ISSN: 2278-0181
ESDST - 2017 Conference Proceedings

Characterization and High Temperature Corrosion Study of FSPed C70 Steel

Aayush Bawdekar
Department of Automobile Engineering
Chandigarh University
Mohali, India

Supreet Singh
Department of Automobile Engineering
Chandigarh University
Mohali, India

Manpreet Kaur
Associate Professor,
Department of Mechanical Engg
Baba Banda Singh Bahadur Engineering College,
Fatehgarh Sahib

Prabhjot Singh
Department of Mechanical Engineering
Chandigarh Group of Colleges
Mohali, India

Iqbal Singh
Department of Mechanical Engineering
Chandigarh Group of Colleges
Mohali, India

***Abstract*— In the present work C70 steel which is used in the manufacturing of automobile connecting rods and bearings was friction stir processed with defined parameters and investigations were made based on microstructure, mechanical properties and high temperature corrosion studies, for comparison unprocessed C70 Steel was also investigated under similar set of conditions. Friction Stir Processing has revealed to be feasible tool for enhancing the mechanical properties of materials like micro hardness, fine grain structures, high tensile strength, improved yield strength, improvement in elongation, and increased corrosion resistance. In detail characterization of both processed and unprocessed steel was carried out using a Scanning Electron Microscope (SEM), an Optical Microscope (OM), and Vickers Micro-Hardness testing. High Temperature Corrosion Studies were conducted for both at 900^{0}C for 50 cycles to see the influence of different parameters on corrosion rate. The microstructure refinement of processed C70 reveals that FSP have contributed to the considerable increase in the microhardness values at 2400 rpm and 60mm/min feed rate with plunge depth of 0.8mm.**
Keywords*— *Friction Stir Processing; medium carbon steel; microhardness; grain refinement

I. INTRODUCTION

C70 are extensively used in the sector of automobile engineering for the fabrication of connecting rods and bearings and shows potential in the field of shipbuilding industries. Friction Stir Processing (FSP) a contemporary surface engineering technique imitated from Friction Stir Welding is being widely used in naval and automobile industry. A non-consumable tool hold on CNC operated milling machine is penetrated inside the specimen of metal or alloys and it is made to rotate on its surface in a stirring motion and in transverse direction. Figure 1 shows the process of FSP. Tools are designed with a pin or pin less usually made up of tungsten carbide (WC) and polycrystalline cubic boron nitride (PCBN) used for FSPed of steel. Studies on a range of materials confirmed that the quality of Friction stir processing generally depends on base material properties as well as tool material, shape and dimensions also on process parameters like rotational speed, transverse speed, axial force, cooling system and feed rate. Frictions stir processing changes the properties of the specimen through intense localized plastic deformation, which results in process mixing the material without changing the phase and creates a microstructure with fine grain and increased hardness.

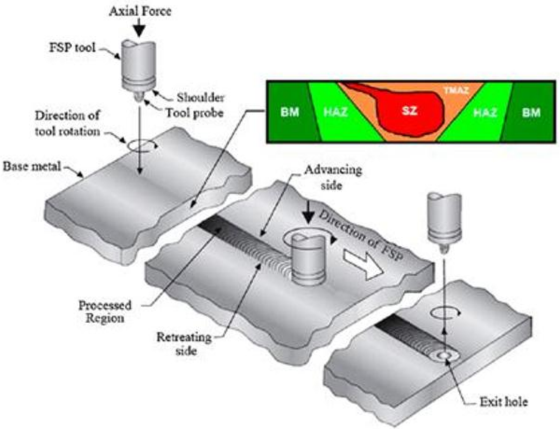

Figure 1 Process for FSP

The attainment of elevated temperatures plays an important role in the development of civilization for many countries [1]. Equipments in many cutting edge high technology areas have to work under elevated temperatures, high pressure and corrosive environment [2]. So, materials are prone to face problems at high temperatures in several high tech industries. Connecting rods in Automobile industry, Gas turbines in aircraft, fossil fueled power plants, refineries, and petrochemical industries, and heating elements for high temperature furnaces are some examples where corrosion limits their use or reduces their life, considerably affecting the efficiency [1].

Friction Stir Processing is the new and fast developing technique which is nowadays used for subsurface structural modification of different materials including not only aluminum and magnesium alloys but steels and other alloys also [3]. Friction stir processing is used for localized modification and control of microstructures in near-surface layers of processed metallic components for specific property enhancement [4]. It has proven to be an effective treatment to achieve major microstructural refinement, densification and homogeneity at the processed zone, as well as elimination of defects from the manufacturing process. Processed surfaces have shown an improvement of mechanical properties, such as hardness and tensile strength, better fatigue, corrosion and wear resistance [5].On the other hand, fine microstructures with equiaxed recrystallized grains improve superplasticity behavior. [6-8]

The main objective of the current investigation is to achieve strengthening of C70 through microstructural refinement by Friction Stir Processing (FSP) and analyze the effect of the same on high temperature corrosion behavior. The high temperature corrosion performance of the unprocessed and the FSPed specimens shall be evaluated in the Na_2SO_4-82%Fe_2 $(SO_4)^3$ molten salt environments in the laboratory and in the actual boiler environments so as to ascertain the usefulness of the FSP technique. The microstructure, mechanical properties, and corrosion resistance of the unprocessed and FSPed materials will be evaluated. The in-depth characterization analysis with an aim to propose mechanisms behind high temperature corrosion behavior of the coating.

II. EXPERIMENTAL PROCEDURE

A. Friction Stir Processing

Friction Stir Processing and preparing samples for metallographic analysis

The unprocessed base material C70 Steel plates with dimensions 76 x 50 x 5 mm were friction stir processed. The varying parameters are shown in table 1.

In the process to minimize the tool wear, selection of tool material i.e. Pin less Tungsten Carbide of 12mm diameter was selected with the sole purpose to minimize wear resistance used for working at high temperature.

The fixture for FSP was mounted on Jyoti VMC 640 CNC operated machine; the tool was held rotating there for about 10 seconds to soften the material and was allowed to travel along the slot. . The chemical composition and mechanical properties of C70 steel is shown in table 2 and table 3. After the procedure, specimens were cooled with dry ice at room temperature.

Table 1 Process parameters for FSP

Process Parameter			
Rotational Speed (RPM)	Transverse Speed (mm/min)	Plunge Depth (mm)	Passes
900	60	0.8	1
1600	60	0.8	1
2400	60	0.8	1

Table 2 Chemical Composition of C70

C	Mn	P	S	Fe
0.65 to 0.75 %	0.6 to 0.9 %	0 to 0.040 %	0 to 0.050 %	98.3 to 98.8 %

Table 3 Mechanical Properties of C70

Mechanical properties	Value
Density	7.8 g/cm³ (490 lb/ft³)
Elastic (Young's, Tensile) Modulus	210 GPa (30 x 10⁶ psi)
Strength to Weight Ratio	82 to 97 kN-m/kg
Tensile Strength: Ultimate	640 to 760 MPa
Tensile Strength: Yield	410 to 570 MPa
Thermal Expansion	12 µm/m-K
Modulus of Resilience	400 to 770 kJ/m³
Poisson's Ratio	0.29

The specimens were cross-sectioned at 90 degree angle to the FSP direction and prepared for metallographic analysis, cut in 5 x 5 x 5mm pieces, hot mounted using mount press machine and polished on SiC emery paper ranging from 220 to 2000 grades followed by velvet cloth polishing as shown in figure 2.

Characterizations of the microstructure were examined by optical microscopy, scanning electron microscopy (SEM). The specimens were etched in a solution containing 98% HNO3 and 2% ethanol. Mechanical characterizations were evaluated using Vickers hardness.

Figure 2 Sample for Metallographic analysis

B. High Temperature Corrosion

a) Sample Preparation

The specimens to be tested were cut and made to specified dimensions of 13 x 10 x 5 mm plates. All six faces of the specimen were polished on Radical DPM-33 double disc polishing machine using SiC emery paper ranging from 200 to 2000 grades and on velvet cloth. The specimen were made dirt free by cleaning them with ethanol and dried, their area was measured using digital vernier caliper and were accurately weighted on Citizon CY 204 electronic balance machine so as to plot weight gain per unit area vs. number of cycle graph.

b) Preheating of Alumina boats and Samples

The alumina boats to be used were preheated in universal oven at 250^0C for 4 hours gradually followed by 20 minutes of cooling at room temperature and again heated in Digital programmable industrial muffle furnace at 900^0C for 2 hour as shown in figure 3 to completely remove moisture from the boats. Samples along with boat were preheated in universal oven at 150^0C for 2 hour and afterwards their weights were measured.

Figure 3 Preheating in Oven

c) Salt mixture Coating and hot corrosion studies

Mixture of salt containing [Na_2SO_4- 82%$Fe_2(SO_4)_3$] was prepared with distilled water and coated on the specimen with salt ranging from 3.0-5.0 mg/cm^2 and further heated in oven at 250^0C for 2.5 hour and weight along the boat were measured. Specimens were then kept in muffle furnace at 900^0C along with boat for 1 hour followed by 20 minutes of immediate cooling at room temperature and weight was calculated on electronic balance as shown in Figure 4. The oxidation cycles were carried out 50 times for each and every sample and their weight was calculated at the end along spalled scale and visual inspections were also made.

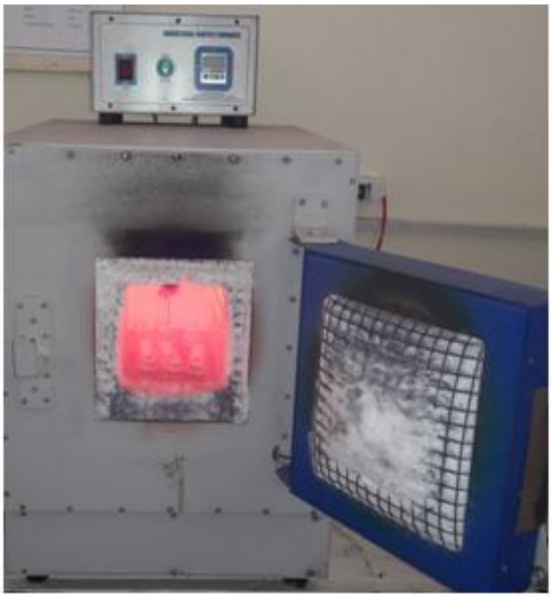

Figure 4 High Temperature Corrosion in Industrial Muffle Furnace

III. RESULTS & DISCUSSION

A. Microstructure

The Microstructures of the specimens were studied on leica inverted microscope available at IIT Ropar. There was a significant decrease in the grain size as the rotational speed was increased with similar feed rate. The images obtained for the different samples are shown in Figure 5.

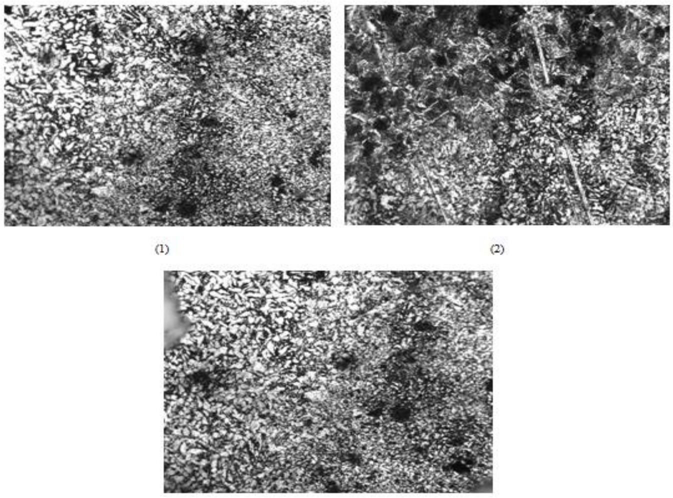

Figure 5 Microstructural developments of FSPed Samples (1) S1 (2) S2 (3) S3

B. Hardness

Hardness values of FSPed C70 specimens were measured on Vickers micro-hardness tester at a load of 0.3kgf. It is observed that grain refinement and reduction in grain size might have contributed to increased hardness value. Beside with grain size refinement, the existence of sub-micron sized precipitates may have also contributed in increasing the hardness of the C70. The microhardness profiles of Friction stir processed C70 along the cross-section as a function of distance from the FSPed interface are plotted in Figure 6. The critical hardness values of the Base Metal were found to be 180 HV. In the case of the FSPed samples, the microhardness values for the FSP Sample 1 named as S1 with defined parameters lie in the range of 427-450 HV. The microhardness values for the sample S2 with defined parameters lie in the range of 490-515 HV. The microhardness values for the sample S3 with defined parameters lie in the range of 559-586. A maximum value of hardness was shown by S3 Friction Stir Processed sample, whereas a minimum of Sample S1.

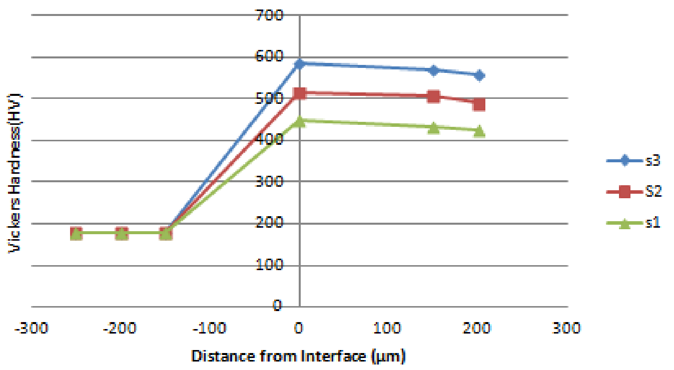

Figure 6 Hardness VS Distance Profile of Sample S1, S2, S3

C. *Scanning Electron Microscopy*

Scanning Electron Microscope analyzer (Jeol JSM -6610 LV) was used to obtain the micrographs and characterize the grain structure and transition zone of the samples at three different varying RPM's. Figure 7 (1), (2), (3) shows the micrograph of specimen at 900 RPM, 1600RPM, 2400 RPM in which the transition zone of ultrafine grain can be seen.
The average grain size of the specimens were observed to be around 2-3µ. The transition zone can be clearly seen in the micrographs.

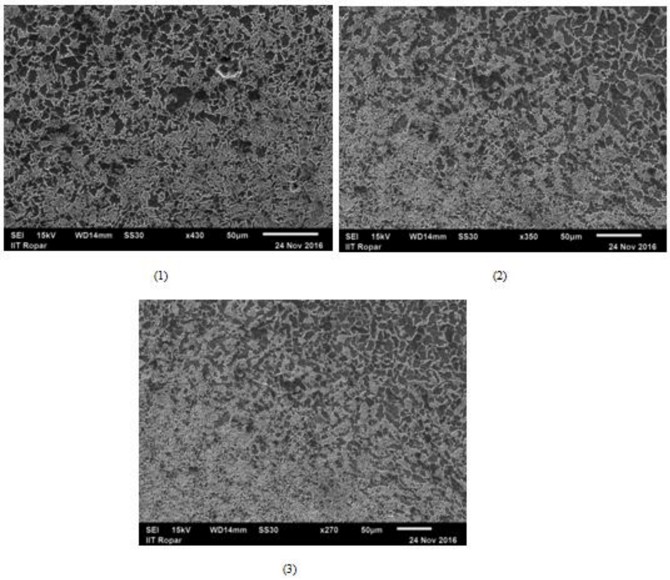

Figure 7 SEM Micrographs of Ultrafine grain of FSPed C70 Specimen (1) S1, (2) S2, (3) S3

D. *High Temperature Corrosion At 900 °C*

(a) Visual Examination

In the case of Sample 1 (S1) steel shown in Fig. 5.1 (a), it was observed that the surface of steel turned dark black with some spots on it after first cycle. The sample turned grayish black after second cycle. Few Minor cracks appeared on the top surface by the end of 5th cycle and remained there till the end of 50 cycles. After 10th cycle separation in fragments from top and sides were seen with a drastic increase in weight. By the end of 25th cycle lustrous layer was seen on the surface.
In the case of Sample 2 (S2) steel shown in Fig. 5.1 (b), the color of the sample changed from black to light gray after the second cycle and later to dark gray by the completion of 10th cycle. Apart from the change in colour, many small cracks started appearing by 13th cycle. Formation of pits with some spallation from the sides were observed by 20th cycle and remained there throughout 50th cycle.
In the case of Sample 3 (S3) steel shown in Fig. 5.1 (c) the color changed from black to grey after 3rd cycle. Some major changes were observed after 20th cycle which includes some cracks and lustrous appearance. The overall weight gain by the end of 50th cycle was least in the case of S3.

(b) Weight Change Data

The weight Gain VS No of cycles graph plot for the base and S1, S2, S3 at 900 °C for 50 cycles. The weight change data is usually used to establish the kinetics of the corrosion process. A higher weight gain represents higher rates of corrosion. In the case of S1 C70 steel, the rate of weight gain was slow for the first 15 cycles. Thereafter weight gain rate continued to increase at a comparatively higher rate till the end of cyclic studies. So from weight gain data it can be inferred that corrosion rate increased after the 15 cycles of study. The S2 C70 steel has shown the tendency to gain weight without showing any indication of steady state corrosion rate comparable to S3, which has shown the tendency to become uniform with least increase in weight.
It can be inferred from the plots that the parameters for Friction Stir Processing of Sample 3 showed protection in high temperature corrosion testing as the weight gain values for the Sample 3 C70 steel is significantly smaller than S2 and S3.

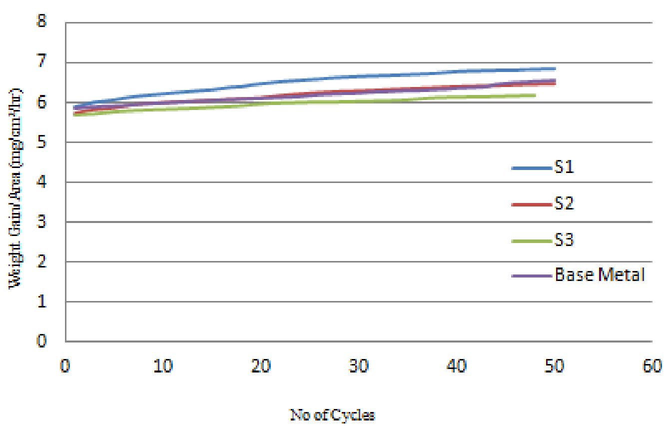

Figure 8 Weight Gain VS No of Cycles Graph for Specimen S1, S2, S3 and Base Metal

E. HALL PETCH EQUATION

Intense heat generation due to friction plastically deforms the metallic material thus resulting in ultra fine grain structure and enhanced mechanical properties such as tensile strength and hardness. To corroborate the relationship between ultrafine grain structure and enhanced mechanical properties Hall-Petch equation is determined to validate the findings.

The grain structure got considerably refined as a result of FSP. The average grain size of S1, S2 and S3 was found to be 8.7 µm, 7.2 µm and 3.9 µm respectively.

According to Hall-Petch, equation, as in (1)

$$\sigma = \sigma_0 + kd^{-1/2}, \quad (1)$$

Where σ is the yield strength, σ_0 is the stress for dislocation movement, k is the strengthening coefficient of material and d is the diameter of average grain size. This equation can be stated for hardness, as in (2)

$$H = H_0 + k_H d^{-1/2}, \quad (2)$$

Where H_0 and k_H are constants

The Hall-Petch equation is usually helpful in determining relationship between grain size and hardness of the material, as the grain size is reduced there is an increase in the hardness of the material.

Experimental values of hardness and observed average grain size of FSPed specimens clearly shows the relationship between both in accordance to Hall-Petch Equation.

Figure 9 (a), (b), (c) shows graphically the relationship between hardness values and grain size of C70 Steel.

The results thus obtained clearly shows that at higher RPM FSP helps in enhancing the mechanical properties of C70 Steel by creating superfine grain structure and increasing the hardness by three times that of base metal.

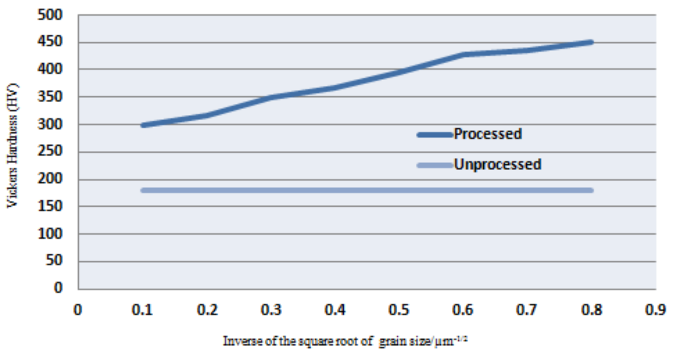

Figure 9(a) Hardness VS Grain Size Graph for S1

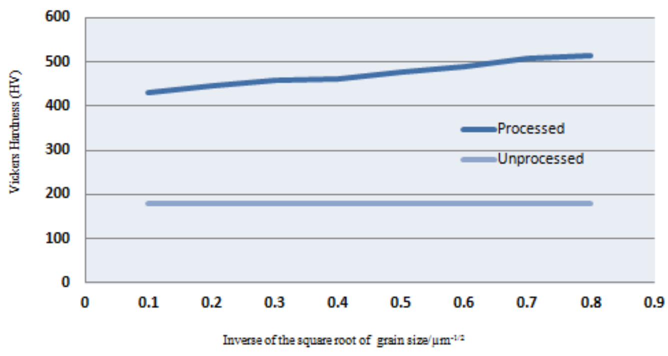

Figure 9(b) Hardness VS Grain Size for S2

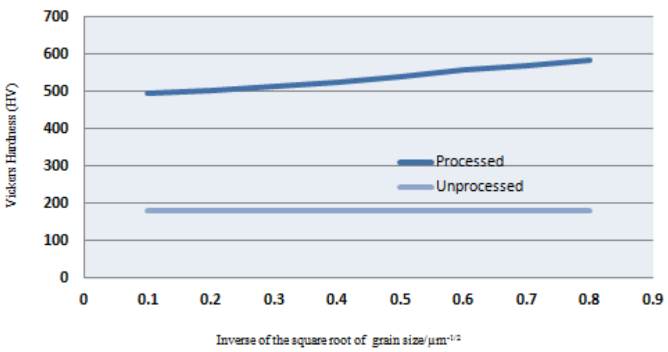

Figure 9(c) Hardness VS Grain Size for S3

V. CONCLUSIONS

The grain structure got considerably refined as a result of FSP. The average grain size of S1, S2 and S3 was found to be 8.7 µm, 7.2 µm and 3.9 µm respectively. It can be inferred from the plots that the parameters for Friction Stir Processing of Sample 3 showed protection in high temperature corrosion testing as the weight gain values for the Sample 3 C70 steel is significantly smaller than S2 and S3. The microhardness values for the sample S3 with defined parameters lie in the range of 559-586. A maximum value of hardness was shown by S3 Friction Stir Processed sample, whereas a minimum of Sample S1.Hence FSP is successful on C70 materials in reference to above results.

REFERENCES

[1] Khanna A.S., (2002): “Introduction to high temperature oxidation and corrosion”, ASM International, ISBN 0-87170-762-4, and SAN: 204-7586, pp.1-322.

[2] Ananthapadmanabhan P.V., Sreekumar K.P., Muraleedharan K.V., Venkatramani N.,(1991) “Plasma-sprayed composite coatings for high temperature applications”, Surface & Coating Technology, Vol. 49, pp. 62-66.

[3] Nicholas E.D. (2003): “Friction Processing Technologies Welding in the World”, Vol. 47, No. 11-12, pp. 2-9.

[4] Ma, Z.Y. (2008): “Friction stir processing technology: A review”, Metallurgical and Materials Transactions A, Vol. 39A, pp.642–58.

[5] Karthikeyan, L., Senthilkumar, V.S., Balasubramanian V. and Natarajan, S. (2009): “Mechanical property and microstructural changes during friction stir processing of cast aluminum 2285 alloy”, Materials and Design, Vol. 30, pp.2237–42.

[6] Mishra, R.S., Mahoney, M.W., McFadden, S.X., Mara, N.A. and Mukherjee, A.K. (2000): “High strain rate superplasticity in a friction stir processed 7075 Al alloy”, Scripta Materialia, Vol .42, pp.163–168.

[7] Ma, Z.Y., Mishra, R.S. and Mahoney, M.W. (2004): “Superplasticity in cast A356 induced via friction stir processing”, Scripta Materialia, Vol. 50, pp. 931–5.

[8] Ma, Z.Y., Liu, F.C. and Mishra, R.S. (2010): “Superplastic deformation mechanism of an ultrafine ne-grained aluminum alloy produced by friction stir processing”, Acta Materialia, Vol. 58: pp. 4693-704.