken with electron beam energy of 15 keV. Microhardness measurements were performed on a Microhardness Tester (Make: Wilson; Model: 401/402 MVD). It was done at 300 g load for a dwell period of 10 s. Tensile Testing was performed at the room temperature on a Universal testing machine (Make: Tenius-Olsen; Model: H50KS) equipped with a computerized data acquisition system. Mini-tensile specimens with 32 mm gauge length, 10 mm width and 3.8 mm thickness were prepared from base and FSPed SA 210 Grade A1 specimen. The stress Vs strain curves for the base and FSPed SA 210 Grade A1 were compared.

#### HIGH TEMPERATURE CORROSION TESTS

For high-temperature corrosion tests, the unprocessed and FSPed specimens each measuring approximately 13 mm x 10 mm x 5 mm were cut from the steels. High-temperature corrosion studies were performed in molten salt  $[Na_2SO_4-82Fe_2(SO_4)_3]$  environment for 50 cycles. Each cycle consisted of 1 hour heating at 900°C in a silicon carbide tube furnace followed by 20 minutes of cooling at room temperature. The specimens were kept in alumina boats and then boats containing the specimens were inserted in the furnace. The aim of cyclic hot corrosion is to create accelerated conditions for testing. The specimens were polished down to 1µm alumina powder on a wheel cloth polishing machine before being subjected to cyclic corrosion. A coating of uniform thickness with 3 to 5 mg/cm<sup>2</sup> of salt was applied with a camel hair brush on the preheated samples (250°C). The weight change measurements were taken at the end of each cycle with the help of an electronic balance with a sensitivity of 1 mg. The spalled scale, if any, was also included at the time of measurements to determine the total rate of corrosion, wherever possible. Efforts were made to formulate the kinetics of the corrosion. The exposed samples were analyzed using SEM/EDS for surface analysis of their scales.

#### **RESULTS AND DISCUSSION**

#### MICROSTRUCTURE

The SEM image of the FSPed specimen is shown in Figure 1. FSP resulted in a considerable refinement in the microstructure especially inside the nugget zone (NZ). The grain structure is completely refined. The coarse ferrite and pearlite grains were fragmented and refined by the effect of severe plastic deformation, dynamic recrystallization and temperature during FSP. Further, the microstructure reveals the reduction in grain size after the friction stir processing of boiler steel. Initially as observed under the optical microscope by the authors in another study, the microstructure of base steel sheet consisted of coarse ferrite and pearlite grains with the average grain size of 14 µm [8]. Figure 1 shows the morphology of the FSPed sample with average grain size of 2-3 µm. This means that the grain size decreases at least 7 times with the effect of FSP inside the NZ. Haijan et al [9] reported in their work that the size of refined grains in the stir zone of friction stir processed steels is inversely proportional to the heat input of the process [10-11]. As the tool rotates at high revolutions that are 900 rpm as in the current investigation, the higher heat input resulted in the decreased grain size. Aldajah et al [12] mentioned in their research work that in the upper stir zone region, the material had been plastically deformed by the friction stir processing tool, and the heat from the process exceeded the austentizing temperature of the base steel resulting in the formation of martensitic phase from the rapid cooling after the FSP tool passes. The results were reported similar to our investigation.



#### Figure 1: SEM image of the nugget region in the FSPed SA 210 Grade A1 Steel sample

#### **MICROHARDNESS**

Results of microhardness analysis are given in Table 2. Microhardness of the base steel was evaluated as 180 Hv and that of FSPed specimen as 555Hv. The results indicate 3 times improvement in the microhardness of the base steel after FSP. Literature studies revealed that the Phenomena responsible for the significant enhancement in the hardness of the steel after FSP, appear to be grain size strengthening in accordance with the well known Hall-Petch equation given

### NIGIS \* CORCON 2017 \* 17-20 September \* Mumbai, India

#### as $\sigma_0 = \sigma_i + KD^{-1/2}$

where  $\sigma_0$  is the Yield stress,  $\sigma_i$  is the "friction stress", representing the overall resistance of the crystal lattice to dislocation movement, K is the "locking parameter", which measures the relative hardening contribution of the grain boundaries, D is the grain diameter [9]. Thus, in accordance with the above equation, as the grain diameter/grain size decreases the yield strength and the hardness of the material increases. The refinement of the microstructure may have contributed to the considerable rise in the micro-hardness values. Along with grain size refinement, the presence of sub-micron sized precipitates might have also contributed in increasing the hardness of the steel [13].

#### Table 2: Hardness and Tensile properties of the base material and FSP steel

Samples	Hardness (Hv)	YS (MPa)	UTS (MPa)	Elongation (%)
Base Metal	180	255	415	28.30
FSPed Sample	555	329	590	23.22

#### **TENSILE STRENGTH**

The representative true stress strain curves for the base steel and the FSPed steel is shown in Figure 2. It was observed that initially, both the specimens exhibit nearly linear elastic deformation. Once the yield point is reached, the plastic deformation sets in. The mechanical properties obtained from the tensile testing for both the investigated cases are listed in Table 2. The results showed that the yield strength and ultimate tensile strength increased and elongation decreased after the FSP. The possible reason for the improved strength of FSPed material may be attributed to the microstructural refinement [14]. Furthermore, the base steel postponed the necking phenomenon nearly up to 18.4% and the total elongation reached to 28.3%. On the other hand the total elongation in the FSPed steel was observed to be 23.22%, which is less than that of base steel. It is clear from the experimentation that as the FSP specimen possessed high hardness value therefore brittle fracture occurred during tensile testing. Xue et al [15] also observed the similar results in their FSPed specimens.



#### Figure 2: Stress Strain Curve for the base steel as well as FSPed SA 210 Grade A1 Steel

# HIGH TEMPERATURE CORROSION BEHAVIOR WEIGHT-CHANGE ANALYSIS

Weight change data expressed in mg/cm<sup>2</sup> versus number of cycles plots for the SA210 Grade A1 base steel and the FSPed steel is shown in Figure 3. The base steel showed a significant weight gain. The rate of oxidation went on increasing with the progress of the oxidation study. The steel has shown an overall weight gain of 343.69 mg/cm<sup>2</sup>, which is substantially higher than its counter FSPed specimen. The overall weight gain values for FSPed GrA1 boiler steel were found to be 172.13 mg/cm<sup>2</sup> respectively. The overall weight gain for the steel got reduced by 50% after the friction stir processing of the steel. Therefore, it can be inferred that the FSP is useful in enhancing the hot corrosion resistance of GrA1 steel.

Further from the parabolic rate constants (Kp) values, Grade A1 boiler steel showed transitions from  $28.4 \times 10^{-8} \text{ g}^2 \text{ cm}^{-4} \text{ sec}^{-1}$  for first six cycles and  $67.67 \times 10^{-8} \text{ g}^2 \text{ cm}^{-4} \text{ sec}^{-1}$  for the range eighth to fiftieth cycle. The deviations from the parabolic rate law have also been observed by Levy et al [16] during their studies on the oxidation and hot corrosion of some Ni-base superalloys at 704 to 1093°C. They attributed these deviations to cracking and spalling of the oxide scales. Whereas, the values of the parabolic rate constants (K<sub>p</sub>) for FSPed specimen are calculated as 17.98 x  $10^{-8} \text{ g}^2 \text{ cm}^{-4} \text{ s}^{-1}$  respectively. It is clear that the K<sub>p</sub> value for the steel has reduced significantly after the FSP of the steel. Hence, it indicates that the friction stir processing were successful to improve the high temperature corrosion resistance of SA 210 grade A1 boiler steel.



# Figure 3: Weight change/area vs. Number of cycles plot for the bare and FSPed SA 210 Grade A1 steel subjected to molten salt environment ( $Na_2SO_4$ - 82% Fe<sub>2</sub>(SO4)<sub>3</sub>) at 900°C for 50 cycles

#### CONCLUSIONS

- 1. Friction Stir processing resulted in microstructure refinement of the SA 210 grade A1 steel which in turn improved the hardness of the base steel. The microhardness of the FSPed material was found to increase by three times as compared to the base material.
- 2. FSPed material showed the increased tensile and yields strength values.
- 3. The oxide scale of GrA1 boiler steel showed a severe tendency of cracking and spalling during the course of high-temperature corrosion study in the molten salt environment. However, FSPed material showed lesser tendency towards cracking or spalling. The relative hot corrosion resistance of FSPed steel was found to be better than base steel.

#### ACKNOWLEDGMENTS

The authors wish to thank Dr. S.S Sehgal Director Chandigarh University (CU), Gharuan, Mohali Punjab (India) for development of Friction Stir Processing on the selected boiler steel material. The support by Indian Institute of Technology Ropar for the use of the Microhardness Tester Machine and universal testing machine facility during this work is highly acknowledged. Moreover, the authors owe special thanks to Dr. Harpreet Singh for extending the necessary facilities and support in conducting the detailed analysis at Indian Institute of Technology Ropar, Roopnagar Punjab. The financial and technical support of Chandigarh University for this work is gratefully ascribed.

NIGIS \* CORCON 2017 \* 17-20 September \* Mumbai, India

#### REFERENCES

- 1. Seong, B.G., Hwang, S.Y. and Kim, K.Y., "High Temperature Corrosion of Recuperators used in Steel Mills," Surface Coating Technology, Vol. 126, 2000, pp. 256-265.
- 2. Ma, Z.Y. (2008): "Friction stir processing technology: A review", Metallurgical and Materials Transactions A, Vol. 39A, pp.642–58.
- 3. E.A. El-Danaf, M.M. El-Rayes, M.S. Soliman, Friction stir processing: an effective technique to refine grain structure and enhance ductility, Materials and Design 31 (2010) 1231–1236.
- 4. D.R. Ni, D. Wang, A.H. Feng, G. Yao, Z.Y. Ma, Enhancing the high-cycle fatiguestrength of Mg–9AI–1Zn casting by friction stir processing, Scripta Materialia 61 (2009) 568–571.
- 5. C.I. Chang, X.H. Du, J.C. Huang, Producing nanograined microstructure in Mg–Al–Zn alloy by two-step friction stir processing, Scripta Materialia 59 (2008) 356–359.
- 6. C.I. Chang, X.H. Du, J.C. Huang, Achieving ultrafine grain size in Mg–Al–Zn alloy by friction stir processing, Scripta Materialia 57 (2007) 209–212.
- 7. P.S. De, R.S. Mishra, C.B. Smith, Effect of microstructure on fatigue life and fracture morphology in an aluminum alloy, Scripta Materialia 60 (2009) 500–503.
- 8. Singh, S and Kaur, M, "Mechanical and microstructural properties of NiCrFeSiBC/Cr3C2 composite coatings-Part I", Surface Engineering Journal, available online; DOI: http://dx.doi.org/10.1179/1743294414Y.0000000416,2014
- Hajian M., Abdollahzadeh A., Rezaei-Nejad S.S., Assadi H., Hadavi S.M.M., Chung K. and Shokouhimehr M., "Microstuxture and mechanical properties of friction stir processed AISI 316L stainless steel" Materials and Design, Vol. 67, 2015,pp.82-94
- 10. Chung YD, Fujii H, Ueji R, Tsuji N. Friction stir welding of high carbon steel with excellent toughness and ductility. Scr Mater 63, 2010, pp. 223–226.
- 11. Miyano Y, Fujii H, Sun Y, Katada Y, Kuroda S, Kamiya O., Mechanical properties of friction stir butt welds of high nitrogen-containing austenitic stainless steel. Material Science Engineering 528, 2011,pp. 2917–2921.
- 12. Aldajah S.H., Ajayi O.O., Fenske G.R. and David S., "Effect of Friction Stir Processing on the Tribological Performance of High Carbon Steel", Wear, Vol. 267, 2009, pp.350-355.
- Arora .S.H, Singh H., Dhindaw B.K., Some observations on microstructural changes in a Mg based AE42 alloy subjected to friction stir processing, Metallurgical and Materials Transactions B: Process Metallurgy and Materials Processing Science 43, 2012, pp.92–108.
- 14. Grewal H.S., Arora H.S. Singh H. and Agrawal A., "Surface modification of hydroturbine steel using friction stir processing", Applied Surface Science, Vol. 268, 2013, pp. 547-555.

## NIGIS \* CORCON 2017 \* 17-20 September \* Mumbai, India

- 15. Xue P., Li W.D., Wang D., Wang W.G, Xiao B.L. , Ma Z.Y., Enhanced mechanical properties of medium carbon steel casting via friction stir processing and subsequent annealing Materials Science&EngineeringA670,2016,153–158
- 16. Levy, M., Huie, R. and Pettit, F, "Oxidation and Hot Corrosion of Some Advanced Superalloys at 1300 to 2000°F (704 to 1093°C)," Corrosion., Vol. 45, No. 8,1989, pp. 661-674.

# NIGIS \* CORCON 2017 \* 17-20 September \* Mumbai, India