Study on the effects of cryogenic treatment on the properties of different types of steel – A review

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***Abstract*— Depending on the thermal treatment used, the atomic structure and microstructure of a material may change due to movement of dislocations, solubility of atoms, variation in grain size, change in the crystal structure, and other mechanisms. Cold treating of steel is widely accepted within the metallurgical profession as a supplementary treatment that can be used to enhance the transformation of austenite to martensite and to improve stress relief. Cryogenics is the science of production and effects of very low temperatures. Cryogenic treatment (CT) is an inexpensive process to conventional heat treatment, which enhance the properties of steels and is considered to be environment friendly. Sub-zero treatments for ferrite steels at temperature of approximately 193K transforms retained austenite left by the heat treatment process to martensite which ensures the property improvement. Cryogenic treatment refines and stabilizes the crystal structure and distributes carbon particles throughout the material resulting in a stronger and hence more durable material. Improvement in properties like hardness, wear resistance, fatigue resistance, dimensional stability, toughness and thermal conductivity are achieved by cryogenic treatment. This paper aims at the comprehensive analysis of Cryogenic treatment and their effects on different types of steels.**

***Keywords*— Cryogenic treatment (CT); austenite; martensite; rotor steel; tool steel; austenitic stainless steel**

I. INTRODUCTION

Cryogenics is defined as the branch of physics and engineering that study very low temperatures, how to produce them, and how materials behave at those temperatures. Rather than the familiar temperature scales of Fahrenheit and Celsius, cryogenicists use the Kelvin and Rankine scales. The word cryogenics literally means "the production of icy cold"; however the term is used today as a synonym for the low-temperature state. It is not well-defined at what point on the temperature scale refrigeration ends and cryogenics begins. The workers at the National Institute of Standards and Technology at Boulder, Colorado have chosen to consider the field of cryogenics as that involving temperature below –180°C. This is a logical dividing line, since the normal boiling points of the so-called permanent gases such as helium, hydrogen, neon, nitrogen, oxygen, and normal air lie below -180°C while the Freon refrigerants, hydrogen sulfide, and other common refrigerants have boiling points above -180°C. Cryogenic temperatures are achieved either by the rapid evaporation of volatile liquids or by the expansion of gases confined initially at pressures of 150 to 200 atmospheres. The expansion may be simple, that is, through a valve to a region of lower pressure, or it may occur in the cylinder of a reciprocating engine, with the gas driving the piston of the engine. The second method is more efficient but is also more difficult to apply.

Cryogenic treatment is a one-time permanent treatment process and it affects the entire cross-section of the material and it is usually done at the end of conventional heat treatment process but before tempering process. Also it is not a substitute process but rather a supplement to conventional heat treatment process. It is believed to improve wear resistance as well the surface hardness and thermal stability of various materials. This treatment is done to make sure there is no retained austenite during quenching process. When steel is at the hardening temperature, there is a solid solution of carbon and iron, known as austenite. The amount of martensite formed at quenching is a function of the lowest temperature encountered. At any given temperature of quenching, there is a certain amount of martensite and the balance is untransformed austenite. This untransformed austenite is very brittle and can cause loss of strength or hardness, dimensional instability, cracking. Quenches are usually done to room temperature. Most medium carbon steels and low alloy steels undergo transformation to 100 % martensite at room temperature. However, high carbon and high alloy steels have retained austenite at room temperature. To eliminate this retained austenite, the temperature has to be lowered.

Liquefied gases, such as liquid nitrogen and liquid helium, are used in many cryogenic applications. Liquid nitrogen is the most commonly used element in cryogenics and is legally purchasable around the world. Liquid helium is also commonly used and allows for the lowest attainable temperatures to be reached. These gases are held in either special containers known as Dewar flasks, which are generally about six feet tall and three feet in diameter, or giant tanks in larger commercial operations. Cryogenic transfer pumps are the pumps used on LNG piers to transfer Liquefied Natural Gas from LNG Carriers to LNG storage tanks.

Cryogenic treatment is a proper operation (treatment) for reducing percent of retained austenite. Cryogenic treatment consists of heating the steel upto austenite temperature, cooling it in quench environment and then immediately putting it in sub- zero centigrade degree and then proceeds to tempering heat treatment. Increased resistance to wear, reduction of internal stresses, consistency of dimensions and deposition of micro carbides in the field can be regarded as the most important privileges of using cryogenic heat treatment. The less the temperature of cryogenic environment, improvement in properties is performed with more rapidity. With deep-cryogenic treatment applied immediately after quenching, residual austenite is reduced, and spots for the nucleation of η-carbides created during tempering are created in martensite. Cryogenic treatments can produce not only transformation of retained austenite to martensite, but also can produce metallurgical changes within the martensite (Harpreet Singh et al, 2013). The objectives of this work are to investigate the effects of cryogenic treatment on the properties of different types of steel.

II. CRYOGENIC TREATMENT

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 1ºC/min, to temperature as low as -84ºC for Shallow Cryogenic Treatment (SCT) and -196ºC for Deep Cryogenic Treatment (DCT), 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 spotty and subjective. 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

Before proceeding for cryogenic treatment the batch of conventionally heat treated specimens has to be cleaned to remove the dirt, impurities and traces of salt layer found on their surface. 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 shown in fig.1. The complete process sequentially consists of the steps austenitizing, quenching, cryoprocessing and tempering.

Fig. 1 Heat treatment sequence for maximizing martensite transformations (P.I.Patil & R.G.Tated, 2012).

A. Shallow Cryogenic Treatment

Shallow cryogenic treatment has been carried at -85ºC with a soaking time of 8 hours. Since rate of cooling is a very sensitive factor and it seriously affects the results of cryogenic treatment, the specimens were very slowly cooled at the rate of -0.5ºC/min, until they reach the final soaking temperature of -85ºC. A soaking period of 8 hours was adopted to allow for transformation reactions to take place after which the cycle was reversed such that temperature builds up at the rate of 0.5ºC/min up to room temperature (D.Candane et al, 2013).

B. Deep Cryogenic Treatment

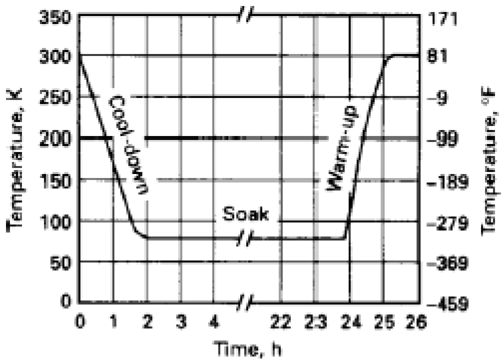

Fig. 2 Plot of temperature versus time for the cryogenic process. Soaking temperature is -196ºC (P.I.Patil & Dr.Bimlesh kumar, 2013)

The diagrammatic representation of cryogenic process is clearly shown in fig. 2. Steels should be hardened using the lowest austenitizing temperature possible in order to achieve the optimal structure for cryotreatment to increase wear resistance. Workers should ramp the cryo processing temperature slowly by 2.5–5 ºC/min. For parts with thick cross sections, it may be desirable to ramp down to an intermediate temperature and allow the temperature to become uniform before continuing with the cool-down. This procedure helps prevent cracking of the parts. Using gaseous nitrogen as the heat transfer medium allows close control of cool-down and warm-up rates.

Research shows that the deep cryogenic treatment (DCT) should start with a slow cooling, continue with a fairly long soak (24 to 72 hours or more hours at temperature), and finally end with a slow warming to room temperature. The sub-zero soak temperature should be close to the liquid nitrogen temperature of –196ºC. The recommended heat-up process warms the material to room temperature at a rate of 1ºC/min in moving air. A tempering cycle similar to that used for cold treatment follows cryogenic treatment since it is likely some retained austenite will have been converted to untempered martensite during the process (P.I.Patil & Dr.Bimlesh kumar, 2013).

III. ADVANTAGES

A wide range of material property improvements has been claimed for steels treated at low temperatures:

A. Wear Resistance

One of the most prevalent claims for cold or cryogenic treatment of metals is an increase in wear resistance (with or without a hardness increase). The practical cost savings from increased wear resistance of low-temperature treating of tooling include:

- Delayed purchase of new tooling
- Decreased resharpen, regrind and rework
- Less scheduled downtime to change tooling
- Lower labor costs
- Decreased loss of production parts when tooling is out of specification
- Reduction of ideal time of machine parts for replacement
- Used for coated as well as uncoated tool steel
- It gives the metal better electrical conductivity

B. Dimensional Stability

The original purpose of cryogenic treatment is to stabilize part dimensions by eliminating the possibility of spontaneous transformation of retained austenite to martensite during fabrication or in service. It transforms almost all soft retained austenite to hard martensite and thereby creates a denser molecular structure and it also decreases residual stresses in the structure.

C. Hardness

Increase in hardness values, tensile strength, toughness and stability occurs because of cryogenic treatment and it also decreases brittleness.

D. Resonant Frequency

Shifts in resonant frequency have been documented and are being applied to musical instruments such as trumpets and trombones (TechCommentary &Ajit Behera and S.C.Mishra, 2012).

IV. EFFECT OF CRYOGENIC TREATMENT ON DIFFERENT TYPES OF STEEL

The effects of cryogenic treatment on the properties of different types of steel are shown below:

A. Tool steel

T.Yugandhar et al, 2002 studied that the only way to reduce the retained austenite percentage is by subjecting the steel to cryogenic treatment (extended quench) immediately after quenching from austenitizing temperature which enhances the precipitation of η-carbides during subsequent tempering. The η-carbides that form is uniformly distributed throughout improved hardness, toughness, wear resistance and resistance to fatigue cracking. The study also identified that the martensite decomposition and precipitation of fine η-carbides as the main mechanisms are responsible for the beneficial effects of deep cryogenics. Mechanical properties of the alloy tool steels subjected to cryogenic treatment are optimized if -196ºC 'extended quench' is followed with single conventional tempering process. The implication is that the multiple tempers commonly incorporated in conventional heat treatments can be eliminated. It has been found that the precipitation of η-carbides in tool steel occurs only during the tempering that follows deep cryogenic treatment, and lengthens the tool life as the amount of η-carbides increases.

B. Austenitic stainless steel

Kyung Jun Lee et al, 2009 conducted a series of uniaxial tensile test under cryogenic temperature for AISI 304 and 316 austenitic stainless steels (ASS). In general, since insulation system of LNG cargo holds or equipment is exposed to cryogenic temperature up to -163 ºC, it is desirable to use austenitic stainless steels because their cryogenic mechanical properties exhibit superior performance, e.g., high strength and ductility and toughness. The main objective of this experiment is the precise investigation of the cryogenic behaviour of austenitic stainless steel at various temperatures. Strength related properties, such as initial yield strength, ultimate tensile strength, hardening ratio exhibit a tendency to increase as temperature decreases. However, the threshold strains corresponding to the beginning of the hardening tend to decrease as temperature decreases. It was found that specific threshold strains for hardening of 304 and 316 ASS for each temperature are in the range of 10–20% elongation.

C. Rotor steel

Fu-Zhen Xuan et al, 2008 carried out three different treatments, including the heat treatment, deep cryogenic treatment and laser surface melting on the 30Cr2Ni4MoV rotor steel. Electrochemical polarization curve and stress corrosion test at the high temperature autoclave are employed to evaluate the corrosion resistance of treated specimens in high temperature water. Results indicate that the conventional heat treatment will increase the value of KIH for specimens with the lower yield strength, and hence reduce the susceptivity of stress corrosion cracking. However, for the deep cryogenic treated specimen, no apparent improvement was observed on the hardness and corrosion resistance due to the limited carbon precipitate and austenite transformation. Superior properties (such as higher hardness and better corrosion resistance) developed by DCT are attributed to the transformation of residual austenite to martensite and the precipitation of fine carbides. However, no significant improvement on SCC resistances in high temperature water was detected for three yield strength of rotor steel treated by DCT. The main reason is that the low carbon content of 30Cr2Ni4MoV steel limited the carbon precipitation and the austenite transformation.

D. Stainless steel

Paolo Baldissera, 2010 performed a study on the effects of deep cryogenic treatment on the static mechanical properties of hardened and solubilized AISI 302 austenitic stainless steel. The results of the tensile and hardness tests were discussed and compared to data and microstructural observations from the previous investigations concerning the same class of steel. In addition, the influence of two important treatment parameters, such as the soaking-time and the minimum temperature, was analysed through a full factorial design of experiments (DOE) and by means of a first approximation model in order to obtain confirmation and suggestions about the possible use of the DCT as a standard practice to improve the mechanical properties of stainless steels. From the results although no significant changes were detected on UTS and on yield stress, an unexpected lowering of the elastic modulus was noticed on some of the treatment groups. Slight but significant improvements were measured on the Rockwell-B hardness of the solubilized material after cryotreating at 8K. On a parallel plane, a pejorative effect was noticed on the Rockwell-C hardness of the hardened AISI 302 with some combinations of the DCT parameters.

Paolo Baldissera et al, 2010 elucidated the effects of DCT on fatigue and corrosion resistance of the AISI 302 austenitic stainless steel for both hardened and solubilized conditions. In both cases, no changes were detected after the DCT on the corrosion resistance and on the capability to reform oxide protective layer. Despite some effects on the fatigue data dispersion, no significant improvements were given by the DCT on the fatigue behaviour of the hardened material. The most interesting results were obtained on the solubilized material, where the DCT proved to be an effective method to improve both the fatigue limit and the fatigue life. The full factorial DOE that was applied to the DCT soaking-time and temperature parameters showed that the treatment does not need for a prolonged exposition and the best results were obtained with a soaking-time of 9h. The fracture surface analysis pointed out the presence of small secondary cracks on the treated material, which could be responsible for the measured improvement by acting as absorber of part of the energy provided by the cyclic loading.

E. Cold work tool steels

A.D.Wale et al, 2013 investigated the effect of cryogenic treatment on mechanical properties of cold work tool steels at various combination of heat treatment cycle (process sequence). The material selected for these processes were AISI D2 and D3. It was seen that for D2 and D3 tool steel conventional heat treated (CHT) specimen has less hardness than cryotreated specimen. But for D2 tool steel there is gradual decrease in hardness observed from Austenizing Quenching Cryogenic (AQC) to Austenizing Quenching Tempering Cryogenic Tempering (AQTCT) specimens. For D3 tool steel there is gradual increase in hardness observed from Austenizing Quenching Cryogenic Tempering (AQCT) to AQTCT and AQC specimen has highest hardness. The multiple tempering decreases hardness in D2 tool steel whereas increases hardness in D3 tool steel. For D2 and D3 tool steel in process sequence AQC massive carbides were not seen and 95% unstable austenitic structure was seen. But both tool steel have maximum hardness value for this process sequence. In D3 tool steel the micro cracks were observed on the untempered samples. The general shape of carbides observed was Globular, Nodular or elliptical. In D2 tool steel retained austenite is not totally converted to martensite where as in D3 tool steel retained austenite is totally converted to martensite.

F. Low carbon steel

Dr.Abbas A. Hussein, 2012 evaluated the performance of low carbon steel (A858) subjected to deep cryogenic treatment (DCT) at liquid nitrogen temperature (-196ºC). The tests specimens were divided in to two groups, the first group was subjected to the conventional heat treatment of normalizing, and the second group was also normalized then subjected to (DCT). It was observed that after DCT, the hardness, tensile properties and the impact energy absorbed were all slightly increased. However the fatigue test showed some positive improvement in fatigue limit by 20(N/mm2), and the volume wear rates at different loads were significantly decreased after DCT. The changes in microstructure due to DCT were clearly noticeable, the grain boundaries were no longer visible, and the pearlite isles globalization was obvious.

G. Mild steel

Harpreet Singh et al, 2013 explored the effect of deep cryogenic treatment at temperature of -193ºC on the corrosion rate and mechanical properties of mild steel. A series of corrosion rate tests have been carried out to cryotreated samples. The specimen were divided into two groups, the first group was subjected to conventional heat treatment process at a temperature of 950ºC for about 1 hour and the other group was subjected to deep cryogenic treatment for about 36 hours at a temperature of -193ºC, followed by tempering at 150ºC for about 1 hour to both the groups. It is seen that after DCT the corrosion rate and mechanical properties of the samples were all improved. The changes in microstructure due to DCT were clearly noticeable, the grain boundaries were no longer visible and the microstructure consists of baninite, martensite and retained austenite.

V. Conclusions

The Conclusions of this review study are as follows:

- Performance of cryogenically treated cutting tools and metal forming tools were improved to 3 times to that of hardened and tempered tools.
- Strength related properties, such as initial yield strength, ultimate tensile strength, hardening ratio exhibit a tendency to increase as temperature decreases. However, the threshold strains corresponding to the beginning of the hardening tend to decrease as temperature decreases.
- Superior properties (such as higher hardness and better corrosion resistance) developed by DCT are attributed to the transformation of residual austenite to martensite and the precipitation of fine carbides. However, no significant improvement on SCC resistances in high temperature water was detected for three yield strength of rotor steel treated by DCT.
- Although no significant changes were detected on UTS and on yield stress, an unexpected lowering of the elastic modulus was noticed on some of the treatment groups, suggesting the need for further investigations focused on this aspect.
- The effect of cryogenic treatment on hardness shows that for D2 and D3 tool steel, CHT specimen has less hardness than cryo treated specimen.
- The hardness, ultimate tensile stress, yield stress, percentage elongation (ϵ) and impact energy for A858 steel were all moderately increased after DCT.
- The deep cryogenic treated mild steel samples are giving better results for wear and mechanical properties like tensile strength and Hardness when compared with conventional and heat treated mild steel samples.

References

[1] A.D.Wale, V.D.Wakchaure, “*Effect of cryogenic treatment on mechanical properties of cold work tool steels*”, International Journal of Modern Engineering, Vol. 03, No. 01, pp.149-154, Jan-Feb 2013.

[2] Ajit Behera and S.C.Mishra, “*Comparative Study of Cryo -Treated Steel*”, International Journal of Scientific & Technology Research, Vol. 01, No. 07, pp.46-49, Aug 2012.

[3] D.Candane, N.Alagumurthi and K.Palaniradja, “*Effect of cryogenic treatment on microstructure and wear characteristics of AISI M35 HSS*”, International Journal of Materials Science and Applications, Vol. 02, No. 02, pp.56-65, Mar 2013.

[4] Dr.Abbas A. Hussein, “*Effect of cryogenic treatment on the properties of low carbon A858 steel*”, Journal of Engineering, Vol. 18, No. 07, July 2012.

[5] Fu-Zhen Xuan, Xiaoqing Huang and Shan-Tung Tu, "*Comparisons of 30Cr2Ni4MoV rotor steel with different treatments on corrosion resistance in high temperature water*", Journal of Materials & Design, Vol. 29, No. 08, pp.1533-1539, Sep 2008.
[6] Harpreet Singh, Er.B.S.Ubhi, Er.Harvinder Lal, "*Improvement in the corrosion rate and mechanical properties of low carbon steel*", International Journal Of Scientific & Technology, Vol. 02, No. 06, June 2013.
[7] Kyung Jun Lee , Min Sung Chun , Myung Hyun Kim and Jae Myung Lee, "*A new constitutive model of austenitic stainless steel for cryogenic applications*", Journal of Computational Materials Science, Vol. 46, No. 04, pp.1152-1162, Oct 2009.
[8] P.I.Patil and Dr.Bimlesh kumar, "*Deep Cryogenic Treatment of Alloy Steels : A Review*", International Journal of Electronics, Communication & Soft Computing Science and Engineering, Vol. 02, No. 08, pp.29-34, Mar 2013.
[9] P.I.Patil and R.G.Tated, "*Comparison of effects of cryogenic treatment on different types of steels: a review*", Proceedings from International Conference in Computational Intelligence, Sandip Institute of Technology & Research Centre, Nasik, Feb 2012.
[10] Paolo Baldissera, "*Deep cryogenic treatment of AISI 302 stainless steel: Part I – Hardness and tensile properties*", Journal of Engineering, Vol. 18, No. 07, July 2012.
[11] Paolo Baldissera, "*Deep cryogenic treatment of AISI 302 stainless steel: Part II – Fatigue and corrosion*", Materials and Design, Vol. 31, pp.4731-4737, 2010.
[12] T.Yugandhar, P.K.Krishnan, C.V.Bhaskar Rao and R.Kalidas, "*Cryogenic treatment and its effect on tool steel*", Proceedings from 6th International Tooling Conference, Karlstad University, 2002.
[13] www.energy.ca.gov/process/pubs/cryog-processing-tc113571.pdf

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