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Corrosion Characterisation of Low Alloy CrMoV Steel Before and After Cryogenic Heat Therapy

S. Sarveswara Reddy[1,*], K.V. Durga Rajesh[2], Francis Luther King M[3]

Abstract

Low alloy CrMoV steel is used in Turbine wheels, Distance pieces, Compressor aft shafts etc of Gas Turbines. Cryogenic heat treatment was used to examine the effects on the corrosion resistance of low alloy CrMoV steel. In order to eliminate residual stresses and boost wear resistance in steels and other metal alloys, cryogenic heat treatment (CHT) employs cryogenic temperatures (i.e., below -190°C) to treat work parts. Cryogenic treatment is desired not only for the benefits it provides in the areas of stress relief and stability or wear resistance, but also because of the improvement it provides in corrosion resistance. The nuclear, pharmaceutical, and food processing sectors all have a vested interest in accurately measuring very low corrosion rates, where minute amounts of contamination and impurities are a problem. In the current investigation electrochemical methods (Tafel Analysis, NOVA software) are used to characterize corrosion mechanisms and predict corrosion rates. To understand the effect of Corrosion rate and Polarization resistance was performed in 3.5% Nacl. The Corrosion resistance of cryogenically treated sample was found to be much better than the un-treated sample and Hardness of the cryo-treated sample is also improved after Cryogenic heat therapy.

Keywords: Cryogenic Treatment, Tafel Analysis, Corrosion rate, Polarization Resistance, NOVA software

INTRODUCTION

The industrial sector was first exposed to cryogenic heat treatment in the 1920s. The right finishing procedure, Which may entail an additional heat treatment, must be carefully chosen for components with a negligible wear rate, low austenite content, and low cost [1, 2]. cryogenic heat treatment for steels primarily achieves the goal of completely removing all trace of austenite. There is also evidence that the distribution of carbide becomes more even when the average size of individual carbides decreases [3]. High strength steel has been rapidly developing in different mechanical engineering applications, including bridges, ships, trains, airplanes, automobiles, and wind energy, during the last few decades, propelled by the extraordinary environmental concerns confronting civilization [4, 5]. superior strength due to its vast range of possible applications, notably in the field of lightweight design methods for buildings, steel is in high demand as a result of the growth of these industries. The "strength-ductility trade-off" is the term sometimes used to describe the trade-off between higher strength and lower ductility [6, 7]. In order to meet the demands for dependability and safety, it is important to develop new high-strength steel in order to avoid the strength-ductility trade-off. Cryogenic therapy, or treatment of materials at

***Author for Correspondence**
S. Sarveswara Reddy

[1]Research Scholar, Department of Mechanical Engineering, Koneru Lakshmaiah Education Foundation, Vaddeswaram, Andhra Pradesh, India
[2]Associate Professor, Department of Mechanical Engineering, Koneru Lakshmaiah Education Foundation, Vaddeswaram, Andhra Pradesh, India
[3]Associate Professor, Department of Mechanical Engineering, Swarnandhra College of Engineering and Technology, Andhra Pradesh, India

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low temperatures, may be classified as either shallow (between 113 and 193 Kelvin) or deep (below 113 Kelvin). Cooling, soaking, and reheating are the three primary stages of cryogenic therapy [8]. It has been demonstrated that cryogenic treatment can increase tool steel’s hardness wear resistance, and dimensional stability. [9, 10]. The deep cryogenic treatment of steel results in Three main alterations to its microstructure: (i) retained austenite is transformed into martensite, (ii) finely distributed carbides are precipitated, and (iii) residual stresses are relaxed [10]. The number of studies examining tempering capacity to raise steel’s strength and hardness has increased in recent years. Researchers have discovered that direct-quenching and tempering low-alloy strip steel at temperatures between 570 and 600 degree Celsius produces an ideal combination of strength and toughness [11]. For Ferrium S53 steel, tempering at 482°C results in the maximum values of 1945 MPa for tensile fracture strength and 1543 MPa for yield strength, as reported by Zhang et al. [12]. An amazing combination of strength (1550 MPa) and impact energy (91.5 J) has been found in tempered 3Mn-Si-Ni martensitic steel at 230°C, however rising the tempering temperature from 320 to 550°C results in tempered martensite embrittlement [13]. Because deep cryogenic treatment (DCT) refines martensite and martensite transforms retained austenite, it is able to improve a material's hardness, toughness, wear resistance, and fatigue behavior in ways that traditional heat treatment methods cannot [14–16].

Adding carbides to steel and cast iron before cryogenic treatment increases the carbide concentration and distributes the carbides more evenly throughout the material [17, 18]. The wear resistance of a tool is an important factor in determining how long it will last. This is especially true of pinion shafts, brakes, rotors, bearings, gears, face gears, and dies. All of these benefit greatly from this improvement. Many types of ferrous materials, including carburized steel [18, 19], high-speed steel [20], and tool steel, are treated with deep cryogenic heat. This study attempts to know corrosion rates in both the cryogenically treated and untreated samples. The term "corrosion" describes any process that leads to the breakdown of metal. Small corrosion rates, a short investigative period, and the theoretical knowledge that is adequately recognized are the main benefits of electrochemical procedures. Electrochemical experiments involve polarizing samples to hasten corrosion and measuring the results in a matter of minutes or hours. Both the oilfield and the research lab use the electrochemical measurements. Tafel extrapolation procedures, potentiodynamic techniques, and polarization resistance approaches are among the Commonly used electrochemical polarization systems [21–24]. Hardness, as measured on the Rockwell C scale, is a useful indicator of overall quality since it can be determined from a very small sample. Beyond its use in measuring hardness, the development of testing procedures using this trait is crucial. There is an exception with the Rockwell C hardness scale. This distinction is sometimes underlined by the statement that size and concentration determinations are measurements whereas Rockwell hardness is a test procedure. Rockwell hardness is an example of how the procedures of a test might be completely unrelated to the features of a product that really matter to consumers. Using the Rockwell C scale to assess hardness during heat treatment may help bring down the standard deviation of the process. Having less variety is a good starting step toward making sure the finished products are up to grade, but it's not a guarantee.

Rockwell hardness tests, are used to evaluate the overall material hardness of metals and plastics. Although it correlates with qualities like strength and wear resistance hardness testing does not offer a direct evaluation of any performance parameters. The relative ease and cheap cost of hardness testing makes it a popular method for evaluating materials. Test specimens are indented using a diamond cone or hardened steel ball indenter. Under an initial minor load of 10 kg, the indenter is pressed into the test material. When equilibrium is achieved, a datum position is established for an indicating device that tracks the indenter's motions and thus responds to variations in the depth to which the indenter penetrates. While the initial minor load is still being applied, a larger load is applied, leading to deeper penetration. When equilibrium is restored, the preliminary minor load is kept in place while the additional major load is eliminated.

MATERIALS AND METHODS

With the inclusion of deep cryogenic treatment in between quenching and tempering phases, CrMoV low alloy steel was selected for this experiment, vacuum heat treated using the recommended methods by steel producers. After being subjected to heat treatments, samples were investigated using conventional metallurgical methods, their hardness was determined, and their corrosion resistance was assessed using the Tafel analysis methodology. The NOVA software (AUTOLAB PGSTAT302N) provides a convenient interface for performing corrosion rate analysis. Heat Treatment and Cryogenic Treatment was performed in PMTLEE Vacuum Technologies, Hyderabad and Corrosion experimentation and Hardness testing were performed in Centre for excellence Nanotechnology (CNT) lab, Bharat Heavy Electrical Limited (BHEL) R&D, Hyderabad.

Selection of Material

Specimens Preparation

CrMoV low alloy steel, used in steam and gas turbines, balance chamber, bushing, pressure seal head etc., were selected for the present study. Specimens were prepared in the shape of rectangle 45 mm × 20 mm × 6 mm from CrMoV low alloy steel forgings. The current research aimed to explore a novel low-alloy, high-strength Cr-Mo-V steel with the chemical composition detailed in Table 1.

Table 1. Chemical Composition of CrMoV Alloy Steel (Wt %)

	C	Mn	Si	P	S	Ni	Cr	Mo	V	C	Fe
CrMoV Alloy Steel	0.33	0.90	0.35	0.012	0.010	0.55	1.35	1.50	0.30	0.35	Balance

Heat and Cryogenic Treatment Procedure

Vacuum hardening and quenching (VH&Q) and a two-stage tempering (T) comprised the conventional heat treatment of the steels under study. In the first stage of the conventional heat treatment, samples were heated in a vacuum hardening furnace (Figure 1). At a rate of 535°C/1 hour and soaked for 1 hour and again heated at 825°C/1 hour and soaked for 1 hour and heated at 1060°C/1 hour and soaked for an hour and then the samples were nitrogen gas quenched to 40°C. After being quenched, the samples were stabilized for 2 hours at 180°C.

Figure 1. Vacuum Hardening (VH).

After the first phase of (VH&Q), the specimens were taken out of the vacuum hardening furnace and put through a second round of deep cryogenic treatment (Figure 2). After being cooled from ambient temperature to -196°C (77.15 K) at a cooling rate of 5 K/min, specimens underwent deep cryogenic treatment by being submerged in liquid nitrogen under strict monitoring. After reaching DCT, samples were immersed in liquid nitrogen for 24 hours, as has been shown to be most efficient in the case of tool steels [25]. The samples were then allowed to reheat to room temperature. In the case of deep cryogenic treatment, tempering is the third and last stage of the heat treatment process. After Cryogenic Treatment samples were taken out from the subzero chamber and placed in a Tempering furnace (Figure 3) and heated at 200°C for 2 hours and again heated at 200°C for 2 hours, two stages of tempering were carried out in this instance.

Figure 2. Sub Zero Chamber (SZ).

Figure 3. Tempering Furnace (TF).

Corrosion Test Procedure

Solution Preparation

Corrosion solution was prepared by collecting table salt or common salt or simply Nacl weighed by using weighing balance in 35.24 grams and mixed in a 1000 ml of distilled water in measuring beaker and obtained NaCl 3.5wt. %.

Electrochemical Tests

The rate of corrosion may be calculated using electrochemical techniques as an alternative to more conventional approaches. Corrosion rates, or the rate at which a specimen corrodes, may be determined with relative ease using only a few basic electrochemical measurements [26–28], such as those provided by linear sweep voltammetry (LSV). With this technique it is possible to measure extremely low corrosion rates and it can be used for continuously monitoring the corrosion rate of a system. In the current investigation, Tafel Extrapolation method is used to determine the Corrosion rate and polarization resistance by using NOVA Software.

EXPERIMENTAL SETUP

In the present investigation sodium chloride (NaCl) 3.5 wt.% was used as a corrosion medium. pH value of the sodium chloride (NaCl) is 7, which is measured by using the multiparameter analyzer (CONSORT 831C). Corrosion tests were carried out at 25°C under static conditions. The CrMoV low alloy steel samples (Cryogenically treated and un-treated) termed the working electrode was fitted one side of the glass corrosion cell. It was filled with Sodium chloride (Nacl) Solution. Silver chloride electrode (Ag-Agcl) was used as the reference electrode and a platinum sheet electrode as the auxiliary electrode. The potential of the working electrode was measured with respect to a reference electrode Ag-Agcl.Tafel analysis was performed with an AUTOLAB PGSTAT302N controlled by a computer which is shown in Figure 4, with the NOVA software. Anode slope (b_a), Cathode slope (b_c) and Corrosion current density (I_{corr}) were determined using dissolution rate analysisby NOVA software.

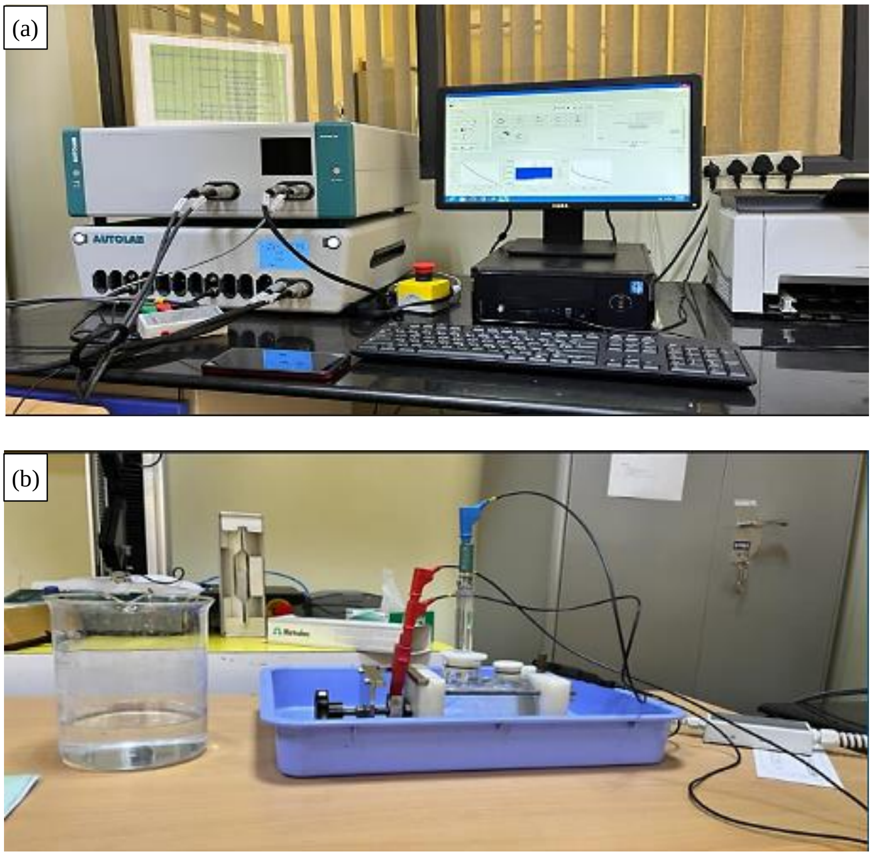


Figure 4. Potentiostat Galvanostat Experimental Setup.

Prediction of Corrosion Rates

The corrosion current, *icorr* (A), is proportional to the metal dissolving rate or corrosion rate, R_M (mm/year), as stated by Faraday's law.

$$R_M = 3.17E - 9\frac{M}{3nF\rho A}i_{corr} \tag{1}$$

Electrochemical Corrosion

The Butler-Volmer (B-V) equation express the connection between the current i and the over potential in charge transfer (kinetic, or activation) control.

$$i = i_{\text{corr}}\left(e^{2.303\frac{n}{b_a}} - e^{-2.303\,\frac{n}{b_c}}\right) \tag{2}$$

For excessive anodic over potentials ($\frac{\eta}{b_a} \gg 1$), the B-V equation shorten as the Tafel equation for the anodic reaction, Equation 3:

$$\eta = b_a.\log\left(\frac{i}{i_{corr}}\right) \tag{3}$$

For excessive cathodic over potentials ($\frac{\eta}{b_c} \ll -1$) the Tafel equation forb the cathodic reaction is given by

$$\eta = -b_c.\log\left|\frac{i}{i_{corr}}\right| \tag{4}$$

For the alteration of the current's logarithm with the potential, the Tafel equations 3 and 4 comes to straight line conclusion. As a result, Tafel charts which are semi logarithmic plots, are frequently used to display currents. Tafel slope analysis is the name given to this kind of analysis. NOVA software provides a convenient interface for performing corrosion rate analysis.

RESULTS & DISCUSSION

Tafel Analysis

The Tafel analysis provides a quick estimation of the corrosion rate or Metal dissolution rate and the polarization resistance. The corrosion rate is calculated with Equation 1. The NOVA software (AUTOLAB PGSTAT302N) provides a convenient interface for performing corrosion rate analysis. Selecting the Corrosion ate analysis Command as shown in Figure 5, inputs are, first go to mode and select drop down button as Tafel analysis or Polarization resistance and second field represents density, and third field represents equivalent weight and last field represents Surface area of the sample. By providing all inputs to corrosion rate analysis command, Tafel plots Current (A) versus Potential applied (V) will be generated. Figure 6(a) & 6(b) represents tafel plot for cryo-treated sample and Figure 7 (a) & 7(b) represents tafel plot for un-treated sample. Figure 6 (a) & 6(b) both plots are same but Figure 6(b) represents the linear regions of the tafel plot are identified by placing markers. The green line represents the linear regressions. The corrosion potential and corrosion current are also shown. Figure 7 (a) & 7(b) both plots are same but Figure 7 (b) represents the linear regions of the tafel plot are identified by placing markers. The green line represents the linear regressions. The corrosion potential and corrosion current are also shown. Corrosion parameters of low alloy CrMoV steel before and after Cryogenic Treatment as shown in Table 2 &3.

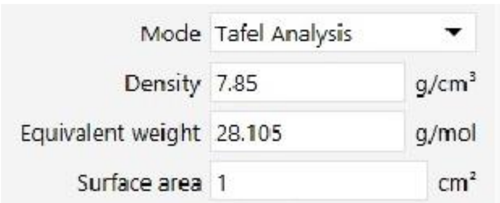


Figure 5. Corrosion rate Analysis Command.

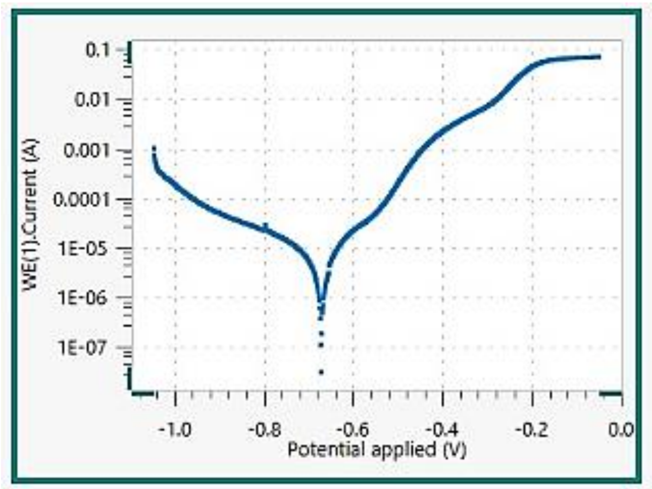
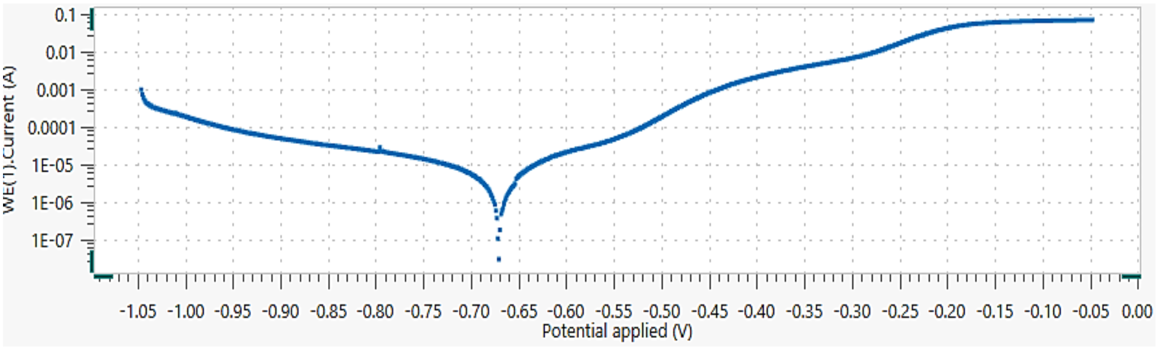


Figure 6. (a) Tafel Graph For Cryogenic Treated Sample.



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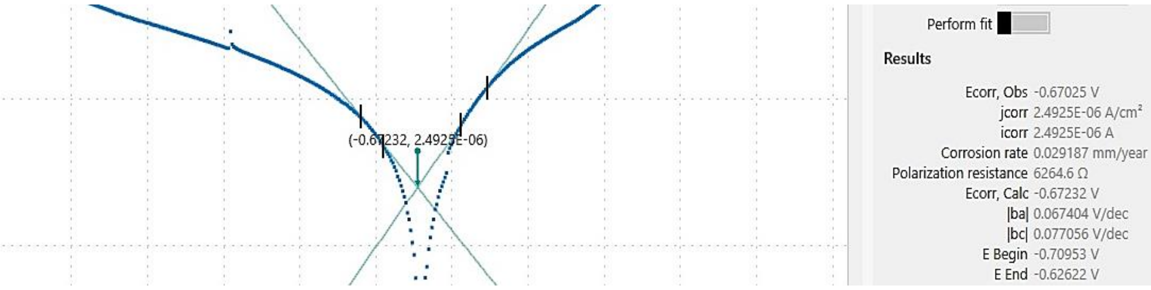

Figure. 6. (b) Tafel Plot For Cryogenic Treated Sample With Linear Regressions.

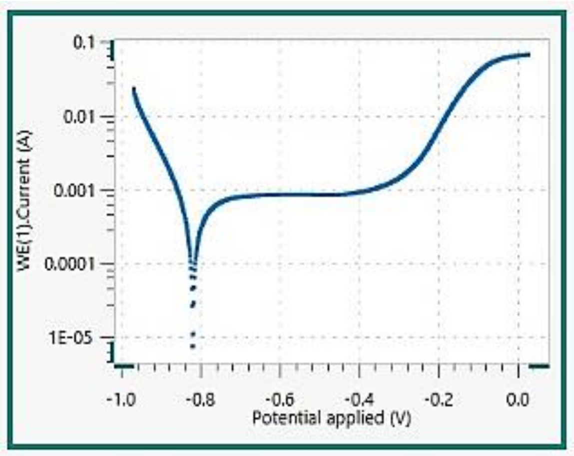

Figure 7. (a) Tafel Graph For Un-Treated Sample

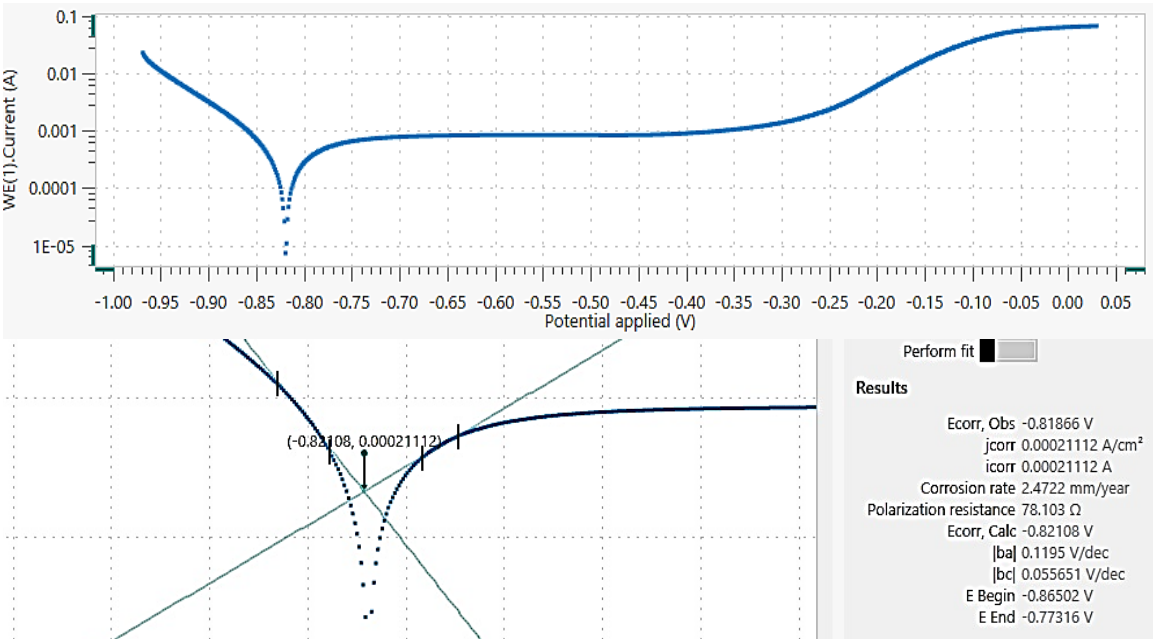

Figure 7. (b) Tafel Plot For Un-treated Sample With Linear Regressions.

Table 2. Corrosion parameters of Un-Treated and Cryogenically treated Samples.

S.N.	CrMoV low alloy Steel	Corrosion potential (E_{corr}) (V)	$\|bc\|$	$\|ba\|$	Corrosion current density (J_{corr}) A/cm^2
			V/dec		
1	Un-Treated Sample	-0.82108	0.055651	00.1195	0.00021112
2	Cryo-Treated Sample	-0.67232	0.077056	0.067404	2.4925E-06

Table 3. Polarization resistance and Corrosion rate of Un-Treated and Cryogenically Treated Samples.

S.N.	CrMoV low alloy Steel	Polarization resistance (R_p) Ω	Corrosion rate (C_R), mm/year
1	Un-Treated Sample	78.103	2.4722
2	Cryogenically Treated Sample	6264.6	0.029187

For cryo treated specimen, values of the corrosion potential (E_{corr}) is -0.67232 (V), Tafel slopes, slope of cathode ($-b_c$) is 0.007056 (v/dec) and slope of anode (b_a) is 0.067404 (v/dec), Corrosion current density (Jc_{orr}) is 2.4925E-06 A/cm^2 are given in Table 2 and polarization resistance (Rp) is 6264.6 Ω as given in Table 3, and corrosion rate (C_R) is 0.029187 mm/year as given in Table 3. For Un-Treated Specimen, values are E_{corr} is -0.82108 (V), Tafel slopes, slope of cathode ($-b_c$) is 0.05565(v/dec) and slope of anode (b_a) is 0.1195(v/dec), Corrosion current density (Jc_{orr}) is 0.00021112 A/cm^2 are given in Table 2 and polarization resistance (Rp) is 78.103 Ω as given in Table 3, and corrosion rate (C_R) is 2.4722 mm/year as given in Table 3. From Figure 6 (a) & Figure 6(b). plot related to Cryogenic treated sample, cathodic and anodic branches met between -0.70 to -0.65 where as in Figure 7 (a) & 7(b) plot related to un-treated sample, cathodic and anodic branches met between -0.85 to -0.80. A slight variation was observed in plots shown in Figure 6(a) & 6(b)(Cryo-treated) and Figure 7 (a) & 7(b) Un-Treated sample, that variation brought enhancement in Metal dissolution rate (M_R) or Corrosion rate which are given in Table 3. It has been observed that when polarization resistance decreases corrosion rate increases in case of Un-treated sample which is given in Table 3, in case of cryogenically treated sample polarization resistance increases, corrosion rate decreases which is given in Table 3 Low alloy CrMoV Steel un-treated before as shown in Figure 8(a) appeared like a shiny surface and after electrochemical analysis specimen surface turn up to rusty circular shape with black in colour, which as shown in Figure 8(b). Similarly, Low alloy CrMoV Steel cryogenic treated before as shown in Figure 9(a) appeared like a satin brown colour and later electrochemical analysis specimen turn up to rusty circular shape with light grey in colour as shown in Figure 9(b). Corrosion rate analysis command is shown in Figure 5.

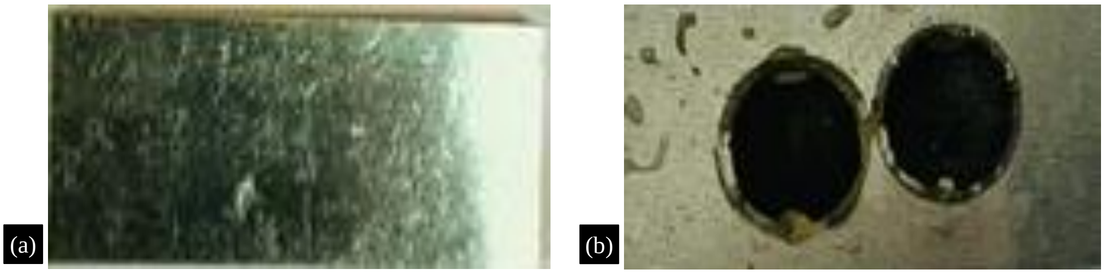

Figure 8. *CrMoV Samples:* (a) Un-Treated Before, (b) After Corrosion

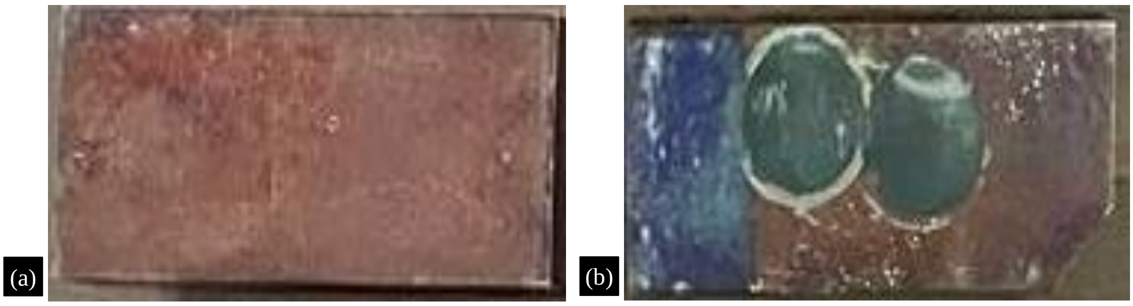

Figure 9. *CrMoV Samples:* **(**a) Cryogenic Before, (b) After Corrosion

Rockwell Hardness

A device that automatically displays a numerical value based on the relative resistance to penetration of a metal or alloy when subjected to a diamond-pointed cone driven into the metal to a predetermined depth.

Table 4. Rockwell Hardness of CrMoV low alloy steel Un-Treated and Cryogenically Treated Samples.

S.N.	CrMoV low alloy Steel	HRC
1	Un-Treated Sample	17.25 HRC
2	Cryogenically Treated Sample	37 HRC

Rockwell Hardness (HRC) values of Low alloy CrMoV steel Samples before and After Cryogenic Heat therapy are listed in Table 4. Cryogenic treatment aids in achieving uniform carbide distribution in the microstructure, improving the material characteristics; deep cryogenic temperatures also reduce the amount of austenite that remains in the material, increasing its hardness, wear resistance and tensile strength. Immediately after vacuum quenching and tempering (Q + T) and before stabilization, the samples averaged 37 HRC in hardness. The majority of increase in hardness attributed to the breakdown of austenite and the precipitation of fine secondary carbides after stabilization. After reaching 200 degrees Celsius in the tempering process, the hardness rises to 37HRC and then lowers somewhat after being heated to 330 degrees Celsius. Once the tempering temperature rises above 380°C, the hardness decreases dramatically. Figure 10 shows the indentations on untreated and Figure 11 shows the indentations on the cryo-treated sample.

Figure 10. Indentations On Un-Treated Sample.

Figure 11. Indentations Cryo-Treated Sample.

The development of the microstructure during tempering at various temperatures is responsible for these alterations in behavior. The martensitic transformation, which is a diffusion less process, is one of the most significant allotropic transformations that may occur in steels. This transformation results in a hierarchical microstructure [29] and a super saturation of solutes, which can result in precipitation during tempering [30]. In many cases, a martensitic microstructure combined with nano-scale precipitates results in exceptional attributes of the steel, including very high levels of strength and hardness, high levels of toughness etc. [31].

CONCLUSIONS

Cryogenic treatment improves corrosion resistance of low alloy CrMoV steels. Cryogenic treated sample Metal dissolution rate (M_R) is 0.029187 mm/year and Un-Treated Sample Metal dissolution rate (M_R) is 2.4722 mm/year. In the current investigation it has been observed that when polarization resistance (6264.6 Ω) increases corrosion rate (0.029187 mm/year) decreases in the case of cryogenic treated sample. In case of Un-treated sample polarization resistance decreases (78.103 Ω) corrosion rate (2.4722 mm/year) increases. Hardness is also increased after Cryogenic heat treatment. Prior to Cryogenic treatment average hardness is 17.25 HRC and after Cryogenic treatment average hardness is 37 HRC. When compared to more traditional approaches to weight loss, Tafel Analysis fares much better. This method is superior to other traditional methods of corrosion analysis, such as weight loss, since it allows for the measurement of very low corrosion rates (less than 0.1 mm/year). The nuclear, pharmaceutical, and food processing sectors all have a vested interest in accurately measuring very low corrosion rates, where minute amounts of contamination and impurities are a problem. Cryogenic therapy was demonstrated to have advantageous effects on both the wear resistance and corrosion resistance of CrMoV low alloy steels.

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