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A Review on the Effect of Cryogenic Treatment on Metals

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Abstract - *Cryogenic treatment is an add-on process to conventional heat treatment process in material processing technology. It is a one-time permanent treatment which affects the whole section of the component. It is not like the coatings of superior materials over other metal surfaces that only affects the surface of components. Cryogenic treatments are proved to be a good way to reduce the retained austenite content and improve the performance of materials by improving its martensite structure. Objectives of cryogenic treatments are to increase material's strength, hardness, wear resistance, ductility, & toughness, to obtain fine grain size, to remove internal stresses, to improve machinability, cutting properties of tools, to improve surface properties, electrical properties & magnetic properties. This paper aims to review about various cryogenic treatments involved in treating various materials so far and their effect on their properties. The summary of this review paper tells about the properties improved due to the cryogenic treatment and the reason behind them.*

Key Words: Cryogenic Treatment, Heat Treatment, Austenite, Martensite, Wear resistance, Ductility

1. INTRODUCTION

All Metals may not possess desired properties in their final product. Alloying and Heat treatments are two methods which are widely used to control material properties. Heat treatment is the controlled heating and cooling operations performed on the material. It is an operation or combination of operations which include heating at a specific rate, soaking at a temperature for some specific time and cooling at some specified rate. The aim of it is to obtain the desired microstructure to achieve some desired properties like physical, mechanical, magnetic & electrical properties. When the material is subjected to heat treatment the atomic structure 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. [16].

Objectives of Heat Treatments are as below:

- To increase strength and hardness
- To increase wear resistance
- To obtain fine grain size
- To increase ductility and toughness
- To remove internal stresses induced by non-uniform cooling from high temperature during casting and welding
- To improve machinability
- To improve cutting properties of tool
- To improve surface properties

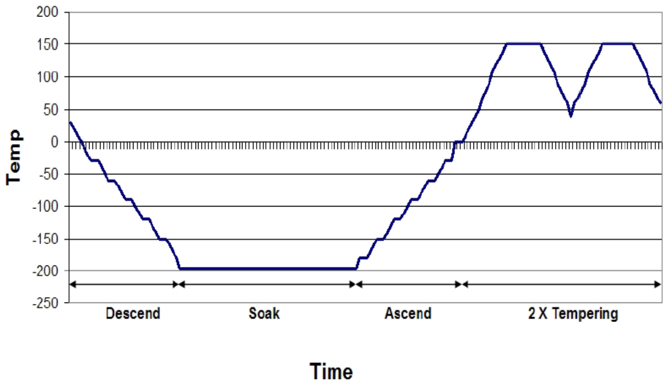

Fig. 1-DCT cycle [7]

1.1 Cryogenic Treatment

Word Cryogenic is made of two Greek words:
Kryo: Which means Very cold (frost)
Genics: Which means to produce [2]

This is a technology where everything we process gets frozen at ultra-low temperatures of -193°C. Then everything is held down at temperatures around of -193 °C for 12-48 hours followed by gradual ascend and tempering.

This process consists of controlled cooling of conventionally hardened materials to a specified temperature followed by controlled heating of the materials back to the ambient temperature for subsequent tempering process.

The cryogenic treatment can be classified into three different temperature regimes [10]:

(1) Cold Treatment (CT, ≥193 K or -80 °C)
(2) Shallow Cryogenic Treatment (SCT, 193–113 K or -80 °C to -160 °C)

(3) Deep Cryogenic Treatment (DCT, 113–77 K or -160 °C to -196 °C)

Two different types of cryogenic solutions used for the treatment of materials:

(1) Liquid Nitrogen (-196 °C)
(2) Liquid Helium(-269 °C)

Liquid nitrogen is a cryogenic liquid. At atmospheric pressure, it boils at −196 °C. When insulated in special containers called Dewar flasks, it can also be transported. Liquid nitrogen is generally used as it is cheaper than liquid helium and can be available easily.

There is no clear understanding of the mechanisms by which cryogenic treatment improves the performance of metals. Most researchers believe that the martensite temperature is below 0 °C due to the higher alloying element in alloyed metals. It means that at the end of the heat treatment, a low percentage of austenite will be retained at room temperature. The retained austenite, as a soft phase in metals, can reduce the product life. Deep cryogenic treatment is used to transform this retained austenite into martensite. As a result, the retained austenite level is reduced and the good working life is obtained. Cryogenic treatment also facilitates the formation of finer secondary carbides in the martensite, thus improving the wear resistance [8].

Many studies have focused on improving the properties of metals by deep cryogenic treatment. Positive effects have been noticed in tool steels, carburized steels, cast irons and other materials, as discussed in a detailed literature review. However, the mechanisms behind this treatment remain unclear, making it difficult to predict the effects of this treatment on a particular alloy. Thus, specific experimental testing is required for each material to be treated [8].

The accepted temperature for cryogenic or sub-zero treatment has been 193K where dry ice can be used for cooling. But, the results of few recent studies suggest that the temperature of cryogenic treatment should be less than 193K in order to obtain the maximum improvement in mechanical properties of metals. The lowest temperature of cryogenic treatments may be 77 K, which is the boiling temperature of liquid nitrogen at normal atmospheric pressure. This is why Deep Cryogenic Treatment is said to be superior to Cold and Shallow treatments as in Deep Cryogenic Treatment temperature 113 K to 77 K is used (-160 °C to -196 °C)

1.2 Process of Cryogenic Heat Treatment

Process of Cryogenic Heat Treatment is as shown in the flow chart:

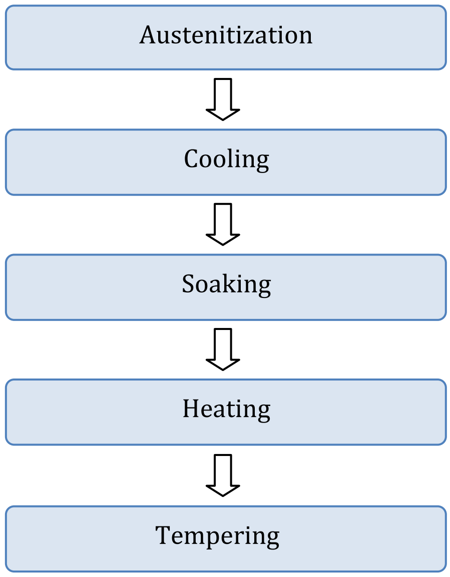

Fig. 2- Process of Cryogenic Heat Treatment

Cryogenic Process Consist four stages, that involves:

1) Austenitization: Heating from room temperature to its austenitizing temperature (around 1100 °C), at an extremely slow rate ranging from 0.5 to 1.5°C /min
2) Cooling: Direct cooling from austenitizing temperature to -196 °C at the rate of 1.5 to 2 °C. It is also called as quenching.
3) Soaking: For a period of time ranging from 24 to 36 hours depends upon which material is to be treated
4) Heating: From -196 °C to room temperature at the rate of 0.5 to 1°C /min
5) Tempering: Reheating the metal at predetermined temperatures which are lower than the transformational temperature (around 150 °C) to obtain different combinations of mechanical properties in the material.

2. LITERATURE SURVEY

K Prudhvi & Mrs. Venkata Vara Lakshmi [1]: They studied about the normal high-speed steel tool for machining. But it is not possible to machine the hardened materials. So they apply cryogenic treatment for a certain time. Hardness is tested for the tool before and after treatment. Hardness for the untreated tool was 64.06 HRC and for a treated tool was 65.83 HRC. Therefore hardness is increased by 1.73 HRC than the untreated tool. They have concluded from the experiments that there are 34.17 seconds decreases in machining time & there is no tool wear when machining EN8 and when machining EN 19 there are 22.04 seconds decrease in machining time & 0.03g increase in tool wear resistance. Deep cryogenic treatment has a significant effect on increment in the wear resistance and correspondingly reduces machining time of steels such as EN8 and EN19.

Haizhi Li, Weiping Tong, Junjun Cui, Hui Zhang, Liqing Chen, Liang Zuo [8]: The cryogenic treatment was carried out by the gradual cooling of the samples to -196 °C and holding the samples at this temperature for different durations or for a different number of cycles, followed by uniform heating to room temperature and then tempering at 560 °C for 1 hr. The effects of deep cryogenic treatment on the wear resistance, hardness, toughness & microstructure of sample of HVAS were studied. Results are as follows:

1) After deep cryogenic treatment, there were a large amount of dispersed carbide precipitation in the HVAS, secondary carbide ever increasing due to the increased processing time and numbers of cycles in this study.
2) DCT decreased the hardness and increased the impact toughness of HVAS compared the conventional treatment (CONT), which is due to the carbon content in the martensite decreasing after DCT.
3) Abrasive wear resistance of DCT samples slightly increases with increasing processing time and numbers of cycles. Micro cracks of the DCT samples were not detected at carbide/matrix interfaces compared to the conventional treatment (CONT).

B. Podgornik, I. Paulin, B. Zajec, S. Jacobson, V. Leskovsek [3]: Material used in this study was a high fatigue strength cold work steel with lower C and high W and Co content. In order to examine the effectiveness of DTC on fracture toughness and load carrying capacity, two more tool steels were used namely high C and V content cold work tool steel and one high-speed steel. After specimens were machined, they were vacuum heat treated using nitrogen gas at a pressure of 5 bar. In order to evaluate the effect of vacuum heat treatment three sets of vacuum heat treatment conditions, resulting in different hardness and fracture toughness combinations were used and combined with deep cryogenic treatment. Following conclusions were made by this study:

1) In case of low carbon cold-work tool steel (A1), DCT results in greatly improved fracture toughness while maintaining high hardness. On the other hand, for high C cold-work tool steel DCT has a negative effect, while for high-speed steel, DCT has practically no effect.
2) DCT produces finer needles like martensite and martensitic transformation accompanied by plastic deformation of primary martensite may be the reason behind the improved property of A1 steel. Alteration in K_{Ic}/HRC ratio affects wear resistance of cold-work tool steel. Hardness is the main parameter affecting abrasive wear resistance.
3) In the case of load-carrying capacity, hardness is the most important parameter. In order to obtain good load-carrying capacity, hardness above 64 HRC is required, with a fracture toughness of over 10 $MPam^{1/2}$ providing further improvement.

M. Perez, C. Rodriguez, F. J. Belzunce [4]: Hardness, strength, and toughness of H13 steel submitted to different heat treatments, including cryogenic treatments, were tested in their research work and the results were explained based on changes in microstructure. Specimens were subjected to different quenched and tempered treatments. Four different thermal treatments were applied called TT1, TT2, TT3 & TT4. They concluded that tensile strength and hardness have hardly changed for the four applied thermal treatments. On the other hand, there is a positive effect of the cryogenic treatments on the fracture toughness of the steels. TT2 (gas) and TT4 (oil) give respectively 22.5% and 24% increment related to their corresponding treatments without cryogenic phase, TT1 and TT3. Quenching media also affected the toughness of the steel. Due to the effect of the cooling rate: oil quenching has higher toughness than gas quenching. Quenched and tempered H13 steel has a martensitic microstructure with well dispersed and finely distributed carbides. SEM analysis concluded that cryogenics generate high internal stress state that activates the carbide nucleation in the first phases of tempering. This results in a much finer and evenly distributed precipitation which also gives rise to a martensite with less carbon.

Marcos Perez, Francisco Javier Belzunce [7]: Cryogenic treatment was carried out on H13 tool steel used for hot forging dies, subsequently determining its mechanical properties tensile, hardness and fracture toughness tests. This paper explains the performance of four different heat treatments applied to H13. Two quenching media (gas and oil) and the effects of a cryogenic stage were studied. Oil quenching by cryogenic treatment was carried out as the best one among all four treatments. The mechanical properties of the H13 steel were measured by tensile, hardness and fracture tests. They concluded that cryogenic treatment notably improves the fracture toughness of H13 steel. Cryogenic with gas and oil quenching generates 22.5% and 24% respectively increase in toughness when compared to without cryogenic. So quenching medium also affects its toughness. Deep cryogenic treatments reduce the retained austenite content in H13 steel.

D Senthil Kumar, I Rajendran [5]: Effect of cryogenic treatment on the wear resistance property of En 19 steel was studied. Also, an analysis on the effect of DCT (-196 °C , 24 h), SCT (-80 °C, 5 h) and CHT was done by dry sliding wear test. Dry sliding wear test for low loading and high loading was observed. The microstructures of CHT, SCT and DCT samples were studied by SEM. They have concluded that both DCT and SCT promote the transformation of retained austenite to martensite, thereby causing a significant increase in wear resistance. Wear resistance was increased by 118.38% for SCT samples and 214.94% for DCT when compared to CHT samples. In addition, the increase in wear resistance of DCT samples is 44.39% with respect to SCT samples. The lowest coefficient of friction is obtained in DCT samples treated at -196 °C for 24 hr.

D. Das, K. K. Ray, A. K. Dutta [10]: Their study examined the effect of the temperature of the treatment on the wear behavior of AISI D2 steel. Samples were subjected to

conventional treatment (CONT), Cold Treatment (CT), Shallow Cryogenic Treatment (SCT) and Deep Cryogenic Processing (DCT) in separate batches. CONT consists of hardening and tempering; while in CT, SCT and DCT, an additional step of controlled sub-zero treatment with the lowest quenching temperature under 198, 148 and 77 K respectively, was incorporated into the curing and quenching treatments. Microstructural examinations were performed using optics and SEM. The hardness was measured by a Vickers hardness tester.
They concluded that:

1) All types of sub-zero treatments appreciably improve the wear resistance of the die steels compared to the CONT ones. Improvement in wear resistance by SCT and DCT is significantly higher than that achieved by CT, and the maximum improvement is obtained by DCT.
2) The obtained hardness of AISI D2 steel for CONT and DCT are 759 and 791 VHN, respectively and typical values of their specific wear rate are 1.03×10^{-6} and 8.26×10^{-9}mm3 $N^{-1}mm^{-1}$.
3) The obtained results lead to the conclusion that lower the temperature of sub-zero treatment higher is the improvement in wear resistance.

A Joseph Vimal, A Bensely, D. Mohanlal [12]: They studied the behavior of Deep Cryogenic Treatment (DCT) on EN31 steel sample work piece used for bearing to improve its wear resistance. The austenitizing temperature in this study is 1039 K (819 °C) after that quenching was done at 40 °C and tempering at 140 °C. They observed wear resistance by pin-on-disc test, microstructure by SEM and hardness test by VHN. They came to conclude that DCT gives rise to hardness with or without tempering. Also, the stated that wear resistance increases as hardness increases. It was observed that by cryogenic treatment, wear can be decreased by a maximum of 75% depending on the service conditions.

A. Akhbarizadeh , A. Shafyei, M. A. Golozar [13]: They studied the effects of cryogenic treatment on the wear behavior of D6 tool steel. For this, two temperatures were used: -63 °C as SCT and -185 °C as DCT. The effects of cryogenic temperature (Shallow and Deep), cryogenic time and stabilization temperature on the wear behavior of D6 tool steel were studied. Hardness and wear test were carried out. Results showed that the cryogenic treatment increases hardness. The samples which were cryogenically treated for a longer time or deep cryogenically treated showed further increase in hardness. The Higher hardness of the shallow cryogenically treated samples was due to the decrease of the retained austenite. It was observed that the cryogenically treated samples have higher wear resistance compared with the conventionally heat-treated samples; this improvement was 5–11% in SCT and 39–68% in the DCT samples.

V. Firouzdor, E. Nejati, F. Khomamizadeh [14]: The effect of deep cryogenic treatment on wear resistance and tool life of M2 HSS drill was studied in their research work. The austenite temperature was 1100 °C and gas quenching was done in nitrogen gas and consequent tempering was done at 600 °C for 2h. Deep cryogenic treatment consisted of slowly cooling drills to approximately −196 °C and holding at this low-temperature for 24h and gradually bringing the specimens back to room temperature. They concluded that Cryogenic treatment profoundly improved the wear resistance of M2 HSS drills in the configuration of high-speed dry drilling of steels. Low-temperature tempering (200 °C) after cryogenic treatment was also found to be highly beneficial. It has been deduced that fine carbide precipitation during cryogenic treatment is the main reason for wear resistance improvement. Transformation of retained austenite to martensite could also contribute to wear resistance improvement, i.e. enhanced hardness value. Cryogenic treatment could not only facilitate the carbide formation and increase the carbide population in martensite matrix, but also make the carbide distribution more homogeneous.

J. Y. Huang, Y.T. Zhu, X. Z. Liao, I. J. Beyerlein [15]: They investigated the reason behind the improvement in hardness and wear resistance after cryogenic treatments. M2 tool steel rod with a diameter of 6.35 mm was used in the experiment. Preheating at 815 °C in a vacuum furnace, then continuously heating to austenite temperature of 1100 °C in a nitrogen atmosphere, holding for 1 h, followed by quenching to an ambient temperature in a cool nitrogen gas. The cryogenic treatment was performed by soaking the samples in liquid nitrogen for 1 week. They came to the conclusion that cryogenic treatment cannot only facilitate the carbide formation and increase the carbide population and volume fraction in the martensite matrix, but can also make the carbide distribution more homogeneous. This shows increases in carbide density and volume fraction, which may be responsible for the improvement in wear resistance.

D. Mohanlal, S. Renganarayanan, A. Kalanidhi [6]: Materials considered for this research were M2, T1 and D3 steel used for dies and punches to check the influence of cryogenic treatment with respect to the carbon percentage. Also, the effect of TiN coating on cryo treated tools and cryo treatment on TiN coated tools were illustrated. TiN coating imparts 48.4%, 42% and 41% improvement while cryogenic treatment imparts 110.2%, 86.6% and 48% improvement in Ti, M2 and D2 steels respectively. Cryogenic treatment to TiN coating is superior also and it provides 45% extended tool life then cryo treatment alone. They also concluded that soaking time is more important than lowering the temperature.

3. SUMMARY

Following summaries have been made from the literature surveys:

- There are three types of Cryogenic Treatment: (1) Cold Treatment, (2) Shallow Treatment & (3) Deep Cryogenic Treatment.

- Cryogenic Treatment enhances hardness, strength, wear resistance, toughness, dimensional stability and also improve fine grain size, machinability, cutting properties of tool steels & surface properties like finishing etc.
- Reason behind the improvement in these properties is the transformation of austenite into martensite.
- Deep Cryogenic Treatment (DCT) has been proved to be the superior one than Shallow and Cold treatment as the segregation of carbon atoms and alloying elements occurred during this temperature and nearly complete transformation of retained austenite into strain induced martensite microstructure (approx. 99%).
- Most affecting parameters for DCT are the rate of cooling soaking temperature, austenite temperature, quenching temperature, soaking period, tempering temperatures and tempering period. The main parameters which contribute to improved wear resistance are soaking temperature, soaking period and rate of cooling.

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