International Journal of Electronics, Communication & Soft Computing Science and Engineering
ISSN: 2277-9477, Volume 2, Issue 8

Deep Cryogenic Treatment of Alloy Steels : A Review

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***Abstract* — The word, "Cryogenics" is taken from two Greek words – "kryos" which means cold or freezing, and "genes" meaning born or generated. Technologically, it means the study and use of materials (or other requirements) at very low temperatures. In general cryogenic treatment consists of slow cooling already heat treated part to approximately -196^{O}C, holding at low temperature up to prescribed period, reheating without thermal shock to room temperature and reheat to a moderately elevated temperature (150 to 315^{O}C) for about 1 or 2 hours, to "temper" the part for the stated purpose of reducing its brittleness. It has been tried with several materials and the results in some cases are fascinating and for certain materials cryogenic treatment did had small changes or no change at all. At present also the initial mistrust about cryogenic treatment has not been cleared up, because it imparts no apparent visible changes to the metal. Alloy steels especially high speed steels, stainless steel, cast iron did show beneficial results where as carbide inserts showed results to certain extent. Even though in case of certain materials due to cryogenic treatment some of the properties are improved, still there is need of systematic investigation to make of the process in commercial sense. In this paper an attempt is made to review the investigations made and published for the effects of the cryogenic treatment on properties of alloy steels.**

I. INTRODUCTION

Cryogenic treatment is an inexpensive process to conventional heat treatment, which improves tribological properties of steels. The deep cryogenic treatment is one time, permanent treatment offering the entire part not just the surface. Tool sharpening will not destroy the treatment. The process has a number of obvious benefits, including increase in tensile strength, toughness, increase in WR, hardness and dimensional stability through the release of internal stresses during tempering.

The exceptional increase in wear resistivity, generally exceeding 200% is the greatest benefit [1].

From over the last few decades, interest has been shown in the effect of low temperatures during the heat treatment cycle on the performance of alloy steels, particularly cold, hot work tool steels, many articles on DCT have been published. To the casual reader, there may appear to be some confusion between the conflicting claims of some of the literature. The purpose of this review is to present bibliography and to summarize the present state of research in this area and point out the underlying mechanisms involved.

In order to avoid confusion, a fundamental distinction among different CT in given by the parameters of cooling – warming cycle. In [2] two families depending on the minimum temperatures reached during the cycle are categorized:

- Shallow Cryogenic Treatment (SCT) or Sub-zero treatment. The samples are placed in freezer at 193 K and then they are exposed to room temperature.
- Deep Cryogenic Treatment (DCT): The samples are slowly cooled to 77 K holding for many hours and gradually warmed to room temperature.

II. PROCESSING CYCLES FOR DEEP CRYOGENIC TREATMENT

Fig. 1 shows cryogenic process. 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 °C/min (4.5 – 9 °F/min) [46]. 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 [46].

Research shows that the deep cryogenic cycle 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 [4,8,14,42]. The sub-zero soak temperature should be close to the liquid nitrogen temperature of –196 °C (–300 °F). The recommended heat-up process warms the material to room temperature at a rate of 1 °C/min in moving air [8]. A tempering cycle similar to that used for cold treatment follows cryo treatment since it is likely some retained austenite will have converted to untempered martensite during the process [46].

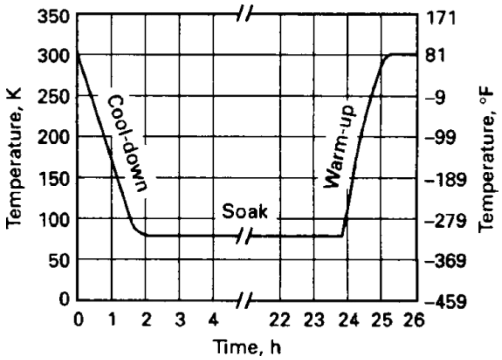

Fig. 1 Plot of temperature versus time for the cryoprocessing process. Soaking temperature is −196°C [7]

III. UNDERSTANDING THE MECHANISMS INVOLVED IN DEEP CRYOGENIC TREATMENT

In order to resolve the apparent anomalies, Collin [3] suggested that a research project was undertaken at University College Dublin on cold-work (D_2) and high speed (ASP 23) tool steels the results indicate that, there are the different phenomena or mechanism involved. These two phenomena have distinctly different effects.

Mechanism1: Transformation of Austenite to Martensite

Akhbarizadeh et.al. [31] studied on wear behavior of D6 tool steels they observed that, in tool steels, a low percentage of austenite is retained after the conventional heat-treatment named ''retained austenite". The retained austenite as a soft phase in steels could reduce the product life and, in working conditions, it can be transformed into martensite [13]. This new martensite could cause several problems for working tools. This new martensite is very brittle and differs from the tempered one, which is used in tools. Furthermore, this martensite causes micro cracks and reduces the product life. Moreover, the retained austenite-to-martensite transformation provides dimensional instability [13].

The transformation from austenite to martensite begins at a well-defined temperature called the martensite start temperature or M_S. For most practical steels, the transformation is isothermal and progresses smoothly as the temperature falls to the martensite finish temperature, or M_F. Some austenite, designated retained austenite, is always present after hardening. Higher martensite contents and carbon percentages increases the hardness of steel. The amount of carbon also affects the temperatures where the martensite transformation begins (M_S) and is completed (M_F). The M_f and M_s temperatures can be lower than room temperature. The steel might only partially transform to martensite with the remaining structure being retained austenite. M_s and M_f temperatures are also depressed with increase in grain size, thus higher autenitizing temperatures can lead to higher level of retained austenite. The retained austenite is always present after heat treatment process and can be alleviated by means of DCT by transferring in to martensite as austenite is soft and unstable at lower temperatures. This treatment alters metallurgical properties and improves the strength of the steel [29,32].

The effect of this deep cryogenic treatment is:

- Dimensional stability
- An increases in hardness
- A reduction in toughness
- Slightly improvement in wear resistance

Mechanism2: Low – Temperature Conditioning of Martensite

Precipitation of Fine η- Carbides

Philip Nash et.al. [26] in their study on M2 tool steel claimed that the advantage of using deep cryogenics is due to an enhancement of the precipitation of fine eta-carbides during the subsequent temper. The strain energy in the martensite lattice increases at a lower temperature. As a consequence Carbon atoms migrate and form a clusters. During the subsequent heating back to the room temperature or even a tempering, these clusters act as nuclei for the formation of the ultra fine eta-carbides. The eta-carbides that form are uniformly distributed throughout a highly decomposed microstructure.

Das et.al. [33] Observed in their study on AISI D2 steel that the sub-zero treatments do not alter the nature of primary and/or secondary carbides. The improvement in population density of small secondary carbides is 193% by deep cryogenic treatment against 80% by cold treatment and 109% by shallow cryogenic treatment, when these are compared with respect to the conventional heat treatment. Results related to the micro-structural analyses, thus, assist to infer that sub-zero treatments not only alter the amount of retained austenite content but also considerably modify the precipitation behavior of secondary carbides which is observed to be increasingly pronounced in the order of cold treatment, shallow cryogenic treatment and deep cryogenic treatment. Das et. al. [34] in their another study on sub-zero treatments of AISID2 steel they inferred that, the degree of precipitation of secondary carbides during tempering of martensite not only depends on the amount of martensite in as-quenched structure but is also controlled by the state of tempering of martensite. Further they also concluded that, sub-zero treatments accelerate the decomposition of martensite and modify the precipitation behavior of secondary carbides on AISI D2 steel. Das et.al. [18] further they concluded that, correlation of the examined microstructures with their wear behaviour unambiguously establishes that substantial modification in the precipitation behaviour of SCs and reduction in $_{\gamma R}$

content are the governing mechanisms for the improved of WR of tool/die steels by DCT.

Paulin [1] also verified the presence of fine precipitated carbide particles and their importance tothe material properties. The precipitated carbides reduce internal tension of the martensite and minimize microcracks susceptibility, while the uniform distribution of fine carbides of high hardness enhances the wear resistance. Collins and Dormer [4] later reiterated the same

view using extended experimental investigations. Low-temperature conditioning of martensitic structure implies crystallographic and microstructural changes which, on reheating, result in the precipitation of a finer distribution of carbides in the tempered microstructure with consequent increase in toughness as well as in wear resistance. Meng *et al.* [35,36] it was envisaged by these authors that cryogenic treatment improves preferential precipitation of fine η-carbides during the primary stage of tempering in a high-carbon alloy steel. These carbides might enhance the strength and toughness of the martensite matrix and thus improve the wear resistance.

The only proposed microstructural mechanism for fine carbides precipitation in tool steels is the martensite contraction, due to thermal stresses during cooling, which leads carbon atoms to segregate near lattice defects [37].

Formation of Eta (η)-Carbides

Senthilkumar et.al [38] observed that the mechanism of this process responsible for stabilization of the structure has considerable impact. This stress is caused by the spatial variation in composition and microstructure which leads to different thermal contraction and also by the transformation of retained austenite to martensite. The martensite wants to be cooled below a certain temperature to develop internal stress sufficient to generate crystal defects. The required long holding time suggests a localized carbon distribution occurring by clustering of carbon atoms to lattice defects (dislocations). The martensite becomes more super-saturated with decreasing temperature. This increases the lattice distortion and thermodynamic instability of the martensite, both of which compel carbon and alloying atoms to segregate nearby defects. These clusters act as or grownup into nuclei for the formation of carbides when tempered subsequently. Above observations are agreement with Das et.al. [39] on their experimental study on the wear resistance of tools steels.

Zhirafar *et.al.* [40] studied the effect of cryogenic treatment on mechanical properties of 4340 steel they observed that, in general, hardness and fatigue strength of the cryogenically treated specimens were a little higher whereas the toughness of the cryogenically treated specimens was lower when compared to that of the conventionally treated steel. Neutron diffraction showed that the transformation of retained austenite to martensite occurred which, along with possible carbide formation during tempering, is a key factor in improving hardness and fatigue resistance of the cryogenically treated specimens.

By reviewing the literature survey, it is observed by Bensely *et.al*. [32] in their study on case carburised steel-815M17 that, micro-structural analysis of the specimen after cryogenic treatment, showed the presence of ultra fine Eta (η) carbides precipitates of size in the range of 10 nm these were characterized as η-carbide. Alexandru *et.al* [41] also observed that cryogenic cooling induced the occurrence of very fine carbides with dimension less than 1 m, which occupy microvoids and contribute to an increase of the density. Meng *et. al.* [35] proposed that greater wear resistance can be obtained with longer soaking periods (~24h) because of the formation of η−carbides which improves the wear resistance to the maximum possible extent. Transformation of austenite to martensite at cryogenic temperature followed by prolonged holding induces micro-internal stresses which results in the formation of crystal defects such as dislocations and twins [1,25,27,20]. While, lattice distortion and thermodynamic instability of martensite at 77 K drive carbon and alloying atoms to segregate at the nearby crystal defects. These segregated regions have been hypothesized as the newer sites for nucleation of SSCs [21].

Homogeneuous Microstructure

Pete Paulin *et.al.* [1]studied on Frozen Gears they observed that ,deep cryogenically treated metals also develop a more uniform, refined microstructure with greater density. Microfine carbide "fillers" are formed, which take up the remaining space in the micro – voids, resulting in a much denser, coherent structure of the tool steel. The end result is increased wear resistance. Huang *et al.* [27] confirmed that cryogenic treatment not only facilitate the carbide formation but can also make the carbide distribution more homogeneous. Alexandru Ailincai and Baciu *et. al.* [41] mentioned that the structure of cryogenically cooled metallic materials has a more uniform and dense microstructure than non-cryogenically treated samples. Das *et. al.* [34] studied on sub-zero treatment of AISI D2 steel they concluded that, in general, sub-zero treatments refine the size of the secondary carbides, increase their amount and population density, and lead to their more uniform distribution in the microstructures. These favorable modifications of secondary carbides are found to be significantly higher in deep cryogenically treated specimens than that in cold treated or shallow cryogenically treated specimens.

Recent investigations [25,27,43,44], however, have established that sub-zero treatments not only substantially lower the retained austenite content but also significantly modify the precipitation behavior of carbide, resulting into considerable change in the characteristics of secondary carbide particles in the microstructure of tool/die steels. The extents of these alterations are reported to be dependent on the types of sub-zero treatments [18]. Microstructural modifications imparted by sub-zero treatments are expected to have considerable influence on the mechanical properties of tool/die steels.

However, the mechanism of microstructure changes in alloys under various treatments, are not yet fully understood [16]. Change in microstructure affects on tool life under certain treatments [45].

IV. Property Improvements Claimed By Cryogenic Treatment

In the literature survey published over the years, a wide range of property improvements have been said to be achieved. These include :

Hardness : In many cases hardness increases of 1-3 HRC have been claimed, although some authors report very little increase in hardness [3].

Toughness: Claims for increases in toughness are widespread [3].

Wear resistance: One of the most prevalent claims is an increase in wear resistance[3]. For example, the improvement in WR of AISID2 steel by deep cryogenic treatment over conventional heat treatment varies from 108% as reported by Collins and Dormer [4] to 817% as reported by Barron[22]. Wilson[24] concluded that, cryogenically treating slitter knives in paper mills increases the lifetime by more than 500%.

Dimensional stability : This was the original purpose of cryogenic treatment, to stabilize dimensions by eliminating the possibility of spontaneous transformation of retained austenite subsequent to the final heat treatment [3]. For applications where extremely precise tolerances required, the austenite decomposition can cause dimensional changes resulting from differences in crystallographic size of phases. Dimensional stability can be improved with repetitive cold treatment cycles [5].

Intergranular corrosion resistance: One author claimed an improvement due to reduced grain-boundary diffusion[3].

V. Ambiguities in Cryogenic Treatment

A. Joseph Vimal *et.al.* [9] have mentioned that, research in cryo treatment has improved in developed countries, but it is still in the dormant level in many other countries. Researchers are still skeptical about the benefits of the cryogenic treatment. All over the world, there are many controversies prevailing on the reported mechanisms and because of such issues in cryo treatment only limited researches has been done on selected alloys.

Dhokey *et.al*.[10] studied and observed that, literature of cryo treatment does not adequately clarify the selection of tempering, cryogenic temperature and soaking time. There is a need to standardize the process for cryo treatment in particular tool steels and understand the underlying mechanism responsible for improvement of mechanical properties like wear, hardness, toughness etc.

In general cryogenic treatment is still in the dormant level as far as to understand the metallurgical mechanisms are concerned.

Cryo treatment technology has not been widely adopted by the industries due to lack of understanding of the fundamental metallurgical mechanisms and due to the wide variation is reported in research findings [5]. Many researchers demonstrated the effect of cryo treatment and the underlying phenomenon, but to understand why this phenomenon occurs, requires sophisticated and analytical equipment and extensive metallurgical knowledge [5].

It has been claimed by several researchers that cryo treatment enhances wear resistance of certain steel [4,2,13,14]. But the reported magnitudes of the enhancement in wear resistance and the governing mechanisms for such enhancement do not provide any unified picture. There are number of treatment processes used for different metals which cause them to behave differently under different conditions [15]. However, the mechanisms of microstructure changes in alloys under various treatments are not yet fully understood [16].

Wayne Reitz *et.al.* [17] have indicated that, the details for successfully conducting each the step in DCT have yet to be determined. Three main factors of the cooling process that are currently being debated are cooling rate, soaking time, and optimal quenching temperature. However, D. Das *et.al*.[18] have concluded that, the underlying mechanisms behind the enhancement of wear resistance of tool/die steels by DCT are still debated and yet to get crystallized.

One of the major uncertainties associated with the earlier investigations related to cryo treatment of tool steels is the duration of cryo treatment at the selected temperatures [19,20,17]. The existing literature does not provide any guideline related to the selection of time duration for cryo treatment [19,21] In [18] the state-of-art of cryo treatment of tool/die steels does not present any coherent information either to accept or to reject any of the propositions due to the absence of systematic investigation on the correlation of microstructure with wear behavior of these steels by DCT.

S.S. Gill *et. al.* [16] indicated that, it is very much comprehensible that the rate of cooling still requires to be debated upon to achieve the desired results. Further they observed that available results in the literature pertaining to structure property relations of tool steels and carbides subjected to cryo processing are not coherent and the underlying postulated mechanisms for achieving improved mechanical properties like wear resistance are not well crystallized. Most researchers believe that deep cryogenic process promotes complete transformation of retained austenite into martensite, and this can be attributed to the enhanced wear resistance of the tool steels [14,22,24,25,26]; however, some researchers suggested there was always the retained austenite after cryo processing [11,12], whereas another school claims that the cause of increased wear resistance is the formation of fine carbides in martensite matrix and their uniform distribution [4,14,25,27,]. The extent of benefits of these emerging processing routes can only be suitably exploited if the underlying mechanisms of these processes are carefully unfolded in an organized manner.

The holding time in cryogenic processing has been varied widely by earlier investigators. For example, holding time employed in the cryo treatment for AISI M2 steel is 1 hour by Leskovsek et al. [28], 20 hours by de Silva *et al.* [29], 35 hours by Molinari et al. [14] and 168 hours by Huang *et al*. [27]. Such wide variation in the selected holding time even for the same material is due to the lack of systematic investigation related to the influence of holding time on the wear resistance of tool/die steels by cryo treatment.

It can be summarized from the above review that lowering down the soaking temperature to the minimum is very important to improve the wear resistance in steel. Lowest temperature CT would enhance wear property to the most. It is believed to be due to complete transformation of the retained austenite to martensite during CT [4,2,9] and this transformation is responsible for improvement in various properties of steel; however, this transformation is claimed to be complete at SCT at around −84^{0}C (189K) [14,30]. So the exact mechanism is still unpredictable due to which improvement in material properties takes place. Further investigation would be needed.

VI. Conclusions

The Conclusions of this review study are as follows:

1 The complete process of Cryo-Heat-Treatment must be as follows :
austenitizing, quenching, DCT and tempering; preferably immediate one-by-one sequentially in a cycle.

2 Prior to DCT austenitizing temperatures plays vital role for improving the properties of the steel like wear resistance, hardness, toughness etc. Each material to be assessed separately for selecting the optimum austenitizing temperature and should be co-relate to the required desired properties after DCT.

3 More useful work has been reported by several researchers, but there are many ambiguities in parameters like austenitizing temperature, quenching temperature, rate of cooling soaking temperature, soaking period, rate of warming-up, tempering temperatures and tempering period needs further investigations and optimize all the parameters of DCT process for various materials. Determination of appropriate level of the above parameters results in to enhance the product quality, productivity and wider acceptance in the industries.

4 With a better understanding of this process a wider acceptance of DCT is possible. Researchers must focus their efforts for complete understanding of the mechanisms behind the formation of the ultra-fine carbides precipitation.

5 Information specific to the effect of DCT on steels and specifically what effects these percentage of retained austenite, hardness, wear and service life must gathered. Engineers and metallurgists must work to standardizing processing cycles including cooling and heating rates, hold times and temperature cycles to optimize the properties of the material. International experts must draft the standards and conduct trials to validate the processes for the more promising alloys. Each material needs to be separately assessed and an individual process route devised for it that will depend on the combination of hardness, toughness, and wear resistance required in service.

6 Beneficial effects of deep cryogenic treatment on wear resistance, hardness, toughness and fatigue behavior have widely confirmed by published papers especially for tool/ alloy steels. With rare exceptions, no noticeable effects on tensile properties have been found in literature.

Acknowledgment

Authors gratefully acknowledge the support of Hon. Dr. Sandip N. Jha, Chairman, Sandip Foundation, to encourage us to carry out a part of this research.

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