National Conference on
"Role of Engineers in Nation Building" organized by VIVA Institute of Technology, Mumbai
(6th and 7th March 2020)
International Journal of Engineering Research & Science (IJOER)
ISBN:[978-93-5391-287-1] ISSN: [2395-6992] [Vol-6, Issue-3, March-2020]

Effect of cryogenic treatment on mechanical properties and wear behavior of high carbon steel

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***Abstract*—*The aim of the study is to evaluate the effect of deep cryogenic treatment with double tempering on mechanical properties and wear behavior of a grade of high carbon steel. In the study, one set of specimens undergoes conventional hardening and tempering process, while other set of specimens undergoes hardening, deep cryogenic treatment and double tempering process. Different sets of specimens were tested for hardness, impact strength, wear resistance and microstructure. The results of the study suggest that double tempering when performed after deep cryogenic treatment can aid into getting advantage of both improved wear resistance and toughness as compared with conventional hardening and tempering process.*

***Keywords*—Cryogenic treatment; hardening; high carbon steel; mechanical properties; tempering; wear resistance.**

I. INTRODUCTION

High carbon steel is a grade of carbon steel that contains between 0.70 % to 1.2 % carbon. High carbon steel possess comparatively more hardness and is used in applications where high wear resistance is required. It is used in applications such as taps, gauges, ball bearings, roller bearings, punches, dies, heavy duty gears, etc. Its applications is mainly present in machine parts where high wear resistance, toughness and high load carrying capacity is required. High carbon steel is generally used in hardened and tempered form to achieve the required combinations of properties for given application.

Hardening process is carried by heating the material around 800°C to 850°C in furnace. At this temperature, the pearlite structure is converted to austenite. After austenization, the material is rapidly quenched in oil. Rapid quenching results in conversion of austenite phase to martensite. Even after reaching room temperature, 100 % austenite doesn't get converted into marteniste. During the conversion of austenite to martensite there is a large volume expansion. As the martensite plates form during quenching, they surround and isolate small pools of austenite, which deforms to accommodate the lower density martensite. However, for the remaining pools of austenite to transform, surrounding martensite must deform. But the martensite resists the transformation and hence either the existing martensite cracks or the austenite remains trapped in the structure. This trapped austenite in the matrix of martensite even after reaching room temperature is called retained austenite.

Tempering is followed by hardening process to relive stresses induced during hardening process and to make it less brittle. But tempering process does not have any significant effect on retained austenite. Although, it improves toughness of material.

Retained austenite present in the matrix of martensite is comparatively softer phase and limits the enhancement of properties at certain limit for hardening and tempering process. Also, if the steel is not thermally stabilized, the retained austenite will over an extended period of time transform into martensite. This transformation is accompanied by an increase in volume that is called metallurgical growth. Metallurgical growth will cause a change in dimension and form of parts such as bearings even at room temperature.

One of the potential solutions to eliminate retained austenite and to enhance the properties of high carbon steel is to treat the material further by cryogenic treatment. Cryogenic treatment is supplement process to conventional heat treatment process. In cryogenic treatment, once the material is hardened conventionally and brought to room temperature it is further cooled to temperatures as low as -190°C at a uniform cooling rate (ramp down rate). After required cryogenic temperature is achieved, the material is held at that temperature for particular amount of time (holding time or soaking time) which may vary from 16 hours to 48 hours. After that, the material is brought back to room temperature with uniform heating rate (ramp up rate) [1]. Tempering is followed by cryogenic treatment to relieve stresses induced during treatment.

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Many studies have already been done to evaluate the effect of cryogenic treatment mainly on tool steel and some typical alloy steels. The literature emphasis that cryogenic treatment results in enhancement of properties like wear resistance and better tool life performance in case of tool steel [2], [3]. Also, the properties like impact strength, wear resistance, dimensional stability is enhanced in EN 353 which is an alloy steel [4], [5], [6]. Study carried out for optimization of parameters suggests that soaking temperature is the most important parameter that influences the output of the test [7]. Tempering temperature is also plays an important role in final results of cryogenic treatment [8], [9]. It is seen from previous studies that although cryogenic treatment results in significant improvement in wear resistance, it has small amount of improvement on impact strength. To achieve the optimum combination of both improved wear resistance and impact strength, the study takes into consideration double tempering of the material after cryogenic treatment.

The aim of the study is to evaluate the effect of deep cryogenic treatment with double tempering on various mechanical properties and wear behavior of high carbon steel.

II. MATERIALS AND METHODS

The material considered in the study was EN 31, which is a grade of high carbon steel. EN 31 material was subjected to conventional heat treatment as well as cryogenic treatment. The raw material was procured in the form of Ø10 mm rod and 10 mm side square rod for making various specimens. The raw material was subjected to chemical testing to confirm the grade of the material. Chemical analysis was done by using optical emission spectrometer. The test standards followed for chemical analysis was ASTM E 415:2015 [10]. The chemical test results are as shown in table 1.

Table 1 – Chemical Analysis Result

Elements	Results	Required value
% carbon	0.96	0.9 – 1.2
% silicon	0.2	0.1 – 0.35
% manganese	0.32	0.3 – 0.75
% phosphorous	0.015	Max. 0.050
% Sulphur	0.004	Max 0.050
% chromium	1.39	1.00 – 1.60

The chemical analysis confirms that the material is EN 31, a grade of high carbon steel. After the chemical test, the test specimens for impact test, wear test and hardness test were prepared as per respective test standards. The specimens of each group were divided into two sets. One set was subjected to conventional hardening and tempering process. The other set of specimens were subjected to hardening, deep cryogenic treatment and double tempering. Differently treated specimens were designated as in table 2.

Table 2 - Specimen designation

Specimen	Type of treatment
M1	Conventionally Heat Treated
M2	Deep Cryogenic Treated

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1.1 Conventional heat treatment.

Initially, M1 specimens were treated by conventional heat treatment process of hardening and tempering. Hardening was carried out by heating the material in furnace at 850°C, then rapidly quenching it in oil. Tempering was followed by conventional hardening process. Tempering was done by heating the material at 300°C, holding at that temperature for 1 hour and then the specimen was air cooled. Figure 1 shows the flow of conventional heat treatment process.

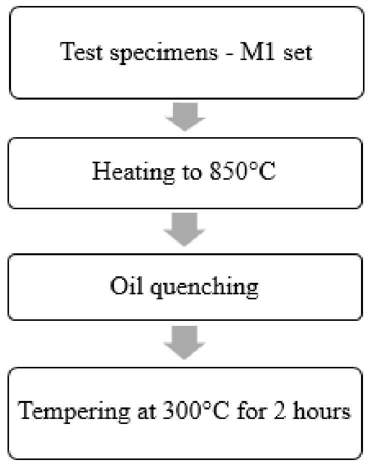

Figure 1 - Conventional heat treatment process

1.2 Deep Cryogenic Treatment

M2 specimens were subjected to deep cryogenic treatment. Cryogenic treatment is the supplement and the substitute of conventional heat treatment. Hence, the material has to undergo conventional hardening process before cryogenic treatment. In the study, the M2 specimens were hardened by heating in furnace at 850°C, then rapidly quenching it in oil. After oil quenching when the material reached to room temperature, it was further cooled till -193°C, with a ramp down rate of 0.5°C/min. Once the material reached to required cryogenic temperature, it was held at that temperature for 24 hours. After that the material was brought back to room temperature with a ramp up rate of 0.5°C/min. Double tempering at 150°C for 1 hour was done after cryogenic treatment. Figure 2 shows the flow of cryogenic treatment process.

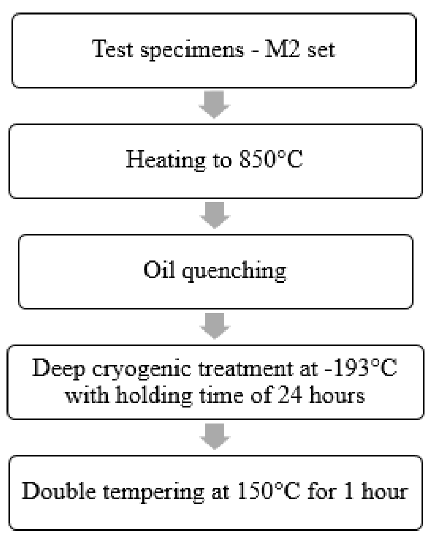

Figure 2 – Deep cryogenic treatment process

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1.3 Tests performed to evaluate the properties of M1 and M2

Flat block of 10mm x 10 mm size was considered for hardness test. Conventionally hardened and tempered block (M1) and cryogenic treated block (M2) were tested for their hardness by Rockwell hardness test set up. The test method used was ISO 6508-1:2015 [11].
The standard test specimen prepared for impact test was as per test method IS 1598(1977) [12]. Izod impact strength was performed on M1 and M2 specimens by same test method to evaluate the impact strength of the respective specimen.
Wear behavior of the conventionally and cryogenic treated material was evaluated by performing pin on disk type wear test on specimens M1 and M2. The standards used for test was ASTM G99 standards [13]. The specimens were prepared as per standards. It consisted of a cylindrical pin of Ø10 mm and length 100 mm with grinded spherical ends. The tests were carried out at three different loads 8N, 10 N and 12N and three different sliding distances of 185 m, 370 m and 555 m. The wear behavior of EN 31 pin specimen was tested against abrasive grinding wheel. Figure 5 and 6 shows the standard and machined test specimen for wear test respectively.
The microscopic examination was carried to determine the changes that occur in microstructure of the cryogenic treated material as compared to conventionally treated material. For examining the microstructure, the sample preparation was done by cutting the cross section of M1 and M2 from the Ø10 mm rod. Later, the cut sections were polished by different grades of silicon carbide papers and lapped with the aid of diamond paste on lapping machine. Samples were then etched with 2% nital solution. After sample preparation, the microstructure of the specimen M1 and M2 were observed under trinocular metallurgical microscope. The test methods adapted for sample preparation, carrying out microstructure examination and interpretation of microstructure were ASTM E112:13 [14] and ASM Vol.9: 2004 [15] respectively.

III. RESULTS AND DISCUSSION

Hardness test results show that there is small amount of improvement in hardness of cryogenic treated specimen. Impact test results show that there is significant improvement in the impact strength of the material which is cryogenic treated and double tempered as compared to hardened and tempered. Table 3 shows results for hardness and impact test of specimens M1 and M2.

Table 3 - Hardness & impact test results

Specimen	Hardness in HRC	Impact strength (N.m)
M1	57, 58, 58	26
M2	59, 60, 60	98

The wear test results were evaluated and compared for M1 and M2. It was found from the wear test results that the wear rate is less for cryogenic treated specimen as compared to conventional treated. Figure 7,8 and 9 shows the plot for wear rate that occurs for M1 and M2 specimens at 185 m, 350 m and 555 m sliding distance respectively. The amount of wear increases with increase in load and sliding distance. From the observations obtained from of the wear test of M1 and M2 specimens, wear resistance was calculated for each case. Table 4, 5 and 6 shows the comparison of wear resistance of M1 and M2 at 185 m, 350 m and 555 m of sliding distance respectively.

From the results of the wear test of M1 and M2 specimens, it can be said that wear resistance of M2 is significantly more than wear resistance of M1. The percentage improvement in wear resistance of cryogenic treated EN 31 specimen is found to be 65.3 % more on an average when compared with conventionally treated EN 31 specimens.

The microstructure as observed under trinocular metallurgical microscope shows that microstructure of the conventionally treated material have more amount of retained austenite. Whereas, deep cryogenic treatment results in decrease in amount of retained austenite. Also, the structure becomes more uniform. Reduction in retained austenite could be the probable reason for

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improvement in wear resistance of EN 31. Also, uniformity in structure might be the reason for improvement in impact strength of EN 31 material. The micrographs at magnification of 100X and 500 X of M1 and M2 specimens of EN 31 are shown in figure 03 and 04.

Table 4 - Wear resistance at 185 m of sliding distance

Load	Wear (Loss of weight in kg)		Wear Resistance		% Improvement in wear resistance
	M1	M2	M1	M2	
8	0.06	0.034	271469	445671	64
10	0.103	0.057	197672	332299	67
12	0.137	0.078	178337	291400	65

Table 5 - Wear resistance at 370 m of sliding distance

Load	Wear (Loss of weight in kg)		Wear Resistance		% Improvement in wear resistance
	M1	M2	M1	M2	
8	0.104	0.058	313234	522510	68
10	0.132	0.073	308488	518932	68
12	0.172	0.097	284096	468643	66

Table 6 - Wear resistance at 555 m of sliding distance

Load	Wear (Loss of weight in kg)		Wear Resistance		% Improvement in wear resistance
	M1	M2	M1	M2	
8	0.1575	0.089	310251	510769	63
10	0.196	0.11	311636	516573	65
12	0.238	0.137	307969	497720	62

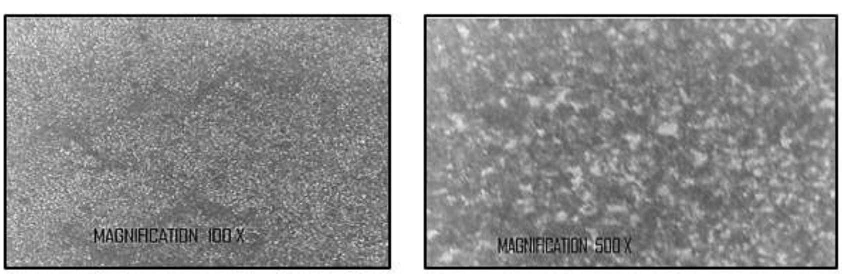

Figure 03 - Microstructure of conventionally treated EN 31 specimen at 100X and 500X magnification

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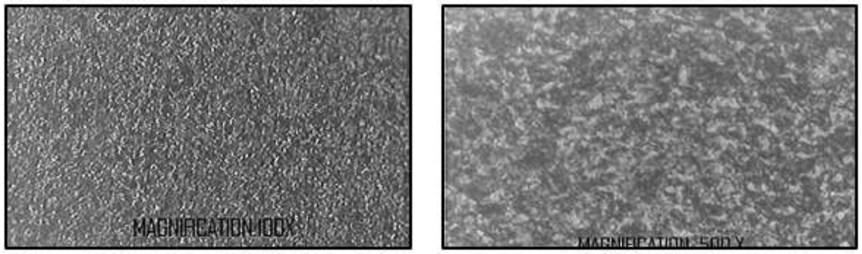

Figure 04 - Microstructure of deep cryogenic treated EN 31 specimen at 100X and 500X magnification

IV. CONCLUSION

In the study conducted, deep cryogenic treatment with double tempering on a grade of high carbon steel material was performed. The properties of cryogenic treated material was compared with conventionally treated material. From the results of the study, following conclusions can be drawn:

- i. Deep cryogenic treatment with double tempering of the material results in balanced improvement in wear resistance as well as impact strength of high carbon steel material.
- ii. The impact strength of deep cryogenic treated and double tempered material is found to be 3.7 times more than conventionally treated material.
- iii. The wear resistance also improves due deep cryogenic treated by 65% on an average when compared with wear resistance of conventionally treated material.
- iv. The microstructure of deep cryogenic treated material reveals reduced amount of retained austenite and more uniformly distributed structure as compared with conventionally treated material.

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