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Research Article

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Experimental Investigation on the Effect of Deep Cryogenic Treatment on 100Cr6 Bearing Steel

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Abstract

Cryogenic treatment is the process of cooling a material to extremely low temperatures to generate enhanced mechanical and physical properties. The present investigation examines the effect of deep cryogenic treatment on the enhancement of mechanical properties, such as wear resistance, corrosion resistance, tensile strength and impact strength of the plunger material 100Cr6 bearing steel. The improvement in the wear resistance, corrosion resistance, tensile strength and impact strength of the deep cryogenically treated samples over the conventionally heat-treated ones, is 50%, 26%, 13% and 27% respectively. This study suggests that the formation of very small carbides dispersed in the tempered martensite structure, can be the main reason for the enhancement of certain mechanical properties, along with the retained austenite transformations.

Keywords: Cryogenic Treatment, Austenite, Mechanical Properties, Wear Resistance Corrosion

Introduction

In recently years, there has been a tremendous increase in the growth of the automobile industry. Much research has been done, with a view to improve the life and performance of automotive components by various treatments. The service failures of the engineering components have always been a challenge to material engineers. The plunger is an important component in a fuel injection pump, as it acts as the heart of an internal combustion engine. The failure that occurs in the fuel injection pump is mainly due to the failure of the plunger. Due to plunger wear, the flow of the fuel increases; this reduces the fuel pressure delivered inside the combustion chamber, and results in incomplete combustion, more smoke, increase in fuel consumption, impurities entering the chamber, etc. Even though new plunger materials and production techniques are constantly being developed, these changes face difficulties in keeping pace with the high demands in a worldwide competitive market. Hence, definite ways and measures are needed to improve the performance and life of the plunger. In order to address the durability and frictional losses in the major tribological components of an automobile engine, an attempt has been made with cryogenic treatment. Cryogenic treatment is one of the ways to improve the mechanical properties, performance and life of the components. It is believed that the life of the components gets extended substantially with deep cryogenic treatment (DCT). Gill et al reported that cryo processing significantly influences the performance of the cutting tool steel, and hence, enhances the productivity and product quality [1]. DCT is a supplementary process to heat treatment, in which the material is subjected to very low temperature in a particular cycle, that consists of well-defined cooling, soaking and heating processes. The basic cryogenic treatment consists of a gradual cooling of the component until the defined temperature, holding it for a given time (soaking time), then progressively heating it back to room temperature, and tempering it to decrease the brittleness of the martensite.

Researchers have observed the effect of cryo-processing on different types of steel and also reported, the most significant and consistent changes in increased wear resistance, tensile strength, impact strength, corrosion resistance, etc. Oppenkowski et al studied the deep cryogenic treatment(−196°C) of tool steels, and reported that the most significant factors influencing the properties of tool steels are the austenitizing and tempering temperatures [2]. Preciado et al investigated the effect of deep cryogenic treatment on the hardness and wear resistance of carburized steels, and reported that the deep cryogenic treatment (−190°C) of quenched and tempered carburized steels improved the wear resistance, being in the low temperature range [3]. Baldissera et al mentioned that the main reason for the improvement in the properties of the cryo- treatment component, is due to the complete transformation of the retained austenite into martensite and precipitation of the fine carbides into the martensitic matrix [4]. Lulay et al reported a slight increase (+12%) in the impact toughness after 48 hrs of DCT on a 7075 aluminum alloy [5]. Chillar et al studied the influence of the cryogenic treatment on mild steel, brass, copper and cast iron, and reported that the torsional property had increased by22% and 66% for mild steel and cast iron respectively [6]. Zhu et al investigated the effect of cryogenic treatment on the corrosion resistance of the medium melting point castable alloy, and found that cryogenic treatment is an effective procedure in enhancing the corrosion resistance [7]. The study pointed out that significant improvements were seen in the stress corrosion cracking performance. Al-Quran and Al-Itawi conducted a study on the effect of the intermediate annealing on the mechanical properties, and corrosion resistance, of low composition of chromium-nickel steel [8]. The study shows that the increase in the corrosion resistance of the material is mainly due to the spheroidal annealing process. Chuang-xian et al reported that the ultimate tensile strength, yield strength and elongation of the cryogenic treated magnesium alloy, added with zirconium, have improved by 38%, 57% and 280% respectively, as compared to those of the same alloy without cryogenic treatment [9]. Jaswin et al investigated the effect of cryogenic treatment on the tensile behaviour of En 52 and 21-4Nvalve steels at room and elevated temperatures, and concluded that deep cryogenic treatment enhances the tensile strength of the valve steels with a marginal reduction in the elongation [10]. Barron reported that the deep cryogenic treatment has many benefits, it provides to the material and improves the wear resistance, corrosion resistance, strength and hardness of the material [11]. The present work is an experimental investigation on the effect of deep cryogenic treatment on the mechanical properties, such as wear resistance, corrosion resistance, tensile strength and impact strength over CHT of the plunger material 100Cr6 bearing steel samples.

Optimisation of Deep Cryogenic Treatment

The preliminary test results of the 100Cr6 bearing steel samples,with the DCT cycle of 1oC/min cooling rate; -185^{o}C soaking temperature; 24 hrs soaking time; and tempering temperature of 200^{o}C, was showed an enhancement in the mechanical properties over those of CHT (Sri Siva etal 2011) [12]. Various researchers used different levels of the treatment parameters (cooling rate, soaking temperature, soaking period, and tempering temperature) in their studies and claimed different degrees of improvement in the mechanical properties of the steel component subjected to DCT. The level of treatment parameters may vary from material to material. In order to identify the optimum treatment parameters, each new material needs to be treated and tested at different temperature levels, holding times, cooling and heating rates, which is quite unmanageable, because of the large number of experiments involved. Hence, it is proposed to optimise the DCT parameters (cooling rate, soaking temperature, soaking period, and tempering temperature) to obtain the maximum enhancement in the mechanical properties of 100Cr6 bearing steel. The GreyTaguchi technique was adopted to maximize the mechanical properties of the 100Cr6 bearing steel through deep cryogenic treatment parameters.The optimal deep cryogenic treatment cycle has been identified, and the benefits are quantified experimentally through a confirmation test (Sri Siva et al 2012) [13]. In the present workthe effects of the DCT at the optimised condition on the allied mechanical properties, such as the wear resistance, corrosion resistance, tensile strength and impact strength were investigated as per the ASTM standards, with respect to the optimal cycle of the cooling rate 1oC/min; soaking temperature -185^{o}C; soaking time 36 hrs; and tempering temperature 200^{o}C.

Experimental Investigation

Conventional Heat Treatment

The heat treatment of the bearing steel is one of the most important factors in determining how it performs in service. The standard process of the conventional heat treatment for the 100Cr6 bearing steel consists of hardening from room temperature to 850ºC, maintaining at that temperature for one hour to equalize the temperature throughout the samples, and after the equalization, oil cooling or quenching, followed by tempering at 200ºC for two hours.

Deep Cryogenic Treatment

The deep cryogenic treatment process consists of slow cooling down from ambient temperature to an ultra-low temperature in a chamber at the rate of 1°C/min. The material reaches approximately -185°C; it was soaked for a period of 36 hrs at that temperature. At the end of the soaking period, the samples are allowed to warm up to room temperature at the rate of 0.6°C/min, to avoid thermal shock and micro cracks. By conducting the cool-down cycle in gaseous liquid nitrogen, the temperature can be controlled accurately using the A.C.I. CP-200vi (Massachusetts, U.S.A) cryogenic treatment processor, and thermal shock to the bearing steel can be avoided. Single-cycle tempering at 200°C for two hours was performed, using a muffle furnace after deep cryogenic treatment, to improve the mechanical properties by the precipitation of carbides and decomposition of the retained austenite to martensite.

Wear Resistance

The wear resistance enhancement in the conventionally heat treated and deep cryogenically treated 100Cr6 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 [14].

Two specimens are required for the test, a round rod and a flat rod. In the fuel injection pump both the plunger and the barrel are made up of 100Cr6 bearing steel. Hence, in the present study, both the surfaces that undergo the reciprocatory wear were made of 100Cr6 bearing steel material. This steel was cylindrically machined to a 6 mm diameter and 10 mm length, and flat plates were machined with dimensions of 25 x 15 x 5 mm as a counterpart, for conducting the wear test. The wear test was conducted on the samples, subjected to the two different treatments, namely the CHT and DCT. Each sample was cleaned with acetone for the removal of moisture, and further cleaned with an ultrasonic cleaner for 10 minutes to remove any other oil or grease contamination.

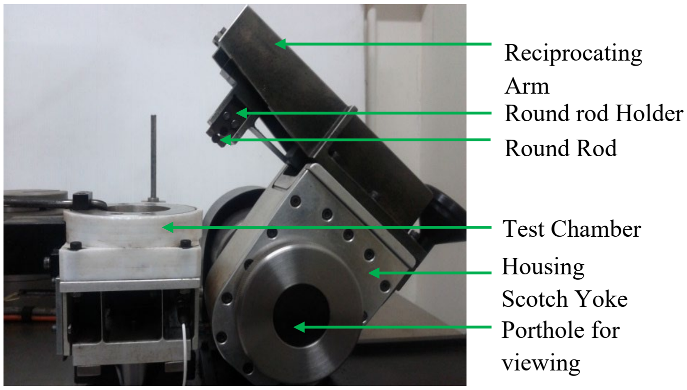


Figure 1: A Close-up Photographic View of the Critical Components in the RFWM

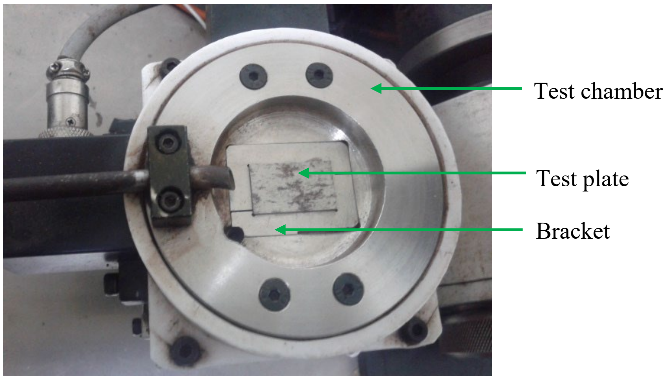


Figure 2: Test Plate Fixed in the Test Chamber

The initial weights of the samples (round rod and flat plate) were noted. Then the round rod was attached to the reciprocating arm of the wear tester, through a specially designed holder, shown in Figure 1, and the flat plate was attached to the test chamber with a specially designed bracket as shown in Figure 2, so that the cylindrical face (round rod) makes a line contact with the counterpart (flat plate). The specimen (round rod) was then allowed to reciprocate over the same hardened 100Cr6 flat rod, with different normal loads of25N, 50N and 75N at a constant frequency of 5 Hz, and the test was conducted for 5 hours. Finally, the specimens were taken out from the holder, cleaned, dried and the weight loss due to wear was estimated using the semi-micro balance. The weight loss (wear loss) was a measure of the wear resistance; the lower the wear loss, the better the wear resistance.

Corrosion Resistance

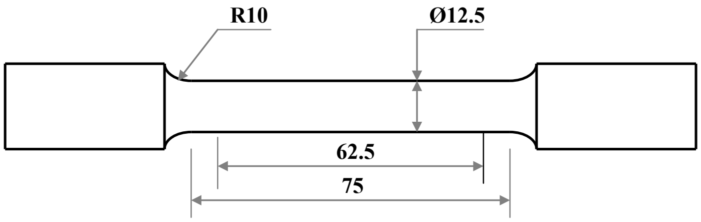
The salt spray corrosion test was carried out by the weight loss method as per the ASTM B117, to measure the corrosion resistance for the CHT and DCT samples [15]. The salt spray test procedure involves the spraying of a salt solution as a very fine fog mist, onto the samples being tested inside a temperature-controlled chamber for 24 hrs. The temperature within the chamber was maintained at a constant level. A 5% Sodium Chloride (NaCl) solution that does not contain more than 200 ppm total solids, and with a pH range of 6.5 to 7.2 when atomized, was used, and the temperature of the salt spray cabinet was maintained in the range of 35 ± 1ºC, within the exposure zone of the closed cabinet. Since the spray was continuous, the samples were constantly wet, and therefore, constantly the test samples were subject to corrosion. The details of the test parameters taken for this study are shown in Table 1.

Table 1: Details of the Salt Spray Test Parameters

S. No	Parameters	Values
1	Concentration of Sodium Chloride	5.3%
2	Chamber temperature range	33.8 – 35.8ºC
3	pH of salt	6.9
4	Total exposure period	24 hrs

Tensile Test

The tensile test was conducted on the 100Cr6 bearing samples, using a 400 KN capacity hydraulic type universal testing machineas per the ASTM standard E8M-04 [16]. Initially the diameter, area and gauge length of a standard tensile test specimen was measured, using a vernier caliper. A cylindrical test specimen of a nominal diameter of 12.5 mm and gauge length 62.5 mm, was used for the tensile test asshown in Figure 3. The specimen was prepared carefully with high quality surface finish of a reduced section, smooth fillets at the ends of the gauge length and oversized grip sections. Fillets with a radius of 10mm were used at the end of the reduced sections.



All dimensions in mm

Figure 3: Tensile Test Specimen

The specimens were grouped into 2 batches, namely, the CHT and DCT, with each batch consisting of 3 samples. The specimen was placed in the grip of the UTM at a specified grip separation; after setting the sample properly, the machine was switched ON, and the load was applied in a unidirectional vertical direction. When the specimen attained the ultimate tensile strength point, it broke with a sound. After removing the broken pieces of the specimen from the holder, the broken pieces were placed together in an elongation gauge, and the final gauge length was measured. The reduced diameter was also measured. Based on the above, the ultimate tensile strength, yield stress and percentage of elongation were calculated.

Impact Test

The samples of 100Cr6 bearing steel for the charpy impact test were milled to a length of 55 mm and a cross section of 10x10 mm, having a V-notch with an angle of 45º and a depth of 2 mm as per the ASTM E23-02a standard [17]. The dimension of the specimen for the test is shown in Figure 4. The machined samples were grouped into two, with three in each group, and subjected to CHT and DCT respectively. The specimen was placed horizontally between the two anvils, so that the knife strikes opposite the notch at the mid-span. The pendulum of the charpy impact machine was raised to its top most position, and held by a latch adjusted to give a constant height of fall. It was released and allowed to fall, and rupture the specimen. The energy required to rupture the specimen is a function of the toughness of the specimen, which is indicated on a semi-circular scale in joules.

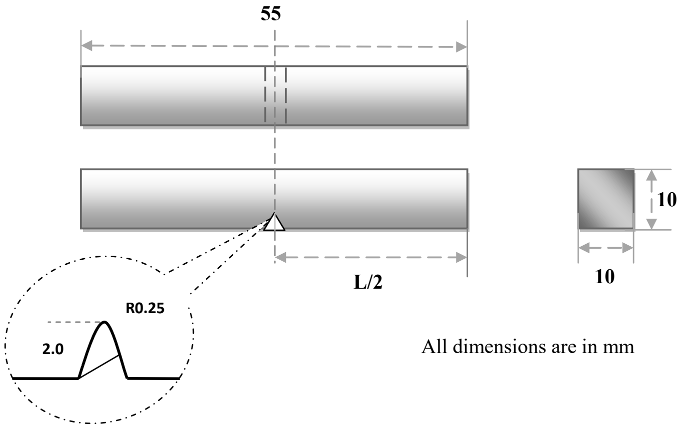


Figure 4: Charpy Impact Test Specimen

The test was carried out in an ambient condition. This helps to identify the best treatment for 100Cr6 bearing steel, with respect to the impact behavior. The study also helps in identifying the metallurgical reason for the increase or decrease in the impact properties, with regard to each treatment.

Result and Discussion

Wear Resistance

It is observed, that the wear resistance of the 100Cr6 bearing steel has improved by 50% due to DCT when compared to the CHT. From figure 5, it can be concluded that the DCT samples have high wear resistance (low wear loss) in all the test conditions.

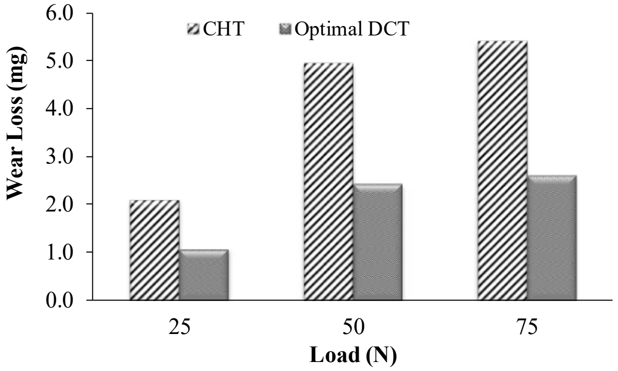


Figure 5: Variation in the Wear Loss of CHT and DCT

The results revealed that the improvement in wear resistance of DCT samples with respect to the conventional heat treatment varies from 49- 51%, when the load is varied as 25, 50 and 75N. The percentage improvement in the wear resistance of the DCT sample is high in the maximum load condition for the 100Cr6 material. The results indicate that the deep cryogenic treatment substantially enhances the wear resistance of the 100Cr6 bearing steel, with respect to the conventional heat treatment. From figure 5 it is also observed, that wear loss increases proportionately when the load increases.

Corrosion Resistance

The salt spray corrosion test is carried out on the CHT and DCT samples, in order to study the corrosion resistance using the weight loss method. The weight of the sample before and after the test is observed and recorded. The results of the salt spray test of the CHT and DCT samples are listed in Table 2.

Table 2: Result of the Salt Spray Test

SI. No	Treatment Condition	Weight before salt spray test (mg)	Weight after salt spray test (mg)	Weight loss (mg)	Average weight loss (mg)
1	CHT	27156.6	27107.2	49.3	47.25
2		26616.4	26571.2	45.2	
3	DCT	27008.4	26975.3	33.1	34.8
4		27086.5	27050.0	36.5	

The percentage of weight loss is calculated for both conventionally heat treated and deep cryogenically treated samples. The most significant improvement is found in the deep cryogenically treated sample.

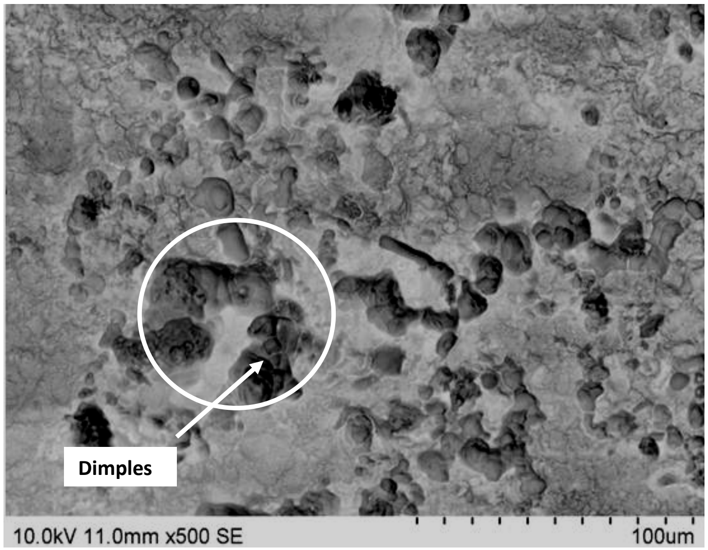


Figure 6: SEM Image of the Sample Subjected to CHT

The result revealed that the conventional heat treated sample suffered more weight loss due to corrosion than the deep cryogenically treated sample; i.e., the corrosion resistance of the deep cryogenic treated samples is comparatively better. The percentage improvement in the corrosion resistance due to the deep cryogenic treatment is 26% over the conventional heat treatment.

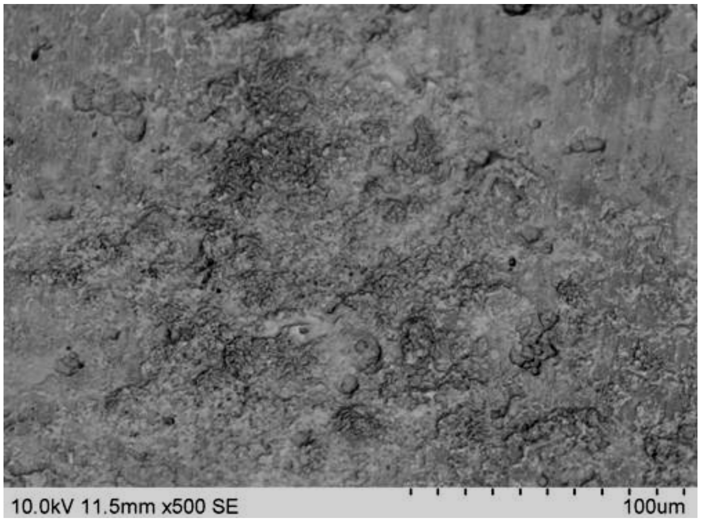


Figure 7: SEM Image of the Sample Subjected to DCT

Figures 6 and 7 show the SEM images of the CHT and DCT samples after the salt spray test respectively. Red rust was noticed after 6 hrs on both the conventionally heat treated and deep cryogenically treated samples. From the figures it is observed, that the CHT sample shows deep corroded surfaces over the DCT samples. This may be due to the presence of a softer phase of the retained austenite. The numbers and size of the dimples present in the CHT samples are larger than those of the deep cryogenically treated samples. The presence of small secondary carbides and the formation of carbide clusters on the surface of the deep cryogenic treated sample of 100Cr6 bearing steel, were the main factors responsible for the improvement in the corrosion resistance. The results show that due to a more uniform carbide distribution in association with the higher carbide percentage, the corrosion behaviour of the deep cryogenically treated samples was improved. The studies show that, chromium carbide and oxides improve the corrosion resistance of 100Cr6 bearing steel. The corrosion resistance may be improved with longer soaking time (36 hrs) and hence, it can be attributed to the uniform carbide distribution. It is inferred that the corrosion resistance is more for the DCT samples.

Tensile Test

The effect of deep cryogenic treatment under conditions on the tensile strength of the 100Cr6 bearing steel is studied, and the test results are summarized in Table 3. The ultimate tensile strength of the sample subjected to the DCT is higher than that of the CHT samples.

Table 3: Tensile Test Result of the Samples Subjected to CHT and DCT

Treatment condition	Trial No	Ultimate tensile strength N/mm^2	Yield strength N/mm^2	% of Elongation
CHT	1	2100.79	1617.03	2.0
	2	2070.54	1568.43	2.0
	3	2084.63	1547.70	1.9
Average		2085.32	1577.72	2.0
DCT	1	2397.40	1952.51	1.6
	2	2389.34	1892.65	1.8
	3	2405.56	1967.49	1.7
Average		2397.44	1937.55	1.7

The ultimate tensile strengths of the CHT and DCT samples are 2085.32 N/mm2 and 2397.44 N/mm^2 respectively. Yield strength refers to the amount of stress a material can withstand without permanent deformation. The yield strengths of the CHT and DCT are 1577.72 N/mm^2 and 1937.55 N/mm^2 respectively. The percent elongation is an indication to the designer, in a general way, of the ability of the metal to flow plastically, before fracture. On comparing the results of the percentage elongation, the DCT samples show a lesser elongation than the CHT samples. The average percentage of elongation of the CHT sample is 2.0%, but the DCT sample shows only 1.7%. This reduction in the percentage elongation of the cryo-treated samples indicates a minor reduction in the ductility of the samples, which can marginally affect the machinability of the components.

When comparing the results, the DCT samples demonstrate a higher tensile strength and yield strength than the CHT samples. It is inferred that the tensile strength and yield strength of the DCT samples exhibit an enhancement of 13% and 18.5% over the CHT samples respectively. The enhancement in the strength can be attributed to the transformation of the retained austenite into martensite, and the precipitation of fine carbides.

Impact Test

The impact energy for the conventionally heat treated and the deep cryogenically treatet samples is tabulated in Table 4.

Table 4: Charpy Impact Test Results for the 100Cr6 Bearing Steel Subjected to CHT and DCT

Conditions	Sample Identification	Absorbed Energy (J)	Average (J)
CHT	A	5	5.33
	B	6	
	C	5	
DCT	A	8	7.33
	B	7	
	C	7	

The toughness of the material is its ability to absorb energy in the plastic range, or its resistance to the propagation of a crack. The effect of the impact energy absorption is carried out, and it is observed that the mean toughness value after deep cryogenic treatment is 7.33 J, whereas that of the conventional heat treatment is5.33 J. The average percentage improvement of the absorbed energy of the DCT sample over CHT is 27%. These results show an increase in the toughness of the DCT sample, when compared with that of the CHT. It is inferred that the transformation of the retained austenite to martensite would eventually cause an increase in the toughness.

Microstructural Analysis

The optical micrographs of the CHT and DCT samples are shown in Figures 8 and 9. The micrograph of the CHT samples exhibited a non-uniform distribution of large, elongated, white regions of primary carbides on the tempered martensite matrix.

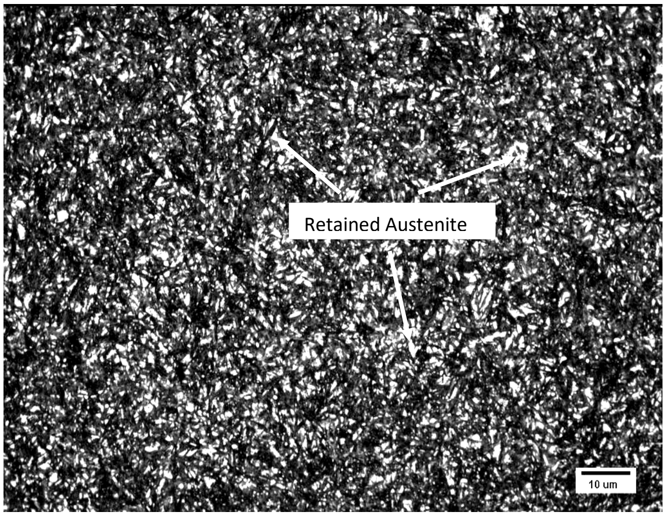


Figure 8: Microstructure of the 100Cr6 CHT Sample

The micrograph of the DCT, shows a more refined microstructure, and the of carbides are distributed more evenly. It is evident that occasional the 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 a spheroidal carbide is observed in DCT samples. It is also noted that the CHT sample shows more number of large-sized carbides when compared to the DCT. The variation in the characteristics of the carbide particles between the CHT and the DCT samples is explained as follows. The transformation of the 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.

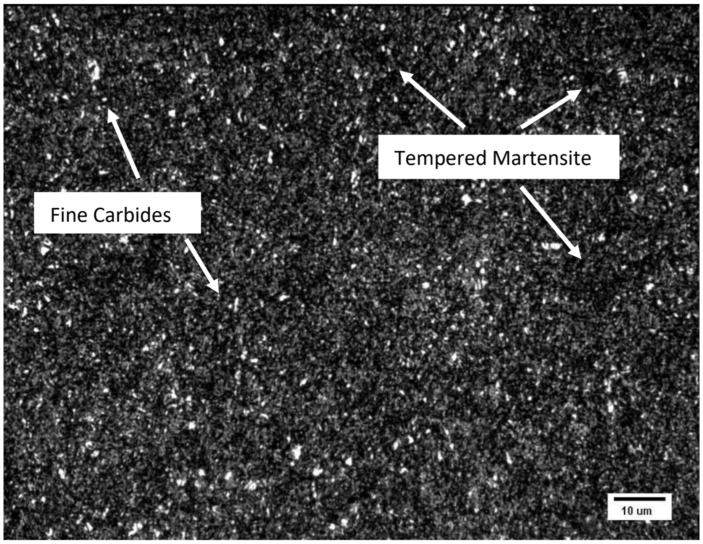


Figure 9: Microstructure of the 100Cr6 DCT Sample

The obtained result suggested that the DCT reduces the retained austenite substantially, as compared to the CHT. This is because the retained austenite is more unstable at lower temperature, and is likely to transform 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 a higher amount of tempered martensite; the increased amount of martensite naturally leads to a higher amount of carbide precipitations.

The microstructure of the DCT specimens exhibits less number of primary carbides but more number of secondary carbides. In addition to small secondary carbides, the microstructure of the DCT specimens reveals finer and more uniformly distributed carbides than the CHT specimen. Finer chromium carbides are precipitated in the martensitic matrix of the DCT specimens.

These are also responsible for the improvement in the properties of the cryo-treated 100Cr6 bearing steel. The refinement and precipitation of more carbide attributed to the long soaking time (36 hrs), enhances the impact energy of the DCT samples. Then, the reheating results in the precipitation of a finer distribution of carbides in the tempered microstructure, with a consequent increase in toughness as well as in wear resistance. The presence of the tempered martensite with very little retained austenite, coarsening of carbide particles, and the percentage of ultra-fine carbide precipitation, are observed to be significantly higher in the DCT sample. The above mentioned characteristics of carbides, and the conversion of the retained austenite exhibits variations in the tensile strength, impact strength, wear resistance and corrosion resistance of the CHT and DCT of 100Cr6 bearing steel.During cryogenic treatment, fine platelets of martensite are formed from the retained austenite, and these platelets promote the precipitation of fine carbides by a diffusion mechanism during tempering; or they may be due to the reduction in the lattice energy during deep cryogenic treatment, which makes the crystal structure perfect.

Conclusion

- The reciprocatory wear test showed that the wear resistance of 100Cr6 bearing steel was improved by 50% due to the DCT compared to the CHT.
- The salt spray test showed that the corrosion resistance of 100Cr6 bearing steel was improved by 26% in the DCT samples compared to the CHT samples.
- The ultimate tensile strength of the 100Cr6 bearing steel DCT samples has improved by 13 % over the CHT samples.
- The impact energy of the 100Cr6 DCT samples has improved by 27% over that of the CHT samples.

The microstructural analysis revealed that the DCT sample produced more martensite transformation and facilitated carbide formation, which made the alloy present a more refined matrix, and produced a fine grain strengthening effect, thereby increasing the impact strength, tensile strength, wear resistance and corrosion resistance of the 100Cr6 bearing steel. This study confirmed that the deep cryogenic treatment imparts changes in the entire cross section of the material.

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