Study the effect of shallow cryogenic treatment on mechanical and microstructural properties of 30CrMnSiA steel heat treated in vacuum furnace and Box Type conventional air furnace.

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I would like to thank my parents and teachers for their support and guidance. Their help and guidance encouraged me and provide me confidence in difficult times during my research. I fully acknowledge their patience. They have contributed the most to my study.

Dedicated to my parents for their support

Abstract

Heat treatment plays a major role in improving the material properties. With the addition of cryogenic treatment these properties can be further improved. In this paper heat treatment is carried out on low carbon low alloy structural steel in two different types of furnaces conventional box type air furnace and vacuum furnace along with shallow cryogenic treatment with the purpose to investigate the outcome of shallow cryogenic treatment on the material properties and to find out which type of furnace is more suitable with shallow cryogenic treatment. For this purpose, structural steel 30CrMnSiA was selected, and heat treatment was carried out in conventional box type air furnace (900°C × 40mins) and vacuum furnaces (900°C × 70mins), shallow cryogenic treatment (-75°C × 125mins) was carried out after the quenching and before tempering. For the physical properties of the material, hardness (HRC) was investigated using Brinell hardness tester HBD-62-5AP. Tensile samples were prepared as per ASTM E8/E8M and investigated using 100kN WAW-100B Electro-Hydraulic Servo Computer Control Universal Testing Machine. Impact toughness was measured by using electronic pendulum impact testing machine CBD-300 and specimens were prepared using ASTM standard E23-12c. Microstructural analysis for grain structure was carried out using Olympus DSX1000 optical microscope.

Key Words: Heat Treatment, Cryogenic, Shallow Cryogenic, 30CrMnSiA, Vacuum Furnace, Box Type Air Furnace

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CHAPTER 1: INTRODUCTION

1.1 Heat treatment

Heat treatment is a set of industrial, thermal, and hot metal working tactics used to improve/change the physical, and microstructural properties of the material. Heat treatment is the material heating process in which material is heated below its melting point as per requirement and cooled in a controlled manner to achieve desired results (physical and microstructural properties). This process is used on metals to make them more strong or ductile, harder, and more resilient. Whatever your preferred properties, it's for the reason that you'll by no means to be capable of getting the whole thing you want. You will make the material brittle while the process of hardening. when you HT to soften the material, the strength is lost. While you enhance a few properties, you get worse others and might make choices primarily based totally on the application of the material.

Every heat treatment process contains heating and cooling of materials, however there are 3 principal variations in the process: the heating temperatures, the cooling rates, and the quenching media and rate used to alter the properties as per requirement.

For the heat treatment of the material, you'll require the right system so you can carefully manage all the elements round heating, cooling, and quenching. For example, the furnace needs to be the right length and sort to govern temperature, such as the gas combination withinside the heating chamber, and appropriate quenching media is required for the controlled cooling of the material.

1.2 Heat treatment stages

Heat treatment is carried out in three different stages

- Heating the material slowly to make certain that the material's temperature is uniform.
- Soak, or hold, the material at a particular temperature for an allocated timed duration.
- Cooling the material at controlled temperature.

During the first stage, the aim is to make certain that the material is heating uniformly. This can be accomplished by heating the material slowly. If the heating of the material is uneven, one phase might also additionally expand quicker than the other one, resulting in the formation of cracks or distortions in the different sections of the material. The heating rate can be decided as per following factors:

- The heat of conductivity of the material. Materials with low heat conductivity of the heat, their heating is slower than those with high heat conductivity.
- Material condition. Tools and components which have been hardened, or stressed, formerly must be heat treated slower than the components that have not.
- Material size and its cross-sectional area. Larger components or components with even cross-sections are required to be heated more slowly than small components to permit the internal temperature to be near the top surface temperature. Otherwise, wrapping can occur in sheets and can also result in the formation of cracks.

Soaking is important phase in heat treatment as it helps to preserve the material at an appropriate temperature till the crystalline structure is achieved. To decide the right duration of time, you'll want the chemical evaluation and mass of the material. For random cross-sections, you could decide the soaking duration can be determined by the biggest section.

In general, the temperature of the material is not increased from ambient temperature to the soaking temperature in a single step. Rather, it is heated slowly to simply underneath the temperature wherein the material structures change, at this temperature, it is held to achieve a uniform temperature throughout the material. This step is known as preheating. After that temperature is raised to the required HT temperature to carry out the process. Multiple preheating steps are involved in the parts that have a complex design to prevent them from wrapping.

In this stage, material is cooled to room temperature, however there are more than one method to which depends on sort of material. It can also additionally require a cooling medium that can be a gas, liquid, or a combination of both. The cooling rate relies upon the material itself and the media of cooling. Cooling is a critical element to achieve the desired material properties.

The process in which rapid cooling of material takes place in the air, water, oil, brine, or some other medium is known as quenching. Usually, quenching is related to hardening since most materials that can be hardened are cooled by the process of quenching, however it isn't continually true that quenching or in any other case speedy cooling results in the hardening of the material. Water quenching, for example, is used in the annealing of copper, and different materials are hardened with gradual cooling.

Some materials cannot be quenched. Cracks or warping of few materials can be the result of quenching. Normally, water or brine can suddenly cool the material, while oil is better for slower cooling. water is used for the hardening of carbon steels, oil is used for the hardening of alloy steels, and water is also used to quench non-ferrous materials. However, as with any treatment, the chosen cooling rate and cooling medium should be the fit for the metal.

1.3 Methods of Heat Treatment

Few different heat treatment techniques are used to get required results. Some of the common techniques are listed below.

- Annealing
- Normalizing
- Tempering
- Carburization
- Hardening
- Ageing
- Stress relieving

1.3.1 Annealing

In this process, the material is heated past the upper critical temperature after which cooling is carried out at a controlled rate. The main purpose of this process is to soften the material. It makes the material suitable for coldworking and other machining processes like forming etc. The ductility, machinability, and toughness of the metal are also enhanced using this process. Annealing is likewise beneficial in relieving stresses withinside the element which was caused due to cold working. At upper critical temperature recrystallization take place which also removes plastic deformation present in the material. Metals can also additionally go through many different annealing strategies which include recrystallization annealing, partial annealing, full annealing, etc.

1.3.2 Normalizing

Normalizing is a HT interaction that is utilized for easing the interior stresses brought about by different cycles like welding, projecting, machining, or extinguishing. In this cycle, the temperature is more than the temperature utilized for solidifying or toughening. Subsequent to holding it at this temperature for a designated span, it is cooled, and cooling happens in the air. Uniform grain sizes are accomplished through normalizing. When contrasted and toughening all the more hard and more grounded materials are created through normalizing. In its standardized structure, steel is harder than some other structures. That is the explanation components that require great strength or need to lift monstrous burdens are constantly standardized.

1.3.3 Tempering

Treating is the technique of diminishing additional hardness, and thus weakness, caused at some stage in the solidifying method. Inside burdens additionally are feeling quite a bit better. Going through this methodology could make steel suitable for uses that need such properties. The temperatures are ordinarily lower than solidifying. Higher the temperature becomes, gentler the material we get. During the most common way of treating for the most part the cooling doesn't influence the material design, material can be cooled in still air or water.

1.3.4 Carburization

In this process of heat treatment, the steel is heated withinside the presence of any other material that decomposes and releases carbon. The surface of the steel absorbs that released carbon. After absorbing the carbon content into the surface, it becomes harder than the internal core.

1.3.5 Hardening

It is one of the most used heat treatment processes in the industries, this process is used to improve or increase the hardness of the material. but sometimes it is only used to increase the hardness of the outer surface of the material. A metal part is hardened in the furnace by heating it to the required temperature, then rapid cooling is carried out by submerging it right into a cooling medium. Oil, water, or brine are used as a cooling medium. As a result, hardness and strength of the parts increase, however, at the same time brittleness of the part increase. Case hardening is a kind of hardening technique that is used to harden the outer layer of the material. The technique used is identical however as the outer layer is subjected to the technique, the final workpiece is soft from the core and has a harder outer layer. This process is suitable for shafts. Wear properties of the material are enhanced. When mounting a bearing to a shaft, it could in any other case damage the surface and dislocate few particles that can boost up the wearing rate. A hardened surface offers safety from that.

1.3.6 Ageing

Aging is the heat treatment process that is used to improve the yield strength of the malleable metals. This technique produces uniformly dispersed grain structure inside the material and alter its properties. Aging simply increases the temperature to medium degrees and cooling can be controlled or uncontrolled as per requirement.

For some materials, aging is carried out at room temperature and for others process of artificially aging is used in which cooling temperature is controlled and mostly carried out inside the furnace.

1.3.7 Stress relieving

Stress relieving is mostly carried out after machining of the parts to remove internal stresses that are generated through cold working. Material is heated at a temperature slightly below its lower critical point and then cooled at slow rate to keep it uniform. some processes that require stress relieving are machining, forming, straightening, and rolling.

1.4 Furnaces

Heat treatment (also known as Heat treating) extensively refers to a collection of thermal tactics in Manufacturing industries and metalworking used to deliberately adjust the physical, microstructural, and sometimes chemical properties of the element (usually a metal). The alterations of these chemical and physical properties are achieved using the process of hardening or softening a metal by heating and cooling at extreme temperatures. Heat treating takes place at temperatures above 550°C and up to 1,200°C for steel. Desired workpiece chemical and physical properties are achieved using different heat treatment processes like annealing, case hardening, tempering, normalizing, precipitation, strengthening, and quenching. To carry out these processes and achieve desired properties thermal processing furnaces are used to incorporate all these heat treatment techniques.

1.4.1 Vacuum Furnace

Heat treatment in vacuum furnaces is specified with the aid of using unique situations regarding the furnace design with temperature control and the level of vacuum during the process of heat treatment. The furnace design typically relies upon the load size, the temperature and the pressure attained, and the cooling medium.

The major elements of a vacuum furnace are:

- Vessel/Container

- Pumping system
- Heating chamber
- Cooling chamber

Vacuum furnace Containers may be classified into hot and cold wall designs. Mostly hot wall furnaces do not have any water-cooling shells or have a retort into which the work is loaded this is normally ceramic or metal depending on the temperature. The heating gadget is usually positioned outside the retort and includes a resistive heating system or an induction coil. Limitations of this retort-kind of the furnace are the restrained dimensions of the heating zones and the restrained range of temperature, normally constrained to a maximum of 1100°C. The vacuum container is cooled with the aid of using a cooling medium (normally water) in the cold-wall furnace and is kept near atmospheric temperature when the high-temperature treatment is carried out.

The capabilities of cold wall furnace in comparison with cold wall furnace are:

- The temperature range is higher up to 1650°C.
- The heat loss is lower, and less energy is released into the environment.
- The heating rate and cooling rate are faster.
- Temperature is more uniform.

The creation of the pumps relies upon the subsequent factors:

- The volume of the container.
- The vessel's surface area and the internal structure of the furnace.
- Release of the load and associated fixturing.
- Time required for pressure removal to normal.

It is vital to observe that the pump keeps the vacuum pressure without any malfunctioning due to the outgassing of loaded parts. Pumping structures are normally divided into subsystems, high vacuum generation pumps and roughing pumps. A single pump can be used for the entire process as per requirement. These pumps are divided into two categories, mechanical and diffusion pumps. There are different kinds of vacuum pumps to generate higher vacuum pressure such as ejectors, cryo-pumps, ion pumps, turbo-molecular pumps, etc. After the heat treatment in a vacuum furnace, the shiny surface of the material is maintained during the cooling. Now a day, many clean gases can be found for cooling with very fewer impurity levels. Nitrogen is mostly used cooling medium because it is less expensive and comparatively safe. Helium is likewise utilized in structures and recycling is important.

1.4.2 Air Furnace (Box Type)

Box type air furnace is also known as batch furnace. These furnaces can process a and large variety of parts, components and tend to contain large, heavy workloads that require long process time. In a batch, the loaded components are commonly stationary which helps in maintaining the equilibrium conditions inside the furnace. Quenching is the most used process in box type air furnace. Heating can be through electric source of through hot gas. When electric coils are used for heating, fans inside the furnace are used to rotate the air inside the furnace which also help to maintain the uniform temperature inside the furnace.

1.5 Cryogenic treatment

Cryogenic treatment is a unique form of heat-treating method that includes cooling the heat-treated metal to temperatures of -196°C.[1] Cryogenic treatment after heat treatment of metals allows their molecules to be introduced to "cryogenic stillness" to enhance their wear characteristics. When steels are cooled to cryogenic temperatures after heat treatment, retained austenite is converted to martensite, finishing the HT process. chipping or cracking can cause if retained austenite is not converted to martensite because it is brittle. Retained austenite can be the nucleation point for metallic fatigue. Many heat-treated parts, mainly for crucial usages like carburized gears or bearings, need deep cryogenic of the elements, and tempering is carried out after that to convert retained austenite into tempered martensite.[2] Cryogenic treatment is done by slowly cooling components in a controlled liquid nitrogen bath or a freezer for shallow cryogenic treatment, retaining the components till equilibrium is achieved with the liquid nitrogen temperature. Nitrogen liquifies at -196°C. Cryogenically treated components are commonly held for one hour per inch of their thickness. Then slowly heated to room temperature. Tempering was carried out after that. Classification of cryogenic treatment is as follow:

Shallow cryogenic treatment (SCT)

When temperature of −40°C to -140°C is applied (dry ice temperature).

Deep cryogenic treatment (DCT)

When temperatures close to liquid nitrogen are reached (−196°C)

There is lots of misunderstanding among the various researchers to specify the cryogenic treatment temperature limits. Many follow this temperature regime.

1. 0°C to -80°C temperature regime for Shallow cryogenic treatment.

2. -80°C to -196°C temperature regime for deep cryogenic treatment.[3][4][5]

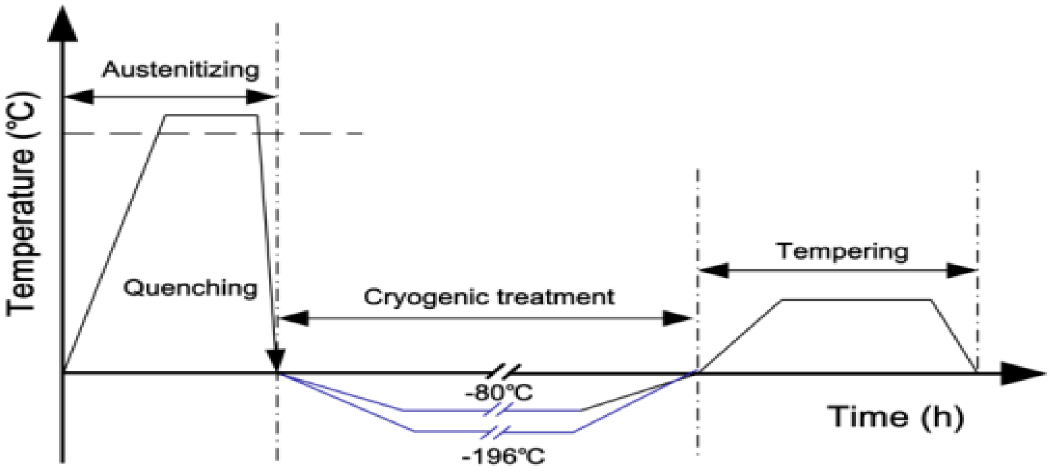


Figure 1: Process diagram of cryogenic heat treatment

For quite some time, material engineers, scientists, and metallurgists are trying to improve the hardness, fatigue, and toughness of metals via way of means of purposely growing compressive residual stresses on the steel surfaces. These are the stresses that remain in the materials even if the original reason for the stresses has been removed. The essential reasons for residual stresses are

1. Failures that took place because of stress corrosion, corrosion fatigue, or embrittlement of hydrogen.
2. Assessment of the ongoing serviceability of parts.
3. Distortion happens in the course of processing a part.
4. In-service or storage distortion

Residual stresses arise from force or thermal gradients or both. These stresses arise during diverse procedures together with shot peening, grinding, welding, straightening, phase transformations, and high-temperature treatment. The improvement of a tensile residual stress on material surfaces especially steel will result in fatigue failure. It has been proven that residual strain performs a crucial role during the design field of the component. It leads to fatigue resistance as well as develops dimensional stability in the component. It also improves the resistance towards cracking because of stress corrosion. These stresses may be labeled into the following types: micro stresses and macro stresses. Macro stresses stay homogenous over a massive quantity of grains, and equilibrium of forces is applied over a massive quantity of crystals. The other stress stays homogenous inside one grain, and with adjoining grains forces are assumed to be in equilibrium. The third type of internal force remains in equilibrium over the crystalline defects these forces are homogenous. micro stresses are formed due to the latter two stresses. According to Scientists, the shallow and deep cryogenic processes supplement techniques to the traditional heat treatment of metals. It may be implemented to develop the properties of metal parts. For shallow cryogenic treatment, metal is processed at -80°C and for deep cryogenic treatment, the material is processed at -196°C. Recently, cryogenic strategies were advanced and are widely implemented in heat treatment industries to enhance the physical properties, corrosion, and other properties of metal components.[6][7][8]

1.6 Aim of Research Work

To investigate the effect on structural properties of the material.

To investigate the effect on the grain structure of the material.

To investigate which furnace gives better results with shallow cryogenic treatment.

1.7 Area of Application

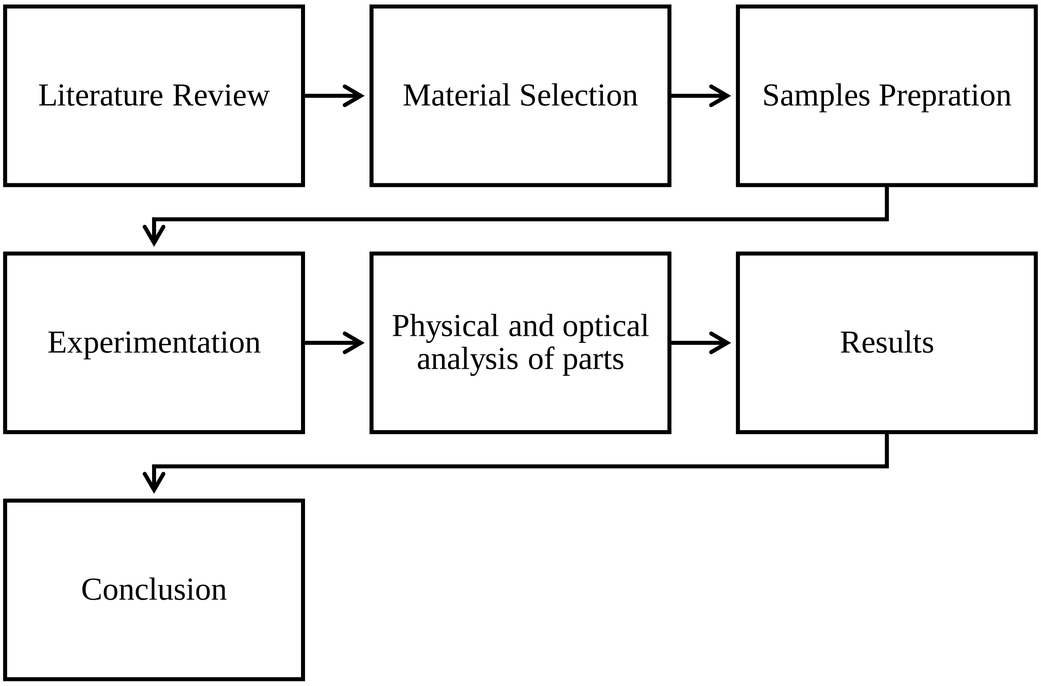
Aircraft small structural parts

Aircraft engine parts

Automotive Industry

Shafts and pistons

1.8 Research Methodology



1.9 Thesis Layout

Chapter 1: This chapter is the introductory to provide information related to heat treatment process and cryogenic process and information about the material used in experimentation. Purpose of research along with its application and research methodology in also mentioned in this chapter.

Chapter 2: discusses about the previous work that is already carried out on cryogenic heat treatment and the results achieved through those research works.

Chapter 3: describes about methodology adopted for this research work, how the experiments are performed, and equipment used to carry out experimentation.

Chapter 4: describes about results and observations obtained through experimentation about physical and optical analysis of the material.

Chapter 5: discusses about the results and conclusions and some future recommendation.

CHAPTER 2: LITERATURE REVIEW

Now a days lot of research is going on in materials industry because of the increased market competition researchers are trying to find new way to improve the overall performance materials as per their applications. Automobile market is growing with time and products with increased performance are more preferred by customers. To increase the life and performance of aircraft components, automobiles, shafts, and other industrial machines research efforts are ongoing since last few decades to improve the materials properties and cryogenic treatment is one of those efforts. Addition of shallow and deep cryogenic treatment along with heat treatment helps in improving the physical as well as microstructural properties of the materials.

In 2007 A. Bensely et al. performed an experimental study to investigate the effect of cryogenic treatment on case carburized steel-815M17 and checked its tensile behavior. 815M17 steel is used to manufacture crown wheel and pinion of heavy vehicles that faces lot of stress during operation. Samples were prepared as pe ASTM E8M and studied under the effect of shallow and deep cryogenic treatment heat treatment was carried out in conventional furnace. For the experimentation test pieces were divided into four categories. Group 1 was remained as it is for original material properties, conventional heat treatment was performed on Group 2, shallow cryogenic treatment was performed on group 3 and deep cryogenic treatment was performed on Group 4. Hardening was performed at 832°C for 1 hour. Shallow cryogenic was carried out at -80°C for 5 hours and deep cryogenic treatment was carried out at -196°C for 24 hours. Tempering temperature was 150°C. It was found that tensile strength was reduces in comparison with conventional heat treatment. For shallow cryogenic treatment (SCT) it is reduced by a factor of 1.5% and for deep cryogenic treatment (DCT) 9.34%, respectively. By observing through SEM more microcracks and micro-voids were found in SCT compared to DCT [9].

In 2008 A. Bensely et al. performed an experimental study to investigate the effect of cryogenic treatment on case carburized En 353 steel and checked distribution of residual stress in the material. X-ray diffraction technique was used to study the stresses on the surface of the material. SCT was carried out at -80°C and deep cryogenic treatment was carried out at -180°C. Experiments were carried out in conventional furnace aided by shallow cryogenic treatment and deep cryogenic treatment. For the experimentation test pieces were divided into 5 categories. After quenching process one test sample was separated without tempering and for remaining test pieces shallow and deep cryogenic treatment was carried out with and without tempering process. It was found that tempering is necessary after SCT and DCT otherwise cracks will generate on the surface of the material that can significantly reduce the life of the material. Tempering performed after shallow cryogenic treatment give the best fatigue results compared to conventional treatment and DCT. In DCT with tempering more carbides ratio was found which means wear resistance was improved as per previous studies. Compressive stresses were found maximum after DCT, and tempering helped in reducing those stresses to a great extent.[10]

In 2009 A. Akhbarizadeh et al. carried out a study on wear behavior of D6 tool steel. For this purpose, shallow cryogenic treatment was performed at -63°C and deep cryogenic treatment was performed at -185°C. wear tests were carried our using the load of 120N and 180N at three different velocities 0.05m/s, 0.1m/s and 0.2m/s. It was found the wear resistance was increased as the retained austenite is decreased. More improvement in wear resistance and hardness was observed in deep cryogenic treatment compared to shallow cryogenic treatment. As a result, carbide distribution was homogenized. More wear resistance and hardness were observed for the sample that were shallow cryogenic treated for 40 hours than 20 hours.[11]

In 2011 D. Senthilkumar et al. figured out the way of behaving of profound cryogenic treatment and shallow cryogenic treatment of 4140 steel remaining state pressure. A bar of

20mm was procured for the material and optical discharge spectroscopy is utilized to get its creation. Regular intensity therapy was done at 875°C socking time was 60 minutes, and the parts were extinguished in the oil. After the extinguishing system parts were cryogenically treated. Shallow cryogenic treatment was done at - 80°C for 5 hours and profound cryogenic treatment was completed at - 196°C for 24 hours. Treating temperature was 200°C for 60 minutes. X-beam diffraction technique was utilized to take advantage of the way that when a metal is feeling the squeeze, applied, or staying, the ensuing flexible strains prompt the atomic planes in the metallic valuable gem development to change hole between them. In this assessment, X-pillar diffraction assessments were finished on X-stress 3000 diffractometer (Stresstech Oy/Finland) to sort out the remaining large-scale anxieties and miniature burdens in the material. The justification for the ongoing assessment is to figure out material remaining pressure after Conventional intensity therapy, Shallow cryogenic intensity therapy, and Deep cryogenic intensity therapy conditions. The waiting anxieties can be requested into three sorts considering the length scale. These weights stay homogenous over a huge number of grains and balance powers are normal over such a large number of precious stones. Here uniform strain occurs over huge distances. Macro stresses are immovably related with normally noticeable plans, for instance, grain limit district, grain shape, grain size, breaks, porosity, and at least a couple components. The data was gained at bar points in the scope of -45° and 45° and the lingering stresses were found by using the Chi-procedure which was acquired by utilizing the old style sin2 technique. It was observed that equivalent readings of compressive large scale burdens were gotten in the extinguished and further Shallow cryogenic treatment and more anxieties were seen after profound cryogenic treatment. The decline in temperature diminishes the grid absconds and works on the thermodynamic soundness of the martensite, which drives carbon and alloying parts to neighborhood flaws. At low temperature it was observed that more martensite was shaped when the examples were not tempered, and higher pace of stresses were found in this state when contrasted and ordinary intensity therapy and shallow cryogenic therapy. Carbide precipitations in tempered SCT and DCT helped in decreasing the burdens from the material. It was additionally observed that greatest anxieties were created after DCT as more martensite was shaped from austenite. There was no unmistakable impact on the hardness and strength of this material was tracked down after shallow and profound cryogenic treatment.[12]

In the year 2012 Foad Farhani et al. performed the experimental study on 1.2542 tool steel to investigate the effect of DCT on its mechanical properties. Three units of specimens had been investigated: 2 units of untreated test pieces, for analyzing the impact of a few hardening

parameters at the steel properties, and a 3rd set along with cryogenic treated specimens. Soaking times were kept steady at temperature of -196°C and tempering temperature was 200°C. Different cryogenic treatment cycles had been applied with the aid of using various holding time (24, 36 and 48 hours) and tempering duration (60mins, 120mins and 180mins). To make sure most efficient remedy conditions, time gaps among numerous remedy steps had been kept to smallest. The sample pieces had been divided into 3 sets. Test specimens had been given codes for easy identification, all through and after the experiments. First set of test pieces is conventionally heat treated at 900°C and quenched using oil as its quenching medium after that tempering was carried out at 45°C. Second sample was heat treated at austenitizing temperature of 900°C and for quenching purpose water was used. Tempering was carried out at 200°C. For third set of test pieces water quenching was carried out after heat treatment at 900°C and tempering was carried out at 200°C between the process of tempering and quenching cryogenic treatment was carried out at -196°C and differ holding times were given to it. After carrying out experimentations successfully a notable difference in hardness and tensile strength was observed 9 to 12 percent increase in the hardness of cryogenically treated specimens was observed and tensile strength improved by 32 to 36 percent in comparison with standard value. Ductility was also improved in DCT. The discovered enhancements withinside the properties of those test pieces are because of the existence of a microstructure, containing extraordinarily small round tungsten and chrome carbide particles, which were dispersed homogeneously all over the metallic matrix with the perfect interface and excessive populace density. It was observed that by increasing the tempering time and soaking time more hardness can be achieved in case of DCT.[13]
In 2013 D. Candane et al. study the effect of cryogenic treatment on wear characteristics of AISI M35 steel. As cryogenic treatment is known for improving their characteristic of materials. Experiments were performed on high-speed steel AISI M35. Experiments were performed using both shallow cryogenic treatment and deep cryogenic treatment. After heat treatment of steel at 1200°C austenitizing temperature. Shallow cryogenic treatment was carried out at -84°C for eight hours and temperature of -195°C was provided for deep cryogenic treatment for 24 hours. After cryogenic treatment double tempering was performed at 200°C. Test were carried out for retained austenite presence. Equipment used for this study was XRD analyzer, scanning electron microscope and pin on disk wear tester to check the wear characteristics. Hardness and toughness were also tested. The amount of restraint austenite dropped after shallow cryogenic treatment as well as after deep cryogenic treatment

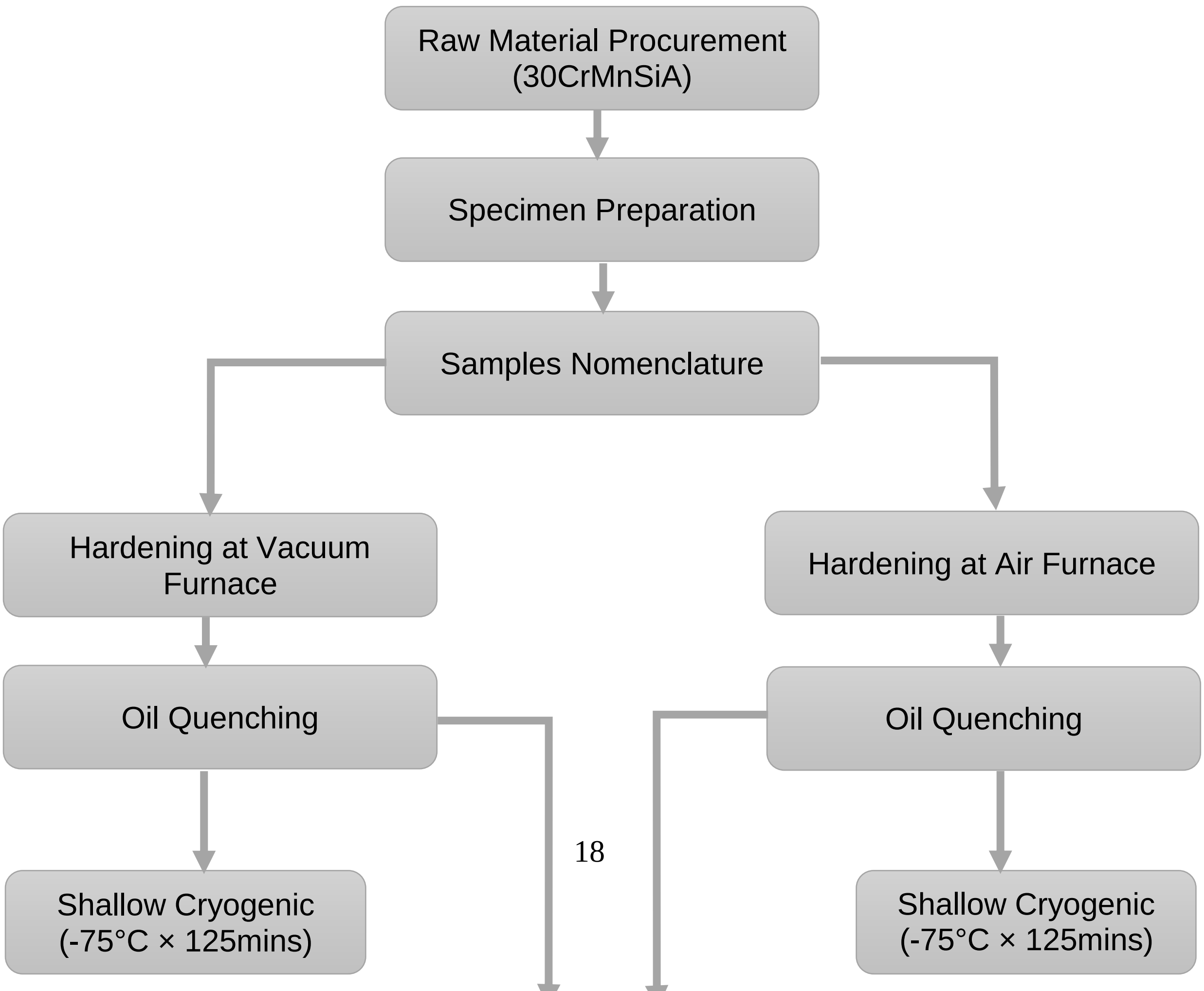
tempering helped in fine precipitation of carbides and martensite softening minor improvement in hardness was also observed.[14]

In 2014 G. Prieto et al. performed cryogenic intensity therapy on AISI 420 tempered steel and attempted to work on its mechanical properties as well as microstructure. This has this examination was done to find out the microstructural changes and the effect of cryogenic cures on influence sturdiness and hardness in AISI 420 tempered steel. X-beam diffraction (XRD) was utilized for segment assessment and portrayal, while carbide degree division, length and sythesis evaluation were estimated with the guide of utilizing the use of examining electron microscopy (SEM-EDX) and Energy Dispersive Spectrometry (EDS). Hardness was checked utilizing Vickers hardness technique and the effect sturdiness was determined via Charpy's V-score try. Crack surfaces have been examined with the guide of utilizing SEM to evaluate the break miniature - systems. In this view, it's been tentatively settled that cryogenic cures favor the precipitation of little carbides, which furthermore framed a more noteworthy homogeneous size circulation. It became found that this microstructural trademark is responsible for further developing these materials mechanical properties. It was found that cryogenic treatment might be executed to a low carbon hardened steel so one can create improved results in microstructural and actual properties. Accordingly, DCT worked on how much strain in the martensite express, the consequence of which is the creation of more modest auxiliary carbides with an additional uniform dissemination. The volume part of held austenite in the wake of treating gave no striking change, in any event, for routinely heat-treated test pieces. 2 hours of dousing time was adequate to accomplish improvement of 5% and 10% in hardness and strength during an effect test. No massive distinction was seen in hardness at a freezing temperature in correlation with traditional intensity therapy of the material. In this way, profound cryogenic temperatures were required. Direct submersion of AISI 420 tempered steel in fluid nitrogen can harm the material despite the fact that they can support a quicker cooling rate than device prepares.[4]

In 2019 Jian Zhu et al. played out a trial concentrate on H13 pass on steel for the improvement in its malleability and strength utilizing pre-treating. Held austenite plays out a vital situation in combination metal to accomplish unnecessary strength and further developed pliability. In this work, the pre-treating strategy was utilized to make a more homogeneous microstructure of H13 metal. improvement in the mechanical properties was seen with the elasticity of 1921 MPa, yield strength of 1534 MPa, Impact sturdiness expanded by 13.8 J· cm-2, tractable prolongation ∼11.8%, and hardness of 53.2 HRC was accomplished. The elasticity and hardness of the examples were kept up with over 83% when kept up with for 50 hours at 550°C, which have been exceptional than the rigidity (1569 MPa) and hardness (48.3 HRC) of tests managed the guide of utilizing customary 600°C treating treatment. It was seen that the treated martensite assisted in working on rigidity with extreme thickness disengagement and scattered carbides in H13 metal. It was guessed that the pre-treating strategy might be done extensively in various die metal for cutting edge execution. This trial utilized Baogang electric controlled heater H13 material. Right away, the examples were isothermally spheroidize warmed to 900°C for 90 minutes, then, at that point, cooled to 740°C for 3 hours and cooled under 450°C at under 50°C each hour withinside the heater, in the end cooled to room temperature. For the extinguishing system, the examples had been warmed to and saved at 1030°C for 30 min after which oil-extinguished to room temperature. The extinguished examples went through the ensuing pre-endlessly treating, the boundaries had been created. The boundaries for treating were expressed as the traditional HT for H13 device steel: the temperature for treating was 600°C and the ideal opportunity for treating was 30 minutes. As bites the dust were utilized at temperatures as much as 550°C, to dissect the transporter in general execution of tests managed through method of method for the pre-treating way, this analysis had researched the warm soundness of examples managed through method of method for the pre-treating and examples managed through method of method for customary treating way through method of method for warming to 550°C and dousing time was 50 hours. The typical pre-treating process was performed at 640°C for 10 min + 600°C for 30 min, and the regular treating was performed at 600°C for 2 h. It was seen that the pre-treating strategy becomes advanced to upgrade the actual properties of H13 apparatus steel in a powerful manner. Treated with the guide of utilizing pre-treating at 640°C for 10 min after which treating at 600°C for 30 min, the H13 metallic got progressed exhibitions with the tractable and yield qualities of ∼1921 MPa and ∼1534 MPa, sturdiness was around 13.8 J· cm-2 and ductile lengthening was 11.8 percent. It very well might be accepted that the high strength of H13 device steel become ascribed to the tempered state martensite with unnecessary separation and the Cr and High appraised carbides scattered withinside the substrate. While over the top malleability was because of the ferrite and strip held austenite, which completed TRIP influence withinside the twisting of H13 metallic. The material strength was decreased while the pre-treating temperature surpasses 700°C or the pre-treating time was over 20 minutes. It could be characterized with the addition of held austenite and carbides in the material, with the improvement of austenite settled factors C, Si, Mo, and V. At the point when the examples had been held at 550°C for fifty hours, the elasticity stayed 83% of the first (~1593 MPa), the yield strength stayed 89% of the first (~1359 MPa) and the hardness was roughly 48.5 HRC that 92% of the first, even as the tractable lengthening advanced to around 13% (116% of the first).[15]

A.keen et al. in 1982 improved the wear resistance of tool steel by 25% with shallow cryogenic temperature at -84°C. hardness of the metal remained same. Wear properties improved by increasing the soaking time [17]. Cryogenic treatment of tool steel after tempering at -140°C give negative results. But favorable results were observed in case of deep cryogenic treatment [18]. A. Bensely study in 2007 on steel-815M17 shows the decrease of tensile strength of 1.5% and 9.34% in both SCT and DCT compared to conventional heat treatment [9][19]. A 6% decrease in retained austenite was observed in case of shallow cryogenic treatment of 80CrMo12 5 cold work tool steel. An increase and homogeneous distribution of carbide precipitation was found and holding time is effective in DCT [20]. In 2011 M.koneshlou performed SCT on AISI H13 tool steel at -73°C and retained austenite is transferred into martensite[21]. In 2016 carbide precipitation of Vanadis tool steel was increased with SCT. Ductility of the tool steel also improved. Wear rate and small amount of impact toughness is also improved. DCT gave better results compared to SCT [22]. Tempering is important after cryogenic treatment as it improves the mechanical properties of the metal[23]. Binzhouli et al. in 2018 performed shallow cryogenic treatment on 20CrNi2MoV steel and observed more refined microstructure, decrease in retained austenite and increase in carbon precipitation. Study also shows that wear characteristics were improved by 17%. Fine carbide precipitation helps in improving wear characteristics of the steel[24][25].

CHAPTER 3: METHODOLOGY



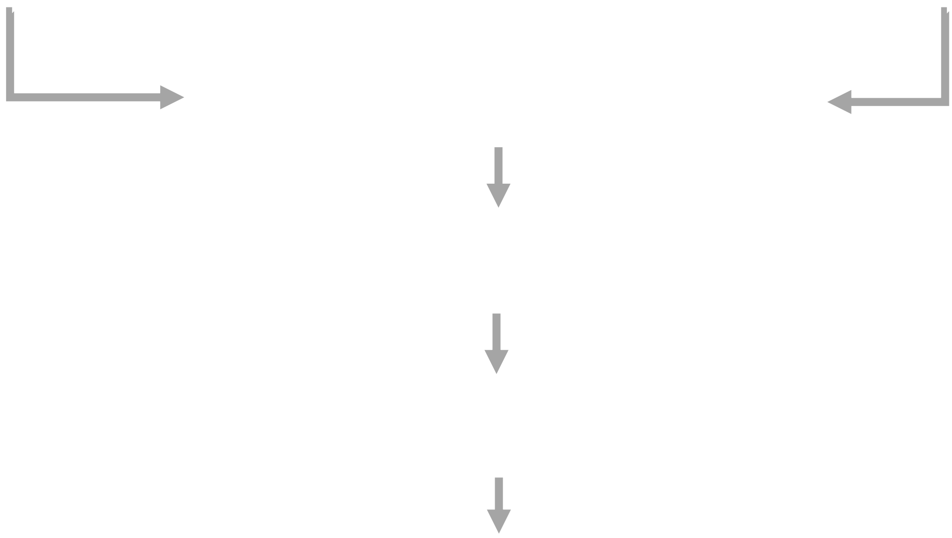


Figure 2: Experimentation Methodology Process Chart

For the experimental purpose above mentioned flow chart is followed. This flow chart gives the view of the major steps followed to perform the experimentation. 30CrMnSiA was procured in the form of a rod. The diameter of the road was 30mm and spectroscopy analysis were carried out using Direct Reading Spectrum Analyzer OBLS, QSG 750 to determine the chemical composition of the material and it’s shown in Table 1.

Table 1: Chemical Composition of 30CrMnSiA

30CrMnSiA chemical composition							
C	Si	Mn	P	S	Cr	Ni	Mo
0.292	0.969	0.86	0.0091	0.0009	0.928	0.021	0.006

Four sets of Specimens were prepared from it, tensile samples were prepared as per ASTM standard E8/E8M. For the better identification and simplicity of work code names were given to the specimens as shown in table 2. First set of specimens (BF-HT) were heated in the box type air furnace at 900°C and oil quenched after that tempering was performed at 505°C and the tempering time was 2 hours. Both graphs were shown in Figure *4* and Figure *5*. Second set of specimens (BF-SCT) was heat treated in Box type air furnace at 900°C for 40 mins and oil quenched shown in Figure *4*. Tempering was carried out at 505°C for 2 hours shown in Figure *5*. Between the process of quenching and tempering shallow cryogenic treatment was carried out at -76°C for 125 minutes shown in Figure *6*. For the third set of specimens (VF-HT) heat treatment was carried out at vacuum furnace at 900°C for 1 hour and oil quenched as shown in Figure *7*. After that tempering was carried out at 505°C for 2 hours as shown in Figure *5*. For the last set of Specimens (VF-SCT) first heat treatment was carried out at vacuum furnace at 900°C for 1 hour and oil quenched shown in Figure *7* after that shallow cryogenic treatment was carried out at -76°C for 125mins and heated at room temperature after cryogenic treatment shown in Figure *6*. After that tempering of 30CrMnSiA was carried out at 505°C for 2 hours shown in Figure *5*. Soaking time of two hours was selected for shallow cryogenic treatment because in case of SCT soaking time of two hours is enough and increasing the time is not much effective.[4]

Figure 3: Specimen ready for HT

Table 2: Description of specimens and applied HT processes

Identification	Description	Temperature and Time
BF-CHT	Box type air furnace heat treatment	900°C for 40mins
BF-SCT	Box type air furnace and shallow cryogenic treatment	900°C for 40mins × -76°C for 125mins
VF-HT	Vacuum furnace heat treatment	900°C for 1 h
VF-SCT	Vacuum furnace and shallow cryogenic treatment	900°C for 1 h × -76°C for 125mins

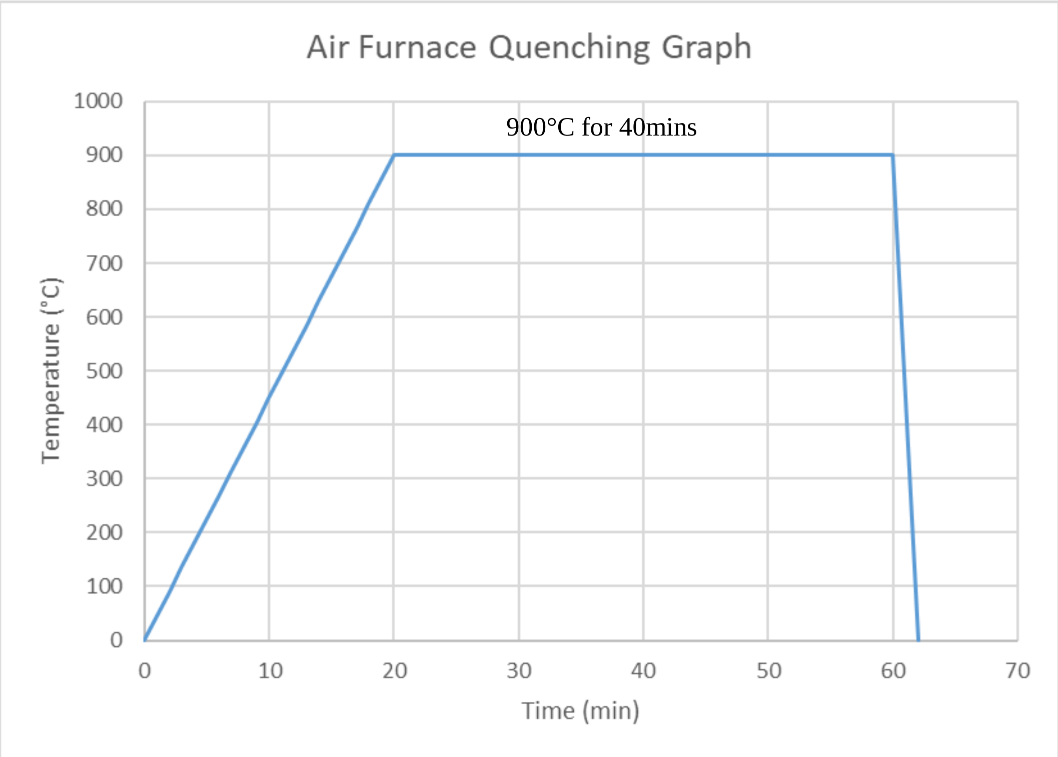


Figure 4: Air furnace Quenching Process Graph

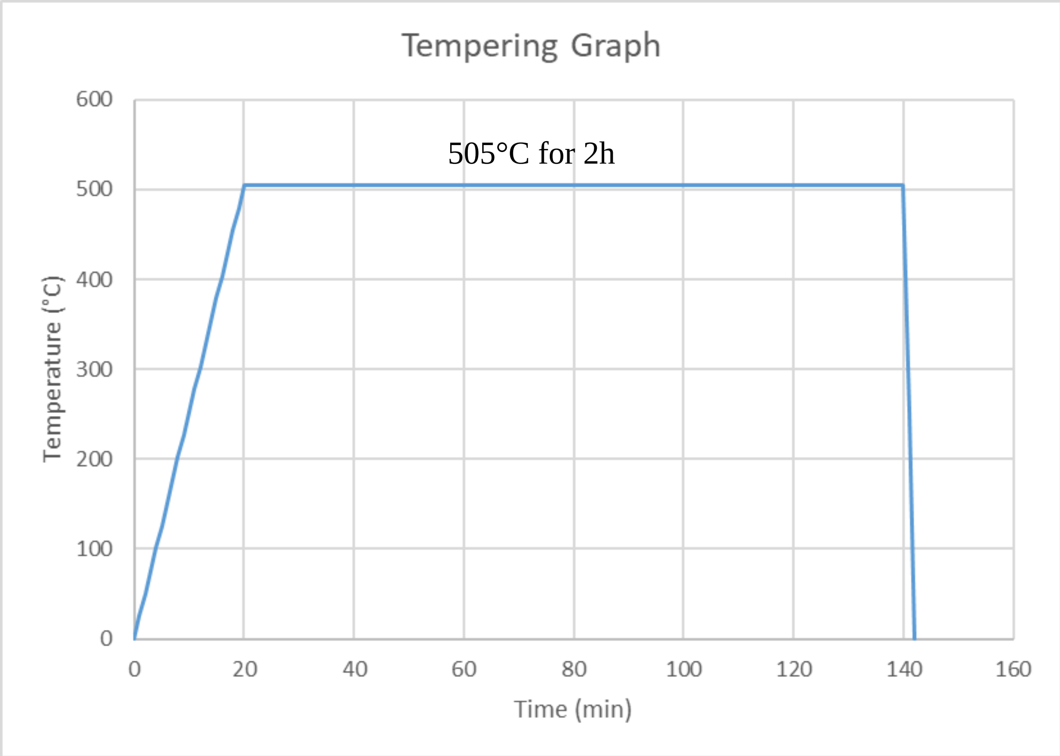


Figure 5: Tempering Process Graph

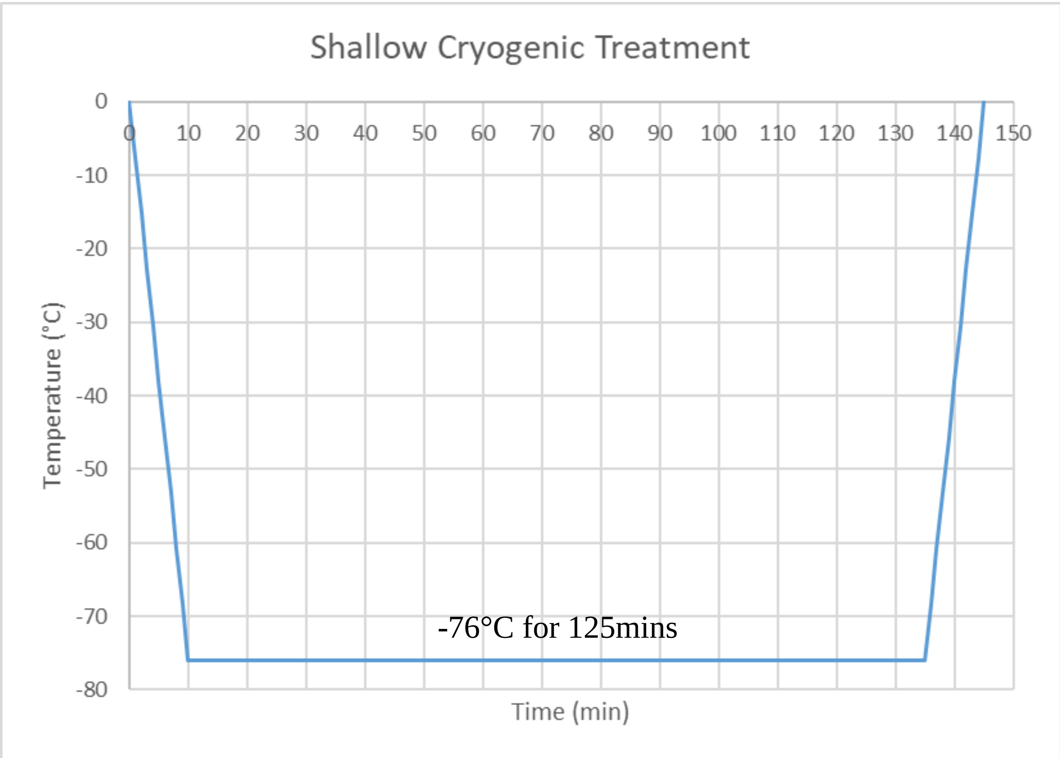


Figure 6: Shallow Cryogenic Treatment Graph

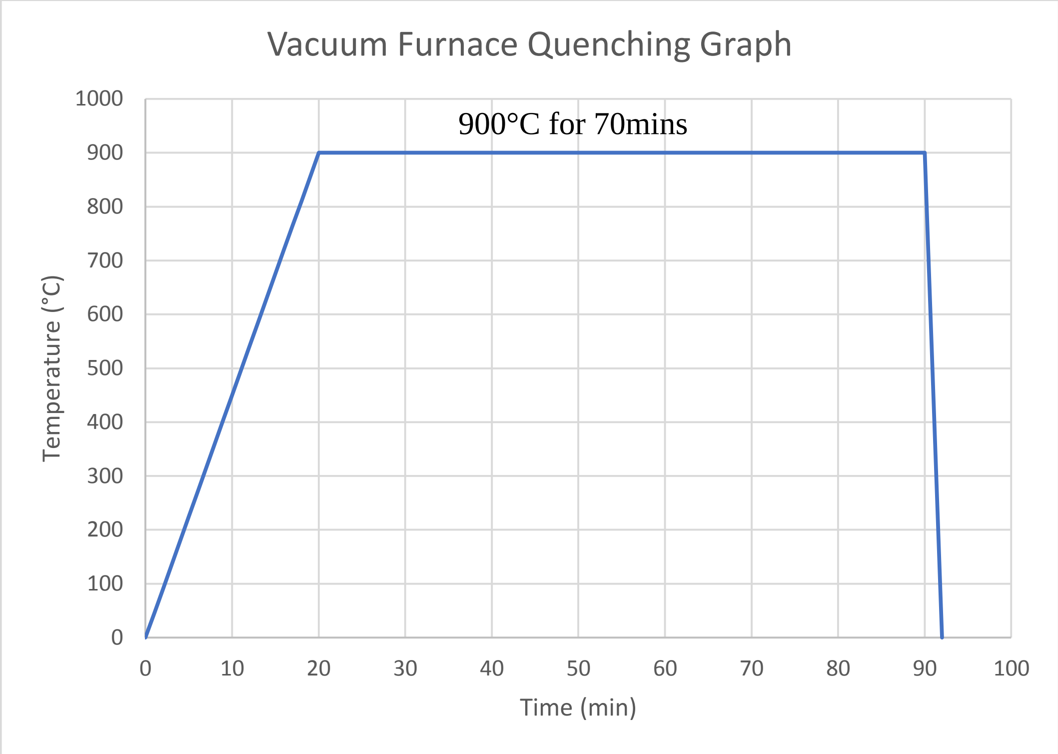


Figure 7: Vacuum Furnace Quenching Graph

Figure 8: Box Type Air Furnace

Figure 9: Vacuum Furnace

Figure 10: Low Temperature Chamber

Combine cycles of all four processes are also given below Figure *11*. represents the complete process of first specimens (BF-CHT) in the form of graph. Figure *12*. represents the complete process of second specimens (BF-SCT) in the form of graph. Figure *13*. represents the complete process of third specimens (VF-HT) in the form of graph. Figure *14*. represents the complete process of forth specimens (VF-SCT) in the form of graph.

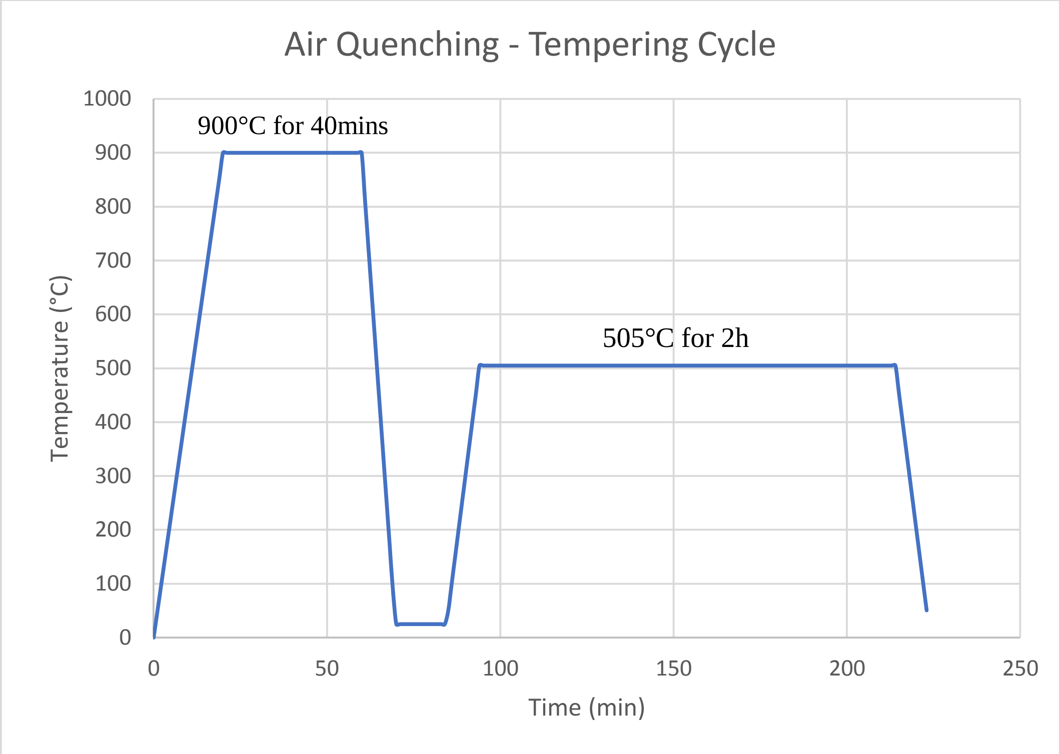


Figure 11: BF-CHT Process Graph

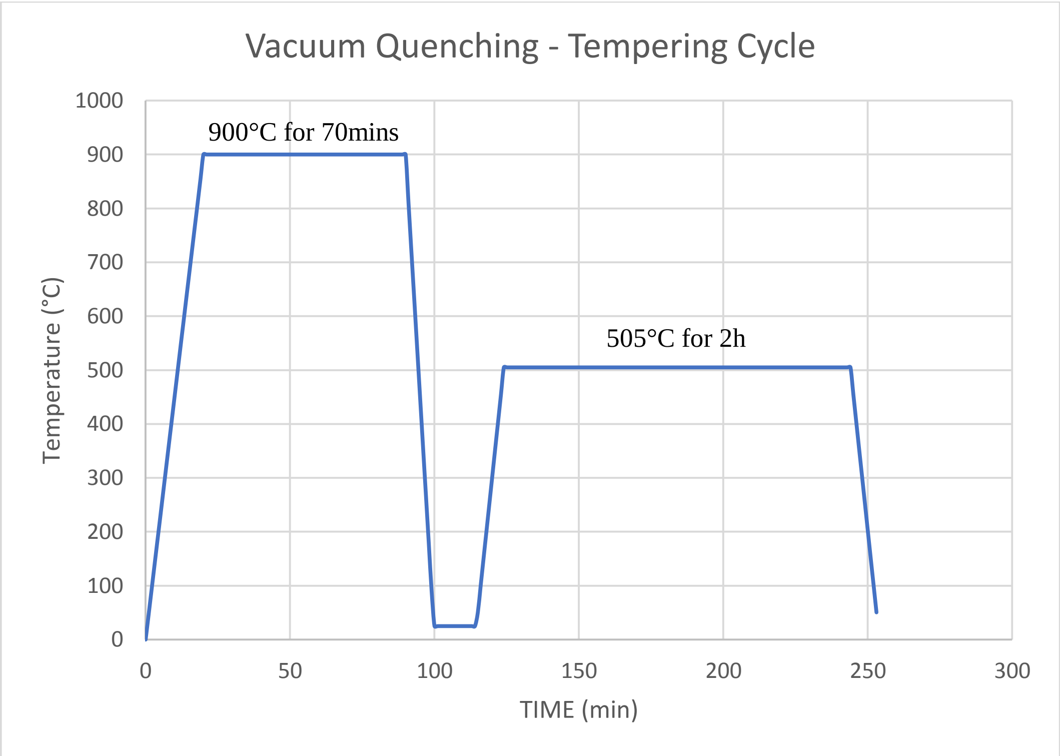


Figure 12: VF-HT Process Graph

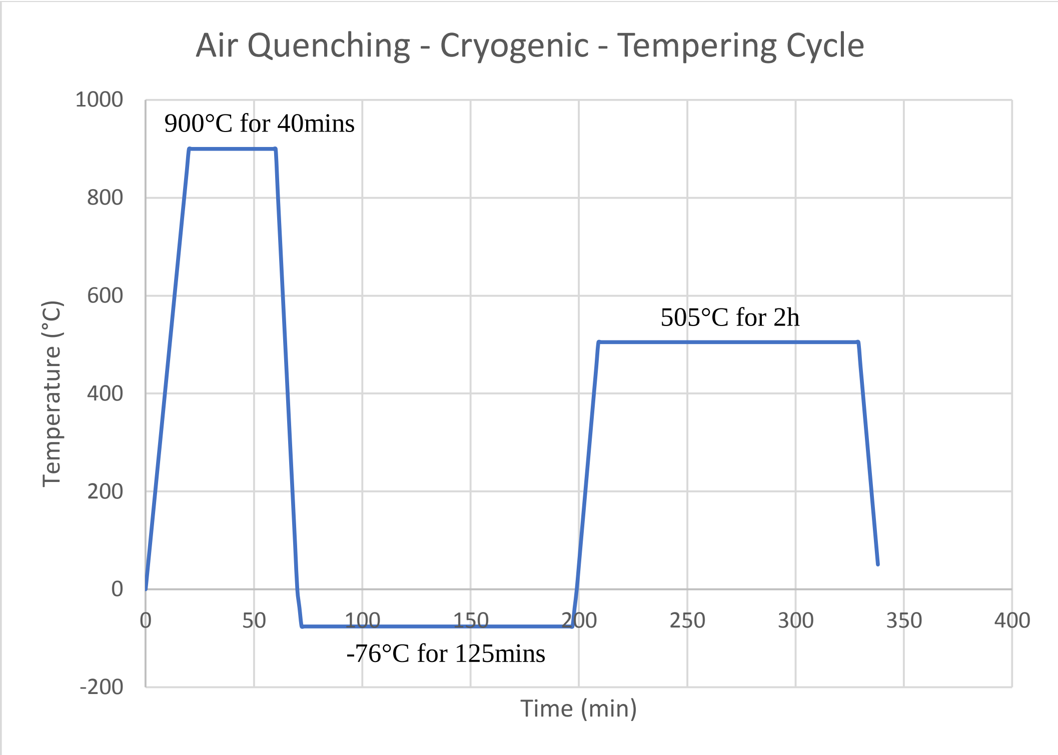


Figure 13: BF-SCT Process Graph

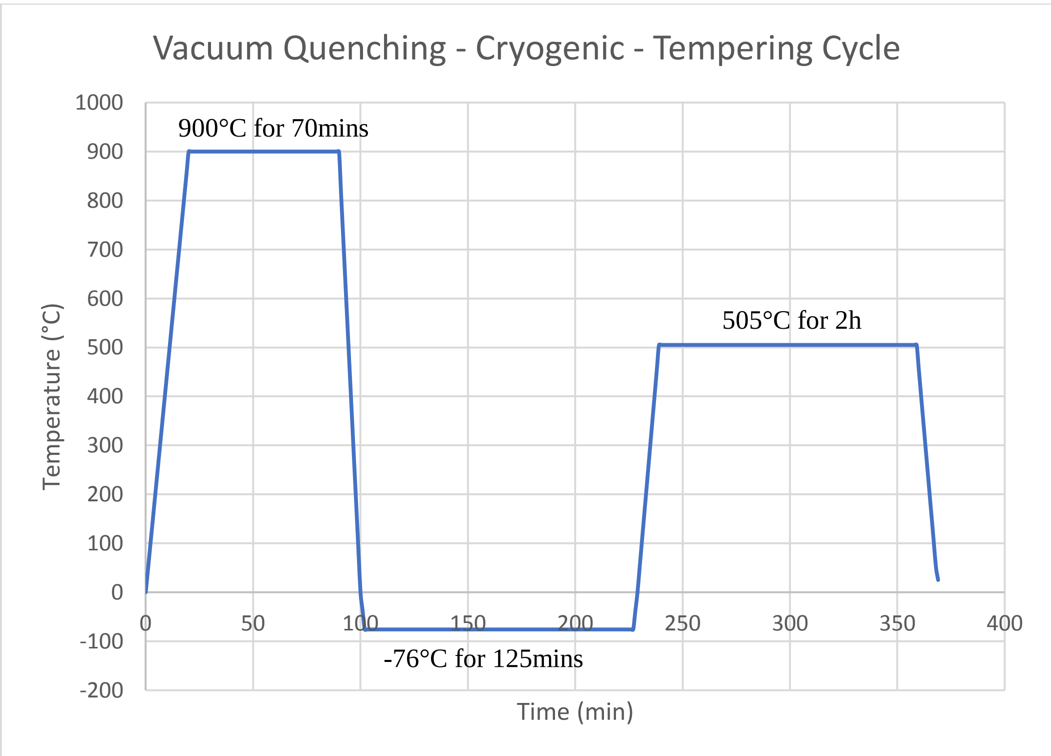


Figure 14: VF-SCT Process Graph

CHAPTER 4: RESULTS AND DISCUSSION

Two types of testing have been carried out on the specimens one is physical or mechanical testing other one is optical microscopic testing.

4.1 Hardness

The purpose of these tests is to find out which furnace gives better results with shallow cryogenic treatment when it is performed on the specimens between quenching and tempering. Brinell hardness tester HBD-62-5AP is used to find out the hardness (HRC) of the specimens. To find out the hardness six tests were carried out on one set of the specimen and average hardness value was calculated as shown in Table 3.

Table 3: Hardness values of the specimens

Sample	S1	S2	S3	S4	S5	S6	Average
BF-CHT	35.5	35.8	35.5	35.5	36.7	36.7	35.95
BF-SCT	35.8	35.7	36.2	36	35.8	35.7	35.87
VF-HT	38.5	38.7	39.1	38.4	38.7	39.5	38.82
VF-SCT	41	40.9	41	39.4	39.9	39.3	40.25

For the heat-treated parts in conventional box type air furnace (BF-CHT) hardness value is 35.95 HRC and with the addition on shallow cryogenic treatment it is found that harness value for Box type air furnace (BF-SCT) is 35.87 HRC. No difference in hardness was observed in case of Box type air furnace with or without shallow cryogenic treatment. For the heat-treated parts in vacuum furnace (VF-HT) hardness value is 38.82 HRC and with the addition on shallow cryogenic treatment it is found that harness value for Vacuum furnace (BF-SCT) is 40.25 HRC. An increase of 3.55% in hardness was observed in case of VF-SCT compared to VF-HT.

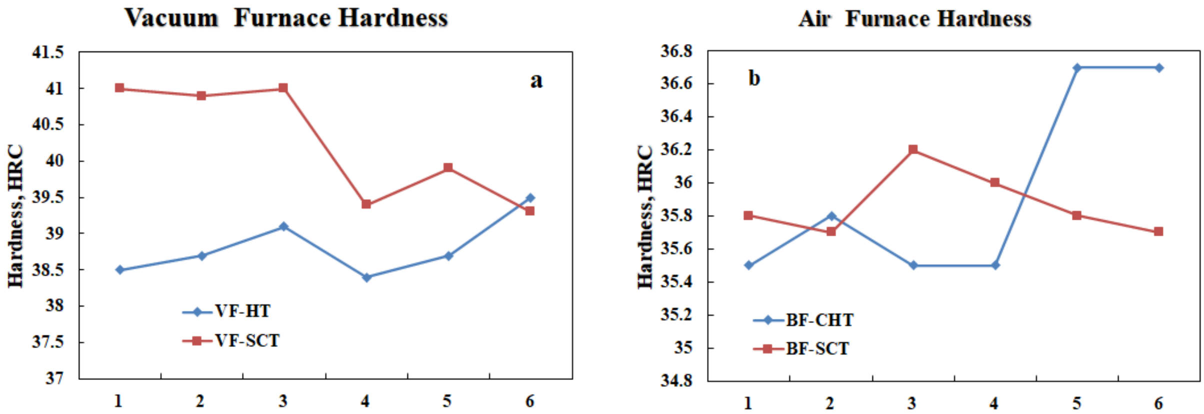


Figure 15: Hardness of the specimens with different furnaces (a) Vacuum furnace specimens and (b) Air furnace specimens

4.2 Impact Toughness

Electronic pendulum impact testing machine CBD-300 was used for the impact toughness testing of 30CrMnSiA specimens. The specimens were prepared according to the ASTM E23-12c, and the specimen dimensions are shown in figure 16. It has been observed that after low temperature treatment the toughness of the material improved. 5% increase in case of BF-SCT compared to BF-CHT was observed and 7.2% increase in toughness was observed in VF-SCT specimens compared to VF-HT specimens results are shown in Table 4.

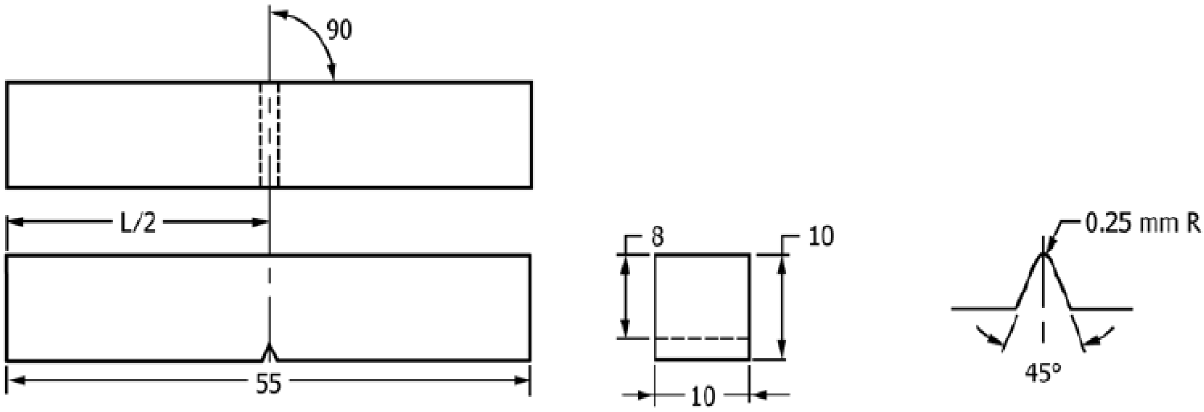


Figure 16: Impact toughness specimen section

Table 4: Impact toughness values of the specimens

Sample	**S1**	**S2**	**S3**	**Average**
BF-CHT	42.78	41.40	41.63	41.94
BF-SCT	44.07	43.97	44.44	44.16
VF-HT	41.27	42.44	42.16	41.96
VF-SCT	44.78	45.40	45.45	45.21

4.3 Tensile Strength

WAW-100B 100kN Computer Control Electro-Hydraulic Servo Universal Testing Machine was used for the tensile testing of the specimens. These specimens were prepared according to the ASTM standard E8/E8M. Gauge length of the specimen was 24mm, Diameter was 6mm, Radius if fillet was 6mm, length of reduced section was 30mm mentioned in Table 5. Figure *17* shows the tensile specimens' sections and Figure *18* is the image taken during tensile testing.

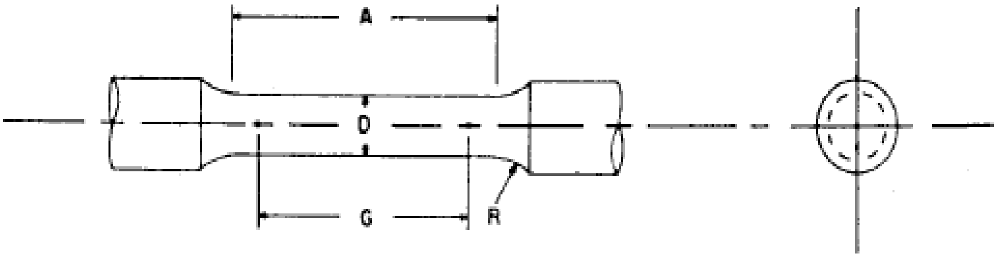


Figure 17: Tensile Specimens Sections

Table 5: Tensile Specimens Dimensions

Dimensions of Tensile Specimens	
Sections	Dimensions (mm)
G—Gauge length	24.0 ± 0.1
D—Diameter	6.0 ± 0.1
R—Radius of fillet	6
A—Length of reduced section	30

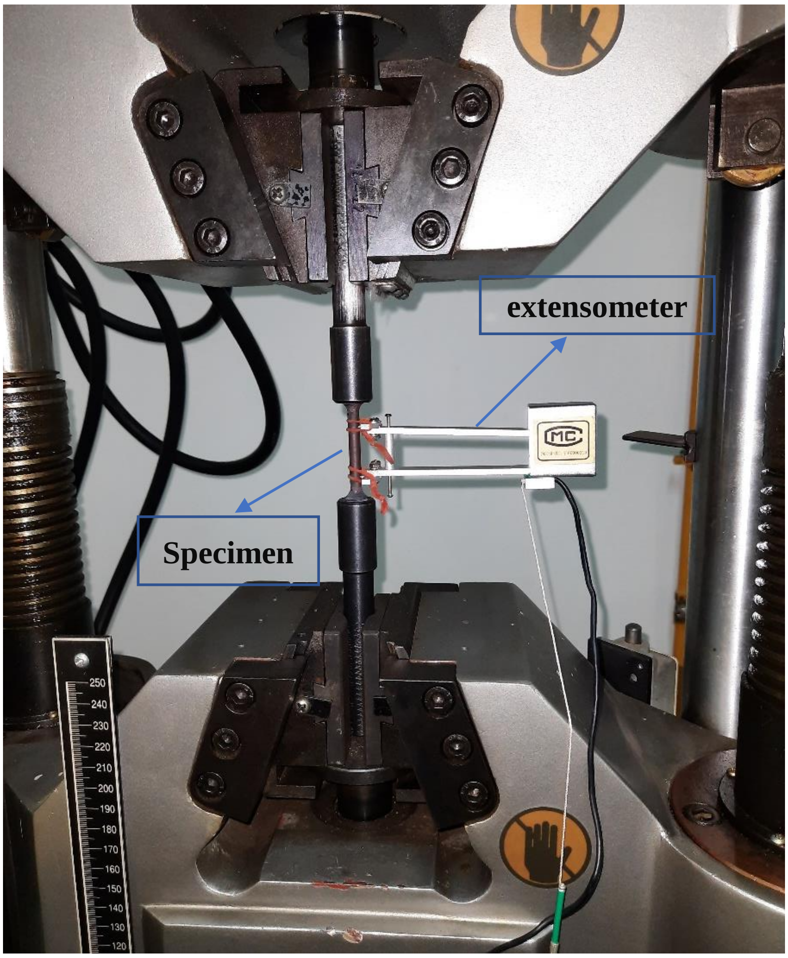


Figure 18: Tensile Testing

Following results have been observed from tensile testing of 30CrMnSiA shown in Table 6.

Table 6: Tensile Test Results

Specimen Name	Gauge Length (mm)	Gauge Area Dia (mm)	Tensile Strength (KN)	Length Before (mm)	Length After (mm)	Percentage Elongation
BF-CHT	30	6	33.56	30	34.7	15.7%
BF-SCT	30	6	30.07	30	35.10	17%
VF-HT	30	6	35.80	30	34.3	14.3%
VF-SCT	30	6	37.59	30	33.90	13%

Tensile strength of the of the specimens that were shallow cryogenically treated after air furnace quenching (BF-SCT) found to have 11.6% less strength compared to the specimen that were heat treated in air furnace without shallow cryogenic treatment (BF-CHT). In case on vacuum furnace, it is observed that strength of the specimen which was vacuum heat treated with shallow cryogenic treatment (VF-SCT) have 5% more strength compared to the VF-HT specimens.

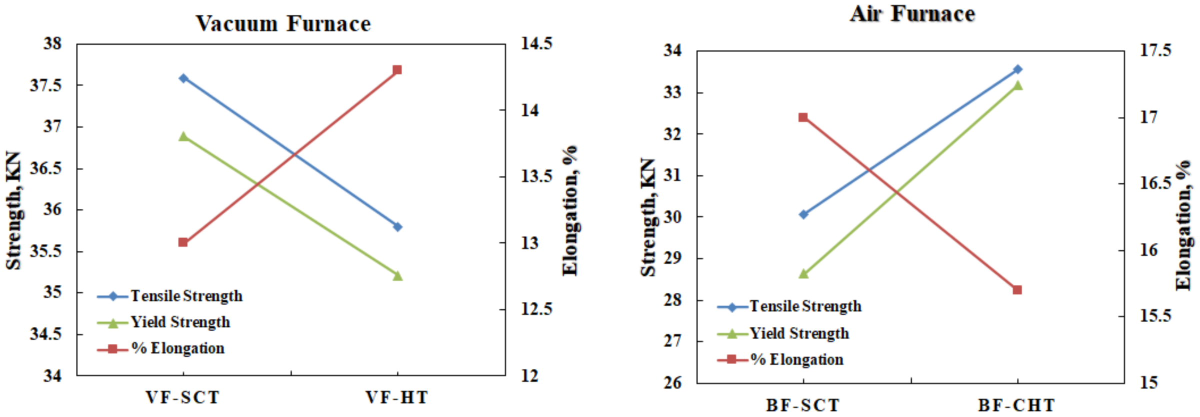


Figure 19: Tensile properties of material treated in different furnaces

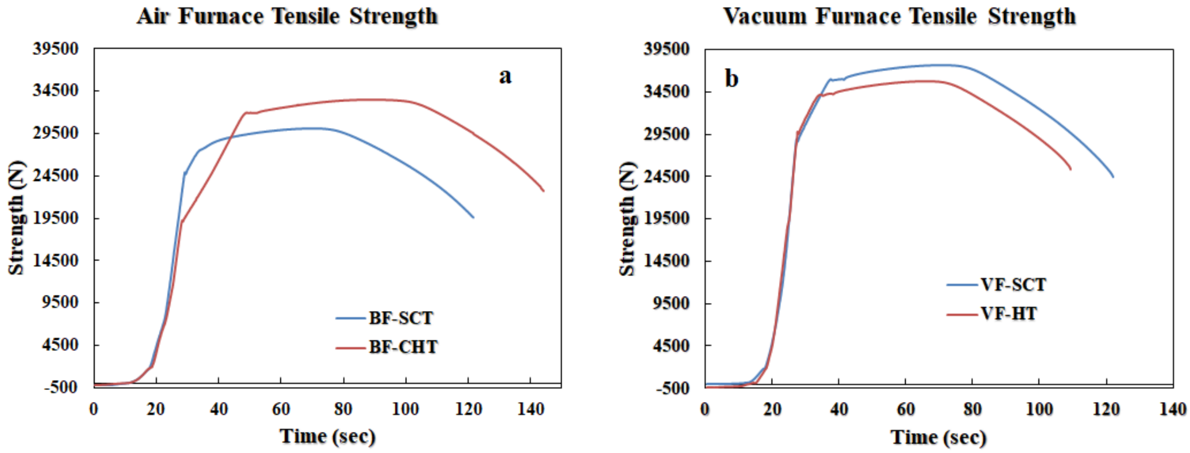


Figure 20:(a) Air furnace tensile results (b) Air furnace tensile results

4.4 Optical Testing

Small pieces of specimens were cut from the main specimens with the help of EDM wire cut then mounted in the mold for grinding and polishing. Specimens were polished very fine up to 6 microns. Number marking was made below the specimens to make sure they do not mix with each other. As 30CrMnSiA is low alloy low carbon steel 10% mixture of nitric acid (HNO3) in 90% mixture of methanol is prepared for the etching process. By dipping the specimens in this solution for 10 seconds grain boundaries and structure became visible. To observe and study the specimen’s structure an optical microscope OLYMPUS DSX1000 was used. Images were taken through microscope are shown in the figures below.

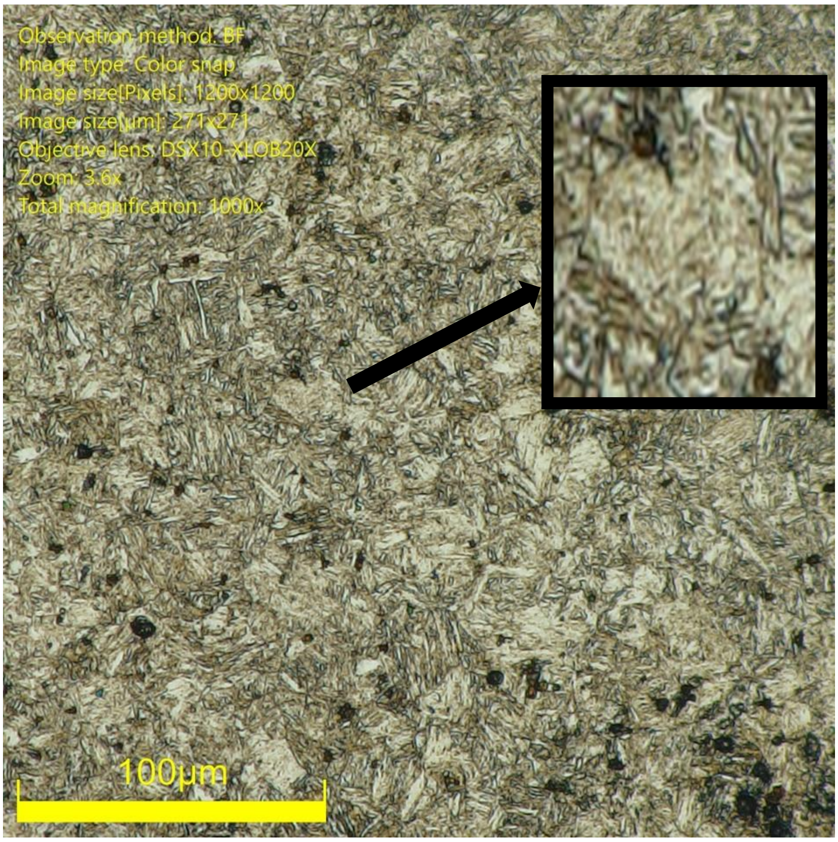


Figure 21: BF-SCT Grain Structure

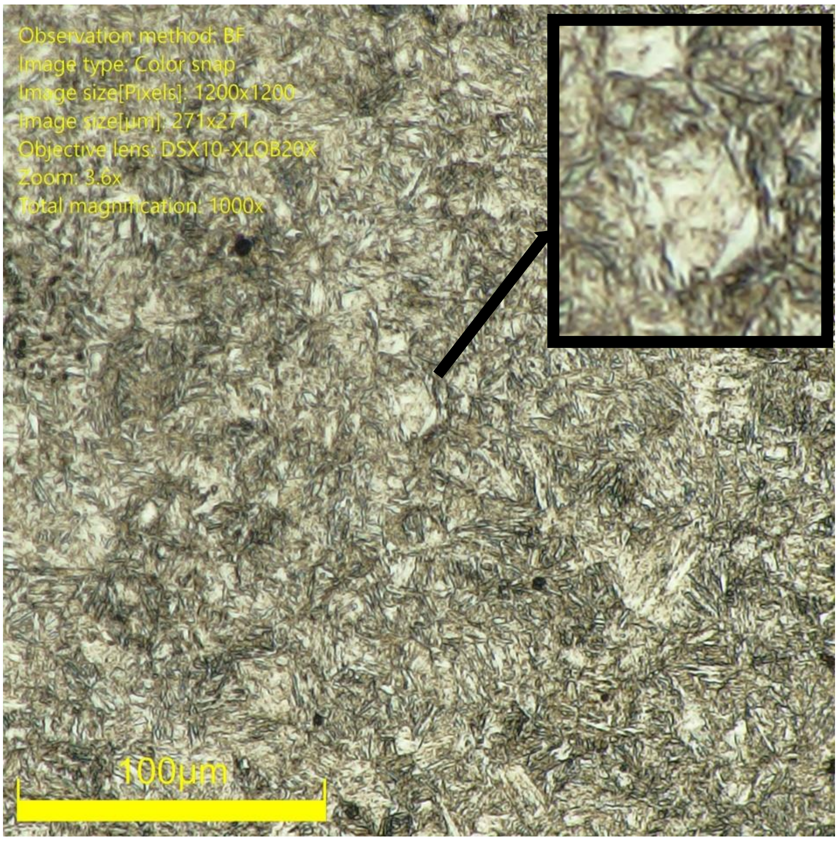


Figure 22: BF-CHT Grain Structure

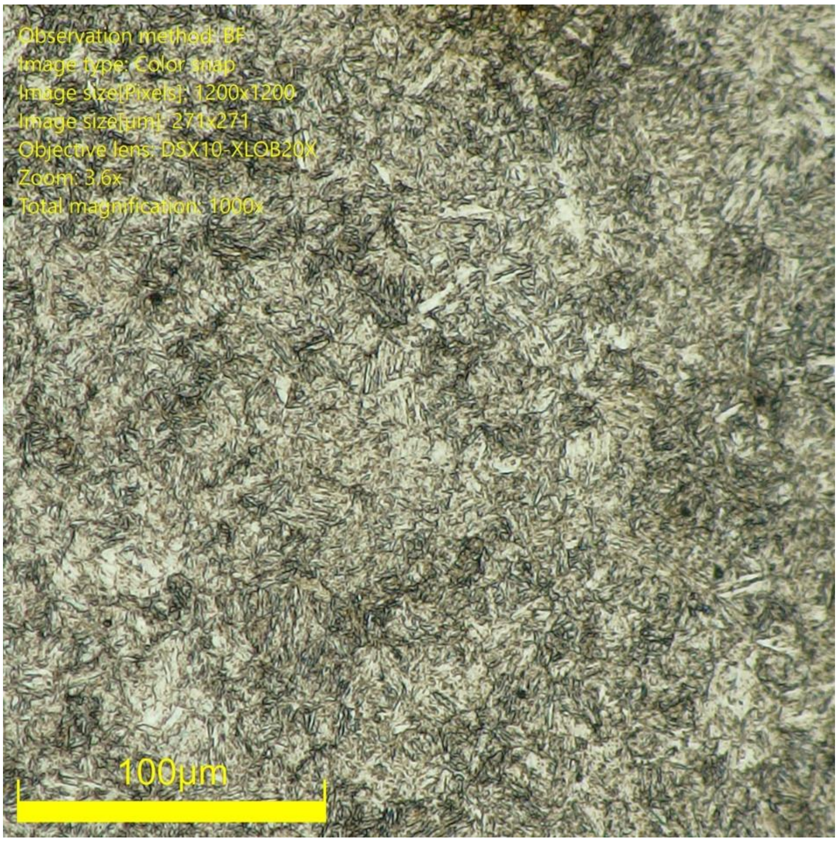


Figure 23: VF-SCT Grain Structure

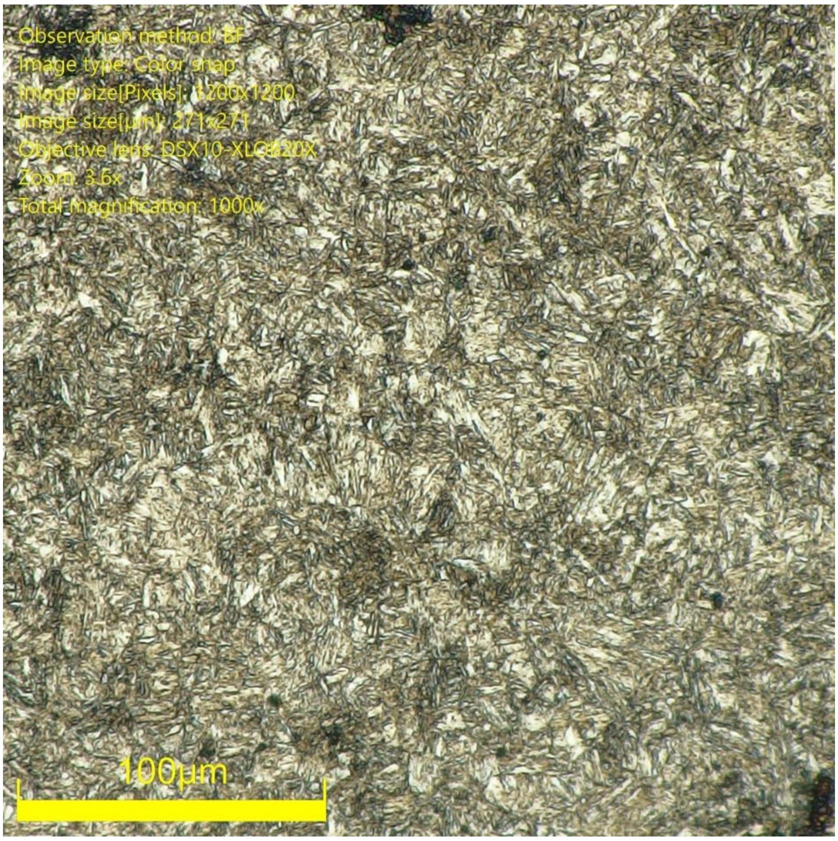


Figure 24: VF-HT Grain Structure

From the 30CrMnSiA structure images taken through microscope it is observed that more homogeneous tempered martensite structure is obtained in VF-SCT compared to VF-HT. In case of Box type air furnace better structural results are obtained in BF-SCT as the structure is more homogenous than BF-CHT. Also, fine carbide precipitation was observed in the shallow cryogenically treated specimens.

CHAPTER 5: CONCLUSIONS

In this study, low carbon alloy structural steel 30CrMnSiA was heat treated in box type air furnace and vacuum furnace. Four sets of specimens were prepared one set was heat treated in air furnace and then tempered second set in heat treated in air furnace and tempered but shallow cryogenic treatment was carried out between quenching and tempering process. Similarly, third and fourth sets of specimens were processed but in vacuum furnace instead of air furnace. Following results were concluded after experimentation.

1. Hardness of the specimens that were treated in vacuum furnace with shallow cryogenic treatment is increased by 3.55%. No difference in observed in case of steel processed in air furnace.
2. Toughness of the specimens after shallow cryogenic treatment increases by 5% in case of air furnace and 7.2% in case of vacuum furnace.
3. In case of heat treatment in vacuum furnace with shallow cryogenic treatment before tempering tensile strength of the material increased by 5%. While in case of heat treatment in box type air furnace with shallow cryogenic treatment before tempering process tensile strength is decreased by 11.6% and percentage elongation is increased by 7.64%.
4. After the optical analysis of grain structure an increase in carbide precipitations is observed in the BF-SCT specimen. Carbide precipitation help in improving the wear properties of the steel. Less retained austenite is observed in VF-SCT specimen.

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