A Review on the Cryogenic Treatment of Stainless Steels, Tool Steels and Carburized Steels

Savyasachi N
Department of Metallurgical and Materials Engineering,
Amal Jyothi College of Engineering,
Kanjirappally, India

Rincy Reji
Department of Metallurgical and Materials Engineering,
Amal Jyothi College of Engineering,
Kanjirappally, India

Jain Anna Sajan
Department of Metallurgical and Materials Engineering,
Amal Jyothi College of Engineering,
Kanjirappally, India

Ajmal Mohamed Rafi
Department of Metallurgical and Materials Engineering,
Amal Jyothi College of Engineering,
Kanjirappally, India

Abstract:- Conventional heat treatment of steel involves heating the material to its austenitizing temperature, followed by quenching and tempering. Rapid cooling transforms austenite to martensite which begins and finishes at the martensite start temperature (M_S) and martensite finish temperature (M_F) respectively. The presence of alloying elements except aluminium and cobalt lowers both the M_S and M_F temperature which can go down to subzero temperatures. Thus CHT cannot fully transform the austenite to martensite resulting in the formation of retained austenite within the martensitic structure. The retained austenite in the steel can transform to martensite under working conditions. This newly formed martensite being untempered is brittle and reduces the product life of the material. Thus, cryogenic treatment is done prior to tempering and after quenching to convert the retained austenite to martensite. This paper aims to review the cryogenic treatments of stainless steels, tool steels and carburized steels so far and their effect on their properties.

Keywords:- *Retained Austenite; Cryogenic Treatment; Stainless Steels; Tool Steels; Carburized Steels.*

I. INTRODUCTION

Heat treatments have always been important in imparting required properties in metals and alloys. This improvement in properties is achieved by changes in microstructure [1]. Heat treatment of metals is not only limited to high temperatures, but also to sub-zero temperatures, which is called cryogenic treatment. Cryogenic treatment involves cooling metals or alloys to sub-zero temperatures to enhance various mechanical properties. Liquid nitrogen is commonly used to cool metals to temperatures as low as -196^0 C [1]. .Quenching of steels from austenitizing temperature transforms austenite to martensite when the cooling rate is higher than the critical cooling rate[2]. Tempering can make this martensite harder and tougher. Quenching cannot transform all the austenite to martensite, even at high cooling rates. Hence, the martensitic structure will also contain some amount of untransformed austenite called retained austenite which can reduce the product life [3]. In order to reduce the amount of retained austenite within the martensitic structure cryogenic treatment is done after quenching and before tempering.

Stainless steels are alloys of iron and carbon and containing a minimum chromium content of 10.5%. Chromium imparts corrosion and heat resistance to the steel. Other elements like nickel, molybdenum, aluminium, titanium, copper, nitrogen may be added to improve certain physical characteristics of the steel. Stainless steels have excellent corrosion resistance and they are 100% recyclable. Due to their excellent physical properties, they find applications in cutlery and kitchenware, medical applications, construction, power generation among others applications [4]. Tool steels are used for cutting, forming or shaping a material [3]. They are carbon-, alloy-, or high-speed steels, capable of being hardened or tempered. Tool steels are used in many applications requiring strength, toughness, hardness and resistance to wear [5] Carburizing steel is produced when low carbon steel is subjected to a carburizing liquid or gas at high temperature. Carburized steels possess more wear resistance, fatigue strength and hardness [6].

The aim of this study is to give a brief introduction on martensitic transformation, cryogenic treatment and its significance, and understanding the effect of cryogenic treatment on stainless steels, tool steels and carburized steels.

A. *Martensitic Transformation*

In the study of quenching of steels, researchers found the transformation of face centered austenite into lens shaped or plate like regions with body centered tetragonal (bct) lattices. Crystals formed by such transformations are called “martensite” and the lattice transformations are called “martensitic transformations” [7]. On quenching, the rate of diffusion is lowered and hence no phase transformations dependent on diffusion take place. Thus martensitic transformations are diffusionless transformations. During the transformation, gamma iron changes to alpha iron. Since no diffusion of carbon and other metallic atoms takes place, the chemical composition of alpha iron and gamma iron will remain the same. The solubility of carbon is greater in gamma iron than in alpha iron. Thus the alpha iron (martensite) formed will be supersaturated.

- *M_S and M_F temperatures*

The formation of martensite begins at the M_S temperature. As the temperature decreases, more and more austenite transforms to martensite and at the M_F temperature the formation of martensite comes to an end. Lowering the temperature below M_F does not result in any further martensitic transformation. The M_S temperature is constant for particular steel and does not change with varying cooling rates. In contrast, M_F temperature can be lowered by decreasing the cooling rates [1].

The M_S lowers with the addition of all alloying elements except aluminium and cobalt [1]. The relation between alloying elements and MS temperature is given by [8]:

$$M_S\ (°C) = 561 - 474(\%C) - 33(\%Mn) - 17(\%Ni) - 17(\%Cr) - 21(\%Mo) \quad (1)$$

Figure 1 shows the effect of carbon content on M_S and M_F temperatures of steel.

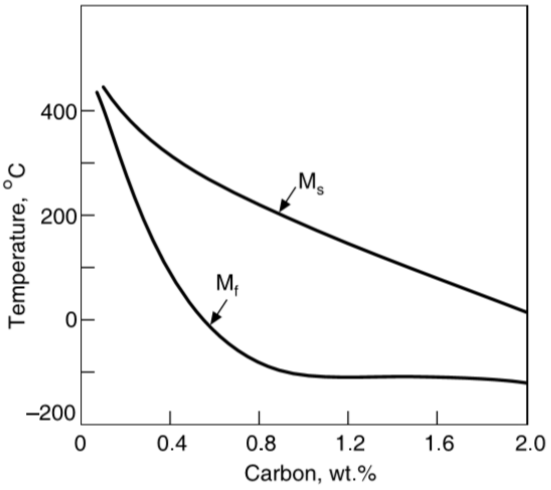

Fig 1:- Effect of carbon content on M_S and M_F temperatures of steel [1]

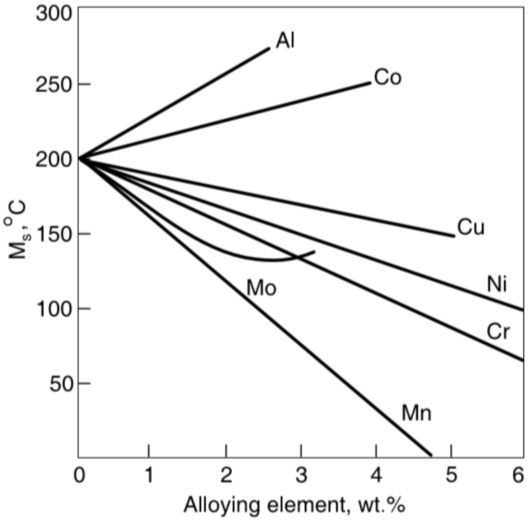

Fig 2:- Effect of alloying elements on M_S temperature of a steel containing 1% carbon [1]

- *Retained austenite*

From figure 2, it is evident that all alloying elements except aluminium and cobalt tend to lower the M_S point of steel. The decrease in M_S point is proportional to the amount of elements dissolved in steel. Of all the alloying elements, carbon has the greatest effect in reducing the M_S temperature. Manganese, chromium and nickel follow this order [9]. High carbon steels and tool steels have their M_S temperatures as low as room temperature.

Thus, conventional heat treatment (CHT) which includes heating the material to its austenitizing temperature for a particular time, followed by quenching and tempering, cannot transform all the austenite to martensite resulting in the formation of retained austenite within the martensitic structure. In other words, retained austenite is the untransformed austenite formed during austenite to martensitic transformation. This retained austenite is detrimental to the life of the product. During the working conditions of the material, the retained austenite can transform to martensite. This newly formed martensite being untempered is brittle and can reduce the product life [10].

The presence of retained austenite can be reduced or eliminated by two heat treatment processes. They are: Cryogenic treatment or sub-zero treatment and Tempering [1].

II. REVIEW ON CRYOGENIC TREATMENT

Cryogenic treatment (CT), also known as sub-zero treatment, consists of cooling the steel to sub-zero temperatures, which is lower than the M_S temperature. This is done after quenching and prior to tempering. Thus, all the retained austenite gets converted to martensite. In the 1930s, German companies used cryogenic treatment for components of aircraft engines [3].

A. Types of cryogenic treatment

Depending on the temperature at which the cryogenic treatment is carried out, it is classified into two types:

- Deep Cryogenic Treatment (DCT), which is carried out at -196°C using liquid nitrogen and slowly brought to the room temperature.
- Shallow Cryogenic Treatment (SCT), which is carried out at -80°C and then soaked in room temperature [3].

B. Cryogenic Heat Treatment Process

The cryogenic heat treatment process for steels consists of:

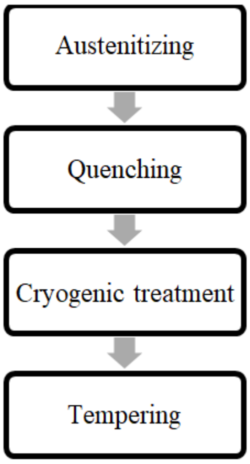

Fig 3:- Cryogenic heat treatment process [3]

The heat treatment process includes heating to austenitizing temperatures, which will produce a microstructure composed of austenitic phase and primary carbides. Austenitizing is followed by rapid cooling, or quenching. This transforms some or most of the austenite into the higher strength martensitic structure with supersaturated carbon. Tempering allows the supersaturated carbon to form carbides, called transition carbides that relieve micro stresses in the martensite matrix and prevent cracking of the part [11].

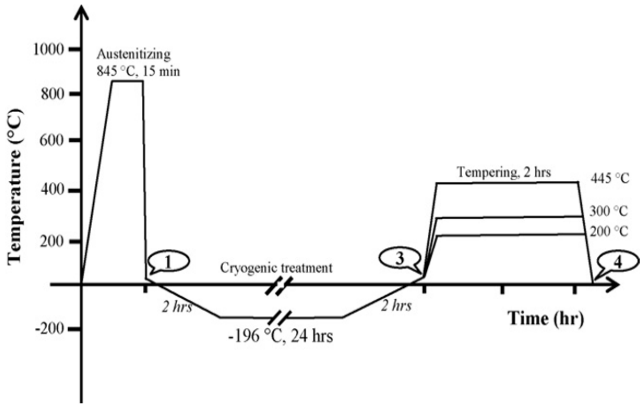

Fig 4:- Cryogenic thermal cycle for the steel [12]

Various advantages of cryogenic treatment are increased hardness, increased wear resistance, reduced residual stresses, fatigue resistance, increased dimensional stability, increased thermal conductivity and toughness. These advantages are the results of transformation of retained austenite to martensite, the metallurgical aspects of eta-carbide formation, precipitation of ultra-fine carbides, and homogeneous crystal structure [11].

C. Treatment Parameters

The typical process parameters in cryogenic treatment are minimum temperature, hold time, cooling and heating rate [3].

J.D. Darwin et al. [13] used Taguchi method to optimize the process parameters of DCT for a 18% Cr martensitic stainless steel. The DCT parameters such as the cooling rate, the soaking temperature, soaking time, tempering temperature, and tempering time, were considered for the optimization study and the optimum levels were found out. The authors showed that soaking temperature is the most significant factor and the optimum soaking temperature for reducing wear resistance is -184°C. The contribution of soaking temperature on wear resistance was found to be 72%.

D. Senthilkumar and I. Rajendran et al. [14] have studied the effect of deep cryogenic treatment parameters of SS 4140 to reduce wear loss. The four process parameters considered for optimization are hardening temperature, soaking period, tempering temperature, and tempering period. They concluded that hardening temperature is the most important factor in the DCT process by ANOVA analysis and the optimum hardening temperature is 880°C. The contribution of hardening temperature to wear loss is 17.34%.

M. Arockia Jaswin et al. [15] used Taguchi method along with the grey relational analysis to optimize the deep cryogenic treatment parameters for En 52 Steel. The factors studied for optimization were cooling rate, soaking temperature, soaking period, and tempering temperature. Soaking period was found to be the most significant parameter with a contribution of 49.8% and the soaking period was 36 hours.

III. EFFECT OF CRYOGENIC TREATMENT ON VARIOUS STAINLESS STEELS

Many researchers worked on the cryogenic treatment of stainless steels to study its effects on their mechanical and microstructural properties. Some have worked to find out the optimum parameters for deep and shallow cryogenic treatment.

R.F. Barron et al. [16] studied various metals to determine which material responds better to shallow and deep cryogenic treatment in improving abrasive wear resistance. Among those samples were martensitic SS440, austenitic SS303 and feritic SS430. The difference in wear resistance between the SCT and DCT samples was found to be less than 10%. The wear resistance for shallow, deep and conventional heat treated specimens is shown in table 1 below.

AISI No	Wear Resistance (R_w)		
	Conventional Heat Treated	*Shallow cryogenic treated*	*Deep cryogenic treated*
SS440 (Martensitic SS)	1447	1853	1763
SS303 (Austenitic SS)	14.3	15.06	15.84
SS430 (Ferritic SS)	11.14	12.94	13.35

Table 1:- Wear test of various Stainless steels [16]

A. *Martensitic Stainless Steels*

G. Prieto et al. [17] investigated the microstructural changes and effect of cryogenic treatment of AISI 420 stainless steel on its hardness and impact energy. X-ray diffraction (XRD) was used for phase analysis and characterization, while scanning electron microscopy (SEM-EDX) and Energy Dispersive Spectrometry (EDS) was used for carbide volume fraction, size and composition evaluation. The fractured surfaces of the charpy impact test specimens were analyzed using scanning electron microscopy. Results showed that cryogenic treatment produces homogeneous precipitation of small carbides and also cryogenic treatment can be done on low carbon steels to improve their microstructural and mechanical properties. The soaking time for the specimens was 2 hours and they were able to increase the hardness and impact energy by 5% and 10% respectively.

A. Idayan et al. [10] showed the influence of deep and shallow cryogenic treatment on the mechanical properties of AISI 440C. The percentage of retained austenite in conventional heat treated, shallow and deep cryogenic treated samples were found out using ImageJ software and they were found to be 29%, 8% and 5.7% respectively. Hardness of DCT and SCT treated samples increased compared to the conventional heat treated specimens. Fractography analysis using SEM clearly depicts the formation of flat facets in SCT specimens, while in DCT specimens' micro cracks were observed.

B. *Austenitic Stainless Steels*

Paolo Baldissera et al. [18] [19] revealed the influence of deep cryogenic treatment on the hardness, tensile properties, fatigue and corrosion resistance of AISI 302 stainless steel. In [13], the tensile and hardness tests results are compared to other martensitic stainless steel literature. The author has also analyzed the influence of two DCT parameters that is soaking time and minimum temperature through a design of experiment (DOE) and by first approximation model. In [14], the authors have discussed the effect of AISI 302 SS on fatigue and corrosion resistance. The test was done for both solubilized and hardened steel. The fatigue resistance was improved in solubilized SS302 while there were no significant changes in the hardened material. However, there was no change in corrosion resistance of both the materials.

P. Johan Singh et al. [20] compared the fatigue life extension of notches in AISI 304L weldments for samples with and without cryogenic treatment. However, it was found that fatigue crack initiation life from notches of cryogenically treated samples was enhanced.

Jitendra Upadhyay et al. [21] tested the effect of deep cryogenic treatment on the mechanical and metallurgical properties of SS316. Deep cryogenic treatment was followed by tempering at two temperatures: 350°C and 250°C. Results showed that the samples which were tempered at 250°C possessed greater hardness and tensile strength. However, the toughness of both the samples negatively affected. Optical microstructural analysis revealed the refinement of grains which could be contributing factor for the decrease in toughness. Micro hardness test was also done for the base sample, and the deep cryogenic treated samples which were tempered at two temperatures.

C. *Ferritic Stainless Steels*

S. Zirafar et al. [12] investigated the effect of DCT on the mechanical properties of 4340 ferritic stainless steel. Rotating fatigue test, hardness and impact test was done. The fatigue strength and hardness of cryogenically treated specimens were higher, while the toughness was lower compared to the conventionally treated specimens. The reason for improvement in fatigue resistance and hardness was shown through neutron diffraction in which the transformation of retained austenite to carbon was found to be the reason

IV. CRYOGENIC TREATMENT OF TOOL STEELS

There are a lot of confusions regarding the various parameters used in cryogenic treatment, they are still widely used in tool steel to improve their product life. An enhanced product life will improve productivity, efficiency and economy.

D. Mohan et al. [22] showed that cryogenic treatment can impart 110% improvement in the tool life. It was noticed that cryogenically treated T1, M2 and D3 samples improved its product life by 3%, 10% and 10.6% respectively. The wear tests of these samples were done by Flank wear test and Sliding wear test. Some samples were TiN coated, but it was found that the cryogenic treated specimens were superior to the TiN coated specimens.

R.F. Baron et al. [16] studied about various tool steels to find out which material responds better to SCT and DCT in improving abrasive wear resistance. The following tool steels were studied:

AISI No	**Wear Resistance (R_w)**		
	Conventional Heat Treated	***Shallow cryogenic treated***	***Deep cryogenic treated***
S-7	5.12	12.38	13.38
P-20	6.25	7.70	6.08
O-1	514	1140	1399
A-2	48.8	47.9	54.4
A-6	2165	1151	2094
A-10	1035	2386	2738
D-2	199	630	1628
H-13	30.2	49.7	63.3
T-1	1613	2287	2843
T-2	5751	5338	4142
M-1	776	1129	1748
M-2	3900	4388	4698

Table 2:- Wear test of various tool steels [16]

It was concluded from the above study that lowering the temperature enhances the wear resistance in steels.

M. Villa et al. [23] subjected D2 tool steel to various austenitization treatments, followed by various cryogenic and tempering treatments. Microstructure and mechanical properties of these samples were studied by X-Ray Diffraction (XRD), scanning electron microscopy (SEM), vibrating sample magnetometry, hardness measurement and tribology tests and found that some amount of retained austenite still remained in the cryogenic treated samples.

Nandakumar Pillai et al. [24] worked on the effect of cryogenic treatment of AISI D series cold worked tool steel. D2, D3, D5, D6, and D7 grades of tool steel were selected for this study. It was observed that retained austenite reduction in D3 tool steel was greater than D2 and D6. The microstructure of conventional heat treated specimens contained retained austenite and martensite, while the cryogenic treated specimens reduced the amount of retained austenite percentage and made the martensite finer.

Marcos Pérez et al. [25] tested the mechanical properties of cryogenically treated AISI H13 tool steel. Tensile, hardness and fracture toughness tests were also done. Better mechanical properties were obtained in cryogenically treated samples compared to conventionally treated samples. SEM and X-ray diffraction analysis were used to study microstructural evolution. Microstructure revealed the presence of dispersed network of fine carbides. It was also concluded that there still exists a small amount of retained austenite within the tool steel which cannot be reduced by heat treatment.

J.Y. Huang et al. [26] performed a microstructure analysis on cryogenically treated M2 tool steel. Both shallow and deep cryogenic treated samples were tested and compared the results with conventional heat treated samples. TEM micrographs were obtained. It was found that carbide formation increased and was more homogeneous after the cryogenic treatment. The authors concluded it to be the reason for the improvement in wear resistance.

V. Leskovsek et al. [27] studied the influence of deep cryogenic treatment on the wear resistance of vacuum heat-treated ESR AISI M2 high-speed steel. Cryogenically treated steels exhibited superior wear resistance compared to vacuum heat-treated steel. Wear resistance was found to improve with increasing tempering temperature. Samples which showed sufficiently high values for both fracture toughness and hardness exhibited superior wear resistance compared to samples which had extremely high values for either fracture toughness or hardness only.

Aleksander CISKI et al. [28] investigated the effects of cryogenic treatment on properties of HS6-5-2 high speed tool steel. DCT of HS6-5-2 caused a reduction in the dimensions of martensite plates, which resulted in the reduced hardness of the material and improved its impact strength. Drills that had been treated to deep cryogenic treatment demonstrated reduced durability during industrial tests, despite showing lower wear intensity during three roller-cone test under 100 MPa load.

Zbigniew Zurecki et al. [29] studied the effects of cryogenic quenching on the microstructure and mechanical properties of A2-grade tool steels. Cryogenic quenching of A2 steel resulted in hardness and wear resistance, at the expense of reduced impact strength. Increased holding time during cryogenic treatment resulted in higher dark carbide number frequency, increased hardness, increased wear resistance, and reduced impact strength. The study showed that cryogenic treatment must be done immediately after quenching from austenitic temperature prior to tempering for the cryogenic treatment to be effective.

D. Das et al. [30] studied the effects of cryogenic treatment on the wear behavior of AISI D2 steel. AISI D2 steel subjected to cryogenic treatment at 77K showed superior wear resistance compared steel subjected to conventional heat treatment. This improvement in wear resistance is due to the reduction in retained austenite content and increased number of finer secondary carbides distributed homogeneously within the microstructure of the steel. Wear resistance is shown to improve with increasing holding time up to 36 hours at 77 K. Further increase in holding time results in monotonic decrease in wear resistance.

V. Firouzdor et al. [31] subjected an M2 HSS drill to austenizing temperature of about 1100^0 C and gradually subjected it to deep cryogenic treatment at about -196^0 C for 24 hours, while another drill was subjected to tempering at 200^{0}C for 1 hour after cryogenic treatment. A reference drill was also taken which was not subjected to any extra heat treatment. Cryogenic treatment was shown to improve the wear resistance of the drill by 77%, while drills that were subjected to both cryogenic treatment and tempering showed improvements in wear resistance by 126%. The improvements in wear resistance are due to the precipitation of fine carbides during cryogenic treatment. Increase in hardness values due to the transformation of retained austenite to martensite also contributes to the improvements in wear resistance.

H.G Naneesa et al. [32] investigated the martensitic transformation of the AISI D2 tool steel which was cooled continuously from its austenitizing temperature using 10 Ks^{-1} or 50 Ks^{-1} cooling rates up to the deep cryogenic temperature. Dilatometry was used for it. SEM revealed the presence of lathe and plate martensite and transmission electron microscope revealed the presence of nano twined martensite. One of the main observations was better dimensional stability.

D. Das et al. [33] examined the fracture toughness of AISI D2 steels using the sub-zero treatments – cold treatment, shallow cryogenic treatment and deep cryogenic treatment comparing with the conventional heat treatment process. They also identified the mechanism of initiation of fracture as cracking of primary carbides and the growth of micro voids by de-cohesion of secondary carbides. The variations in fracture toughness values have also been noted.

N.W. Khun et al. [34] investigated the effects on mechanical and tribological properties of AISI D3 tool steel due to deep cryogenic treatment with a consolidated connection between their wear resistance and hardness. It was found that an additional DCT process to the conventionally heat treated samples increased the hardness with the elimination of more retained austenite and formation of homogeneous carbide formation.

R. H. Naravade et al. [35] examined the wear behavior of D6 tool steel subjected to deep cryogenic treatment for 36 hours. It was identified that the presence of retained austenite was decreased after the cryogenic treatment and hence the wear resistance and hardness is improved compared to the conventional heat treatment process. The authors also investigated the significance of multiple tempering before and after cryogenic treatment on friction and wear behavior of the samples. A variety of heat treatments were carried out and finally concluded that cryogenic treatment improved the wear resistance and hardness the most.

N.B. Dhokey and S. Nirbhavane et al. [36] did a test using pin-on-disc machine to find out the role of multiple tempering on D3 tool steel which has undergone cryogenic treatment. Wear rate deteriorates during subsequent tempering and this process is an influential function of fine carbides and its distribution. The mechanism behind improving wear resistance of the particular material was identified using the results from SEM analysis, hardness data and microstructures of the worn out sample.

A. Akhbarizadeh et al. [37] studied how deep cryogenic treatment affected the austenite grain size, hardness and wear resistance of D6 tool steel. The microstructural properties and phases present in the heat treated samples were examined using scanning electron microscopy (SEM) and x-ray diffraction (XRD) method. From the results it can be concluded that during the cryogenic treatment the retained austenite is almost completely transformed to martensite.

K. Moore and D.N Collins et al. [38] determined the effect of various factors affecting the hardness of steels which has undergone three types of cryogenic treatment. The main observation was the increase in hardness greatly dependent on cryogenic temperature and independent of soaking time. The three materials selected for this study was H13, D2 and D17.

V. CRYOGENIC TREATMENT OF CARBURIZED STEELS

Paolo Baldissera et al. [39] investigated the effect of deep cryogenic treatment on the static mechanical properties of 18NiCrMo5 carburized steel, which is used commercially for manufacturing gears. Deep cryogenic treatment after tempering showed an increase in hardness without much change in its ultimate tensile strength. Increase in the soaking time during deep cryogenic treatment corresponds to an increase in hardness values. Tempering of steel after deep cryogenic treatment results in increased hardness as well as increased values of ultimate tensile strength by up to 11%. Tensile test of cryogenically treated groups shows a significant increase in Young’s modulus of the material. Fractographic analysis of the steel specimens shows no changes in fracture mechanisms for different heat treatment methods.

A. Bensely et al. [40] studied the effects of shallow cryogenic treatment and deep cryogenic treatment on the tensile properties of case carburized 815M17 steel. The investigation revealed a reduction in tensile strength by a factor of 9.34% for DCT and 1.5% for SCT over conventional heat treatment. Fractograph analysis using SEM revealed presence of dimples and flat fracture regions in SCT samples than for CHT and DCT samples.

M. Preciado et al. [41] investigated the effects of tempering prior to DCT on the hardness and wear resistance of carburized steel. Tempering prior to DCT was found to improve the wear resistance of carburized steel. However, only those samples that had been tempered at 200^0 C showed significant improvements in hardness values, which was due to the segregation of alloying elements and carbon atoms during the transformation of retained austenite to martensite.

A. Bensely et al. [42] studied the effects of shallow cryogenic treatment and deep cryogenic treatment on the distribution of residual stresses in case carburized En 353 steel using XRD technique. The study concluded that cryogenic treatment should be followed by tempering to achieve the best mechanical properties. Cracks were observes on the surface of untempered samples, which is detrimental to the life of the component. Samples subjected to CHT and SCT+tempering showed better fatigue properties as compared to samples subjected to DCT+tempering.

VI. CONCLUSIONS

The conclusions of this review study are as follows:

- The process of cryogenic treatment involves austenitizing, quenching, cryogenic treatment (Shallow or deep cryogenic treatment) and finally tempering. Multiple tempering can be done to completely relieve the stresses.
- It is important to determine the optimum heat treatment parameters for the cryogenic treatment of each material to obtain maximum desirable properties for that material. This will help to implement it in the further cryogenic treatment of the material and improve its properties.
- Minimum soaking temperature is an important factor in improving the material properties. Hence, deep cryogenic treatment is more effective than shallow cryogenic treatment.
- There is a lot of scope for engineers and researchers to work on the cryogenic treatment of alloys, especially to optimize the treatment parameters for each material and to standardize it.
- Though cryogenic treatment is widely used in steels to improve its product life. But still it is clear from this review that in some materials it does not cause a significant change.
- The wear resistance is enhanced by cryogenic treatment not only by the transformation of retained austenite to martensite but also due to a homogeneous carbide precipitation.

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