Comprehensive analysis on the effect of deep cryogenic treatment on the mechanical behaviour of Martensitic valve steel

*[1]M. Arockia Jaswin, [2]D. Mohan Lal

[1]Department of Mechanical Engineering, Velammal Engineering College, Chennai - 66.

[2]Department of Mechanical Engineering, College of Engineering Guindy, Anna University Chennai, Chennai – 25, India.

* Corresponding author / Email: winn.au@gmail.com Tel. +919444522847.

ABSTRACT

The behaviour of the cryogenically treated En52 martensitic valve steel has been experimentally analyzed in this paper. Material samples are subjected to deep cryogenic treatment after completing the regular heat treatment. The critical properties of the valve steel like wear resistance, hardness, tensile strength and impact strength are evaluated for the cryo treated En52 valve steel samples as per the ASTM standards. The microstructural changes and the mechanism behind the enhancement of the properties are examined and reported. The precipitation of fine carbides, transformation of retained austenite and refinement of carbides were the reasons behind the improvement of the mechanical properties. Deep cryogenic treatment process parameters are optimized for better wear resistance, hardness and tensile strength using grey Taguchi technique. Deep cryogenic treatment process greatly influences the wear resistance, a maximum enhancement of 54% is observed.

Keywords: Cryogenic treatment, Valve steels, Carbides, Optimization, Microstructure, Martensite.

1. INTRODUCTION

The service failures of engineering components have always been a challenge to the materials engineer. Other than various production methods, the failure of the components can be enhanced by using various processes. Through advances in cryogenics, it has been discovered, and proved that the life of the cryo-treated components is enhanced to a great extent [1].

Researchers made attempt to treat the tool steels at different condition and reported remarkable improvement in the mechanical properties. Darwin et al [2] performed a study to optimize the process parameters of the cryogenic treatment for a commercial piston ring, made up of 18 % Cr martensitic stainless steel (SR34) to obtain the maximum wear resistance using Taguchi's design of experiment technique. Das et al [3] conducted a study on the refinement of carbide precipitates by the cryotreatment of AISI D2 steel. It was inferred from the microstructural analysis that the population density of small secondary carbides is significantly higher in the DCT specimen and the diffraction peaks of austenite are almost invisible for the DCT specimen suggesting that DCT almost completely transforms all retained austenite (γ_R) to martensite. Yong et al [4] treated tungsten carbide tools at 89 K in a chamber. The temperature was lowered to 89 K in a period of 6hrs from room temperature and held steady for about 18hrs and heated back to room temperature in 6hrs. The cryogenically treated tools perform better when compared with untreated tools. Hong-Shan Yang [5] studied the effect of deep cryogenic treatment on the matrix structure and abrasion resistance of high chromium cast iron subjected to destabilization heat treatment. Vadivel and Rudramoorthy [6] investigated the performance of cryogenically treated coated carbide inserts. It was identified that the cryogenic treatment improves the hardness of the coated inserts, and provides more wear resistance that reduces greater flank and abrasive wear. Paolo Baldissera [7] investigated the fatigue scatter reduction through deep cryogenic treatment on the 18NiCrMo5 carburized steel. It was observed that the DCT has an evident influence of fatigue behaviour on the carburized 18NiCrMo5 steel. Both fine carbides precipitation and residual stress changes are suspected to be at the root of the obtained results. Jun Wang et al [8] investigated the effects of deep cryogenic treatment on the microstructure, hardening and abrasion resistance behavior of high chromium cast iron subjected to the destabilization treatment. El Mehtedi [9] analyzed the effect of Deep Cryogenic Treatment on the hardness and microstructure of X30 CrMoN 15 1 steel. An equation based on the composite model was developed to quantify the contributions of the various constituents (martensite, retained austenite, carbides and carbonitrides) to hardness. YongLiu, et al [10] studied the effect of cryogenic treatment on the microstructure and mechanical properties of Mg-1.5Zn-0.15Gd magnesium alloy. The result showed that numerous W phase particles precipitated from the Mg matrix after cryogenic treatment. Above studies shows that deep cryogenic treatment draws much attention due to its significant advantages. For cryogenic treatment to be implemented as a regular treatment process, a good understanding of the process parameters and the underlying mechanism is necessary.

The valve and valve seat are the key parts installed in the cylinder head of an engine to ensure the sealing of the combustion chamber. Engine valve wear is one of the important factors which reduce the engine performance. Xia Yong et al [11] reported that many faults of engines occur on the valve train. Although new valve materials and production techniques are continuously being developed, these advances have been outpaced by demands for increased engine performance, and wear related problems and failure of engine valves remain an issue. The En 52 valve steel has been widely used for the intake valves and exhaust valve stems in internal combustion engines. In this paper an attempt has been made to study the effect of cryogenic treatment on the mechanical properties of En52 valve steel and the treatment parameters are optimized. The mechanisms behind the enhancement in the properties are also examined through a characterization study.

2. EXPERIMENTATION

The heat treatment procedure for En 52 valve steel consists of austenitizing followed by oil quench (quench-hardening) and then tempering. In the quench-hardening process of the En 52 valve steel, the specimens were heated to 1248 K (975 ºC), soaked for 1 hour and then quenched in oil. En 52 valve steel material which has undergone the conventional hardening were slowly cooled from room temperature to 85K at a rate of 1 K/min, soaked for 24 hours, and heated back to the room temperature at a rate of 0.6 K/min. The cryogenic treatment parameters like soaking time, temperature and cooling rate were controlled using a programmable temperature controller. The En 52 samples taken out from the processor were tempered at 923 K (650 ^{o}C) for an hour. The results obtained by the chemical analysis using optical emission spectroscope for the raw material is given in Table1.

Table 1 Chemical composition of the En 52 valve steel

Material	**Carbon (%)**	**Silicon (%)**	**Manganese (%)**	**Chromium (%)**	**Nickel (%)**	**Nitrogen (%)**	**Sulphur (%)**	**Phosphorus (%)**
En52	0.45	3.25	0.50	8.50	0.40	-	0.03	0.04
Typical composition	0.4-0.5	2.7-3.3	0.6 max	8-10	0.5 max	-	0.03 max	0.04 max

Microstructural examination is carried out to identify the possible mechanism brought in by the cryogenic treatment in improving the properties En52 valve steel. The samples for the metallographic examination are prepared as per the ASTM E 3-01. The wear resistance enhancement in the cryo-treated valve steel samples are experimentally measured using a reciprocatory friction and wear monitor (DUCOM TR-281M-M4) by weight loss method, as per the ASTM standard G-133-95. The loss in weight (wear loss) is a measure of wear resistance, lower wear loss for better wear resistance. As per the ASTM designation E 92-82 (standard test method for Vickers hardness of materials), the hardness test is carried out.

Tensile test is carried out as per the ASTM standard E8M-04 (standard test method for the tensile strength of a material at room temperature) and ASTM standard E21-03a (standard test method for high temperature tensile strength of a material) with round bar test specimens. Since the size of the material available was not sufficient for making the standard tensile test specimen, sub-sized specimens as per the standard are used for conducting the tensile test. Charpy impact test is carried out as per the ASTM E 23-02a standard.

After performing the above experimental analysis Grey-Taguchi optimization is carried out to optimize the deep cryogenic treatment process to maximize the wear resistance, hardness and tensile strength of En 52 valve steel. Grey relational analysis and the L_9 orthogonal table are integrated to analyze the experimental results of the deep cryo-treated En 52 valve steel samples. The main parameters involved in the DCT are cooling rate, soaking temperature, soaking time, and tempering temperature. Various researchers used different levels of the above said parameters in their studies and claimed different percentages of improvements in the mechanical properties of steel components [12].

3. RESULTS AND DISCUSSION

En 52 valve steel is cryogenically treated to improve the life and performances. The capability of these materials to meet the requirements is determined by the mechanical and physical properties of the material. The enhancement in the properties is analyzed by conducting the reciprocatory wear test, hardness test, tensile test at room and elevated temperature and impact test. A microstructural analysis is conducted to study the mechanism underlying the deep cryogenic treatment. The grey-Taguchi method is used to optimize the DCT process for the maximum enhancement in wear resistance, hardness and tensile strength.

3.1 Microstructural Analysis

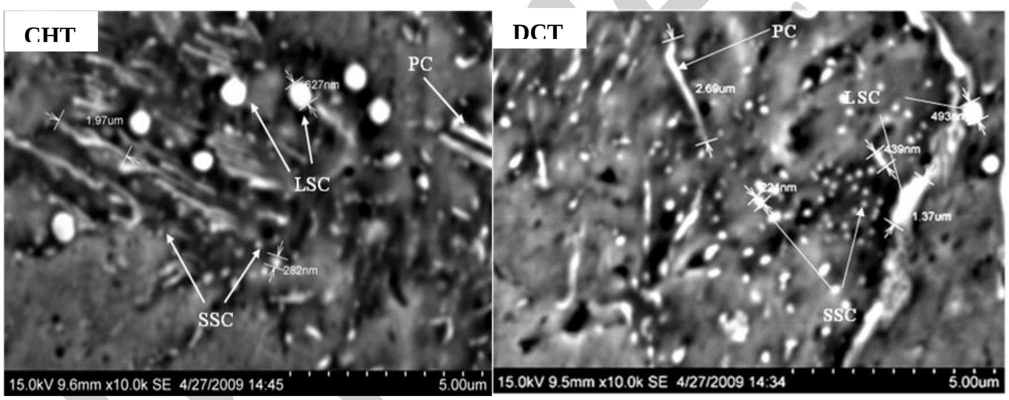

Figure 1 SEM micrograph of the cryogenic treated and non cryogenic treated specimen

Microstructural examination is carried out to study the changes that influence the enhancement in the properties of the En 52 valve steel material, when subjected to cryogenic treatment. The microstructure of the En 52 CHT and DCT samples are shown in Figure 1. The morphology and the particle size of the carbides seen in the micrograph are more similar however, more distribution of fine carbides on the martensitic matrices in the cryogenically treated samples than that in the CHT samples. Under deep cryogenic treatment the volume of martensite contracts and its lattice parameters shrink, which can reinforce the precipitation of carbon atoms. It is observed that the ability of diffusion of carbon atoms is less in low temperature and the diffused distance is shorter, therefore, fine carbides precipitated from the matrix. Refinement of carbides is also visible in the DCT specimen. It is clearly seen from the micrograph of the DCT sample that a large amount of fine carbides of micron size are precipitated throughout the structure. The fine carbides precipitated through the cryogenic treatment strengthen the structure and tie up with certain elements and restrict the promotion of instability during service. Yang Gao et al. [13] stated that the change of the properties can be primarily attributed to the fact that the martensite phase transformation from γ-(Fe,Ni) to α-(Fe,Ni) and precipitation of W particles in the binder phase improve the hardness and strengthen the binder phase during the deep cryogenic treatment. Kaikai Wang et al [14] infer that contraction of unit cell at low temperature promotes the precipitation of fine dispersed carbides and contributes to wear resistance.

Table 2 Comparison of mechanical properties of En 52 valve steel with CHT & DCT

Sl.No	Mechanical Properties	CHT	DCT
1	Wear loss	6.84 mg	3.94 mg
2	Hardness	345 HV	378 HV
3	Impact Strength	2.9 J	3.53J
4	Tensile strength @ room temp.	1063 MPa	1093 MPa
5	Tensile strength @ elevated temp.	863 MPa	937 MPa

3.2. Reciprocatory Wear & Hardness Test

The improvement of wear resistance of the cryogenically treated En52 valve steel is accessed with respect to various loads and frequencies. The wear test results for a maximum load of 30N and 10 Hz frequency is tested and shown in figure 2 . The precipitation of carbides and the conversion of retained austenite results the materials to resist wear during the reciprocatory wear test.

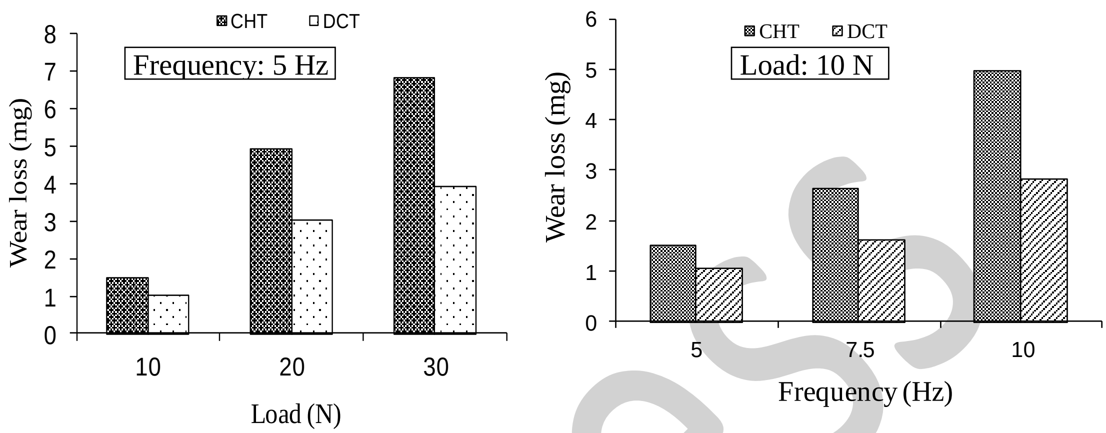

Figure 2 Wear loss of the En 52 steel for different load and frequency

The wear resistance of the cryogenically treated samples is always better than that of the non-cryogenically treated samples in the whole range of test conditions. The wear failure of the engine valve will greatly reduce and improve the engine breathing and life of the engine valve. To account for the observed improvement of the wear resistance in the cryogenic treatment there are three important factors to be considered. The first one is the conversion of abundant retained austenite into martensite during the cryogenic treatment, which can offer stronger support for carbides to inhibit their spalling and prevent large grooves forming during the abrasion course. Secondly, the precipitation of finer carbides and their more homogeneous distribution as a result of the cryogenic treatment is responsible for the improved wear resistance. The distribution of the carbides in the cryogenically treated samples is more homogeneous than that in the non-cryogenically treated samples, and the carbide volume fraction in the cryogenically treated samples is more than that in the non-cryogenically treated samples. Thirdly, cryogenic treatment can produce more martensite transformation and make the alloy present a refiner matrix, and a refiner matrix implies a fine grain strengthening effect, which would contribute to the wear resistance improvement. The presence of numerous ultrafine carbides in cryo-treated steels assists in attaining the micro-stress distribution of the material in a way that results in favorable crack growth resistance of the material. This, in turn, leads to higher wear resistance. The Vickers hardness test result shows a fractional improvement in the hardness of En 52 valve steel due to the cryogenic treatment. On comparing the CHT with DCT the Vickers hardness has improved to a value of 30 HV which is given in table 2. The cryogenic treatment is more effective in reducing the amount of austenite and can make a larger number of fine secondary carbides precipitate which can increase the dispersion strengthening effect. Both the above reasons are beneficial to increase hardness. Additionally, as is well known, the decrease of grain size will improve the hardness.

3.3 Tensile test

Tensile strength is the important measure of the material's ability to perform in service. The treated materials tested under the room condition shows an improvement of 30 MPa in the ultimate tensile strength when compared to non-treated material. The yield strength of the DCT samples exhibit an enhancement of 11 % over the CHT samples. The elongation of the material is tested and noticed a small reduction in the percentage elongation which indicates minor reduction of ductility in the samples. The transformation of retained austenite into martensite and precipitation of fine secondary carbide through the DCT are the probable reasons behind the improved strength and the reduction in elongation.

To know the behaviour of material at elevated temperature the material is tested at its operating temperature 673 K (400^{o}C). The ultimate tensile strength shows an improvement of 8% compared to CHT samples and the yield strength of the treated samples shows an improvement of 11% over the CHT samples. The fine secondary carbides precipitated during the DCT reveal good strength at the high temperature. It is inferred that the material undergoes the DCT has better enhancement in the strength at high temperature.

Table 3 Tensile Test results of the En 52 valve steel

Sl. No	Ultimate Tensile Strength (MPa)			Yield Strength (MPa)			% Elongation		
	CHT	DCT	Optimized DCT	CHT	DCT	Optimized DCT	CHT	DCT	Optimized DCT
1	857.1	924.7	940.48	695.5	796.6	785.03	30	30	32
2	871.8	951.3	958.5	695.5	746.2	776.43	30	27.5	25
3	853.7	948.2	924.04	682.9	732.7	815.61	27	25	30
4	866.9	940	982.63	685.7	801.4	770.36	32	27	30
5	865.4	919.8	947.41	688.4	753.3	794.52	32	25	25
Avg.	863	937	951	690	766	788	30	27	28
SD	6.6	12.4	19.5	5	27.7	16	1.8	1.8	2.9

Tensile fractograph of the En 52 CHT & DCT specimens are shown in figure 3. In the CHT specimen, scattered secondary cracks appear and are so deep that they give more elongation. The fractography study conducted on the fractured surface of the DCT specimen shows dimple rupture in some regions that suggests local quasi-cleavage fracture. The formation of carbides has significantly reduced the amount of porosity in the DCT samples, and increases the yield strength and tensile strength.

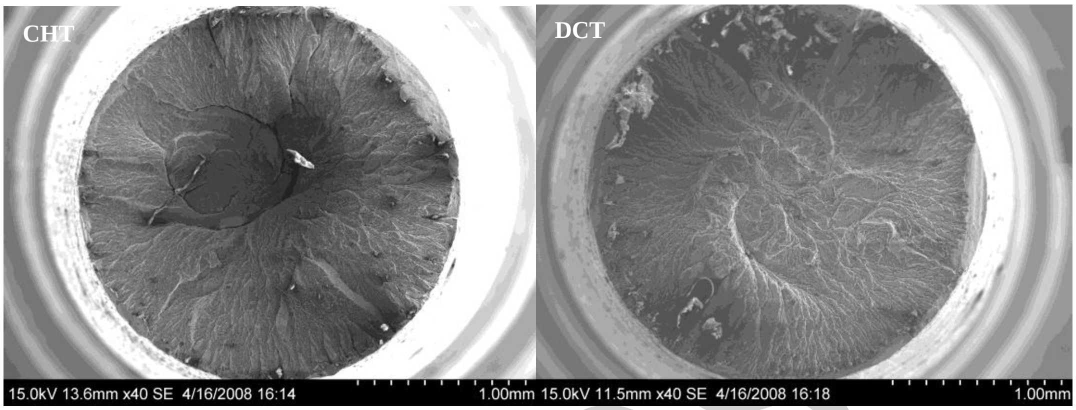

Figure 3 Tensile fractograph of the Cryo-treated and non treated specimens

3.4 Optimization of DCT process parameters

Grey – Taguchi technique is used to optimize the DCT process parameters of the En52 valve steel. Based on the L_9 orthogonal array tests have been conducted and the responses obtained are transformed to a grey relational grade for further analysis. The best experimental run for the DCT is selected from the response table and graph with a cooling rate of 1 ºC/min, a soaking temperature of -130 ºC, a soaking period of 36 hrs and a tempering temperature of 650 ºC. The test results of the optimized DCT and the CHT samples listed in the table 6 and shows good improvement.

Table 6 Test results of the En 52 valve steel with optimized DCT & CHT

Response	**Optimal DCT**	**CHT**	**% improvement**
Tensile strength (MPa)	1153	1063	8
Hardness (HV)	394	345	14
Wear loss (mg)	0.706	1.32	47

The results of the confirmation test indicates the improvement in all performance characteristics when compared to that of the typical DCT examined on the En52 valve steel samples.

4. CONCLUSION

The test results of the En52 cryo treated valve steel showed enhancement in the tensile strength, wear resistance, hardness and impact strength. The cryogenic treatment responded well for the wear resistance by exhibiting 47% improvement. From the microstructural analysis it is observed that finer and more uniformly distributed carbides are visible in the DCT specimen. Precipitation of secondary carbide was primarily due to the expansion resulting from transformation of the retained austenite into martensite and secondly caused by the difference in thermal contraction of the phases. It is concluded that the fine carbides precipitated through the cryogenic treatment strengthen the structure and tie up with certain elements and restrict the promotion of instability during service. The ultimate tensile strength and the yield strength of the valve steel tested at room and elevated temperatures get enhanced through the DCT. The DCT process parameters are optimized for better hardness, wear resistance and tensile strength using grey-Taguchi technique. From the experimental study it is concluded that the DCT cycle can be carried out along the conventional heat treatment to obtain optimal performance during the service.

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