Chapter

Optimization of Retained Austenite and Corrosion Properties on EN-31 Bearing Steel by Cryogenic Treatment Process

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Abstract

In this work, the percentage of retained austenite and corrosion rate presented on EN 31 bearing steel was identified by which cryogenic treatment processes. Further investigation carried out the possible mechanism brought in by which treatment has significantly improving the properties of the EN-31 bearing steel. The hardness values of CHT and DCT were compared by using the microstructure view of the CHT and DCT samples. The optimised cryotreated samples were prepared for metallographic examination as per ASTM E3-01. Then, the specimen were subjected to factor level settings such as cooling rate, soaking period, soaking temperature and tempering temperature at various conditions. Moreover, the precipitation of fine carbides and the transformation of retained austenite to martensite showed considerable variations in the hardness of the optimised DCT samples compared with the CHT samples. The mean hardness value of this sample is 861 HV and 19.20%, 847 HV and 17.25%, 838 HV and 17.10%, 857 HV and 18.40%, 790 HV and 13.45% improvement in the hardness compared with CHT.

Keywords: cryogenic treatment, EN 31 steel, retained austenite, corrosion, optimization

1. Introduction

EN-31 Bearing steel is a chromium-alloy steel, which is suitable for most applications such as bearing, plunger, barrel, etc. According to Ashish Bhateja et al. [1], the effect on the hardness of three sample grades of steel i.e. EN31, EN-8 and D3 after heat treatment processes such as annealing, normalising, hardening and tempering were conducted. According to their study after annealing specimen of EN31 it becomes softer than untreated specimen. After normalising hardness is more as compared to untreated specimen. Amey et al. [2], studied the effect of heat treatment on the degree of distortion due to phase transformation on 100Cr6 material. It was inferred that cooling rate plays a major role in decreasing distortion. It also suggested that the presence of retained austenite, in the large amount, on the material can lead to distortion/.failure of the component. Sri Siva et al. [3], conducted a study to examine the effect of cryogenic treatment on the enhancement of wear resistance of 100Cr6 bearing steel. The study also aims to reveal the underlying mechanisms responsible for the enhancement of wear resistance by deep cryogenic treatment. It was found that the wear resistance was increased by 37% due to DCT when compared with that of conventional heat treatment (CHT). However, DCT may also be employed due to the increased benefits reported in terms of wear resistance and compressive residual stress compared with SCT. The presence of dimples and fractured surface are more in conventional heat treatment than the cryogenic treatment reported by Bensely et al. [4]. Author reported the carbon clusters increases during the heat treatment the cardide resulted improving wear resistance in the steels by Huang et al. [5]. JosephVimal et al. [6], conducted a Deep cryogenic treatment improves wear resistance of EN-31 steel. The bearing steel was selected and cryogenically treated to improve the physical properties of EN-31 steel. Before treatment EN-31 steel hardness is 18HRC hardness of untreated material is less. After done the heat treatments the hardness of the specimen is improved. Das et al. [7], the amount of carbide was decided the properties such as mechanical and wear resistance on D2 steel and also the carbide precipitation employed the behaviour of the steel materials. Harish et al. [8], conducted a comparative study of EN-31 bearing steel samples after CHT, SCT and DCT. The study reveals the presence of equi axed dimples and flat facets in the SCT specimen, micro cracks and wide ranged dimples in DCT specimen with respect to CHT samples. Hao-huai Liu et al. [9], investigated the characterisation on CrMnB high-chromium cast iron with the help of optical microscopy (OM), scanning electron microscopy (SEM) and X-ray diffraction (XRD), which can be improved due to the precipitation of carbides, the martensite transformation, and a refined microstructure resulting from cryogenic treatment [10]. The refrigeration of metals to improve their performance was generally classified as either shallow cryogenic treatment, sometimes referred to as subzero treatment, or deep cryogenic treatment (DCT) based on the treatment temperature. Fadare et al. [11], conducted an Effect of heat treatment on mechanical properties and micro structure of NST 37-2 steel. This study based upon the empirical study which means it is derived from experiment and observation rather than theory. The heat treatment operation is controlled heating and cooling rates but also determine microstructure and the grain size. The main aim of heat treatment is control the properties of a metal or alloy through the alternation of structure of metal or alloy. Harish S et al. [12], conducted a Micro structural study of cryogenically treated EN-31 bearing steel. The bearing steel was selected and cryogenically treated to improve the physical properties of EN-31 bearing steel. Before treatment EN-31 hardness is 18HRC hardness of untreated material is less. After done the heat treatments the hardness of the specimen is improved. Vadivel and Rangasamy [13], conducted Performance analysis of cryogenically treated coated carbide inserts. This performance analysis is produce micro structural view of two or more carbides. Images of micro structure taken at 50× magnification shows lot difference between conventional heat treatment and cryogenic treatment. Especially around the grain boundaries the fine precipitates carbides of size 0.1–0.3 micron. Based on the above literature survey, it was observed that the mechanical properties like hardness for the bearing steels. This paper has been carried out to study the effect and microstructure analysis of various heat treatments and cold treatment on EN-31 bearing steel and its influence on hardness of bearing steel.

2. Experimental investigation

2.1 Chemical composition

The optical emission spectroscopy (OES) was used to identify the chemical composition of the material. Moreover, the chemical composition was employed by using the spark analyser software to determining the weight percentage of the elements from the sample. Normally, the size of the specimen sized as per the standard used for the test is cylindrical, 20 mm in diameter and 10 mm in height. From the test which was identified the quantitative elements from the samples in ambient condition by optical spectrometer mentioned in the **Table 1**.

2.2 Experimental procedure

EN-31 bearing steel samples was machined as per ASTM standards for various mechanical tests. The experimental procedure adopted in the present work is shown in **Figure 1**. According to the flow chart, the composition of the raw material was analysed in optical emission spectroscopy (OES). The material considered for the study was obtained in the form of 6 mm diameter rod. The test was carried out separately for different treatments. Then the sample was subjected to conventional heat treatment and deep cryogenic treatment as per orthogonal array. Followed by the thermal treatment hardness test and microstructural study has been carried out. Finally the comparison of microstructure and the influence of the microstructure the hardness were studied.

2.3 Conventional heat treatment

The Conventional Heat treatment (CHT) was given to the EN31 bearing steel specimens as per the procedure prescribed in the ASM standards. The materials were subjected to hardening (austenitizing) at 850°C for 1 hour, followed by an oil quench, and tempered immediately after quenching at 200°C for 2 hours. The process graph for conventional heat treatment is shown in **Figures 2** and **3** respectively.

Figure 2 represented the procedure of conventional heat treatment on martensitic steels has employed at various heat treatment process starting from desired temperature at 850°C. The following quenching medium such as air, oil and water involve the martensitic steel which was decided the properties of steels behaviour. Moreover, the properties of steel tempered depend on the rapid cooling with various time and temperatures. The martensite steel surface became soft and ductile nature due to spheroidite dispersed present in materials during cryogenic treatment conditions. Most of the applications of steel materials resulted either pearlitic and bainitic nature during the production in lower cost. The EN 31 steels must tempered at microstructure of martensitic due to their significant brittleness. The microstructure and mechanical

Element	C	Mn	Si	S	P	Cr
Range (%)	0.95–1.10	0.25–0.40	0.15–0.35	<0.03	<0.03	1.40–1.70

Table 1.
Chemical composition of EN-31 bearing steel.

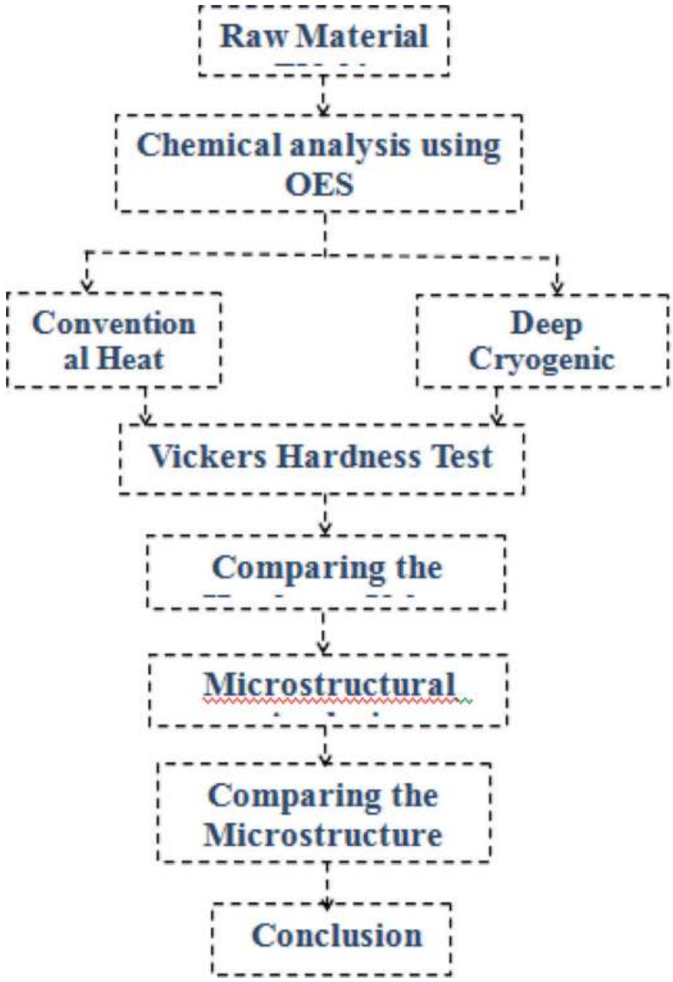

Figure 1.
Experimental procedure.

properties which are decided based on the elevated treatment and also being getting the retained of carbide in the materials. The samples revealed various microstructures such as Pearlite, Bainite and Martensite was decided the properties during the heat treatment process. The following heat treatment Annealing, Normalising, Quench Hardening, Tempering, and Austempering often used to change the properties of steels surface significantly. Moreover, the surface resulted solid strengthening also being desired the properties of the materials.

2.4 Deep cryogenic treatment

Deep cryogenic treatment was performed as per the factor level setting of the taguchi's OA. The chemical composition of the material should be confirmed by optical emission spectroscopy. Then the samples for mechanical testing were as machined as per the ASTM standard and the machined samples were subjected to hardening at 850°C for 1 hour, followed by a rapid oil quench, and treated in an A.C.I.CP-200vi cryogenic treatment processor(Applied Cryogenic Inc., Burlington, MA, USA) as required for the Taguchi orthogonal array. The cryogenic processor consists of a treatment chamber, which is connected to a liquid nitrogen tank through an insulated

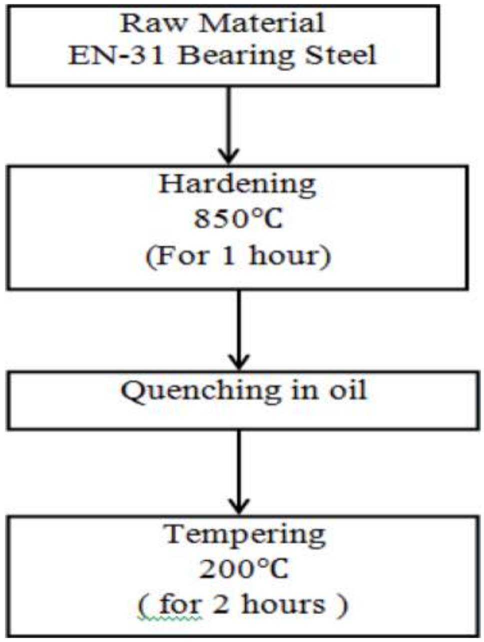

Figure 2.
Process chart for conventional heat treatment.

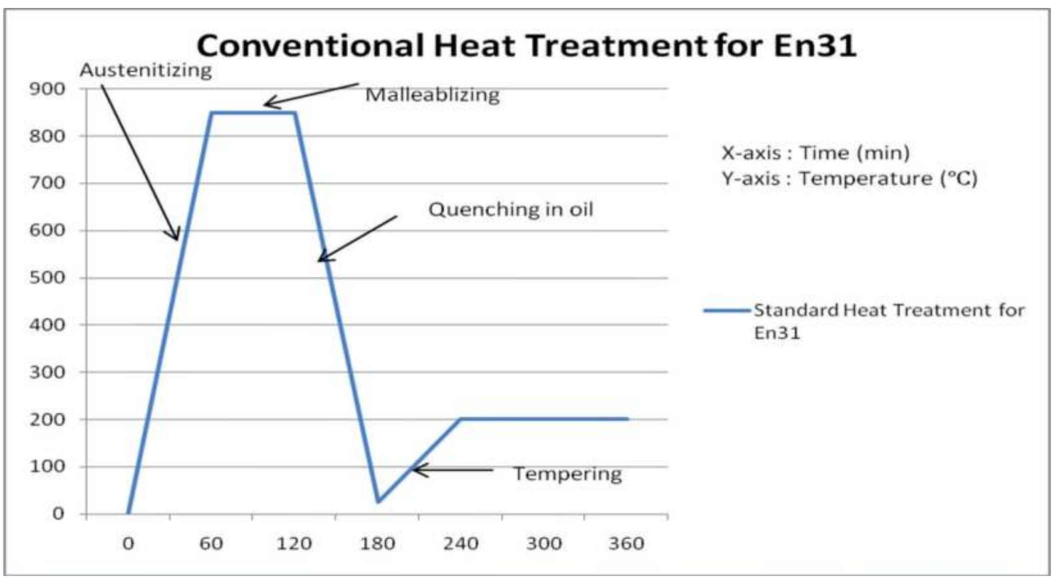

Figure 3.
Process graph for conventional heat treatment.

hose. The thermocouple inside the chamber senses the temperature, and accordingly the proportional –integral-derivative (PID) temperature controller operates the solenoid valve to regulate the liquid nitrogen flow. The liquid nitrogen passes through the spiral heat exchanger and enters the duct leading to the bottom of the chamber as nitrogen gas. The blower at the top of the chamber sucks the gas coming out at the bottom and makes it circulate inside the chamber. The programmable temperature controller of the cryogenic processor is used to set the cryogenic treatment parameters, as per the Taguchi orthogonal array. Following DCT, the samples were prepared for 2 h and the performance characteristics of the samples were evaluated by conducting a reciprocating hardness test. At the time of the experiment, as per the Taguchi factor-level settings, three replications were performed for each test. The cryogenic processor consists of a treatment chamber, which is connected to a liquid nitrogen tank through an insulated hose. The thermocouple inside the chamber senses the temperature, and accordingly the proportional-integral-derivative (PID) temperature controller operates the solenoid valve to regulate the liquid nitrogen flow. The liquid nitrogen passes through the spiral heat exchanger and enters the duct leading to the bottom of the chamber as nitrogen gas. The blower at the top of the chamber sucks the gas coming out at the bottom and makes it circulate inside the chamber. The programmable temperature controller of the cryogenic processor is used to set the cryogenic treatment parameters, as per the Taguchi orthogonal array. Following DCT, the samples were prepared for 2 h and the performance characteristics of the samples were evaluated by conducting a reciprocating hardness test. At the time of the experiment, as per the Taguchi factor-level settings, three replications were performed for each test. DCT Process factors and their levels were shown in the **Tables 2** and **3**.

2.5 Specimen preparation and microstructure analysis

When grinding manually, the specimen should be a moved back and forth across the paper to allow for even wear. Between grinding steps, the specimen should be rotated 45O-90O. The moulded specimens were first polished using emery paper of grits 80, 120, 200, 600, 800 and followed by polishing using on a rotating linen disc,

Symbol	Factors	Level 1	Level 2	Level 3
A	Cooling rate (°C/min)	1	1.5	2
B	Soaking temperature (°C)	−130	−150	−185
C	Soaking period (hr.)	24	36	48
D	Tempering temperature (°C)	150	200	250

Table 2.
DCT Process factors and their levels.

Exp. No	Cooling rate (A) (°C/min)	Soaking temperature (B) (°C)	Soaking period (C) (hr.)	Tempering temperature (D) (°C)
1	1	−130	24	150
2	1	−150	36	200
3	1	−185	48	250
4	1.5	−130	36	250
5	1.5	−150	48	150
6	1.5	−185	24	200
7	2	−130	48	200
8	2	−150	24	250
9	2	−185	36	150

Table 3.
Orthogonal array.

and finished on a velvet cloth using alumina powder and water as a coolant. After polishing, the moulded samples were subjected to microstructure analysis. A microstructural study was conducted to explain the improvement in hardness of EN-31 bearing steel subjected to CHT and DCT. The samples were prepared as per the ASTM E3-01 for metallographic examination. These samples were etched with 2 vol% Nital and dried in air. The etched samples were examined using Metallurgical microscope at 50× magnification to study the changes in hardness. High resolution digital micrographs were taken randomly at different regions of the specimens.

3. Results and discussion

3.1 Chemical composition

The measured values of the sample were tabulated in the **Table 4**. The result of the chemical analysis confirms the chemical composition of EN-31 bearing steel.

With the above results of chemical composition it is proved that the selected material is EN-31 alloy bearing steel, according to BS 450 ASTM standard.

3.2 Vickers's hardness test

The result obtained from the Vickers's hardness of the EN-31 alloy bearing steels is given as below in the **Table 5**. From the results of Vickers hardness test, the hardness value of Conventional Heat Treatment sample is compared to the hardness value of

Element	C	Mn	Si	S	Ph	Cr
Measured value (%)	0.97	0.27	0.28	0.002	0.006	1.43

Table 4.
Chemical composition of EN-31 Bearing steel.

Process	Exp. No	Vicker's hardness			Mean
		Y1	Y2	Y3	
CHT	I	698	696	706	700
DCT	1	866	853	854	858
	2	850	845	843	846
	3	864	854	864	861
	4	851	839	850	847
	5	838	837	840	838
	6	804	802	800	802
	7	850	857	863	857
	8	784	786	799	790
	9	846	864	846	850

Table 5.
Hardness values of EN-31 bearing steel.

Deep Cryogenic Treatment samples using orthogonal array. It is significantly increase in the hardness of DCT sample. It is clear from the table that the raw material has the lower hardness.

The DCT samples have the highest hardness when compared to SCT and CHT samples. DCT followed by tempering improves the hardness by 14% and sub-zero treatment followed by tempering improves hardness by 13% when compared to the CHT. The untemper structure has the highest hardness in all the cases but the material is more brittle due to presence of untemper martensite which is seen in the microstructure. Martensite is a highly supersaturated solid solution of carbon in iron. Hence, tempering should be done to reduce the brittleness by scarifying some hardness and tensile strength to relieve internal stresses and to increase toughness and ductility. It results in a desired combination of hardness, ductility, toughness and structural stability. During tempering, marten site rejects carbon in the form of finely divided carbide phases. The end result of tempering is a fine dispersion of carbides in the iron matrix, which bears little structural similarity to the original as-quenched martensite.

3.3 Microstructure analysis

A microstructural investigation was carried out to identifying the possible mechanism brought in by the thermal treatment in improving the hardness of the EN-31 bearing steel. The hardness values of CHT and DCT were compared by using the microstructure view of the CHT and DCT samples. The micrograph view of the CHT sample is given in below **Figure 4**. The CHT sample was prepared for metallographic examination as per ASTM E3-01. The specimen was investigated using a Metallographic microscope at 50X magnification to study the changes influence the hardness. The CHT sample exhibited non-uniform distribution of large, elongated carbides on the tempered martensite matrix and a notable amount of retained austenite in the specimen. The mean hardness value of CHT sample is 700 HV. The optimised cryotreated samples were prepared for metallographic examination as per ASTM E3-01. Then the specimen were subjected to factor level setting like cooling rate, soaking period, soaking temperature and tempering temperature for experiment-1 in OA table. After the specimens were investigated using a Metallurgical microscope at 50× magnification to study the changes that influence the hardness. The micrograph of the optimised DCT samples shown given below. The micrograph of the optimised DCT sample-01 revealed a marked reduction in the amount of retained austenite and an increase in the amount of fine secondary carbides. At cryogenic temperature, the amount of retained austenite decreased, resulting in a greater amount of tempered martensite; the increased amount of martensite led to more uniform distribution of fine carbides throughout the structure. The precipitation of fine carbides and the transformation of retained austenite to martensite showed considerable variations in the hardness of the optimised DCT sample-01 compared to the CHT samples. The mean hardness value of this sample is 858 HV and 18.43% improvement in the hardness compared to CHT. The micrograph of the optimised DCT samples shown given below. The optimised cryotreated samples were prepared for metallographic examination as per ASTM E3-01. Then the specimen were subjected to factor level setting like cooling rate, soaking period, soaking temperature and tempering temperature for experiment-2 in OA table. After, the specimens were investigated using a Metallurgical microscope at 50× magnification to study the

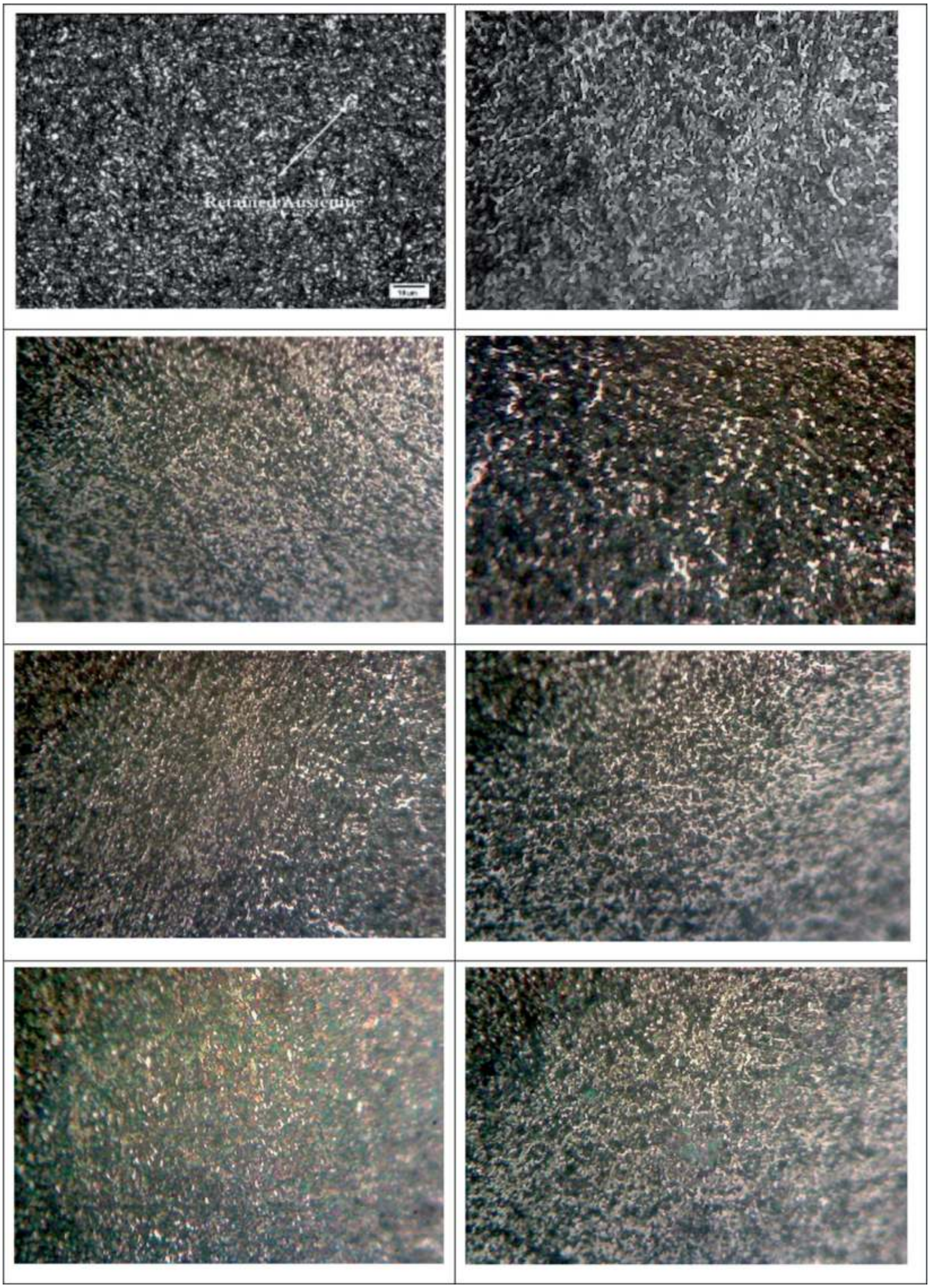

Figure 4.
Microstructure of the optimised DCT sample at 50× magnification.

changes that influence the hardness. The CHT sample exhibited non-uniform distribution of large, elongated carbides on the tempered martensite matrix and a notable amount of retained austenite in the specimen. The mean hardness value of CHT sample is 700 HV.

The optimised cryotreated samples were prepared for metallographic examination as per ASTM E3-01. Then the specimen were subjected to factor level setting like cooling rate, soaking period, soaking temperature and tempering temperature for experiment-1 in OA table. After the specimens were investigated using a Metallurgical microscope at 50× magnification to study the changes that influence the hardness. The micrographs of the optimised DCT sample-01 were shown in below. The SEM micrograph images found the wear debris of the CHT samples as shown in Figure has large platelets and flake shaped particles worn out and further determined the particle size which is comparatively smaller than the optimised DCT samples. The SEM micrographs were seen the worn morphology DCT samples considerably smoother than that of the CHT samples at significant loading conditions. Further it shows the SEM images more delamination lips and small considerably cracks could be seen in the CHT samples than the DCT samples. The precipitation of fine carbides and the transformation of retained austenite to martensite showed considerable variations in the hardness of the optimised DCT sample-01 compared to the CHT samples. The mean hardness value of this sample is 858 HV and 18.43% improvement in the hardness compared to CHT.

The micrographs of the optimised DCT sample-02 were shown in below. The optimised cryotreated samples were prepared for metallographic examination as per ASTM E3-01. Then the specimen were subjected to factor level setting like cooling rate, soaking period, soaking temperature and tempering temperature for experiment-2 in OA table. After, the specimens were investigated using a Metallurgical microscope at 50X magnification to study the changes that influence the hardness. The mean hardness value of this sample is46 and 17.23% improvement in the hardness compared to CHT. The micrograph of the optimised DCT samples was shown in below. The optimised cryotreated samples were prepared for metallographic examination as per ASTM E3-01. Then the specimen were subjected to factor level setting like cooling rate, soaking period, soaking temperature and tempering temperature for experiment-3 in OA table. After the specimens were investigated using a Metallurgical microscope at 50× magnification to study the changes that influence the hardness. The DCT samples examined the surface after the heat treatment revealed the carbides produce good mechanical properties which is the help of secondary carbides. Sometimes the surface was marked as a condition is homogeneity; it may be resulted in better surface during the treatment process. From the micrograph of the optimised DCT sample which was observed the fine secondary carbides in martensitic steels. The optimised DCT sample-03 compared to the CHT samples. The mean hardness value of this sample is 861 HV and 19.20% improvement in the hardness compared to CHT. The optimised cryotreated samples were prepared for metallographic examination as per ASTM E3-01. Then the specimen were subjected to factor level setting like cooling rate, soaking period, soaking temperature and tempering temperature for experiment in OA table. After the specimens were investigated using a Metallurgical microscope at 50X magnification to study the changes that influence the hardness. The micrograph of the optimised DCT sample revealed a marked reduction in the amount of retained austenite and an increase in the amount of fine secondary carbides. At cryogenic temperature, the amount of retained austenite decreased, resulting in a greater amount of tempered martensite; the increased amount of martensite led to more uniform distribution of fine carbides throughout the structure. The micrograph of the optimised DCT sample retained carbide dispersed on homogenous condition of the microstructure.

Moreover, the optimised DCT sample-7 compared to the CHT samples which is resulting the better hardness due to the transformation of retained austenite to martensite. The mean hardness value of this sample is 857 HV and 18.40% improvement in the hardness compared to CHT. The optimised DCT sample-08 compared to the CHT samples. The mean hardness value of this sample is 790 HV and 13.45% improvement in the hardness compared to CHT. The micrograph of the optimised DCT sample estimated the percentage of retained austenite fine secondary carbides. During the cryogenic temperature resulted fine carbides in the form of closed packed and hardened. Some sample of DCT has revealed carbides presented in homogeneity structure during the treatment process. Moreover, the carbides which are employed better mechanical properties due to the solid precipitation. The transformation of retained austenite to martensite due to precipitate the fine carbides in the surface of DCT samples significantly. Finally, the hardness of the optimised DCT sample-09 compared to the CHT samples. The mean hardness value of this sample is 850 HV and 17.54% improvement in the hardness compared to CHT.

3.4 Corrosion behaviour

Figure 5 shows the potentiodynamic polarisation curves for the bulk of the CHT and DCT samples in 1 M Na_2CO_3 solution. It can be noted that the DCT show lower Icorr than the CHT and SCT (Icorr for DCT is about one order the magnitude lower than that of CHT) indicating more general corrosion resistance. In this experiments were taken three samples showing the passive state conditions. The Figure resulted the current has moving the positive terminal is to identify the low corrosion tendency in martensite samples in the same potential. Moreover, the corrosion peak identified all three samples at passivation behaviour in a slow manner.

Sometimes the increasing the anodic scan limit to 2 V (versus Ag/$AgCl_2$ basic line) and breakdown potential is observed at almost the same potential (about 0.75 V). Normally the alkali media were resulted the properties of low corrosion resistance of martensite materials at conventional treatment especially for the deep cryogenic treatment (DCT). However, it seems the samples of CHT and DCT the passivation layer forms at slower rate than in the case of the SCT as observed with the lower corrosion currents.

For the En 52 valve steel material, the potential of the DCT specimen shows −0.485 mV and the CHT specimen shows −0.54 mV at the initial condition. The potential of the CHT specimen decreases continuously and reaches a stable value, and for the DCT specimen the potential decreases up to around 1300 sec and increases to some extent and gets stable at the end. The increase in the potential for the DCT specimen is due to the repassivation effect; this may be due to the presence of more chromium carbides in the martensitic structure. From **Figure 6** the initial potential for the 21-4 N valve steel at the CHT condition is −0.195 mV and at the DCT it shows a value of −0.205 mV. The potential value for both the specimen decreases continuously with time and the DCT specimen reaches a stable value at the end; the potential of the CHT specimen increases after around 1500 sec and reaches a stable condition. The higher potential value in the OCP curve indicates the higher corrosion resistance of the En 52 DCT and 21-4 N CHT specimen. The polarisation curves for the CHT and optimised DCT specimens of both the materials are shown in **Figures 6** and **7**. The corrosion potential Ecorr and corrosion current Icorr are determined by the Tafel extrapolation method, by carrying out scans in both the positive and negative directions.

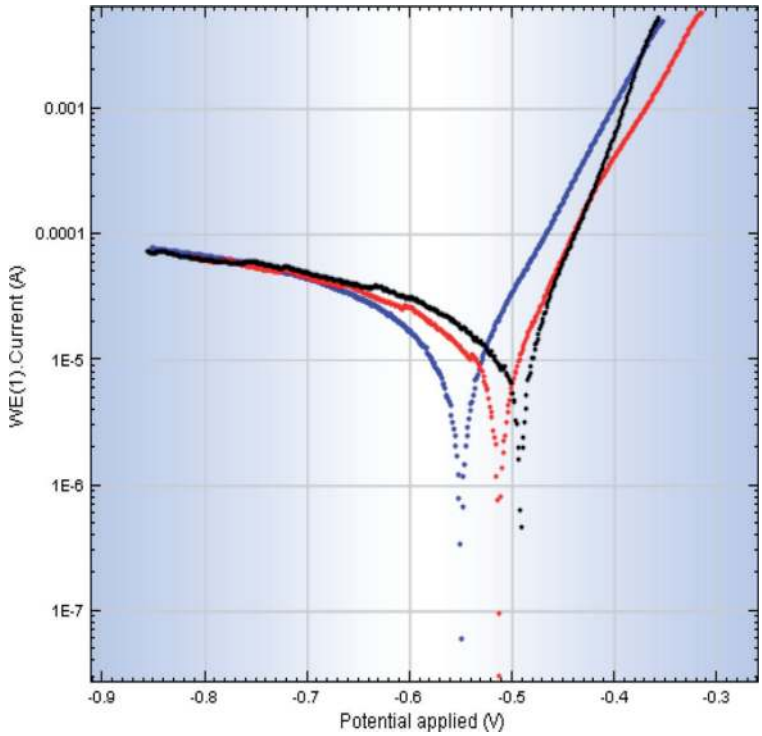

Figure 5.
Polarisation curves recorded in 1 M Na2CO3 solution at 10 mv/s for the Bulk of CHT and DCT samples.

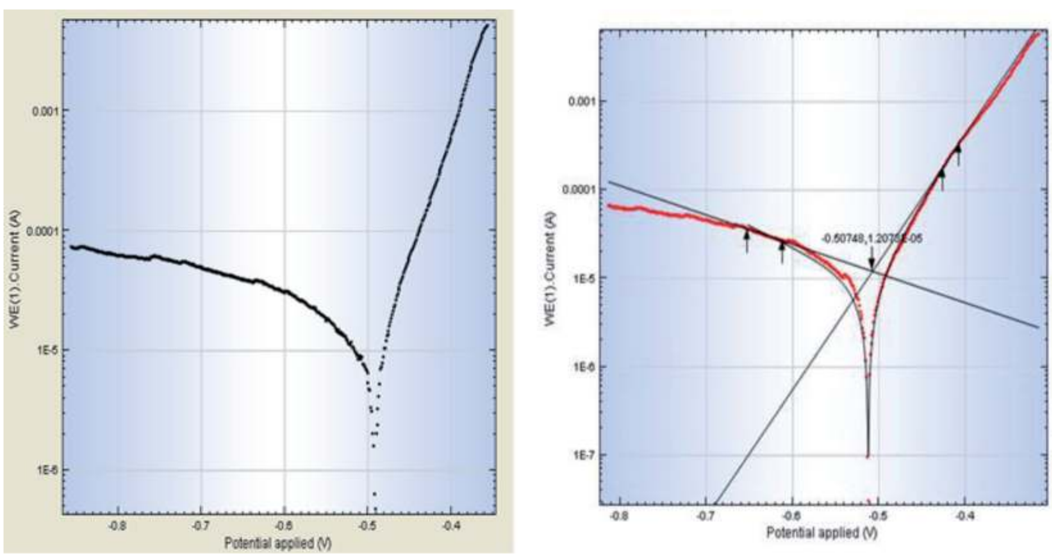

Figure 6.
Normal corrosion tafel plot for DCT samples.

4. Conclusion

The hardness of the EN-31 bearing steel almost varied after both CHT and DCT which is evident from the mean VICKERS'S hardness test. The microstructural analysis reveals that the precipitation of fine carbides and transformation of the retained austenite to martensite enhanced the hardness. The hardness of the DCT samples is

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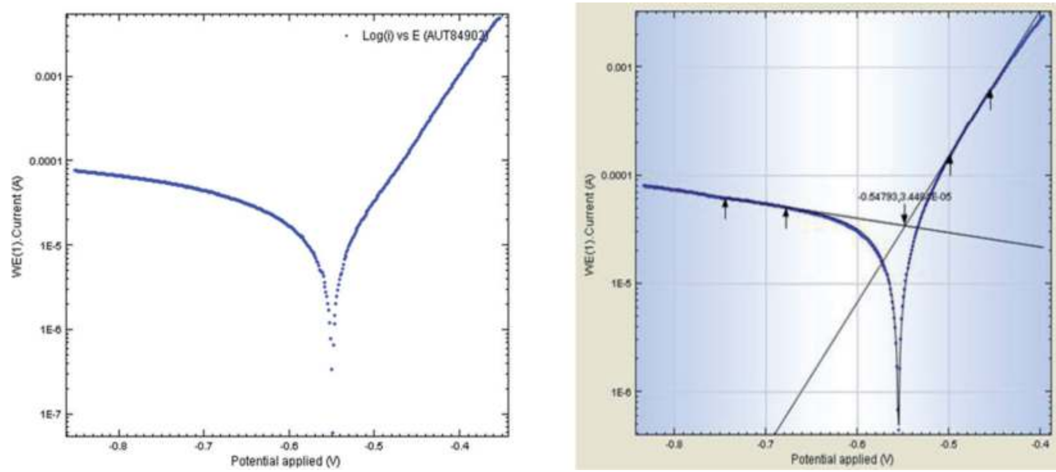

Figure 7.
Normal corrosion tafel plot for CHT samples.

higher than the CHT. The Images of microstructure were taken at 50X magnification source lot of differences between CHT and DCT. Fine carbides are found in that treatment CHT and DCT. It was very fining nature with reasonable hardness. The DCT seems to offer higher corrosion resistance than CHT samples.

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