CHAPTER - I

INTRODUCTION

1.1 INTRODUCTION

The word Cryogenics is derived from two Greek words: "kryos", which means cold or freezing, and "genes", which means being born of or generated. The word Cryogenics means, the production of icy cold [1]. However the term is used today as a synonym for low temperatures. The point of low temperature on the temperature scale at which refrigeration in the ordinary sense of term ends and cryogenics begins is not sharply defined. The national bureau of standards at Boulder, Colorado, have chosen to consider the field of cryogenics as that involving temperatures below $-150^\circ$C as a logical line. Because the boiling temperature of the permanent gases, such as helium, hydrogen, neon, nitrogen, oxygen and air lie below $-150^\circ$C. While the Freon and other refrigerants boil at temperatures above $-150^\circ$C.

Low temperature Cryogenics started in the late 1800's when Sir James Dewar perfected a technique for compressing and storage of gases from the atmosphere into liquids. These compressed gases were super cold and any metal that came in contact with the ultra low temperatures showed some interesting changes in their characteristics. Now that these super cold liquids could be stored and transported. Experimentation with the cryogenic liquids could expand.

Cryogenic engineering involves the design and development of systems and equipments, which produce, maintain or utilize low temperatures. Experimentation with cryogenic temperatures continued and at the beginning of World War II scientists discovered
that material subjected to super low temperature showed signs of increased resistance to wear.

In the early 1960's, aerospace engineers applied the benefits of the cryogenic temperatures to stress relieve parts for use in space. Soon after, the military used the super cold treatment on aircraft applications. In the early 1970's, scientists from universities and research centers began documenting test results on industrial tooling as well as other applications. In the 1980's, cost associated with the process dropped and commercialization of the process began.

Cryogenic processing can be split into two categories; wet and deep dry cryogenic tempering. The wet process involves immersion of the material in liquid nitrogen, where direct exposure to the cryogenic liquid occurs. This process is believed to be inferior to the deep dry process because of the risk of the thermal shock and material cracking from extremely rapid cooling. The deep, dry process, however, does not result in any thermal shock, as the material ever is being exposed to the cryogenic liquids, and instead the liquids are converted to gas before they are inserted into the cryo chamber. The process then calls for a computer controlled cooling, where the material temperature is decreased by about 1° per minute to avoid shock. Typical soak times vary from 20-60 hours depending on the material type and the thickness of the sample. The time taken to raise the temperature of the material back up to room temperature affects the effectiveness of the process. This aspect of the process is some times computer controlled, especially for deep cryogenics. Materials may often require tempering after the cryogenic process is completed in order to restore the
fracture toughness, which is destroyed by the cryogenic process. Without tempering the product may be extremely susceptible to failures resulting due to impact loads experienced afterwards.

Heat-treatment is the process that gives steel its hardness, toughness, wear resistance and ductility. Even performed properly, heat-treating cannot transform all of the retained austenite from steel. Proper heat-treating is a key process in increasing part toughness, durability, wear resistance, strength and hardness.

The beneficial changes that occur as a result of the heat treat process do not actually take place during the heating, but rather from the rapid process of cooling or "quenching" from its high temperature. The benefits of the quench do not stop at room temperature, as many alloys will continue to show significant improvements if the quench temperature is reduced further to subzero levels approaching absolute zero [35]. While it is impossible to actually achieve −273 °C, (a molecular zero movement state that eliminates all stress), deep cryogenic treatments are very efficient and cost effective in increasing dimensional stability, wear resistance and performance of most alloys.

1.2 APPLICATIONS OF CRYOGENICS

Present day applications of cryogenic technology are widely varied [1], both in scope and magnitude. Some of the areas involving cryogenic engineering include the following.

1.2.1 ROCKET PROPULSION SYSTEMS

All the space vehicles use liquid oxygen as oxidizer. The space shuttle propulsion system uses cryogenic fluids, liquid oxygen and liquid nitrogen.
1.2.2 STUDIES IN HIGH-ENERGY PHYSICS

The hydrogen bubble chamber uses liquid hydrogen in the detection and study of high-energy particles produced in large particle accelerators.

1.2.3 ELECTRONICS

Sensitive microwave amplifiers, called masers, are cooled to liquid nitrogen or liquid helium temperatures so that thermal vibrations of the atoms of the amplifier element do not seriously interface with absorption and emission of microwave energy. Cryogenically cooled masers have been used in missile detectors, in radio astronomy to listen to far away galaxies and in space communication systems.

The superconducting electronic elements called Squids (Superconducting quantum interface devices) have been used as extremely sensitive digital magnetometers and voltmeters. These devices are based on superconducting phenomenon, called the Josephson effect, which involves quantum mechanical tunneling of electrons from one super conductor to another through an insulating barrier.

1.2.4 MECHANICAL DESIGN

Using the Meissner effect associated with superconductivity, practically zero friction bearings have been constructed that use a magnetic field as lubricant instead of oil or air. Super conducting motors have been constructed with practically zero electrical losses for such applications as ship propulsion systems. Super conducting gyroscopes with extremely small drift have been developed.
1.2.5 SPACE SIMULATION AND HIGH VACUUM TECHNOLOGY

To produce a vacuum that approaches that of space (from $10^{-12}$ torr to $10^{-14}$ torr), one of the more effective methods involving low temperature, Cryopumping or freezing out the residual gases, is used to produce the ultra high vacuum required in space simulation chambers and in test chambers for space propulsion systems. Cooling the shroud with in the environmental chamber by means of liquid nitrogen simulates the cold of open space. Dense gaseous helium at less than 20 K or liquid helium is used to cool the cryopanels that freeze out the residual gases.

1.2.6 BIOLOGICAL AND MEDICAL APPLICATIONS

The use of cryogenics in biology or cryobiology has aroused much interest. Liquid nitrogen cooled containers are used to preserve whole blood, tissue, bone marrow and animal semen for long periods of time. Cryogenic surgery (cryosurgery) has been used for the treatment of Parkinson's disease, eye surgery and treatment of various lesions. This surgical procedure has many advantages over conventional surgery in several applications.

1.2.7 FOOD PROCESSING

Freezing as a means of preserving food was used as far back as 1840. Today frozen foods are prepared by placing cartons on a conveyor belt through liquid nitrogen bath or gaseous nitrogen cooled tunnel. Initial contact with liquid nitrogen freezes all exposed surfaces and seals in flavor and aroma. The cryogenic process requires 7 to 8 minutes compared to 30-48 minutes required by conventional methods. Liquid nitrogen has also been used as the refrigerant in frozen food transport trucks and railway cars.
1.2.8 MANUFACTURING PROCESS

Oxygen is used to perform several important functions in the steel manufacturing process. Cryogenic systems are used in making ammonia. Pressure vessels have been formed by placing a formed cylinder in a die cooled to liquid nitrogen temperatures. High pressure nitrogen gas is admitted into the vessel until the container stretches about 15 percent and the vessel is removed from the die and allowed to warm to room temperature. Through the use of this method the yield strength has been increased from 400 to 500 percent.

Cryogenic Processing of materials is the latest technology developed to enhance the wear resistance of materials, to ensure dimensional stability and to give better life to the tools used by manufacturing industry.

1.2.9 RECYCLING OF MATERIALS

One of the more difficult items to recycle is the automobile or truck tire. By freezing the tire in liquid nitrogen, the rubber is made brittle and can be crushed into small particles. The tire cord and the metal components in the original tire can be separated easily from the rubber, and the rubber particles can be used again for other items. At present the cryogenic technique is the only effective way to recover the rubber from steel radial tire.

1.3 CRYOGENIC TREATMENT

Cryogenic treatment is the process based on a predetermined thermal cycle that involves cooling of the tools/parts in a totally controlled cryogenic chamber. The material is slowly cooled to $-190 \, ^\circ C$ and "soaked" at that deep cryogenic temperature for 20-40 hours.
The material is then allowed to return very slowly to ambient temperature. The whole cryogenic cycle can take up to 40-75 hours to complete. This procedure of precisely controlled temperature profiles avoids any possibility of thermal shock and thermal stress that is experienced when a tool or part is subjected to abrupt or extreme temperature changes. In this process liquid nitrogen is used as a refrigerant.

Cryogenic processing is not a substitute for heat treatment, but rather an extension of the heating, quenching and tempering cycle. In most instances a heat tempering procedure follows the cryogenic cycle to temper the untempered martensite that was formed during cryogenic processing. As all alloys do not have the same chemical constituents, the tempering procedure varies according to the materials chemical composition, thermal history and/or a tool’s particular service application.

1.3.1 THE EFFECT OF COLD TEMPERATURE ON METALS

Simply chilling metals to sub zero temperatures for stress relief and stabilization is a very old technique. About 150 years ago [11,16], Swiss watchmakers noticed that extreme cold changed the properties of their metal clock parts for the better. So after manufacturing their gears some watchmakers would then store the parts in caves during the cold Swiss winter and let them freeze. Unwittingly, that has given birth to what is now commonly known as cryogenic processing.

NASA engineers discovered the effects of cold temperatures on metals when they analyzed spacecraft that had returned from the cold vacuum of space. They noticed that many
of the metal parts in the spacecraft came back stronger than they were before flight. NASA developed Cryogenic Processing in the 1960's. Every vessel sent into outer space was processed to protect it from the extreme cold of outer space and the extreme heat of re-entry. Subzero treatment process (down to about -80 °C) have been used for many years for tool steels such as M-2 and D-2, but with the inconsistencies in their results.

1.3.2 CRYOGENIC PROCESSING USING LIQUID NITROGEN

The process of experimentation and understanding of the cryogenic treatment of steels really got under way during World War II at the Watertown Arsenal in Watertown, Mass. It was under the direction of Clarence Zener who would later go on to develop the Zener diode. At that time there were no computer controls so the steel tooling would be immersed in liquid nitrogen for a brief period of time, allowed to warm up, then placed into service. This method was crude and uncontrolled. Many of the tools would chip and break immediately upon use because the immersion process would create a very high thermal gradient in the tool and this would produce micro-cracks in the body. It was also later learned that the cryo-treatment would convert the retained austenite into un-tempered martensite. But the tools that would not break would experience a greatly enhanced service life.

In the 1960's cryogenic processors would use multi-stage mechanical coolers along with insulated 'cold-boxes' to gently remove the latent heat from tooling thereby achieving a much slower cooling rate, concurrent with longer wear lives. Performing a standard wear test (pin-on-disk) showed that the wear resistance for these steels could be increased by more
than 600%. At this time it was theorized that the increase in wear resistance was a direct result of the reduction in the amount of retained austenite.

The cryogenic liquids, which can be used for cryogenic treatment, are liquid nitrogen, liquid helium and hydrogen. But the later two are far more expensive and have not proven to be needed for the type of work presently being processed on ferrous and nonferrous applications. Liquid nitrogen is relatively inexpensive and performs the final steps of the heat-treatment transformation very well, and still colder treatment is not known to be necessary.

1.4 NEED FOR CRYOGENIC TREATMENT

Manufacturing companies around the world replace thousands of worn out steel tools every year. A leading Deep Cryogenic Company states, “Research has shown that the original cost of a tool is about ten percent of the total cost of using the tool”. It also shows that the greatest costs of using a tool are tooling failures. Tooling failures create down time, scrap, lost production, meeting delays, product failures, finger pointing and so on and on.

Making the tool more wear resistant and stable, cryogenic treatment can minimize the tooling failures.
1.5 HEAT TREATMENT

To understand the effects of cryogenic processing it is essential that one be acquainted with the heat-treating of metals. The primary reason for heat-treating steel is to improve its wear resistance through hardening. Gears, bearings, and tooling for example are hardened because they need excellent wear resistance for extended reliability and performance. The steps in heat-treating are frequently explained in a simplistic manner but it takes significant skill and experience to execute heat treatments successfully.

1.5.1 DEFINITION OF HEAT-TREATMENT

A combination of heating and cooling operations applied to a metal or alloy in the solid state to obtain desired conditions or properties.

1.5.2 NEED FOR HEAT TREATMENT

Tool steel is normally delivered in the soft annealed condition. This is to make the material easy to machine with cutting tools and to give it a microstructure suitable for hardening. The microstructure consists of a soft matrix in which carbides are embedded. In carbon steel, these carbides consist of iron carbide, while in the alloyed steel they are chromium (Cr), tungsten (W), molybdenum (Mo) or vanadium (V) carbides, depending on the composition of the steel. Carbides are compounds of carbon and these alloying elements and are characterized by very high hardness. Higher carbide content means higher resistance to wear. In alloy steels, it is important that the carbides are evenly distributed. Other alloying elements are also used in tool steel, such as cobalt (Co) and nickel (Ni), but these do not form
carbides. Cobalt is normally used to improve red hardness in high-speed steels, nickel to improve through-hardening properties.

Heat Treatment is often associated with increasing the strength of material, but it can also be used to alter certain manufacturability objectives such as improve machining, improve formability, and restore ductility after a cold working operation. Thus it is a very enabling manufacturing process that not only helps other manufacturing process, but can also improve product performance by increasing strength or other desirable characteristics. Steels are particularly suitable for heat treatment, since they respond well to heat treatment and the commercial use of steels exceeds that of any other material.

Hardening of steels is done to increase the strength and wear properties. One of the pre-requisites for hardening is sufficient carbon and alloy content. If there is sufficient Carbon content then the steel can be directly hardened.

1.5.3 FORMATION OF AUSTENITE

In soft annealed tool steel, most of the alloying elements are bound up with carbon in carbides. In addition to these there are the alloying elements cobalt and nickel, which do not form carbides but are instead dissolved in the matrix. When the steel is heated for hardening, the basic idea is to dissolve the carbides to such a degree that the matrix acquires an alloying content that gives the hardening effect without becoming coarse-grained and brittle. Note that the carbides are partially dissolved. This means that the matrix becomes alloyed with carbon and carbide-forming elements. When the steel is heated to the hardening temperature
(austenitizing temperature), the carbides are partially dissolved, and the matrix is also altered. It is transformed from ferrite to austenite.

1.5.4 FORMATION OF MARTENSITE

This means that the iron atoms change their position in the atomic lattice and make room for atoms of carbon and alloying elements. The carbon and alloying elements from the carbides are dissolved in the matrix. If the steel is quenched sufficiently rapid in the hardening process, the carbon atoms do not have time to reposition themselves to allow the re-forming of ferrite from austenite, i.e. as in annealing. Instead, they are fixed in positions where they really do not have enough room, and the result is high microstresses that can be defined as increased hardness. This hard structure is called martensite. Thus, martensite can be seen as a forced bonded solution of carbon in ferrite.

A close analogy to this is coal and diamond. Primarily carbon atoms make them both. In coal the carbon atoms are in a very loose arrangement and have very few cross-links to one another, this makes them readily available to be taken away by wear or some other reaction like heat. In a diamond the carbon atoms have a very different bonding arrangement making it the hardest substance known.

1.5.5 TEMPERING

When steel is hardened, the matrix is not completely converted into martensite. Some austenite is always left and is called "retained austenite". The amount increases with increasing alloying content, higher hardening temperature and longer soaking times. After
Quenching, the steel has a microstructure consisting of martensite, retained austenite and carbidies. This structure contains inherent stresses that can easily cause cracking. But reheating the steel to a certain temperature, reducing the stresses and transforming the retained austenite to an extent that depends upon the reheating temperature can prevent this. This reheating after hardening is called tempering. Hardening of tool steel should always be followed immediately by tempering. It should be noted that tempering at low temperatures only affects the martensite, while tempering at high temperature also affects the retained austenite. After one tempering at high temperature, the microstructure consists of tempered martensite, newly formed martensite, some retained austenite and carbidies. Precipitated secondary (newly-formed) carbidies and newly formed martensite increase hardness during high-temperature tempering. Typical of this is the so-called secondary hardening of e.g. high-speed steel and high-alloyed tool steels.

1.5.6 LIMITATIONS OF HEAT TREATMENT

The problem with heat treatment is that when the steel is heated up to a phase called austenite and then quenched with oil, air or water it is usually not a 100% martensite because the steel must reach a much lower temperature than room temperature to successfully transform. Another problem that is associated with heat treatment is the internal stresses and the dimensional instability due to the retained austenite.

1.5.7 RETAINED AUSTENITE

Retained austenite [12] is a softer grain structure always presents after heat treatment, which may transform into the harder and more durable grain structure – martensite at a later
stage. The range of retained austenite in a material after heat-treating may be as high as 50% or as low as 3%. The amount depends on the heat-treating operator and the accuracy of the heat-treating equipment. The retained austenite also depends upon tempering cycle. For high-speed steels (HSS) the amount of retained austenite is proportional to the number of tempers.

To minimize the amount of retained austenite triple temper is necessary [35]

1.5.8 EFFECT OF RETAINED AUSTENITE

The retained austenite content of steel can have a pronounced effect on the performance and life of steel products. Especially where dimensional stability is critical, any retained austenite can be detrimental to the useful life and performance of the part.

Austenite is the face-centered cubic phase in steels formed at high temperatures. During quenching and other heat-treating operations, austenite can be transformed into other phases such as martensite (a body-centered tetragonal phase). The volume of the austenitic unit cells greater than either the martensitic or ferritic unit cells. Therefore, if the austenite transformation is not 100 percent, the retained austenite that remains after heat-treating can further transform during the service life of the product into other phases, providing the potential for changes in the dimension of the part. In addition, other physical properties, such as hardness and strength, vary with the different phases, so that if the part is transformed during service, the physical properties may also change.

It should be noted that freshly formed martensite is brittle and only tempered martensite is acceptable. The transformation of austenite in to martensite yields 4% volume
expansion causing distortion, which cannot be ignored. Therefore the retained austenite should be transformed to the maximum possible extent before any component or tool is put into service.

1.6 LITERATURE SURVEY

Dr. Randall F Barron is the most noted academic authority on the subject of cryotreatment today. He holds both masters and doctorate degrees in Mechanical Engineering from the Ohio State University. His area of expertise is in heat transfer, and he is Professor in the Mechanical and Industrial Engineering Department at Louisiana Tech University, Ruston LA. His dissertation was on "Heat and Mass Transfer to a Cryo surface in Natural Convection." Dr. Barron is cited by many of the cryotreatment companies for studies of the effects of cryotreatment. For example, an article that appeared in the August 1982 issue of Cryogenics, "Cryogenic treatment of metals to improve wear resistance," studied nineteen metals, including 12 tool steels, 3 stainless steels and 4 other steels, subjected to cryogenic treatments to determine the difference between a 189K soak and a 77K soak in improving the abrasive wear resistance. The tool steels exhibited a significant increase in wear resistance after the 77K soak and a less dramatic increase after the 189K soak. There was an increase in wear resistance after the cryotreatment for the stainless steels, but the difference between the two treatments was less than 10%. The plain carbon steel and the cast iron showed no improvement after either cryotreatment.

An article in the January 1992 issue of Modern Machine Shop summarizes Dr. Barron's explanations of what happens and why: "Researchers are not certain about what
happens to materials at 190 degrees below zero (so-called deep cryogenic treatment). Alloy tool steels used to make end mills, twist drills, reamers and other cutting tools have been the object of the most study in this area. Results vary, but a two to seven fold improvement over the normal life of these tools is typical.

One of the best explanations was put forth by Dr. Randall Barron, one of the foremost researchers in cryogenic treatment of metal. Two primary mechanisms are at work, he wrote. First, super-cold treatment apparently converts any retained austenite into martensite, and the martensite is to be tempered as the metal returns to room temperature to transform the untempered martensite to acceptable tempered martensite [2].

The martensite structure resists plastic deformation much better than the austenite structure, because the small carbon atoms in the martensite lattice 'lock together' the iron atoms more effectively than in the more open-centered cubic austenite lattice. Tempering the martensite makes it tougher and better able to resist impact than untempered martensite.

Second, the cryogenic treatment of high-alloy steels, such as tool steels, results in the formation of very small carbide particles dispersed in the martensite structure between the larger carbide particles present in the steel. This strengthening mechanism is analogous to the fact that concrete made of cement and large rock is not as strong as a concrete made of cement, large rocks, and very small rocks (coarse sand). The small, hard carbide particles within the martensite matrix help support the matrix and resist penetration by foreign particles in abrasive wear.
R F Barrow and C R Mulharan [3] investigated the relationship between the wear resistance improvement and the factors involved in cryogenic treatment for tool steel. They considered AISI T8 and C 1045 materials. A set of five samples of each material were tested with no cryogenic treatment, and 16 samples were subjected to a cryogenic treatment involving four different levels of cool down rate, soak temperature and soaking time. The most rapid cool down rate was achieved by immersing the sample in liquid nitrogen, and other cool down rates are obtained by cooling the specimens in gaseous nitrogen. Abrasive wear tests were run by pressing the end of the sample against an aluminum oxide grinding wheel, which was rotated at a constant speed of 77 r.p.m. They observed that cryogenic treatment consisting of slow cool down from ambient to liquid nitrogen temperatures followed by 24 hour soak at the cryogenic temperature resulted the maximum improvement by a factor 2.4 for the AISI T8 steel and a factor 1.4 for the C 1045 steel.

J.Y. Huang, Y.T. Zhu, X.Z. Liao, I.J. Beyerlein, M.A. Bourke and T.E. Mitchell [4] studied the microstructure of M2 tool steel to analyze the underlying mechanism remains unclear. They studied the microstructure changes of M2 tool steel before and after cryogenic treatment. They found that cryogenic treatment could facilitate the formation of carbon clustering and increase the carbide density in the subsequent heat treatment, thus improving the wear resistance of steels. A commercial M2 tool steel rod with a diameter of 6.35 mm was used in the experiment. The composition of sample is (weight%) 0.85 - 1 C, 6 W, 5 Mo, 4 Cr, 2 V. Their investigation in M2 tool steel favors the hypothesis for two reasons, (1) the distribution of the carbides in the cryogenic treated samples are more
homogeneous than that in the non-cryogenic treated samples, and (2) carbide volume fraction in the cryogenically treated samples is almost twice as much as that in the non-cryogenically treated ones. The precipitation of more hard carbides in the cryogenically treated samples can reduce the carbon and alloy contents in the matrix, which improves the toughness of the matrix. We believe the combination of higher carbide content and tougher matrix enhanced the wear resistance. It is unclear how the cryogenic treatment promotes the precipitation of carbide in martensite. The only clues they have are two engineering observations first, martensite needs to be cooled well below the temperature at which transformation of austenite to martensite starts during cooling; and second, the longer the holding time, the higher the carbide population and volume fraction. They summarized 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.

D. Mohan Lal, S. Renganarayan and A. Kalanidhi [5] studied the improvements in wear resistance of M2 and T1 steels, also studied D3 steel to check the influence of cryogenic treatment with respect to carbon percentage. The experiments were conducted with different types of samples like untempered and cryogenically treated, tempered and cryogenically treated. Treatment is carried at two different temperatures with different soaking periods. They observed that high improvement is obtained with the samples treated at 93 K and opinioned that the soaking time is more important than lowering the temperature.
Fanju MENG, Kohsuke TAGASHIRA, Ryo AZUMA and Hideaki SOHMA [6]

investigated the improvement in wear resistance of an Fe-12.2wt%Cr-0.84wt%Mo-
0.43wt%V-1.44wt%C alloy tool steel after cold treatment at 223 K and after cryogenic
treatment at 93 K. The effects of cryogenic treatment on the microstructure were also studied
by means of X-ray diffraction and transmission electron microscopy methods. The wear rate
of specimens after cryogenic treatment is smaller than that of as-quenched specimens
(without any subzero treatment) for whole sliding speeds. Furthermore, it decreases
dramatically at high sliding speed. The cryogenic treatment results show 110 to 600% improvements. The volume fraction of retained austenite is 12% for all quenched specimens
after austenitzation at 1293K, and approximately 6% for specimens after cold and cryogenic
treatment. However, it decreases with treating temperature going down for specimens
austenitzed at 1373K. Cold treatments reduce the volume fraction of retained austenite
drastically. Nevertheless, cryogenic treatment reduces it slightly relative to cold treatment.
They concluded that Cryogenic Treatment increases wear resistance dramatically, especially
at high sliding speed. The specimens after cryogenic treatment show a minimum of wear rate,
unlike cold treatment, cryogenic treatment promotes preferential precipitation of fine n-
carbides, The formation mechanism of n-carbides is supposed to be as follows: iron or
substitutional atoms expand and contract, and carbon atoms shift slightly due to lattice
deformation as a result of cryogenic treatment. The mechanism that cryogenic treatment
contributes to wear resistance is through the precipitation of fine carbides, which enhances
strength and toughness of martensite matrix, rather than the removal of the retained austenite.
PEN-LI YEN [7] studied the “Effect of cryogenic treatment on the wear resistance to tool steels” as partial fulfillment for the requirements for the degree of Doctor of Philosophy at the Pennsylvania State University. He studied the improvement in wear resistance of two tool steels. One AISI 4340 material and the other T 15 tool steel. He conducted block on disk wear resistance test for AISI 4340 material and a machining test (Turning operation) for the tool steel T 15. He opinioned that untempered tool inserts experience multiple tool wear problems, which may cause tool failure before flank wear reaches its failure criterion. He found that eta carbides precipitate during the tempering process after prior cryogenic treatment. Cryogenic treatment without tempering causes a transformation of retained austenite to martensite. No eta carbides precipitate during this process. He also found that longer tempering time and higher tempering temperature yields a higher content of eta carbide precipitation. Experimental results also showed that for higher machining speeds eta carbide precipitation may not be able to enhance wear properties as it does at low speeds due to higher temperatures generated during cutting, which may approach the hot hardness of the metal. He also found that cryogenic treatment created a tough, stable microstructure after single temper, where as conventional heat treatment or sub zero treatment requires multiple tempering to achieve a similar microstructure.

Huang Luguang, Yia Yanguo and Hu Xianguo [8] investigated the wear resistance improvement of a D2 steel by cryogenic treatment and found that the life is increased by 6-8 times. They concluded that the main reason for increasing the useful life of D2 tool and die at -160 °C cryogenic treatment is that most residual austenite in steel are transformed into martensite, texture micro fined, carbide dispersion educed and the combining strength
between carbide and matrix increased. They also opined that cryogenic treatment can fully exploit the material potentialities of most tools and die and therefore can increase the use life of tool and die by a big margin.

Richard N. Wurzbach and William DeFelice [9] Maintenance Reliability Group, Brogue, Pennsylvania dealt a problem from a paper mill that the paper mill was experiencing extremely low operating life of a water pump impeller. Because of head conditions in the system, the impeller was subjected to significant cavitation forces, which rendered unusable after every 3 months in service. The mill was forced to shut down the pump and swap out the impellers for every 3 months. With system redesign not cost-feasible, the only option seemed to be the purchase of expensive stainless-steel impellers.

As an alternative, the existing silicate bronze alloy impellers were prepared with a combination of surface treatment and cryo-treating. The result was an increase in hardness and wear resistance of the alloy, and an indefinite increase in the life of the impeller in the same conditions.

Lauren Gallagher [10] studied the enhancement in strength and durability that would be gained by cryogenically treating titanium lacrosse shafts. A standard Brine Titanium defenseman’s shaft measuring 5 feet long with a 11/8th inch outer diameter and a wall thickness of 1/16th inch, was sectioned to provide titanium lacrosse shaft samples that could be tested with the cryogenic treatment. The Brine shaft used was advertised to have a high strength to weight ratio, and a strength exceeding 120 kpsi. Sections taken directly from
the shaft were analyzed in order to determine the initial strength characteristics and microstructure of the untreated material. One section of the shaft was used to make a metallographic mount in bakelite, which was then ground and polished. The titanium alloy was then etched using Kroll's Reagent, a 2% hydrochloric acid, 6% nitric acid, 92% distilled water mixture. Initially, the etchant did not produce significant results revealing the microstructure of the untreated titanium. The sample mount was then let to dip in the etchant for a period of 15-20 minutes. This resulted in a more distinct microstructure that was discernable under 5-20X magnifications. He opinioned that the process of investigating the cryogenic process and finally materializing a test on titanium alloy was overall a valuable learning experience. There was no concrete evidence discovered that the process enhanced the properties of titanium alloy lacrosse shaft.

C.H. Gur, B.O. Tucee [11] studied the relationships among sound velocity, sound attenuation, microstructure and hardness. AISI/SAE 1040 and 4140 steels in the form of hot rolled bars of 30 mm diameter were cut perpendicular to the rolling direction to 5 mm thick specimens, and then, the specimens were ground to obtain appropriate roughness and surface parallelity. Since some differences in grain size and bonding have been observed in the initial microstructures, all specimens were water-quenched after austenitizing at 900°C for 0.5 h. As a result, initial microstructures of all specimens became identically martensitic. After austenitization at 850°C for 0.5 h the specimens have been heat-treated.

In this study, in order to be able to determine only the effect of phases on sound velocity, the average grain size tried to be kept constant. Before the isothermal heat
treatments, all the specimens have been austenized and quenched in water following the same procedure to obtain martensitic structure, next for various isothermal treatments, each specimen has been austenized at 850°C for 0.5h. Then, the isothermal heat treatments have been performed leading to the desired microstructure. Thus, the average size of austenite grains of all specimens maintained constant, ensuring the only parameter affecting the ultrasonic measurement to be the differing microstructures.

Tempering of martensite resulted in an increase in the sound velocity. After tempering at 200°C, sound velocity increased very slightly compared to martensite. During tempering, the dissolved carbon precipitates into carbides. This is associated with a decrease of the specific volume of martensite and complete relief of micro residual stresses. Together with this, partial loss of tetragonality and very small drop of dislocation density seem to be the reasons of the velocity increase. Tempering at 600°C resulted in a marked reduction in dislocation density, which directly reduces micro residual stresses. Those combined with the changes in the morphology of the microstructure result in a remarkable increase in the sound velocity.

R. Colaço and R. Vilar [12] Investigated the effect of retained austenite and martensite on wear resistance. In this work, the influence of the proportion of retained austenite in the abrasive wear resistance of a laser surface melted martensitic stainless tool steel is analyzed. Samples of a 0.5 wt.% C, 13 wt.% Cr + Fe martensitic stainless tool steel were surface melted using a CW CO₂ laser. By changing the processing parameters, samples with proportions of retained austenite varying from 15 to 100% were produced. Microscale abrasive wear tests were performed on the samples. The wear behaviour of the laser-melted
samples was compared with that of a sample of the same steel in the conventional quenched and tempered condition. It was observed that the wear behaviour of the material depends not only on the proportion of retained austenite but also on the test conditions, namely the applied load. For the lower test loads, it was observed that the material submitted to conventional heat treatment presents higher wear resistance; on the contrary, for the higher test loads, it was observed that some of the laser melted samples present a wear resistance higher than that of the conventionally treated material. The analysis of the worn surfaces reveals that, in the laser surface melted samples, the stress-induced transformation of austenite into martensite plays an important role in the increase of the wear resistance of the material with increasing applied load. They concluded that at lower loads, the material with a microstructure formed of martensite and carbide particles exhibited the higher wear resistance. Conversely, at higher loads, a microstructure formed of martensite and 15–25% of retained austenite exhibited the higher wear resistance.

Po Chen., Tina Malone., Robert Bond and Pablo Torres [13] studied the benefits of cryogenic processing for aerospace aluminum alloys. Cryogenic treatment was applied to aluminum alloy that had been heat treated or welded. They found that the residual stress was reduced, significant improvements in SCC performance were seen, minor increase in tensile strength and hardness were noted for parent metal and no significant changes were found in tensile properties for weld specimens or in fatigue properties.

1.7 SUMMARY OF LITERATURE SURVEY

In the literature survey, several investigations have been reported to quantify the improvements with cryogenic processing of metals with different materials. All the works
have been carried out only at two different temperature treatments. The subzero processing or shallow cryogenic processing at a temperature of -78 °C and deep cryogenic processing at a temperature of -190 °C. The traditional idea behind the processing only at two intervals is -78 °C is the dry ice (Solid Carbon dioxide) temperature and -190 °C corresponds to liquid nitrogen temperature. But no investigation is carried out in the middle of the two treatments.

In summery, first, it is believed that during cryogenic processing most of the retained austenite would transform into martensite and increases wear resistance. Second, fine carbides will precipitate during deep cryogenic soaking which will enhance the wear resistance significantly than the sub zero processing.

No work has been reported to quantify the improvements in wear resistance at different temperature intervals. Very few investigations are observed with Metallography studies to prove the dispersion of fine carbides, which are expected to precipitate during deep cryogenic soak and tempering after cryogenic treatment.

Therefore there is a need to investigate and quantify the improvements in wear resistance of metals at different temperature intervals with the Metallography studies to understand the mechanism of wear resistance improvements and the structural changes what happens to the metals at every temperature interval.
1.8 OBJECTIVES OF THE PRESENT WORK

The present work is concentrated on quantifying the improvements in wear resistance of three selected materials at different temperature treatments starting from normal heat-treated, treated at -40 °C, -78 °C, -150 °C and -190 °C. A cryogenic system has been designed for the purpose. Efforts are also made to record the microstructure of the metal at every stage of treatment to identify the mechanism of improvements in wear resistance.

The objectives of the present work are

1. To design a cryogenic system for economical processing of metals.
2. To process the selected metals at different temperature intervals.
3. To quantify the improvements in wear resistance at each of the processing stage.
4. To record and analyse microstructure at every stage of processing.

1.9 ORGANIZATION OF THE THESIS

The introduction, literature survey of cryogenic processing, summary of literature survey and the objectives of present work are presented in the first chapter.

Second chapter deals with the literature and history of cryogenic processors and design of a cryogenic system for cryogenic processing of metals using liquid nitrogen with minimum consumption of liquid nitrogen for economy of operation and to obtain the slow cooling rate required for cryogenic processing. The design methodology also aimed at variable rate of cooling if required, by varying the mass flow rate of liquid nitrogen.
The third chapter presents the history and methods of cryogenic processing, benefits of cryogenic processing, applications of cryogenic processing, materials selected & their composition. The experimentation method is discussed in detail.

In the fourth chapter, the mechanism of wear, different types of wear and their effects, the importance of abrasive wear, need for improving the wear resistance and method for investigating the wear resistance have been discussed. General methods other than cryogenic processing used for enhancing the wear resistance are also discussed. The abrasive wear of all the specimens are tabulated at different sliding speeds, different loads and for different abrading periods for every specimen individually.

The fifth chapter deals with the importance of Metallography, history of Metallography, step-by-step procedure to be followed in polishing the specimen to reveal the microstructure of the metals. The microstructures of all the specimens are recorded at a magnification of 500X.

Results are presented in detail in the sixth chapter. The experimental data recorded in the fourth chapter are analyzed and tabulated individually for every specimen. The average wear resistance is calculated initially for all the loads and then summarized to the average of all abrading periods and speeds. All the results are presented graphically also for easy visualization. Microstructures recorded in the fifth chapter are analyzed.

Conclusions and future scope of the present work is discussed in the seventh chapter.