

Residual Stress and Wear Studies of Deep Cryogenically Treated SAE 52100 Bearing Steel

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Abstract

The effect of cryogenic treatment on the enhancement of tensile residual stress resistance in the SAE 52100 bearing steel was studied using X-Ray diffraction technique. The tensile residual stress behaviour is assessed by X-Ray diffractometer as per the ASTM standard E2860-12. Refinement of carbide particle by deep cryogenic treatment (DCT) is often proposed as the improvement for the residual stress in the bearing steel. X-Ray diffractometer technique include the identification and quantitative analysis of crystalline chemical compounds (phase analysis and quantification, e.g. retained austenite determination), residual macro- and micro-stress analysis. It was found that the tensile residual stress was increased by +30Mpa due to deep cryogenic treatment when compared with that of conventional heat treatment (CHT). Moreover, the hardness of the DCT samples shows an improvement of 15% over the CHT samples. Copyright © VBRI Press.

Keywords: Cryogenic treatment, residual stress, wear, micro-hardness, corrosion.

Introduction

Bearing is one of the machine elements that constrain relative motion between moving parts to only the desired motion. Bearings allow smooth, low-friction motion between two surfaces loaded against each other. The motion can be either rotary (such as a shaft turning with in housing) or linear (one machine element moving back and forth across another). Bearings are the important component for all forms of rotating and reciprocating machinery. SAE 52100 bearing steel is used in the manufacturing of the bearings. Bearings are basically used to ease friction between moving parts. They also carry load in certain industries such as those that handle materials. The automobile industry is the major consumer of bearings followed by general engineering, heavy industries and railway. Since the bearing is subjected to high stress, these parts must be strong enough to withstand high stress, including fatigue and impact stress. At the same time, they have to maintain tensile strength, resist to corrosion and wear resistance. Common ball bearings are constructed by SAE 52100 chromium-alloy, high-carbon bearing steel, which is suitable for most applications. It offers satisfactory operation at temperatures approaching 125°C with no adverse effect on load capacity. X-Ray diffraction stress measurement can be a powerful tool for failure analysis or process development studies. Quantifying the residual stresses present in a component, which may either accelerate or arrest

fatigue or stress corrosion cracking, is frequently crucial to understanding the cause of failure. The failure that occurs in the bearing is mainly due to the high friction in the bearing balls. Some of the rolling bearings failures are due to rolling contact fatigue (RCF) and are defined as the mechanism of crack propagation caused by the near surface alternating load cycle within the rolling-contact bodies, which eventually leads to material removal by cracking or pitting/delamination Stewart and Ahmed (2002). Cryogenic treatment is a one-time permanent treatment and it imparts changes in the entire cross-section in the component. The basic cryogenic treatment consists, slow cooling of the component until the defined temperature, holding it for a particular time (freezing time) and then progressively heating it back to the room temperature. It is evident that cryogenic treatment improves the mechanical properties of ferrous and non-ferrous alloy and increase the performance life of the components. Cajner *et al.* (2009), Podgornik *et al.* (2009), Arockia Jaswin *et al.* (2011) and Liu *et al.* (2006) studied the effects of deep cryogenic treatment on the microstructure, hardening behaviour, and abrasion resistance of 3Cr13Mo1V1.5 high chromium cast iron. The results showed that deep cryogenically treated specimens after sub-critical treatment had an increase in hardness and abrasion resistance. This was due to abundant retained austenite transforming into martensite and secondary carbides precipitation. Huang

et al. (2003) studied the micro structural changes of M2 tool steel before and after cryogenic treatment. It was concluded that cryogenic treatment has facilitate the formation of carbon clustering and increase the carbide density in the subsequent heat treatment, thus improving the wear resistance of steels. Pellizzari *et al.* (2001) mentioned that cryogenic treatment improves the mechanical properties of material by allowing the molecules of the material to compress and expand in a uniform, thus reducing internal stress and thereby increasing the life of components. Alexandru Ailincăi and Băciu (1999) mentioned that the structure of cryogenically cooled metallic materials has a more uniform and dense microstructure than non-cryogenically treated samples. In addition, cryogenic cooling induced the formation of very fine carbides with less dimensions than 1 μ m, which occupy micro voids and contribute to an increase of the density. Barron (1974) conducted preliminary tests to determine the effect of cryogenic treatment on lathe tools, end mills, and zone punches which had been soaked in liquid nitrogen for 12 hrs and reported that tool life has been increased from 50% 200%. Wilson (1971) concluded that cryogenically treating slitter knives in paper mills increases the lifetime by more than 500%. The improvement in wear life is due to complete transformation of the retained austenite to martensite. Mohan Lal *et al.* (2001) conducted a study on the improvement in the wear resistance and significance of the treatment parameters in D3, M2 and T1 tool and die steel in various treatment conditions. It was found that cryogenic treatment imparted nearly 110% improvement in tool life. Ioan Alexandru and Vasile Bălăncea (2002) have concluded that the residual stresses play a very important role both during and after the cryogenic cooling. These stresses constitute the main cause for the continuation of the transformation of retained austenite to martensite, within the cryogenic field. It is surprising to see the fact that after cryogenic cooling the value of the residual stresses becomes even lower compared with classical quenching, either in water or in oil, to the ambient temperature. When quenched steel is cryogenically treated, the retained austenite will transform to martensite. Then the size of the component will have only a little expansion and the stability of the component will increase. Mack Alder and Olsson (2000) have observed that after case hardening, the gear tooth would experience compressive residual stresses, which are beneficial in maintaining an appreciable endurance limit. But these beneficial stresses are counter acted by detrimental tensile residual stresses in the core. The explanation of this phenomenon is based on the uneven volumetric expansion in the core and at the surface layer during phase transformation. Hence, it is an important consideration in the heat treatment of steel. Based on the above literature it is believed that cryogenic treatment improves the mechanical properties of the material and increases the life of the material. The

objective of the present work is to examine the distribution of tensile residual stress in SAE 52100 bearing steels due to cryogenic treatment, with respect to cryogenic treatment time, temperature, pre and post treatment condition of the bearing material.

Experimental investigation

The composition of the SAE 52100 was confirmed using optical emission spectroscopy (OES). The spark analyzer software is used in estimating the weight percentage of the elements in the sample. The specimens used for the test are cylindrical, 18 mm in diameter and 10 mm in height. The chemical analysis of the samples helps to identify the chemical composition of the materials used for preparing the samples to conduct various tests. The signal recorded by an optical spectrometer finally provides a quantitative elemental analysis of the metal samples. The composition of the SAE 52100 bearing steel was tabulated in **Table 1**.

Table 1. Chemical composition of SAE 52100 Bearing Steel.

| Material | C | Cr | Mn | Si | S | P | Iron |
|-----------|------|-------|------|------|-------|-------|------|
| SAE 52100 | 0.90 | 1.469 | 0.31 | 0.23 | 0.001 | 0.003 | Bal. |
| Wt. % | | | | | | | |

Thermal treatment

The samples were heat-treated as per the procedure prescribed in the ASM standards (1995). The machined specimens were formed in to two groups, Group 1 (Conventional heat treatment) and Group 2 (Deep cryogenic treatment) and subjected to two different treatment processes. The following thermal treatment was given to the SAE 52100 bearing steel specimens: The Group 1 samples were subjected to hardening (austenitizing) at 850 °C for 1 hr, followed by an oil quench, and tempered immediately after quenching at 200 °C for 2 hrs. The Group 2 samples were subjected to hardened were slowly cooled from room temperature to -185 °C in 3.5 hr at 1°C/min, soaked at -185 °C for 24 hrs, and then heated back to the room temperature. Finally, the samples were tempered immediately at 200°C temperature for 2 hrs.

The cryogenic processor consists of a treatment chamber, which is connected to a liquid nitrogen tank through a vacuum insulated hose. The temperature sensors inside the chamber sense the temperature and accordingly the 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 effectively inside the chamber and reduces the chamber temperature. The programmable temperature controller of the cryogenic processor is used to set the deep cryogenic treatment

parameters, which in turn, control the process parameters like soaking time, temperature and cooling rate. Through the data acquisition system, the deep cryogenic treatment processes are recorded and stored.

Residual stress analysis

The test samples for the residual stress analysis were machined as per the ASTM standard E2860–12 (2013). The samples were taken from each group and subjected to the residual stress analysis using X-ray diffractometer. X-Ray analyzer is based on solid state linear sensor technique. Diffraction effects are produced when a beam of X-rays of specific wavelength passes through the three dimensional array of atoms, which constitutes the crystal. Each atom scatters a fraction of the incident beams, and if the required conditions are fulfilled then the scattered waves reinforce to give a diffracted beam. Light weight, compact field proven performer, the XRD has set the standard for portable residual stress analysis instrumentation. Built in high voltage generator, self-contained re-circulating high efficiency liquid to air heat exchanger and the necessary electronics to run the goniometer and field stand. The samples are machined for X-ray diffraction test was fine polished with silicon carbide sheets and also cleaned by using electroplating process. Residual stress present in the SAE 52100 bearing steel subjected to CHT and DCT was compared in this study. The residual stress can be determined by passing high intensity chromium rays to the samples with the help of ceramic X-ray tubes. Residual stress analysis was determined by room temperature and it was calculated with the help of PROTO software. The residual stress was calculated as to given two input parameters namely Bragg angle and $\frac{1}{2}S_2$, based on the ASTM standard and directly giving the input parameter to the XRD.

Vicker's hardness test

Test specimens for the Vickers hardness test were machined as per ASTM standard E92-82 (2004). Totally three numbers of samples are taken from each group for hardness test. Hardness measurement was made with a 30kgf load with a dwell time of 15 seconds. The Vickers hardness test was carried out in such a way that three indentations were made in each test sample.

Reciprocatory wear test

The wear resistance enhancement in the conventionally heat treated and deep cryogenically treated bearing steel samples was experimentally measured, using a reciprocatory friction and wear monitor (DUCOM TR-281M-M4) by the weight loss method, as per the ASTM standard; G-133. Photograph of the Reciprocatory Friction and Wear Monitor (RFWM) as shown in **Fig. 1**.



Fig. 1. Photographic view of the Reciprocatory Friction and Wear Monitor.

Corrosion test

The salt spray corrosion test was carried out as per the ASTM B117 [17], to measure the corrosion resistance for the CHT and optimized DCT samples. Sample was taken from each group for conducting the test. This is the most commonly used method to determine the corrosion resistance of steel. The salt spray test procedure involves the spraying of a salt solution as very fine fog mist onto the samples being tested inside a temperature-controlled chamber for 24 hrs.

Characterisation study using optical microscope

The sample for the given test are prepared as per the ASTM standard E3-01 [19]. The SAE 52100 bearing steel specimens are cylindrically machined to a length of 10 mm and a diameter of 10 mm for the micro-structural studies. These specimens are subjected to the CHT and DCT. Then the samples were polished on SiC water proof abrasive papers of different grid sizes (200, 300, 600, 800, 1000, 1200) followed by fine polishing using diamond paste of 1 μ m size, with the help of an automatic polishing machine. Finally, the samples were mirror polished on velvet cloth, using white kerosene as a coolant. The polished surface was etched with 2% Nital solution, and then drying was performed with a jet of hot air for revealing the microstructure. The specimens were examined using the optical microscope (Nikon EPIPHOT 200) at 1000 X magnification.

Results and discussion

Residual stress analysis

The result obtained from the residual stress analysis of the bearing steel show an equivalent tensile residual stress of +84.6 Mpa for CHT samples and for DCT samples it has increased to +113.8 Mpa. It is observed

that the cool down process of deep cryogenic treatment causes the transformation of retained austenite to martensite, such stress improving behavior was due to the precipitation of fine carbides in specimens subjected to DCT with tempering the tensile residual stress level have been increased due to DCT when compared to CHT. Cryogenic treatment subsequent tempering is absolutely necessary to achieve fine carbide precipitation. Tempering improves tensile residual stresses, increases ductility, toughness and ensures dimensional stability.

The typical X-ray diffractograms of two specimens of the bearing steel of the optimised DCT and CHT samples are shown in **Fig. 2** and **Fig. 3**. It is seen that the (200) and (220) peaks of retained austenite (R) is more prominent in the case of the CHT sample than in the optimized DCT sample. The XRD spectrum of the cryogenically treated samples exhibits diffraction peaks only from the planes of martensite and carbide particles. It is estimated that the average volume fraction of the retained austenite in the conventionally heat treated sample is 14%, and in the case of optimized deep cryogenically treated samples, it is 3%. It is understood that the retained austenite percentage decreases due to the cryogenic treatment.

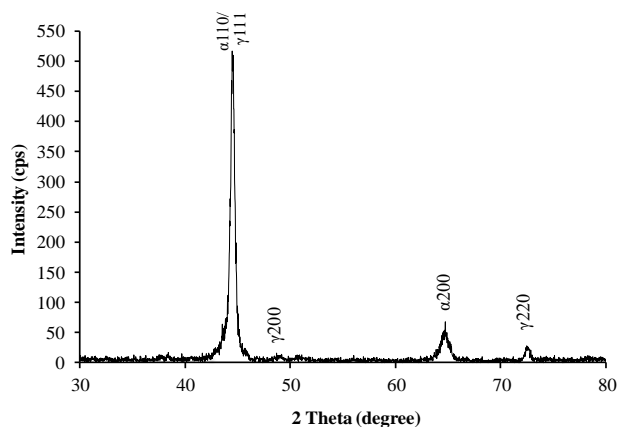


Fig. 2. X-ray diffraction pattern of the sample subjected to CHT.

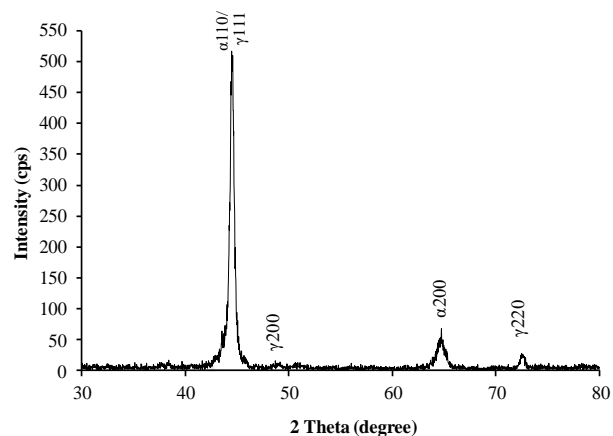


Fig. 3. X-Ray Diffraction Pattern of the Sample Subjected to optimized DCT.

SEM analysis of wear morphology on treated samples

The worn surfaces and wear debris generated at the end of the reciprocating wear test on conventionally heat treated and optimized deep cryogenically treated samples under different normal loads and constant frequency, have been examined for surface morphology using the scanning electron microscope, in order to identify the operative wear mechanisms of the reciprocating wear test. Moreover, **Fig. 4** presents the wear loss for various load conditions of 25 N, 50 N and 75 N at a frequency of 5 Hz. It can be inferred that the wear loss increases proportionately when the load increases. And it is clear that the wear resistance of optimized deep cryogenically treated samples are significantly higher than that of the conventionally heat treated samples at all loads. While assessing the wear behavior, in the most severe condition of the test (maximum load 75 N and frequency 5 Hz), it revealed that the bearing steel experienced a wear loss of 5.398 mg and 2.601 mg in the CHT and optimized DCT samples respectively. This study confirms that the wear resistances of the optimised DCT samples are always better than that of the CHT samples, in the whole range of test conditions. The difference in the wear loss between samples subjected to conventional heat treatment and optimised deep cryogenic treatment was more significant at higher normal load, and less significant at lower normal load. SEM micrographs (**Fig. 5**) showed that the morphology of the worn surface and wear debris of different treatment/testing conditions.

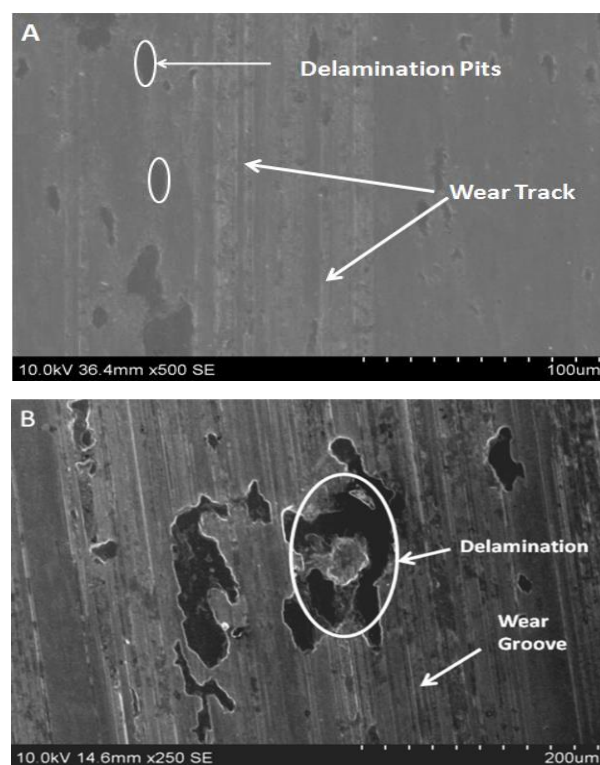


Fig. 4. SEM images of wear track morphology (A) DCT samples (B) CHT samples.

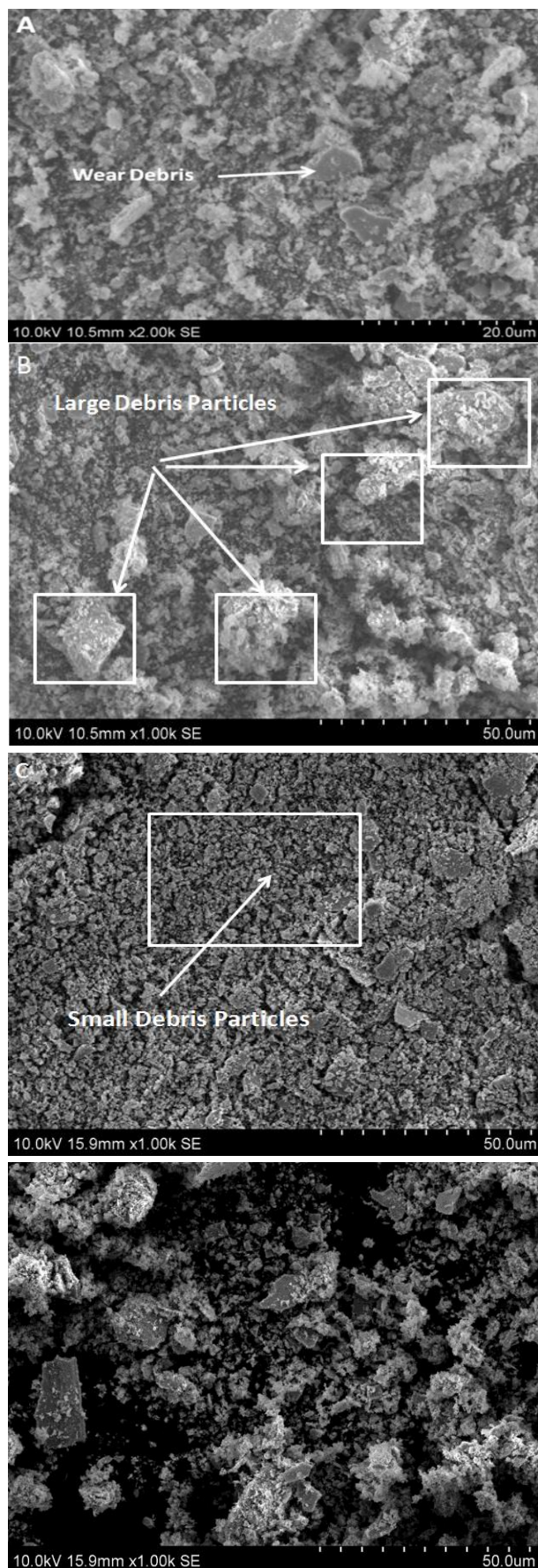


Fig. 5. Wear debris particles extracted by wear test at loading conditions (A), (B), (C) and (D).

The micrographs for all the three loading conditions viz; 25 N, 50 N, 75 N reveal that the worn surface of the optimised DCT sample is considerably smoother than that of the CHT sample. More dimples and deformation lips could be seen predominantly on the worn surface of CHT samples while such features are less pronounced on the worn surface of the optimised DCT samples. The above features of operating failure mechanisms are predominant at higher loads for CHT samples and such characteristics are not observed in optimised DCT samples. The worn surface of the CHT sample subjected to the test at normal load of 75 N has more surface damage and large delamination of carbides than those subjected to the normal loads 25 N and 50 N. The microstructure obtained by the SEM reveals that the severity of wear is high in the CHT sample and very low in the optimized DCT samples, for any selected applied load. The wear debris of the CHT sample seen in Figure 5 has large platelets and flake shaped appearance, which is comparatively smaller in the optimised DCT sample. Thus it is inferred that the advantage of optimised DCT are well pronounced at severe conditions which has far reaching significance in the context of product quality and productivity enhancement. These observations indicate that the wear mechanism for the CHT specimen induces severe plastic deformation, which causes the deformation lips and fractures, whereas that for the optimised deep cryogenically treated specimen induces predominantly low deformation lips and fractures. This confirms that the wear resistance of deep cryogenically treated specimen is significantly higher than that of the conventionally heat treated specimen.

Vicker’s hardness test

The hardness of the SAE 52100 bearing steel show an equivalent hardness of 725 HV for the CHT samples and for the DCT samples it has increased to 838 HV. The hardness of the DCT samples shows an improvement of 15% over the CHT samples. Improvement in the hardness is due to the conversion of retained austenite to the martensite. **Table 2** shows the hardness value of SAE 52100 bearing steel. From the table has reported the hardness been tested at room conditions CHT and DCT samples. It was observed the maximum hardness value of DCT is higher than CHT samples at cryogenic treatment. Due to precipitation of carbon consists in the samples DCT has shown better hardness than CHT samples at room conditions. The trails (I, II and III) were carried out in all the samples were revealed same value of hardness reported in the **Table 2**.

Table 2. Hardness Value of the SAE 52100 Bearing Steel.

| Sl.No | Condition | Vicker’s Hardness, HV | | | Mean |
|-------|-----------|-----------------------|----------|-----------|------|
| | | Trial I | Trial II | Trial III | |
| 1 | CHT | 724 | 726 | 724 | 725 |
| 2 | DCT | 842 | 836 | 836 | 838 |

Corrosion test

The salt spray test of the THT and DCT samples shows how much time to take the metal become rust. The DCT samples shows very less time to take corrode the given material compared to THT samples. The main reason is the precipitation of fine carbides due to deep cryogenic treatment. Carbides reduce corrosive nature and improve strength. The results are tabulated in the **Table 3**.

Table 3. Corrosion Test Result of the SAE 52100 Bearing Steel.

| Sl. No | Condition | Time (Hrs) | Percentage of Rusted (%) | Time (Hrs) | Percentage of Rusted (%) |
|--------|-----------|------------|--------------------------|------------|--------------------------|
| 1 | THT | 14 | 70 | 24 | 100 |
| 2 | DCT | 14 | 35 | 24 | 80 |

The Optical micrographs of the CHT and DCT samples are shown in **Fig. 6** and **Fig. 7**. The micrograph of the CHT samples shown in **Fig. 6** exhibited non-uniform distribution of large, elongated, white regions of primary carbides on the tempered martensite matrix. It is also observed that the presence of bainite (black area), austenite (white area), tempered martensite (grey area) and the white dots denotes carbide particles not dissolved during austenitizing.

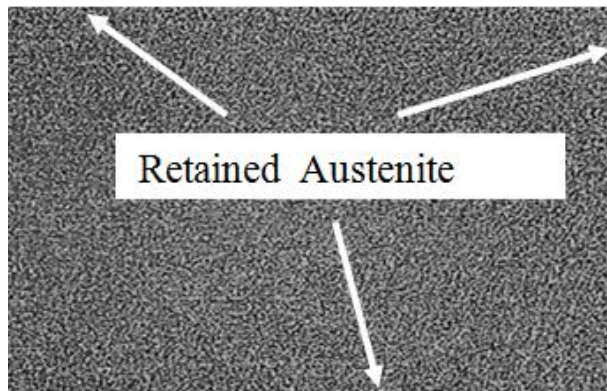


Fig. 6. Microstructure of the Sample Subjected to CHT.

The microstructure obtained after DCT, which is shown in **Fig. 7**, shows a martensitic structure and fine non-dissolved carbides. It is evident that occasional white patches of retained austenite have been detected more in the CHT than in the micrograph of the DCT samples. Moreover, a clear martensitic structure, characterized by the dispersion of spheroidal carbide is observed in DCT samples. It is also noted that the CHT sample shows large number of large-sized carbides when compared to the DCT samples. The variation in the characteristics of the carbide particles between CHT over the DCT samples is explained as follows. The transformation of retained austenite to martensite at deep cryogenic temperature, followed by prolonged holding, induces micro-internal stresses, which results in the formation of crystal defects such as dislocation and twins.

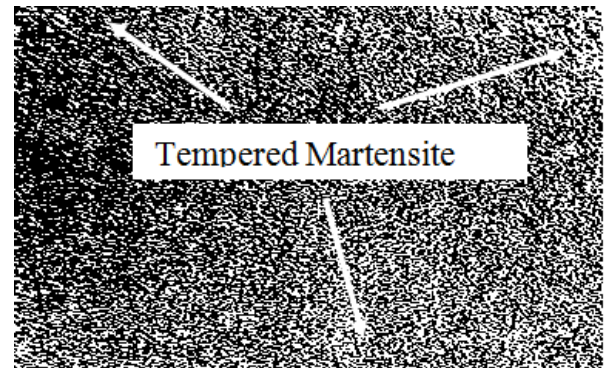


Fig. 7. Microstructure of the Sample Subjected to DCT.

From these results, it is suggested that the typical DCT and optimised DCT reduce the retained austenite substantially, as compared to the CHT. This is because the retained austenite is more unstable at lower temperature, and transforms into martensite. It is interesting to note that the application of deep cryogenic treatment in between conventional hardening and tempering increases the percentage of secondary carbides. At cryogenic temperature, the amount of retained austenite decreases, resulting in higher amount of tempered martensite; the increased amount of martensite naturally leads to higher amount of carbide precipitations. The microstructure of the typical DCT and optimised DCT specimens exhibits fewer primary carbides but more secondary carbides. In addition to small secondary carbides, the microstructure of the typical DCT and optimised DCT specimens reveal finer and more uniformly distributed Tempered Martensite Fine Carbides carbides than the CHT specimen. Finer chromium carbides are precipitated in the martensitic matrix of the typical DCT and optimised DCT specimens. These are also responsible for the improvement in the properties of the cryogenically treated bearing steel. There is a reduction of the retained austenite present in the typical DCT, and much more in the optimised DCT compared to the CHT specimens. The presence of tempered martensite with very little retained austenite, and the percentage of ultra-fine carbide precipitation is observed to be significantly higher in the optimised DCT sample, than in the typical DCT and CHT. The improvements in hardness, dimensional stability and wear resistance experienced could be attributed to the above mentioned variation in the microstructure.

Conclusions

- Tensile residual stress of the DCT samples shows an improvement in 36% compared to CHT samples. Improved in tensile residual stress is mainly due to the precipitation of fine carbides in the specimens subjected to DCT with tempering.
- Hardness of the DCT samples shows an improvement of 15% compared to CHT samples. The improve in hardness is mainly due to the transformation of retained austenite to martensite.

- Cryogenic treatment reduces the corrosive nature of the bearing material compared to the conventional heat treated samples.
- This study suggests that cryogenic treatment improves the tensile residual stress and improving the life of the component.

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