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Wear evaluation and microstructure analysis of cryogenically treated AISI 440C Bearing steel

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Abstract

In this research work, two types of cryogenic treatment such as deep cryogenic treatment (-196°C) and shallow cryogenic treatment (-80°C) have been adopted for wear resistance to increase in AISI 440C bearing steel. This paper has been focused to increase Wear Resistance (WR) through deep micro structural analyses, and also attention has been made to correlate the microstructure with the wear character of Deep Cryogenic treated (DCT) specimens, Conventional Heat Treated (CHT) specimens and Shallow Cryogenic Treated (SCT) specimens. Micro structural examinations have been carried out in the specimens through Scanning Electron Microscopy (SEM), Energy Dispersive Analysis of X-ray (EDAX) and X-Ray Diffraction (XRD). Wear characteristics of AISI 440C bearing steel has been studied. The outcome of the research disclosed that the DCT specimens have higher wear resistance than SCT and CHT specimens. The effective wear mechanisms recognized were the constitution of white layers and delamination of white layers. The microstructures of the materials have been varied through heat treatment process. The modification of Secondary Carbides (SCs) precipitation characteristics and its reduction of retained austenite in the microstructure have been correlated with wear character and these are the liable mechanism to raise the wear resistance of bearing steels through DCT.

Key words: Bearing steel, Cryogenic treatment, Conventional heat treated specimen, Wear rate, Microstructure, SEM and XRD.

1. Introduction

Cryogenic treatment is a process which has been conducted for increasing the wear resistance of the bearing steels by increasing the amount of hard martensite through the transformation of soft retained austenite. It is an ancillary process which has been done immediately after hardening and prior to tempering process, which acts over entire section of the steel. During cryogenic treatment, conventionally heat treated specimens were cooled in a moderate cooling rate for selected temperatures (148-77K) and soaked for 12 - 24 hours and then it was heated at a uniform heating rate to reach the atmospheric temperature (Leskovsek et al.2006, Idayan et al. 2014). From several study of research, it has been promulgated that DCT undoubtedly increased the wear resistance (Akhbarizadeh et al. 2009, Podgornik et al.2016, Yusuf Arslan 2020, De Silva 2006) dimensional stability (Sona and Lomte(2005), Idayan et al.2016) of the bearing steels and reduction in residual stresses (Bensely et al.2008) induced during heat treatment than CHT and SCT. The heat treatment factors are responsible for the increase in life of the components which are manufactured through steels (Yong et al.2007).

Researchers generally suggested that DCT initiates retained austenite (γ_R) conversion into martensite in CHT specimen and it does improve the wear resistance of the steels (Satish Kumar et al.2017, Firouzdor et al.2008). The wear resistance of the steel has been increased through cryogenic treatment (Barron 1982). The cryogenic experiment was conducted on AISI D2 steel to found the effective mechanism involved for the increment of wear resistance through deep analysis of microstructure. The micro structural correlation with the wear of DCT specimen with reference to CHT and SCT specimens were reported. The mechanism accountable for the improvement in wear resistance of tool steels mainly depend on the precipitation properties of the secondary carbides and the exhaustion in amount of retained austenite by DCT and SCT (Das et al. 2009).The investigations of microstructure and wear behavior of Fe- Cr bearing steel under cryogenic treatment have been studied. The predicted wear resistance has been enhanced during cryogenic treatment due to favorable distribution of carbides and nominal recovery of martensite, instead of retained austenite removal (Fanju Meng et al.1994). The effective mechanism was used to liable for the increment in WR of M2 tool steel through micro structural analysis with and without the influence of cryogenic treatment (Huang et al.2003).

It has concluded that the WR of cryogenically treated tool steel has greater WR due to retained austenite available in the micro structure. The perforated hole and circularity have been investigated in tool steel (Arslan et al.2016). The role of DCT and SCT on the WR of case carburized steel (EN 353) was studied and it was found that the WR increased considerably than CHT. The major responsible factors are martensite transformation and fine distribution of carbides (Bensely et al 2006). Some experimental investigations were conducted on tool steels to change the characteristics after cryogenic treatment and it was stated that hardness, wear resistance and toughness have been increased through cryogenic treatment (Molinari et al.2001). The mechanical properties and microstructure of AISI 4340 steel were analyzed after cryogenic treatment and it was concluded that hardness and fatigue strength have been increased than CHT specimen (Zhiraifar et al.2007). The wear property of AISI D2 steel during sub-zero temperatures was examined. The wear resistance was improved due to retained austenite reduction, refinement in size, and increase in quantity of secondary carbides (Debdulal Das et al.2010). The precipitation characteristics of carbides of tool steels were studied after cryogenic treatment. The wear resistance was enhanced by the reduction of retained austenite and generation of nano-size fine η carbides (Young Mok Rhyim et al.2006).The importance of DCT on WR of 100 Cr6 bearing steel was examined and it was studied the basic mechanism acts for the WR enhancement. It was concluded that WR of DCT specimen enhanced by 37% (Sri Siva et al.2012). During DCT, amount of martensite presented was enhanced through the transformation of retained austenite to martensite and facilitation of carbide formation. These were provided the alloy with more refined matrix to produce fine grain strengthening which causes the WR. The improvement of WR in AISI 440C bearing steel after cryogenic treatment has been analyzed. This steel comes under the classification of martensitic stainless steel with high corrosion resistance. It was mostly adopted for making small bearings, chisels, ball, needle valves and surgical knives etc. The AISI 440C bearing steel withstands high temperatures without altering their properties. Hence, it can be used in high temperature applications. Carbon atoms are static at temperature below -50°C but carbon agglomeration takes place in the virgin martensite during heating above this temperature. Due to the increase in the density dislocation, the strain induces partial dissolution of the carbide phase makes virgin martensite enough ductile (Tyshchenko et al.2010). Three types of heat treatment were done in X153CrMoV12 to study the carbide precipitation. Quenching at 77 K or 123 K (-196 ° C or -150 ° C) at RT and deep cryogenic treatment, DCT. No fine carbide precipitation is observed after long-term holding at

cryogenic temperatures, in contrast to some previous studies. The transient $\epsilon(\epsilon')$ carbide was precipitated between 373 K and 473 K (100 ° C and 200 ° C) after quenching at room temperature and converted to cementite starting from 573 K (300 ° C) (Gavriljuk et al. 2014). The laser heat treatment process leads in the formation of a microstructure consisting of sub micronic ferrite / austenite grains, martensitic needles and secondary carbides, whereas the combined laser heat treatment and ultrasonic treatment contributes to the creation of an ultra-fine grain structure with SC grain boundaries (20 nm) and some martensitic feather areas. In dynamic conditions with respect to the initial surface after the laser heat treatment, ultrasonic treatment and combined laser heat treatment ultrasonic treatment processes, the altered layers show nearly double, triple and four times decrease in wear losses. Using the Abbott-Firestone curves and Kragelsky-Kombalov formula, the wear losses observed after short (15 min) tests correlate well with the modifications in experimental surface roughness R_a and bearing capability parameters respectively (Lesyk et al. 2017). In comparison with the initial state, wear losses of the treated specimens after long-term wear tests were decreased by 68, 41, and 77% at the laser heat treatment, ultrasonic impact treatment, and combined laser heat treatment + ultrasonic impact treatment processes, respectively. The wear losses are in good agreement with the reciprocal surface micro hardness HV and with the wear parameters after the long-term (45 min) studies, respectively, calculated using the Archard-Rabinowicz formula and the complex roughness-and-strength approach. Nano twins, thick dislocations net and dislocation cells with fine carbides were observed as result of laser heat treatment, ultra impact treatment, combined treatment respectively which were formed in the surface layers of the steel helped to increase the vickers hardness. Due to increase in hardness, wear resistance was improved (Lesyk et al. 2018). A nano scale grain structure of heterogeneous nature (α' and γ phases) was formed in the outmost surface layer simultaneously with the areas filled with networks of deformation twins and stacking faults cryo treatment. The minimum grain size of α' -martensite $D_{\alpha'}$ and austenite D_{γ} was respectively 25 and 45 nm, and twin thickness/spacing was of 60–120 nm. Both types of the microstructure contribute to the material strength and result in higher hardness of the cryo-UIT processed specimens (~ 5–5.66 GPa) compared to that of the argon-UIT processed ones (~ 4.3 GPa) (Vasylyev et al. 2018).

The present work describes wear evaluation and microstructure analysis of cryogenically treated AISI 440C Bearing steel.

2. Experimental details

2.1 Material Selection

The martensitic stainless steel was the work piece which its containing 0.93% of C, 16.94 % of Cr, 0.40 % of Mn, 0.74% of Si, 0.45 % of Mo and remaining was iron (Fe). The above composition was confirmed by optical emission spectroscopy. The blank of 10x10x10mm, ϕ 10x20 mm and 25x25x5 mm have been taken as the specimens for micro structural analysis, Dry sliding wear experiment and XRD analysis respectively. The specimens grouped as CHT, DCT and SCT subjected to have undergone respective treatments were exhibited in Fig.1.

Fig.1. Heat treatment cycle of AISI440 C bearing steel

In CHT, the specimens were preheated at 760°C. Then it was heated to austenite temperature at 1010°C for 60 minutes. For hardening, the specimens were oil quenched which was maintained at atmospheric temperature. Finally, the specimens were involved in tempering process at 200°C for about 30 minutes. The SCT and DCT process was conducted after hardening process. During SCT process the set of specimens were placed in a freezer at -80°C and soaking at the same temperature for 5 hours. Finally, tempering done at 200°C. During DCT process the set of specimens were cooled from ambient temperature to -196°C in a constant cooling rate of 1.25°C/min, after soaking of 24 hours, the specimens were bring back to ambient temperature through heating of 0.625°C/min at constant heating rate. Finally, tempering was done at 200°C.

2.2. Micro structural characterization

The micro structural examination was done by using on both optical microscope and SEM (JEOL, JSM-6390). Initially, the Bakelite moulds were polished for about 1mm depth by emery papers of 80,120, 200, 600, 800, and 1000 grit sizes. Then the moulds were polished in rotating liner disc. Finally, the specimens were polished by velvet cloth with white kerosene. Then the polished specimens were subjected to involve in micro structural analysis at 500X. The microstructure exhibits the carbide particles available in tempered martensite matrix. Carbide particles of size $>5\mu\text{m}$ are designated as Primary Carbides (PCs) and the carbide particles of size $\leq 5\mu\text{m}$ are designated as Secondary Carbides (SCs). The secondary carbides are further designated as the carbides of size $1\mu\text{m}$ to $5\mu\text{m}$ are Large Secondary Carbides (LSCs) and sizes of $0.1\mu\text{m}$ to $1\mu\text{m}$ were Small Secondary Carbides (SSCs). Image software was used for analysis of micrographs for finding of size, volume

fraction of PCs, LSCs, and SSCs, population density and inter-particle spaces of LSCs and SSCs. The X-ray diffractometer (make: SHIMADZU, made in Japan, Model: XRD 6000) with Mo- Cu radiation with 0.01 deg/min scan rate was used. The volume of retained austenite (γ_R) was estimated as per E975-00 ASTM standard with the diffraction peaks taken into consideration.

2.3. Hardness measurement

The micro and macro hardness values of CHT, SCT and DCT specimens were found by Vickers indentation technique at 50Kgf (0.5N) and 60Kgf (589N), respectively. During micro hardness measurement, the determined value was decided by SCs even though the indentations of the matrix were carefully made to avoid the PCs. At least 50 readings have been taken to find the mean value of micro hardness and 10 readings have been taken for finding the macro-hardness.

2.4. Evaluation of wear behavior

Dry sliding wear tests were conducted by using through pin-on-disc apparatus (TR 20LE: DUCOM made in India) as per G99-05ASTM standard. Specimens of $\phi 10 \times 20$ mm were used as static pins. Moreover, alignment of circular profile was very easy as compared to other profiles. The disc was a rotating member having hardness of 61HRC with the roughness value of $R_a < 12.0 \mu\text{m}$. The material of the disc is IN600 whose hardness is 65HRC. Pin material is martensite stainless steel whose hardness is 45HRC. By using $1 \mu\text{m}$ diamond paste, the contact area of the pins were polished and cleaning was done by using acetone in ultrasonic cleaner, finally the specimens were dried. Dry sliding wear test was executed for three distinct normal loads (F_N): 10 kgf (98.1N), 12 kgf (117.7 N) and 14 kgf (137.3N), constant linear sliding velocity (S_V) of 1.6m/s in dry condition, the atmospheric temperature of 26°C and 62 % relative humidity. Minimum contact pressure applied over pins was 7.80GPa, 9.36GPa and 10.92 GPa. The wear test continued up to 1000m sliding distance. The sliding distance as 1000m was considered for wear test of aluminium (Alagarsamy et al. 2019). At least three results of the tests have been taken under identical conditions to found the mean wear rate (W_R)

2.5. Analysis of worn surfaces, wear debris and sub surfaces

Micro hardness of worn specimens were measured by Vickers micro hardness tester with 50gf (0.5N) applied load. Minimum fifty indentations were taken for each surface area for the

calculation of mean hardness value. A minimum of one specimen at normal loads has been taken for the analysis of the sub-surface features through SEM. Before SEM analysis, the size of the specimen was reduced through cutting the specimen using wire cut EDM. Examination of the sub-surface of worn specimen discloses the existence of white layer. Evaluation of white layer thickness has been done from the sub-surfaces micrographs of each worn specimen by image analysis.

3. Results and Discussion

3.1 Micro structural evaluation

Micrograph of DCT obtained from optical microscopic examination was shown that the big elongated, white zones of dendritic PCs which were non-uniformly distributed and uniformly distributed SCs in spherical shape in tempered-martensite matrix (Fig.2).

Fig.2. Optical micrograph of DCT specimen

The optical microstructure of SCT and CHT are same as DCT specimens. It was shown that PCs were in white phases and SCs in either a small black phases. The variation in size, distribution and amount of SCs in DCT, SCT and CHT are clearly visible in SEM micrograph and EDAX (Fig.3). Fig. 3 clearly indicates that the population densities of SSCs of DCT specimen were comparatively higher than CHT and SCT specimens.

The XRD profiles of DCT, SCT and CHT specimens of AISI 440 C bearing steel were exhibited in Fig.4. Compared to CHT, the SCT specimen has intensity diffraction with lesser peaks for austenite and its concurrent increment in the intensity diffraction peaks for martensite. Diffraction peaks of austenite almost vanished in DCT specimen and it was concluded that the retained austenite presented in CHT specimen was fully converted into martensite during DCT. The EDAX profiles of CHT, SCT and DCT specimens were exhibited in Fig.5.

Fig. 3. Micrographs of SEM (a) CHT(c) SCT and (e) DCT specimens and EDAX graph of the specimens (b) CHT, (d) SCT and (f) DCT

Fig. 4. XRD profiles of bulk specimens and in vertical manner exhibits the 2θ positions of austenite (γ), martensite (α'), M_7C_3 , Cr_3C_7 carbides.

Fig.5. EDAX analysis of worn surfaces
(a) CHT (b) SCT and (c) DCT specimens at FN 137.3N

The amount of micro structural constituents was calculated and it was furnished in the table

1. The tempered martensite has been calculated by subtracting the available retained austenite and carbides. It can be inferred that there was a considerable decrease in the quantity of γ_R during SCT from CHT. There was a complete elimination of γ_R in DCT. The quantity of SCs and martensite have been accelerated both in SCT and DCT specimen due to control or almost vanishing of γ_R available in CHT specimen,

DCT was accelerated through SCs quantity by 52% and 42% over CHT and SCT respectively. The features of SSCs and LSCs for CHT, SCT and DCT specimens were indicated by bar diagrams in Fig.6. The precipitation behaviors of SCs have been modified by both SCT and DCT. However, the modifications were prevalent in DCT than SCT (Fig.6). DCT helps appreciably for the refinement of SCs, enhancement in population-density, quantity and reduction in mean inter particle spacing. Population-density of SSCs and LSCs of DCT was increased by 208% and 156% respectively to CHT, while the inter particle spacing of SSCs and LSCs have been reduced up to 41% and 38%, respectively through DCT over SCT.

Fig.6. Result of image analyses of CHT, SCT and DCT specimens (a) amount of SSCs LSCs (b) mean spherical-diameter of SSCs LSCs (c) mean population-density of SSCs LSCs and (d) mean inter particle-spacing of SSCs and LSCs.

3.2 Hardness variation

The readings of Vicker'shardness (HV_{60}) and micro hardness ($HV_{0.05}$) of specimens of DCT, SCT and CHT were tabulated in table.1. Macro-hardness values of SCT, DCT specimen was increased by 4.8% and 11.7%, respectively over CHT specimens. The Macro-hardness values of the specimen which undergoes various heat treatment was the key factor for the reduction of soft γ_R percentage and it was definite increase in quantity of tempered martensite and hard SCs (Table.1). The micro hardness was noticeable evidence in the matrix strength because of impact of SCs, particularly SSCs.It is obvious that the harder surface also leads to a higher resistance to deformation to prevent the particles from pressing into it and preventing it from being worn out, adhering to the softer surface. In turn, the thickness of the compound layer affects the behavior of the wear rate [44].

Table1. Phase compositions and Hardness results

3.3 Wear Resistance and Wear Rate

The volume of material lost per meter sliding distance with respect to uniform wear region has been taken for calculating the wear rate (W_R). The W_R calculated for DCT, SCT and CHT specimens during tests which were conducted in range of normal loads (F_N) from 98.1N to 137.3N (with reasonable contact pressure of 7.80 to 10.92GPa) were shown in Fig.7. These results infer that W_R of CHT, SCT, DCT were ascending for any F_N . Due to the maximum hardness attained by DCT, the wear rate was less compared to the other two heat treatment process. At $F_N = 140\text{N}$, the wear rate for DCT, SCT and CHT specimens are $0.11 \times 10^{-9} \text{ mm}^2/\text{m}$, $0.142 \times 10^{-9} \text{ mm}^2/\text{m}$ and $0.178 \times 10^{-9} \text{ mm}^2/\text{m}$ respectively. Wear resistance decreases 33.3 % sharply for DCT when load changes from 100N to 140N. From Fig.7, it was confirmed that wear resistance for DCT has more influence towards wear rate. The above statement was coincides with few of the earlier reports. The results in Fig.7 also accept that was an increase of W_R in linear manner with increasing of F_N for each type of specimen. These results acknowledge the Archard law of Wear (Archard 1953), which has been expressed as

$$W_R = C \frac{F_N}{HV} \quad (1)$$

Where, W_R is wear rate in (m^3/m), F_N is normal load (N), HV (N/m^2) is hardness of the Pin and C is a non-dimensional parameter wear coefficient. The inverse of C in the Eq. (1) is termed as WR of the given material (Rabinowicz 1995). The specimens with different values were arrived and exposed in Fig.8. It was clearly shown that for each specimen, the type of variation of WR and F_N was non-linear type with W_R and F_N (Fig.7). From Fig.8, WR was varied in the order of DCT, SCT and CHT for any F_N . There was a considerable increase of WR in DCT and minimum increase in SCT.

Fig. 7. Comparison of measured Wear Rate with load F_N for the specimens involved different treatment.

Fig. 8. Comparison of measured Wear Resistance with load F_N for the specimens involved different treatment

3.4 Morphology of Worn-Surfaces and Wear-Debris

Fig.9 illustrates the micrograph (SEM) of worn-surface and wear-debris of CHT, SCT and DCT pins tested at $F_N=137.3\text{N}$ and 98.1N . Worn surfaces were coarse and metallic with fracture ridges. Wear debris reveals shiny and metallic nature with a platelet or flake shape. The EDAX (Fig. 5) analysis results deduce that the composition admissible for the worn-surface and wear-debris were same as the base material. The silicon traces which has been displaced from the rotating steel disc to the pin surface by adhesion. The wear track also reveals some removal of material by abrasion.

The study of morphology of worn surfaces and debris infer that for all the specimens, the basic factor responsible for occurrence of wear was heavy delamination for all testing conditions (Amini et al.2016). The micrograph (Fig.9) disclosed that in higher F_N , the amount of damage to worn surface and the size of generated debris got increased in the order of DCT, SCT and CHT.

Fig.9. SEM micrograph of worn surface area of DCT (e, f), SCT (c, d) and CHT (a, b) specimens tested at F_N 98.1N (a, c, e) and 137.3 N (b, d, f) in sets indicates the wear debris produced in the uniform wear zone to the respective specimens and the respective test conditions.

3.5 Sub surface Features

The sub-surfaces presents a featureless region called as white-etched layer (Liang Tang et al.2020, Wang and Lei (1996)). This layer is not affected by any metallurgical etchants commonly available and it appears in white colour in optical microscope. The result shows that the white layer comprises of cracks, which starts beneath a critical distance of the worn-surface and propagates parallel to the worn-surface. The sub-surfaces tested at $F_N=98.1$ and 137.34N of CHT specimens have been compared and they have suggested that rise in F_N was responsible for the formation of many layers with parallel cracks.

The sub-surface through the variation in white layer thickness (d_{WL}) happened due to the nature of heat treatment. Thickness of the white layer (d_{WL}) was found through image analysis. The amount of hardness was increased due to wear (ΔH_W). It was found by the differences in hardness between worn specimen (H_{ws}) and unworn specimen. The values of d_{WL} , H_{ws} and ΔH_W are shown in Table2. These results also infer that:

- The values of H_{WS} , ΔH_W and d_{WL} for all the specimens were improved due to the increase in F_N
- For any constant F_N , the values H_{ws} for CHT, SCT and DCT are ascending; nevertheless, the ΔHW values are descending.

From the observation, it was disclosed that in alike wear conditions, the wear rate is higher in the sequence of CHT, SCT and DCT.

- The d_{WL} values of CHT specimens are considerably higher when compared to DCT specimens, and the d_{WL} values of SCT lies in between CHT, and DCT.
- H_{ws} and d_{WL} values were shown in Table 2 have better correlation with calculated wear properties (Figs.7, 8)

The effort has been taken to correlate the microstructure with wear of DCT, SCT and CHT and through the complete elimination of γ_R , amount of SCs available in the microstructures and white layers presented in the sub-surface of worn specimen. During this study, in conventionally, the first two factors like γ_R and SCs have been taken in to account for the improvement of WR by SCT and DCT and the calculation of white layer is restricted .For sub-sections like conventional approach the white-layer has been taken into account.

4. Effects of microstructure

4.1 Effects of microstructure on wear behavior: conventional approach

For quantifying the improvement of WR through SCT, DCT than CHT the following formula was used.

$$(\alpha\%) = \left(\frac{WR^{DCT \text{ or } SCT}}{WR^{CHT}} - 1 \right) \times 100 \quad (2)$$

Where, the superscripts denote the type of treatment involved in the specimen. The α values have been found in all the specimens of with different conditions. By using these values the graph drawn with the applied load is shown in Fig.11, which delineates that within the selected range of F_N both SCT and DCT were improved the WR of AISI 440C steel as 44-20% and 92-42% respectively compared to CHT. The performances of SCT and DCT in the enhancement in WR of steels have been delineated by several researchers. The researcher

(Mohan Lal et al.2001) suggested that α values for CT (at 163K) and DCT(at 93 K) specimens are ≈ 5.0 and 29.0% respectively for AISI D3 steel at $F_N = 50\text{N}$ and $SV = 0.18\text{m/s}$. The result shown in Fig.10 was also concluding that if the Normal load F_N increases, the magnitude of α for DCT and SCT specimens decreases. This statement concurs with the report of another researcher (Joseph Vimal et al.2008) the α quantity of EN 31 steel after DCT decreased from 97% to 70% when F_N enhanced from 20.0N to 50.0N. The wear properties were correlated with the result of micro structural analyses (Table1and Fig.6) and it was shown that the generated microstructures of various heat treatments were the major phenomena for the rise of WR in steels.

It can be restated that the major factors accountable for the rise of WR during DCT than CHT and SCT were γ_R reduction, size modification and even distribution of SCs. The α values primarily based on F_N connected to uniform variation with W_R and F_N (Fig.7) or non-uniform variation of WR and F_N (Fig.8). It was obvious that the W_R of any type of specimen should increase in linear pattern with F_N or S_V got increased, the wear mechanism responsible under the testing conditions found was the delimitative wear which was induced by deformation (Wang and Lei (1996)). WR of any material was primarily relied on dynamical modifications due to its microstructure and material properties. The acquired results exhibited that, for any specimen, the value of H_{WS} increases only because of wear and degree of ΔH_W (Table 2).

Table 2. Effect of wear on the material

Magnitude of H_{WS} has fix the amount of strengthening of worn surface through plastic deformation observed in wear test and which was governed by strain hardening capability of the microstructure. Features acquired from the microstructure of the differently heat treated specimens disclosed that the soft γ_R quantity decreased (Table 1) with simultaneous increment in the quantity of SCs (Fig.6a), comprises with their refinement in size (Fig.6b), inter-particle space reduction (Fig.6d) and development in population-density(Fig.6c), in the sequence of CHT, SCT and DCT. Hence, the strain hardening capability of DCT, SCT and CHT specimens should be in ascending order and its being observed from the calculated values of ΔH_W for all F_N (Table 2). The specimens have been higher strain hardening capability with higher amount of augmentation of H_{WS} and also ΔH_W with F_N . Hence, the hardness variation between any two specimens reduces because of wear and decreases in hardness differences. For instance, the variation in micro-hardness values ($HV_{0.05}$) of DCT and CHT got reduced from 1.2GPa to 0.49 and 0.36GPa in the unworn condition and after wear tests of $F_N = 98.1$ and 137.3N, respectively. This statement clearly explains the

reduction in the degree of α of DCT and SCT specimens in higher values of F_N (Fig.11).

Fig.10. Comparison of calculated degree of enhancement of WR of DCT and SCT with CHT - function of F_N .

4.2 Influence of microstructure on wear: a white-layer approach

Evaluation of sub-surfaces, depiction of worn out surfaces and wear-debris disclosed that the white layer formation. White layer formed in hard steels due to adhesive sliding wear without using lubricants (Sipos et al.2008). Under dry condition, the high hardness and grain refinement of the white layer increase the strength of the machined surface, thereby improving the adhesive wear resistance. The residual stress does not affect the wear resistance (Fangyuan et al.2019). An attempt was made to correlate the determined values of dWL with wear properties and it was found that the magnitude of dWL increases with increasing F_N , in the order of DCT, SCT and CHT. The generation of white layer during wear was stated in various researches (Rigney and Glaeser (1978), Baumann et al.1996). The phenomenon's connected with the generation of white layer was found such as heavy plastic deformation in sub-surface, the heat generated due to friction, induced phase transformations which has undergone rapid heating, quenching and reaction of surrounding gas or liquid over the rubbing surface (Torrance and Cameron (1974)). It has been proved that the refinement of microstructure of the sub-surface of the worn specimen, particularly, grain size was up to an extent which was not determined optically done by above phenomena only or in combination. These sets of consequences were described in the optimum hardness and white layer. The formation of white layer happened mostly by the reaction of the rubbing surface with surrounding gas or liquid, which was generated during wear test by using the lubricant with hydro carbon (Schöfer et al.2001). Very high temperature to be generated at the contact area which was needed for the constitution of white layer 'by phase transformations' or by 'melting', was possible when wear tests were conducted at higher F_N and/or S_V . The quantity of resistive force developed against plastic deformation due to minimum wear in CHT than DCT specimens because of differences in microstructure (Table 1); at any F_N , the determined value of dWL was high for CHT compared to DCT (Table 2). Generation of high thickness white layer induces severe multi-layer cracking (Fig.11); this was liable for the fastest removal of over-sized wear-debris (Fig.9) from the sub-surface. Figure 9 shows the intensity of damage in worn surface and debris size was greatly influenced by sliding distance. The compound layer (white layer) was central for wear and load capacity. This type of plastic deformation occurs due to the long sliding distances. The predominant wear mechanism for short sliding distances was plastic deformation and abrasion wear, while plastic deformation

controlled the wear mechanism and, to a certain degree, oxidative wear for long sliding distances (Solis Romero et al.2013) The white layers were produced during wear test. It causes the delamination of materials and it's gradually affects the wear loss (Yang et al.1996). The hardness and yield strength of material have been increased due to the presence of white layer during wear test on AISI 52100 steel (Dae-Hyun Cho et al.2011).

5. Conclusions

The results of the experiment connected to micro structural analyses and wear analysis of CHT, SCT and DCT specimens of AISI 440C bearing steel and their relevant studies helps to understand the following conclusions:

- For every type of specimen, the W_R increases in a linear way with increasing normal load (F_N). The W_R of CHT specimen, SCT specimen and DCT specimens were kept in descending order. The W_R of CHT was higher when related to the other two type specimens. The W_R of SCT was lesser than CHT and higher than DCT specimen for all F_N . The calculated W_R for DCT, SCT and CHT specimens were $0.133 \times 10^{-9} \text{m}^3/\text{m}^{-1}$, $0.156 \times 10^{-9} \text{m}^3/\text{m}^{-1}$ and $0.189 \times 10^{-9} \text{m}^3/\text{m}^{-1}$ respectively.
- Amount of increase in WR through SCT and DCT over CHT varies from 44.0 to 20.0% and from 92.0 to 42.0% respectively, for F_N at 98.1N and 131.3N. The enhancement of WR of DCT specimen over CHT and SCT specimens reduces with raise in applied F_N . The feature of area of worn surface and the wear-debris produced in the examined wear conditions were shown that for each specimen, the delamination which was activated by deformation. The features of the subsurface of the worn specimens were recommended that because of fracturing of the white layer constituted by heavy plastic deformation of sub-surfaces.
- For every specimen, if F_N increases, worn surface hardness and white layer thickness have been increased. The quantity of raise in worn surface hardness and the white layer thickness mainly depends on the microstructure of the material.
- SCT greatly decreases the amount of retained austenite (γ_R) available and DCT completely vanished with simultaneous improvement in secondary carbides (SCs). Correlation of microstructures with wear characteristics undoubtedly confirms that the significant transformation in the SCs precipitation property and reduction in γ_R content which were the steering factors for WR enrichment of bearing steels through DCT.

Nomenclature

AISI	American Iron and Steel Institute
EDAX	Energy Dispersive Analysis X-ray
SCT	Shallow Cryogenic Treatment
CHT.	Conventional Heat Treatment
SCs	Secondary Carbides, size $\leq 5\mu\text{m}$
LSCs	Large Secondary-Carbides, $1\mu\text{m} < \text{size} \leq 5\mu\text{m}$
DCT	Deep Cryogenic Treatment
SSCs	Small Secondary-Carbides, Size ($0.1 \leq 1\mu\text{m}$)
PCs	Primary Carbides, size $> 5\mu\text{m}$
SEM	Scanning Electron Microscopy
XRD	X-ray diffraction
HV0.05	Vicker micro-hardness of load 50 gf (N/mm ²)
HV60	Vicker macro-hardness of load 60 kgf (N/mm ²)
HWS	Vickers micro-hardness observed from worn surfaces of load 50gf (N/mm ²)
C	Coefficient(Wear), a non- dimensional parameter
SV	Velocity of Sliding (m/s)
FN	Normal load (N)
α	Increase in wear resistance after DCT or SCT than CHT
γ_R	Retained austenite content
ΔHW	Difference between Vickers micro-hardness of worn-surfaces and unworn specimens at load 50gf (N/mm ²)
dWL	White layer thickness (μm)
α'	Martensite

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Fig.1. Heat treatment cycle of AISI440 C bearing steel

Fig.2. Optical micrograph of DCT specimen

Fig.3. Micrographs of SEM (a) CHT(c) SCT and (e) DCT specimens and EDAX graph of the specimens (b) CHT, (d) SCT and (f) DCT

Fig.4. XRD profiles of bulk specimens and in vertical manner exhibits the 2θ positions of austenite (γ), martensite (α'), M_7C_3 , Cr_3C_7 carbides.

Fig.5. EDAX analysis of worn surfaces
(a) CHT (b) SCT and (c) DCT specimens at FN 137.3N

Fig.6. Result of image analyses of CHT, SCT and DCT specimens (a) amount of SSCs LSCs (b) mean spherical-diameter of SSCs LSCs (c) mean population-density of SSCs LSCs and (d) mean inter particle-spacing of SSCs and LSCs.

Fig. 7. Comparison of measured Wear Rate with load F_N for the specimens involved different treatment.

Fig. 8. Comparison of measured Wear Resistance with load F_N for the specimens involved different treatment

Fig.9. SEM micrograph of worn surface area of DCT (e, f), SCT (c, d) and CHT (a, b) specimens tested at F_N 98.1N (a, c, e) and 137.3 N (b, d, f) in sets indicates the wear debris produced in the uniform wear zone to the respective specimens and the respective test conditions.

Fig.10. Comparison of calculated degree of enhancement of WR of DCT and SCT with CHT - function of F_N .

Table1. Phase compositions and Hardness results

Specimen	Phase values (vol. %)				Vicker's hardness value (GPa)	
	γ_R	SCs	PCs	TM	Macro-hardness (HV60)	Micro-Hardness(HV _{0.05})
CHT	10.43 ± 0.6	6.72 ± 0.2	6.79 ± 0.3	76.06	6.94 ± 0.03	8.92 ± 0.05
SCT	3.67 ± 0.4	7.23 ± 0.6	7.43 ± 0.3	81.67	7.27 ± 0.04	9.27 ± 0.06
DCT	Negligible	10.25 ± 0.4	6.54 ± 0.5	83.21	7.75 ± 0.05	10.12 ± 0.07

Table2. Effect of wear on the material

Specimens	Hardness of worn surface, <i>HWS</i> (GPa)		Rise in hardness due to wear, ΔHW (GPa)		Thickness of white layer, <i>dWL</i> (mm)	
	<i>FN</i> = 98.1N	<i>FN</i> = 137.3 N	<i>FN</i> = 98.1 N	<i>FN</i> = 137.3 N	<i>FN</i> = 98.1 N	<i>FN</i> = 137.3 N
CHT	10.93 ± 0.17	11.72 ± 0.24	1.37	2.51	28.1 ± 0.6	37.4 ± 1.3
SCT	11.24 ± 0.28	11.89 ± 0.23	1.13	2.24	21.53 ± 0.6	32.87 ± 1.2
DCT	11.73 ± 0.27	12.08 ± 0.20	0.64	1.75	17.69 ± 0.5	24.64 ± 0.6