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REVIEW ARTICLE

APPLICATION OF CRYOGENICS TREATMENT AS AN ENGINEERING TOOL FOR TEXTILE APPARELS AND METALS: A REVIEW

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ABSTRACT

Cryogenics is the study of very low temperature (below -150 °C, -238 °F or 123 K). Cryogenic treatment is a treatment process widely used in recent years to enhance the material properties without sacrificing other properties at the same time. Cryogenic treatment, a supplementary process to conventional heat treatment, involves deep freezing of materials at cryogenic temperatures to enhance the mechanical and physical properties. It is a onetime 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 marten site 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 mach inability, cutting properties of tools, to improve surface properties, electrical properties, magnetic properties, and reduces tool consumption and down time for the machine tool set up, thus leading to cost reductions. This paper reviews the use of cryogenics as an engineering tool for textile apparels and metals. The summary of this review paper tells about the properties improved due to the cryogenic treatment and the reason behind them.

Key words: Photocatalysis, Immobilization, Titanium dioxide, Solar irradiation, and Inclined six steps fixed bed.

INTRODUCTION

Recently, application of cryogenic treatment in enhancing properties of tool materials has received wide acceptance by researchers and industries. Research has shown that cryogenic treatment increases product life, and provides additional qualities to the product, such as stress relieving (Firouzdor *et al.,* 2008, and Flávio *et al.,* 2006). Tool life is a major factor that is considered in production of finished product in manufacturing industry as any improvement in tool life will have a direct impact on the cost of production, tool changing time and maximum production target. Cryogenic treatment has been acknowledged by some researchers as means of extending tool life of many cutting tool materials, thus improving productivity significantly. Cryogenic treatment, a supplementary process to conventional heat treatment, involves deep freezing of materials at cryogenic temperatures to enhance the mechanical and physical properties. Cryogenics is the branch of physics and engineering that involve the study of very low temperature, how to produce it, and how materials behave at these temperatures. The word "cryogenic" comes from the Greek word "kryos", which means cold, and is simply the study of materials at low temperature such as 77 K (Kalia *et al.,* 2010). Cryogenics is important because rocket fuel (oxygen, hydrogen) must be loaded in as liquids at cryogenic temperatures. It is also used as good preservation of body tissues by cooling (Parthiban *et al.,* 2013). It is not well defined at what point on the temperature scale refrigeration ends and cryogenics begins (Bilstein *et al.,*1996), but scientists assume a gas to be cryogenic if it can be liquefied at or below −150°C (123.15K; −238.00°F). The U.S. National Institute of Standards and Technology has chosen to consider the field of cryogenics as that involving temperature below −180°C (93.15K; −292.00°F). Whenever material is subjected to any manufacturing operation, it is subjected to stresses. The stress manifests itself in the nature of defects in the crystal structure of materials. The most commonly observed defects are in the form of vacancies, dislocations, stacking faults etc. As the level of stress increases, the density of these defects increases, leading to increase in inter atomic spacing. When the distance between the atoms exceeds a certain critical distance, cracks develop and failure takes place. Deep subzero treatment uses third law of thermodynamics which states that entropy is zero at absolute zero temperature. This principle is to relieve stresses in the material. The materials are subjected to extremely low temperatures for a prolonged period of time leading to development of equilibrium conditions. This leads

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to ironing out of the defects in the material and also attainment of the minimum entropy state (Patil *et al.,* 2012). A person who studies elements that have been subjected to extremely cold temperatures is called a cryogenicist. The cryogenic processing on materials increases wear resistance, hardness, and dimensional stability, and reduces tool consumption and downtime for the machine tool set-up, thus leading to cost reductions (Gill *et al.,* 2010).The technique of cryogenic processing is a method that improves the physical and mechanical properties of materials such as metals, plastics and composites. Po *et al.* studied the effect of cryogenic treatment on the residual stresses and mechanical properties of aerospace aluminium. In his work, the cryogenic treatment was applied to the Al alloy used for aerospace application that had already been heat treated. It was slowly cooled without thermal shocks to approximately 89K, held at this temperature for 24 hours and reheated slowly, it was observed that after the cryogenic treatment the residual stresses was reduced by up to 9ksi in the parent metal. Significant enhancement was observed in Stress Corrosion Cracking performance was observed, very small increase in the value of the micro hardness fatigue and tensile properties were noted after the treatment. Venkateswara *et al.* studied on Cryogenic Toughness of Commercial Aluminum Lithium Alloys: Role of De lamination toughening. Based on a study of the fracture toughness and tensile behavior of commercial aluminum lithium alloys, 2090 and 8090 heat treated and cryogenic (77 K) temperatures, the following conclusions were drawn: All commercial alloys displayed increases in strength, uniaxial tensile ductility, and strain hardening rates with decrease in temperature from 298 to 77 K. The observed increase in uniaxial tensile ductility with decrease in temperature also appeared to be associated with loss of constraint from enhanced short transverse delamination at 77 K.

Eswara *et al.* studied Mechanical behavior of aluminium lithium alloys. It reveals significant information about the Aluminum Lithium alloys. These alloys are prime candidate materials to replace traditionally used Al alloys. Despite their numerous property advantages, low tensile ductility and inadequate fracture toughness, especially in the through thickness directions, militate against their acceptability. Xian *et al.,* investigated the Effect of Mechanical Properties and Microstructures of 3102 Al Alloy. In his work, the mechanical and microstructure properties of cryogenic treated Al 3102 H19, H26 or O state, were studied. The outcome of the result was that after deep cryogenic treatment, the strength of H 19 state increased and the elongation to failure decreased but in the O state the yield strength increased but the breaking strength, elongation decreased. Ish *et al.,* also studied the cryogenic processing of HSS M2: Mechanical Properties and XRD Analysis. He discovered that Deep cryogenic treatment practically removed all traces ofaustenite in the sample. The superior performance of cryogenically treated HSS can be attributed to the transformation of almost all retained austenite into martensite, a harder structure and precipitation of fine andhard carbides. Mohan *et al.* considered a research of 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 D3 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. Senthilkumer *et al.* studied the Effect of cryogenic treatment on the wear resistance property of En 19 steel. 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.

History of cryogenic

Scientists have been experimenting with the use of extreme cold to strengthen metals since the mid-1800s, but it was not until the advent of space travel that cryogenic processing really came into own. NASA engineers analyzed spacecraft that had returned from the cold vacuum of space and discovered that many of metal parts came back stronger than they were before spending time in space. Cryogenics and refrigeration technologies share a common history. The most obvious difference between the two is the temperature range. Cryogenics had its beginning in the mid of nineteenth century when for the first time man learned to cool objects to a temperature lower than had ever existed naturally on the surface of earth. First practical vapour compression refrigerator was invented by James Harrison in 1855. In 1872, Sir James Dewar invented the vacuum flask. The air first liquefied in 1883 by Polish scientist named Olszewski. Ten years later Olszewski and a British scientist Sir James Dewar liquefied hydrogen. In 1902, Georges Claude improved the efficiency of air liquefaction by including reciprocating expansion engine. The Dutch Physicist Kamerlingh Onnes finally liquefied helium in 1908. Thus, by the beginning of twentieth century, the door had been opened to a strange new world of experimentation (Foerg *et al.,* 2002, Scurlock *et al.,* 1990, Steckelmacher *et al.,* 1993, Richardson *et al.,* 2003, and Timmerhaus *et al.,* 1982).The method of cryogenic processing materials at sub-zero temperatures was first acknowledged when metal parts that were transported via train had been crammed with dry ice (at -79^{o}C), resulting in perceptible increases in wear resistance (Brown *et al.,* 1995). Work is being done to confirm that cooling below the temperature of dry ice (-79 ^{o}C), at boiling temperature of liquid nitrogen (-196 ^{o}C), would further improve the physical properties of materials (Sweeney *et al.,* 1986).

Method of cryogenic treatment

Liquid nitrogen is generated from anitrogen plant, and stored in storage vessels. With the help of transfer lines, it is directed to a closed vacuum evacuated chamber called cryogenic freezer through a nozzle. The supply of liquid nitrogen into the cryo-freezer is operated with the help of solenoid valves. Inside the chamber, gradual cooling occurs at a rate of 2 ^{o}C /min from the room temperature to a temperature of -196 ^{o}C. Once the sub-zero temperature is reached, specimens are transferred to the nitrogen chamber or soaking chamber where they are stored for 24 to 48 hours with continues supply of liquid nitrogen. Figure 1illustrates the set up for cryogenic treatment (Amrita *et al.,* 2007).

Figure 1. Set-up of cryogenic treatment in the temperature of 77 – 450 K, which consists of a well-insulated treatment chamber (right), liquid nitrogen in a Dewar vacuum flask (left), (*Linde gas, sub-zero treatment of steels, technology/processes/equipment*)

Cryogenic processing is capable of treating a wide variety of materials such as metals, alloys, polymers, carbides, ceramics and composites. Cryogenics is a dry process in which liquid nitrogen is converted to a gas before it enters the chamber so that it does not come into contact with the parts assuring that the dangers of cracking from too fast cooling are eliminated. The risk of thermal shock is eliminated as there is no exposure to cryogenic liquids. The whole process takes between 24to 74 h depending on the type and weight of material under treatment. Cryogenic processing must be done correctly in order to be successful. The basic steps in a cryogenic process are as follows (Kalia *et al.,* 2010, Singh *et.al*., 2003, and Gulyaev *et al.,* 1937).

- **Ramp Down:** Cryogenics involves slow cooling of the material from room temperature to 77 K and ramp down time is in the 4 – 10 h range.
- **Hold:** The material is soaked or held at 77 K for 20 – 30 h which depends upon the volume of the part. This is the part of the treatment in which the micro-structural changes are realized.
- **Ramp Up:** Finally, the material is brought back to room temperature. The ramp up time can be from 10 to 20 h range (Sandip *et al.,* 2012).

The ramp down time is 9 hours. A temperature of –196 °C is achieved in 9 hours. After ramping down to (-196 °C), material is soaked (hold) at this minimum temperature for 24 hours. It is again brought up to the room temperature in 9 hours known as the ramp up temperature. The total duration of the cryogenic treatment is about 42 hours. After the material is cryogenic treated, it is tempered to 150 °C. The temperature of 150 °C is achieved in 1.5 hours, kept it at this temperature for 4 hours. The material is brought back to room temperature in the next 1.5 hours. The total duration of tempering cycle is about 7 hours. Tempering is done in order to remove the stresses developed during cryogenic cooling (Rupinder *et al.,* 2010).

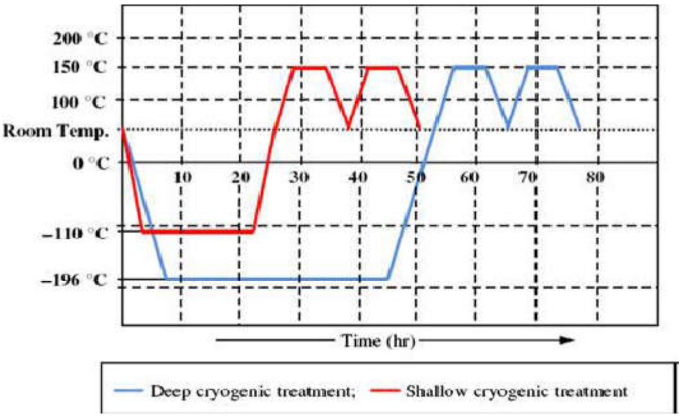

Figure 2. Time Vs temperature for shallow cryogenic & deep cryogenic treatment (Gill *et al.,* 2011).

Principle methods of cooling

There is a variety of methods to bring parts down to the desired processing temperature. However, all methods work on the same thermodynamic principles of heat transfer. All sub-zero equipment falls into two broad categories; direct or indirect cooling.

- **Direct Cooling:** Processors can use liquid nitrogen effectively to achieve the temperatures necessary for cryo treatment and to get quick cool down rates for cold treatment. One of the most common techniques is to use a spray header system with atomizing nozzles that convert the liquid nitrogen (LIN) to very cold gas, cooling the parts as the liquid nitrogen turns into vapor and warms up. The LIN is directly converted to cold gas to cool the parts.
- **Indirect Cooling:** Mechanical freezers are an example of indirect cooling. Nitrogen and mechanical means can both be used to cool an alcohol tank where parts could be submerged for cold treatment. Carbon dioxide in the form of dry ice has also been used to attain low temperatures for cold treatment. Since the temperatures of these techniques cannot go below about –120 °C or –185°F, they cannot be used for cryo treatment processes.

Types of cryogenic processes

Generally the cryogenic treatment is classified into two types;

- **Shallow Cryogenic Treatment:** In this treatment, the material is subjected at -110°C and held at this temperature for 18 - 25 hours and gradually brought back to the room temperature (Gill *et al.,* 2011).
- **Deep Cryogenic Treatment:** In this treatment, the material is subjected at -196°C and held at this temperature for 24 - 72 hours and gradually brought back to the room temperature (Gill *et al.,* 2011).

Effect of cryogenic treatment

Cryogenic treatment will significantly affect the microstructure of the material. These changes in microstructure enhance the properties of the material such as hardness and wear strength of the material (Gill *et al.,* 2008). Two different types of cryogenic solutions are mostly used for the treatment of materials:

- Liquid Nitrogen (-196 °C).
- Liquid Helium (-269 °C).

Liquid nitrogen is a cryogenic liquid that can be transported easily when insulated in a special container called Dewar flasks at atmospheric pressure, it boils at −196 °C. Liquid nitrogen is generally used as it is cheaper than liquid helium and is more available. The mechanism by which cryogenic treatment improves the performance of metals has no good clear understanding. Most researchers believe that the martensite temperature is below 0 °C due to the higher alloying element in alloyed metals. This 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. Therefore 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 (Haizhi *et al.,* 2016). 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. Specific experimental testing is required for each material to be treated after treatment (Haizhi *et al.,* 2016). 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 Shallow treatments (Chitrang *et al.,* 2017).

Table 1. Common Cryogenic Fluids with Their Boiling Points

Cryogen	(K)	(°C)	(°R)	(°F)
Methane	111.7	-161.5	201.1	-258.6
Oxygen	90.2	-183.0	162.4	-297.3
Nitrogen	77.4	-196.0	139.3	-320.4
Hydrogen	20.3	-252.9	36.5	-423.2
Helium	4.2	-269.0	7.6	-452.1
Absolute zero	0	-273.15	0	-459.67

Process of cryogenic heat treatment

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

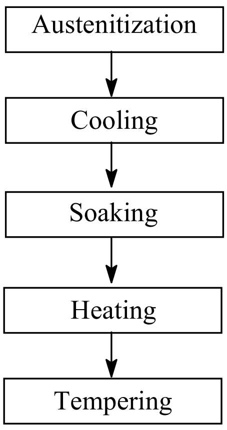

Figure 3. Process of Cryogenic Heat Treatment

Cryogenic Process Consist five stages, that involves:

- **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.
- **Cooling:** Direct cooling from austenitizing temperature to -196 °C at the rate of 1.5 to 2 °C. It is also called as quenching.
- **Soaking:** For a period of time ranging from 24 to 36 hours depends upon which material is to be treated.
- **Heating:** From -196 °C to room temperature at the rate of 0.5 to 1°C /min.
- **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.

Cryogenic application in textile and apparels

Liquid Ammonia Mercerization: Liquid ammonia mercerizing refers to the process that truly revives the cotton through the expression of liquid ammonia at an ultra-low temperature inside the fiber. When the fiber is treated at -33°C liquid ammonia, the ammonia at ultra-low temperature will permeate immediately into the crystallographic structure of the fiber. Stress will be released through interior expansion, which makes the fiber cavity round and smooth, and rearranges the molecular structure, thus the crystallographic structure becomes slack and stable. This physical change makes the surface of the entire fabric smooth and bright, with solid and soft feel, so elasticity and wash-and-wear is fully achieved. (Parthiban *et al.,* 2013).

Cryogenic Treatment in Garment Manufacturing: Garment cutting knives and sewing needles are given cryogenic treatment, which is the process of converting the austenite state (malleable) to the martensite state (tough), such that the processed materials have increased wear, toughness, and reduced brittleness (Fu *et al.,* 2002).

Cry Cooling Applications in the Garment Cutting Knives: Austenite is a soft allotropic form of iron that forms at high temperature. During cooling, it gets transformed to other structures of which martensite are the desirable harder phase. But rote of cooling plays a major role in the formation of martensite. The cryogenic process can be applied on garment cutting knives and sewing needles (Satish *et al.,*2004).

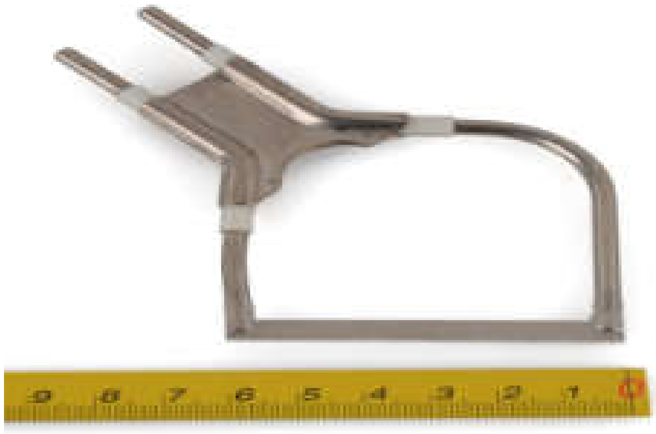

Figure 4. Cryogenic treated garment cutting knives

Cryogenic Treatment on Sewing Needles: Sewing needles are classified by their length and thickness. The numbering system is not directly related to the length or thickness of the needles, it serves only to distinguish one needle from another. These sewing needles are cryogenically treated, and the wear resistance of garment cutting knives and sewing needles improved (Parthiban *et al.,* 2013).

Figure 5. Cryogenically treated sewing needles

Industrial applications of cryogenic processing: There are several categories of industrial applications and representative parts (*Singh et al.,* 2006).

- **Paper and Corrugated Board Industry:** Chipper knives, slitter knives, tape cutters, paper drills, trimmers, tissue perforators.
- **Cutting Tools:** Mill, cutters, ball screws, punches, drills, broaches.
- **Performance Vehicles:** Crankshafts, brake rotors, push rods, heads, pistons, blocks, cams.
- **Plastics Industry:** Trimmers, mill knives, granulating blades, dies feed screws.
- **Other Applications of Cryogenics:**
- **Fuels:** Cryogenic fuels are used in rockets with liquid hydrogen as the most widely used example. Liquid oxygen (LOX) is even more widely used but as an oxidizer, not a fuel.
- **Frozen Food:** Cryogenic gases are used in transportation of large masses of frozen food. When very large quantities of food must be transported to regions like war zones, earthquake hit regions, etc., they must be stored for a long time, so cryogenic food freezing is used. Cryogenic food freezing is also helpful for large scale food processing industries.
- **Blood Banking:** Certain rare blood groups are stored at low temperatures, such as −165 °C.
- **It is also Applicable in:** Copper, electrode, gun barrels, sporting goods, razor blades, musical instruments.

Conclusion

Cryogenically treated materials show a marked increase in wear resistance without any desirable change in dimensional or volumetric integrity. The material shows little or no change in yield or tensile strength. The treated material becomes less brittle, without a change in original hardness. The most significant and consistent change is the increased toughness, stability, and wear resistance. Therefore, it has less maintenance and change over, which allows for lower production cost.

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