REVIEW ON CRYOGENIC TREARMENT OF STEELS

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Abstract - *Cryogenic treatment (CT) is the supplementary process in the heat treatment process; by which the materials were subjected to sub-zero temperatures to improve mechanical and physical properties. Various advantages like increase in hardness, increase in wear resistance, reduced residual stresses, increase in fatigue Resistance, increased dimensional stability, increased thermal conductivity, toughness were absorbed by the transformation of retained austenite to martensite, eta-carbide formation, precipitation of ultrafine carbides, and homogeneous crystal structure. CT used to treat many type of steels like tool steels, die steels etc. This paper aims to review about various CT involved in treating various steels and their impact on their properties. Optimum parameters to develop maximum wear resistance is discussed. The conclusion tells about the properties improved due to cryogenic treatment.*

Key Words: Cryogenic treatment (CT), Deep cryogenic treatment (DCT), Shallow cryogenic treatment (SCT), Heat treatment (HT), Austenite, Martensite, Wear resistance.

1.INTRODUCTION

Heat treatment is the controlled heating and cooling operations performed on the material. When the material is subjected to heat treatment the atomic structure microstructure may change due to movement of dislocations, increases or decrease in solubility of atoms, increase in grain size, formation of new grains, formation of new different phase and change in crystal structure etc., [1]. Conventional heat treatment include annealing, normalizing, quenching, tempering etc., these process are used to increase the hardness of the material by converting the austenite to martensite structure austenite is the soft phase of metal whereas the martensite is the hard phase of the metal by converting the austenite to martensite hardness increases which will increase the wear resistance of the material [1,10,12, 13]. But the problem in conventional heat treatment is that all the austenite is not converted in to martensite there will be some retained austenite present in the metal in order to eliminate the retained austenite content in the material, subzero treatment is used[1,3,4,5,7].

The complete treatment process of the steels consists of hardening that is austenitizing and quenching, cryo-treatment or deep cryogenic treatment (DCT), and tempering. To achieve better microstructure of the steel and to get most desired properties, it is recommended by the most researchers to execute DCT after completion of quenching and before tempering in conventional heat-treatment cycle as. The complete process sequentially consists of the steps austenitizing, quenching, cryoprocessing and tempering. Fig 1 shows the time taken vs temperature curve.

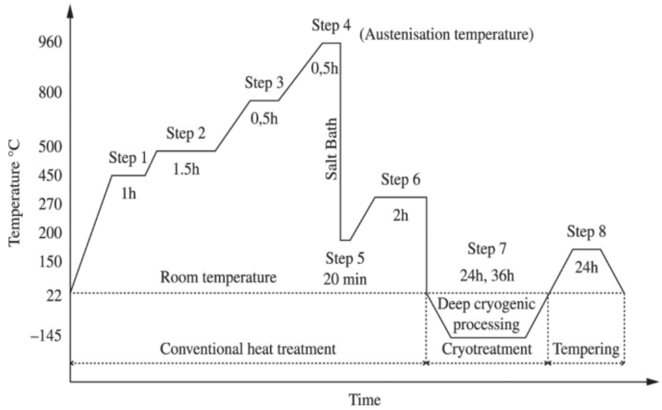

Fig -1 A schematic presentation of the heat treatment [20].

Cryogenic treatment (CT) is the supplement for conventional heat treatment process in which materials are subjected to very low temperatures, subzero temperatures to enhance mechanical properties of material which is treated. Various advantages like improved wear resistance, reduced residual stress, increase in hardness, dimensional stability, fatigue resistance, toughness by transformation of retained austenite in to martensite and precipitation of ultrafine carbides[2,5,7,10,12,] were absorbed. Das [16] reported that deep cryogenic treatment leads to a permanent change in the carbide precipitation kinetics. According to his report, the material upon deep cryogenic treatment contains 22% more carbides per unit volume than the conventionally quenched and tempered one. It is a onetime permanent process in which entire mass is treated so that its benefit can be achieved even after machining [11]. [17] Depending upon the temperature by which the material is treated it is divide in to two types

- Deep cryogenic treatment (DCT) (-196°c).
- Shallow cryogenic treatment (SCT) (-80°c).

DCT is mostly preferred than SCT because of

- Total conversion of retained austenite in to martensite [5,7],
- Increase in carbide density and their uniform distribution[8,20],
- Precipitation of fine η-carbides[4],
- The mechanism by which cryogenic treatment can improve the properties of steel is the formation of very small carbides dispersed in the tempered martensite structure [14, 15].

Deep cryogenic treatment (DCT) is a onetime permanent process, carried out on steel components in such a way that the material is slowly cooled down to the cryogenic temperature, after which it is held at that temperature for a specified period of time and is heated back to room temperature at a slow rate followed by low temperature tempering. The main advantage of DCT is to enhance the wear resistance. Soaking period and soaking temperature plays the vital role in increasing wear resistance with 24% contribution in increasing wear resistance [7]. The parameters which affect the CT are cooling rate, soaking temperature, soaking time, and tempering[1,7].

Mainly the soaking temperature which varies from -80°c to -196⁰c which contributes about 72% in increase of wear resistance, another parameter which contributed 24% in increase of wear resistance is soaking period which varies from 1hr to 48hr [1, 4, 7]. The optimized parameters for improving maximum wear resistance is found to be cooling rate: 1°C/min, soaking temperature: -184°C, soaking period: 36h, tempering temperature: 250°C [7]. Venses et al [1] said in his experiment that the optimum parameter to increase wear resistance for the 100Cr6 bearing steel is the cooling rate of 1.5°C/min, soaking temperature of -150°C, soaking period of 48hrs and tempering temperature of 150°C.

1.1 CRYOGENIC TREATMENT PROCESS

Cryogenic Treatment (CT) of tool materials consists of three stages, that involves cooling of tool material from room temperature, at an extremely slow rate ranging from 0.5 to 1.5ºC/min, followed by soaking for a period ranging from 24 to 36 hours and finally heating up at the rate of 0.5 to 1ºC/min, to room temperature. Though Cryogenic Treatment has been around for many years it is truly in its infancy when compared to heat-treating. Scientific publications on the use of CT on tool materials are rare. Fig no 2 shows the Therefore it requires rigorous experimentations and investigations to ascertain and evaluate the process before commercial exploitation could begin. Cryogenic treatment involves the following sequence:

1. Slow cooling to predetermined low temperature
2. Soaking for predetermined amount of time
3. Slow heating to room temperature
4. Tempering

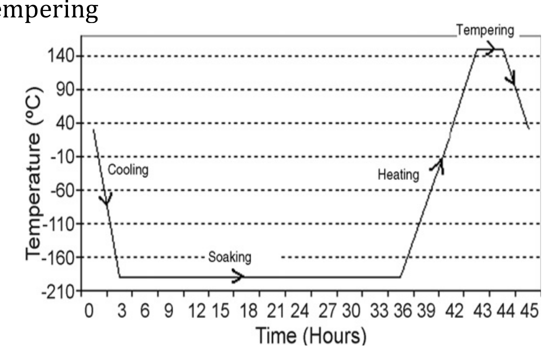

Fig. 2 – Standard DCT cycle [7].

Mostly samples were cryotreated in a Cryogenic processor. The cryogenic processor consists of a treatment chamber, which is connected to a liquid nitrogen tank through a vacuum insulated hose. The thermocouple inside the chamber senses the temperature and accordingly the temperature controller operates the solenoid valve to regulate the liquid nitrogen flow. The programmable temperature controller of the cryogenic processor can be used to set the cryogenic treatment parameters, which in turn controls the process parameters. Fig. 3 shows the schematic diagram of the Cryogenic processor [7].

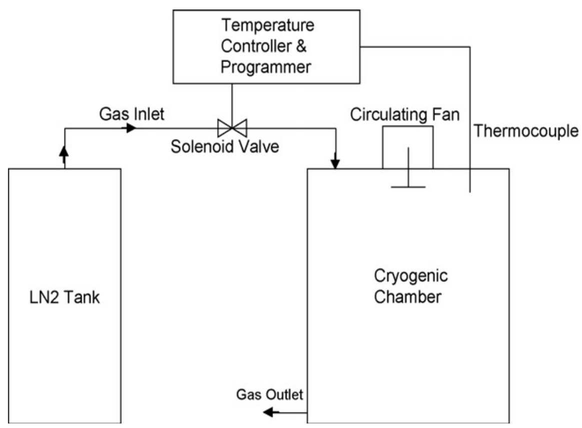

Fig. 3 – Schematic diagram of cryogenic processor [7].

2. LITERATURE SURVEY

Idayan et al [23], In this paper, effect of cryogenic treatment is compared to the conventional heat treatment. Effect of cryogenic treatment on the mechanical Properties of AISI 440C Bearing Steel was studied. Cryo treated samples show improved properties then heat treated samples and hardness of the sample is improved by 7% when subjected to DCT and by 4% when subjected to SCT compared to heat treated samples. The retained austenite percentages of these samples were 29%, 8% and 5.7%. In the overall analysis DCT shown the higher reduction in the retained austenite and comparably higher hardness than that of SCT.

Ilyas et al., [20] In this paper, effect of CT on the corrosion of AISI D3 Steel was studied. The alloy AISI D3 is a high-carbon, high-chromium steel developed for various applications which require high resistance to wear, severe pressure and abrasion. Four types of uncoated AISI D3 steel used in the study were conventionally heat treated, 24 h cryogenically treated, 36 h cryogenically treated and 36 h cryogenically treated and then 2 h tempered at 150 °C. Due to a more uniform carbide distribution in association with a higher carbide percentage, the corrosion behavior of deep cryogenically treated samples had been enhanced, for the longer holding durations (e.g., 36 h), the corrosion resistance has been improved as a result of the increase in the carbide percentage by 3% and a more uniform carbide distribution. Cryogenic treatment causes the formation of martensite, which can improve the mechanical properties, but unfortunately can also reduce the good resistance to corrosion. In steels, martensite is more susceptible to corrosion than austenite. All the samples were corroded in 3.5% NaCl, the corrosion resistance of these samples is as follows HT > CT24hr > CT36hr > CT36hr2hrtemp.

Arvind et al [10]In this paper, effect of CT on AISI-D2 Tool steel is studied. The samples were cryo treated at -196°c for 24 hours. After the CT it showed that the carbon bonding the carbon atoms are very closed to iron atoms thus they give strong Bonding characteristics to the steel, austenite phase is gradually reduced to martensite phase and the hardness is improved from 806 h.v to 827 h.v.

Hasan et al [9] In this paper, effect of CT CBN inserts is studied. The materials used in the study are Titanium and AISI 440 C hard Martensitic Stainless steel for testing the machining parameters of cryo treated inserts. The parameters used for testing are cutting velocity, feed rate, depth of cut. The performance such as tool wear, surface roughness were evaluated. The material is slowly cooled to -196°C and soaked at deep cryogenic temperature for 20 hours. Cryogenically treated CBN inserts produced less tool wear on titanium than AISI 440 C Martensitic stainless steel. Flank wear in turning Titanium alloy was low than AISI 440 C stainless steel, high flank wear and formation of built up edges was due to hard carbide present in the material. Titanium produced low surface roughness where it seem to have high plastic behavior deformation than stainless steel and always produced low surface roughness for all parameters at higher depth of cut. Even though the inserts were strengthened by cryogenic process to resist wear, the temperature at tool tip - work piece interface destroys the special property induced in the inserts. More crater wear formation found during turning of AISI 440 C stainless steel than Titanium turning.

P.SUCHMANN et al [4] in this paper impact of deep cryogenic treatment on the wear resistance and microstructure of the X37CrMoV5-1 hot work steel is studied. Four samples used were heating and double tempering, heating, DCT (6h, 12h, 20h) and double tempering. The inferred result showed that the deep cryogenic treatment dramatically improves the material's wear resistance. The wear resistances increases with the holding time at the deep cryogenic temperature but after increasing further resulted in the decline of the wear resistance. Final results in the study showed that deep cryogenic treatment of the X37CrMoV5-1 (H11) steel dramatically improves the resistance to wear at high temperatures.

Pugh et al [5] In this paper, the effect of DCT on the mechanical properties of AISI 4340 steel was interpreted. To compare the effects of DCT on the samples, conventional hardening was used as a reference. A group of specimens were conventional hardened which included, austenitizing followed by oil quenching. Then tempering was carried out at three temperatures of 200, 300, and 450°C. The DCT treatment is of slowly cooling oil-quenched specimens to approximately −196°C and holding at this low temperature for 24 h and gradually the specimens were brought back to room temperature. The austenite percentage decreased from 5.7 to 4.2% due to cryogenic treatment, the cryogenically treated samples show a slightly higher level of hardness compared to the conventional heat treatment. The cryo-treated samples produced a slight increase in hardness 2.4% but reduced the toughness to 14.3%.

Yi He et al[22] In this paper, the 2800-Mpa grade maraging steel is strengthened by CT. Cryogenic treatment at 200 K, transformed full of low-carbon martensite structure without retained austenite, a complete transformation to martensite also makes the steel an obvious increase of hardness. The strength of the material reached up to 2700 MPa due to the removal of the retained austenite by CT before aging. Though the hardness increased fracture toughness doesn't reduced it still remained at a relatively high level, above 30 MPa m1/2. This is achieved in the study by modification of Ni, Co, Mo and Ti contents based on 13Ni(400), as well as a cryogenic treatment before aging.

Paolo et al [6] This paper focuses on the comparison between the results given by different sequences of DCT and tempering performed after the conventional case hardening. The results point out substantial hardness increases of hardness from +0.6 HRC to +2.4 HRC for all the cryotreated groups and a remarkable enhancement of the tensile strength up to 11%. The results point out that the hardness of the 18NiCrMo5 steel has been increased by the DCT, the hardness enhancement is greater when the treatment is performed between the case hardening and the final tempering. The pre-tempering DCT allows to obtain substantial increases of the superficial hardness, up to 2.4 HRC points from the initial value. Over all hardness of the samples were improved due to DCT.

Valmik et al [8] In this paper DCT and nitrocarburized AISI H-13 tool steel is compared to DCT only and nitro carburized only samples. The commercial grade of AISI H-13 tool steel was used in the study, the microstructure of HT sample showed mainly primary and secondary carbides but cryogenically treated sample showed very fine carbides along with primary and secondary carbides which increase carbide density. The carbide density was 4.3 % for HT sample. In cryogenically treated samples it increased up to 5.7 %. The initial hardness of all samples were in the range of 49-50 HRC. But after conventional nitro-carburizing increased this hardness to 60 HRC at surface, core material hardness reduced to 40 HRC because of thermal softening. Whereas the combination of treatment where deep cryogenic treatment followed by nitro-carburizing (DCT+NC), the surface hardness increases up to level of 70 HRC and core material hardness reduced. Nitro-carburizing on cryogenically treated samples results in higher surface hardness and reduction in wear rate as compared to nitrocarburizing of conventional samples. The performance of nitro-carburizing was enhanced by carrying out deep cryogenic treatment before it. The result showed that samples which were cryogenically treated before nitro-carburizing (DCT+NC) exhibits better performance as compared to other sample treatment conditions.

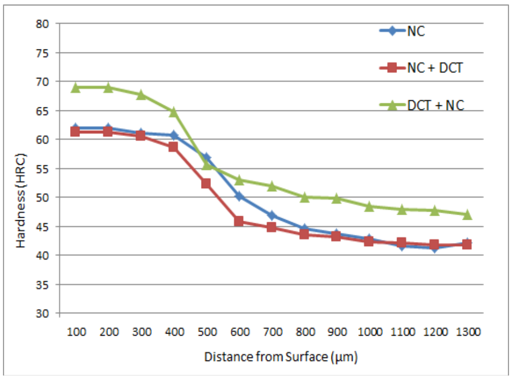

Chart-1: Hardness distribution along with surface layers[8].

Venses et al [1] This paper studies about the optimization of the deep cryogenic treatment process parameters for the improvement of wear resistance of 100Cr6 bearing steel using Taguchi technique. The process parameters considered in the experiment for the optimization are the cooling rate, soaking temperature, soaking time and tempering temperature with the target of achieving increased wear resistance. The experiment is as follows the material is soaked at temperatures of approximately -130°C,-150°C and -185°C and it is soaked for a period of 16,24, 36 and 48 hrs the samples were tempered at 150°C, 200°C and 250°C. The optimum parameters for the 100Cr6 bearing steel was found to be the cooling rate of 1.5°C/min, soaking temperature of -150°C, soaking period of 48hrs and tempering temperature of 150°C, based on the Taguchi technique, at these parameters the material samples showed maximum wear resistance and minimum wear loss.

Darwin et al [7] In this paper, the Taguchi method has been used to optimize the process parameters of DCT for a commercial piston ring, made up of 18% Cr martensitic stainless steel (SR34) to obtain maximum wear resistance. The DCT parameters considered for optimization were: the cooling rate, the soaking temperature, the soaking time, the tempering temperature and the tempering time. Cryogenically treated SR34 piston ring shown improvement in the wear resistance by 43.8% at optimum levels. Soaking temperature is the most significant factor and the maximum percentage contribution of soaking temperature on the wear resistance of the material was 72% and the best soaking temperature in the possible range is −184 °C. The second significant factor is the soaking period and it contributes 24% for the improvement of wear resistance and the best level for this factor is 36 h. The third significant factor is the cooling rate. The maximum percentage contribution of cooling rate on the wear resistance was 10% and the best level was determined as 1 °C/min. The tempering temperature shows only little significance and its contribution on the wear resistance was 2% only. The optimum level of tempering temperature was arrived at 250 °C. Wear tests were conducted on untreated samples and the results were compared with that of the confirmation test results. This shows that the cryogenically treated SR34 samples as per the arrived optimum conditions improve in wear resistance by 43.8%. The order of importance is as follows (1) soaking temperature; (2) soaking period; (3) cooling rate; (4) tempering temperature. Finally the optimum levels are found to be soaking temperature,−184°C; soaking period, 24 h; cooling rate, 1°C/min; tempering temperature, 200°C and to maximize the wear resistance were, cooling rate: 1°C/min, soaking temperature:−184°C, soaking period: 36h, tempering temperature: 250°C.

S. T. Liu et al [2] this paper studies about the detailed analysis and brief summary of the influence of cryogenic treatment on microstructure after quenching process or quenching plus tempering process, first and second carbides, content of retained austenite, surface hardness, mechanical properties and antiwear ability of die steels. Cryogenic treatment helps to reduce the hardness fluctuation range and increased hardness up to 10 - 14 HRC. Experiments in the study revealed that the cryogenic treatment reduces the volume of retained austenite to improve the sizestability and antiwear ability, the cryogenic process also reduces the width of martensite laths and thus improves the life of the die steels.

3. CONCLUSIONS

The conclusion of the review study is as follows

- Hardness of the CT samples increased than conventionally heat treated samples, this is due to the transformation of retained austenite into martensite.

- Life of the cryo-treated tool steels and die steels is improved as compared to the HT steels.
- Deep cryogenic treatment is mostly preferred than shallow cryogenic treatment because of its lower deep freezing temperatures which showed more improved properties like , improved wear resistances, increased hardness, dimensional stability and precipitation of ultrafine carbides.
- The parameters to be considered during DCT are austenitizing temperature, quenching temperature, rate of cooling soaking temperature, soaking period, rate of warming-up, tempering temperatures and tempering period. The main parameters which contribute for improved wear resistance are soaking temperature, soaking period and rate of cooling.
- In tool steels high flank wear is found due to the formation of hard carbine in tool steels.
- The impact energy for the conventional, shallow cryogenic and deep cryogenic treated samples were comparable and there is minor difference in impact energies and the mode of fracture was brittle for all the specimens.

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