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A Project Report
On

"EFFECT OF SOAKING TEMPERATURE ON MACHANICAL PROPERTIES (HARDNESS) OF MARTENSIC STEEL AISI 440C''

Submitted in partial fulfillment of the requirements as a part of the curriculum,

Bachelors of Engineering in Mechanical Engineering

Submitted by

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CERTIFICATE

Certified that the project work entitled "**EFFECT OF SOAKING TEMPERATURE ON MECHANICAL PROPERTIES (HARDNESS) OF MARTENSITIC STEEL AISI 440C"** is a bonafide work carried out by **Mr. AMAN CHANDRA R and Mr. KARTIKEY BHARDWAJ,** bonafide students of **CMR Institute of Technology** in partial fulfillment for of the requirements as a part of the curriculum, **Bachelors of Engineering in Mechanical Engineering,** of **Visvesvaraya Technological University, Belagavi** during the year **2019-20**. It is certified that all correction/suggestion indicated for Internal Assessment have been incorporated in the report deposited in the departmental library. The project report has been approved as it satisfies the academic requirements in respect of the project work prescribed for the bachelor of engineering degree.

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DECLARATION

We, students of Eighth Semester, B.E, Mechanical Engineering, CMR Institute of Technology, declare that the project work titled **"Effect of soaking temperature on mechanical properties (Hardness) of martensitic steel AISI 440C"** has been carried out by us and submitted in partial fulfillment of the course requirements for the award of degree in **Bachelor of Engineering in Mechanical Engineering** of **Visvesvaraya Technological University, Belagavi,** during the academic year 2019-2020.

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EFFECT OF SOAKING TEMPERATURE ON MECHANICAL PROPERTIES (HARDNESS) OF MARTENSITIC STAINLESS STEEL AISI 440C

ABSTRACT

To study the effect of soaking temperature on mechanical properties (hardness) of martensitic stainless steel AISI 440C and to examine the hardness variation and microstructure with the aim of supplying heat treatment guidelines to the user that will ensure a martensitic structure with minimal retained austenite, evenly dispersed carbide and a hardness between 58-60 HRC after quenching. Composition of AISI 440C was checked using X-ray fluorescence. Steel samples were soaked at temperatures of about 1020°C, 1050ºC, 1100ºC, 1150ºC and 1200ºC for constant time of 1 hour for each sample followed by quenching. Microhardness of samples from case to core was obtained using micro hardness tester. Macro hardness of the samples were also evaluated using Rockwell Hardness tester in C scale.

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CHAPTER 1

1.INTRODUCTION

The aim of the training is to learn about the various types of materials used in aircraft engine:

- By conducting failure analysis
- By performing composition analysis and
- By concluding the cause of failure of components.

1.1. ABOUT THE ORGANIZATION

1.1.1. DRDO

Defence Research and Development Organisation (DRDO) was established in 1958 by amalgamating Defence Science Organisation and some of the technical development establishments. A separate Department of Defence Research and Development was formed in 1980 which now administers DRDO and its 50 laboratories/establishments. DDRO is currently directed by A.P.J. Abdul Kalam, who was previously director of the Defence Research and Development Laboratory (DRDL) responsible for India's missile development program.

The Department of Defence Research and Development formulates and executes programs of scientific research, design and development in the fields of relevance to national security leading to the induction of new weapons, platforms and other equipment's required by the Armed Forces. It also functions as the nodal agency for the execution of major development programs of relevance to Defence through integration of research, development, testing and production facilities with the national scientific institutions, public sector undertakings and other agencies. It functions under the control of Scientific Advisor to Raksha Mantri who is also Secretary, Defence Research and Development.

Research and development activities at DRDO cover important demarcated disciplines like aeronautics, rockets and missiles, electronics and instrumentation, combat vehicles, engineering, naval systems, armament technology including explosives research, terrain research, advanced computing, artificial intelligence, robotics, works study, systems analysis and life sciences including

high-altitude agriculture, physiology, food technology and nuclear medicine. In addition to undertaking research and

development activities, DRDO also assists the Services by rendering technical advice regarding formulation of requirements, evaluation of systems to be acquired, fire and explosive safety and mathematical and statistical analysis of operational problems.

DRDO has made significant achievements in its efforts to meet the requirements of the three Services. The notable developmental successes include flight simulators for aircrafts, 68mm reusable rocket pod, brake parachute for fighter aircrafts, mini remotely piloted vehicle, light field gun, new family of light weight small arms systems, charge line mine clearing vehicle for safe passage of vehicles in battlefield and illuminating ammunitions for enhancing night fighting capabilities. Cluster weapon systems for fighter aircraft, naval mines, next generation bombs for high speed aircraft and low-level bombing, mountain gun, 130 mm SP gun, low-level tracking radar Indra-I and II for Army and Air Force, light field artillery radar, battlefield surveillance radar, secondary surveillance radar have also been achieved. Bridge layer-tank Kartik, military bridging systems capable of withstanding tank load, advanced ship sonar systems, advanced son buoys, naval decoys. naval simulators, torpedo launchers, advanced materials and composites for military applications and parallel processing computer for aerodynamic computations have also been developed.

Several high-technology projects are in various stages of design and development. The main battle tank Arjun, incorporating state-of-art tank technologies with superior fire power, high mobility and excellent protection has been developed. A limited number of tanks are being produced as pre-production series which are in final stages of evaluation by the Army.

A light combat aircraft which would be lighter than any other combat aircraft and would incorporate modern design concepts and several state-of-the art technologies, is under full scale engineering development.

The Integrated Guided Missile Development program is in progress. The program comprises of five missile systems: Prithvi - surface-to-surface tactical battlefield missile, Akash medium range surface-to-air - missile systems, Trishul -a short range surface-to-air missile and Nag - third generation anti-

tank missile, and Agni, an intermediate range ballistic missile.

DRDO offers specialized training at its two premier training institutions called Institute of Armament Technology, Pune and Defence Institute of Work Study, Mussoorie. The courses at these institutions have been evolved primarily to meet the needs of DRDO, Department of Defence Production.

1.1.2. GTRE

Gas Turbine Research Centre (GTRC) took its birth at No.4 BRD Air Force Station, Kanpur in 1959 with 8 Engineers/Scientists and about 20 Technicians. First indigenously developed centrifugal type gas turbine engine of 1000 kg thrust was tested in 1961. GTRC was moved to Bangalore, brought under the banner of DRDO, and was re-named as Gas Turbine Research Establishment (GTRE) in November 1961.

Gas Turbine Research Establishment is one of the pioneering Research and Development Organizations under the Ministry of Defence, Government of India. The main charter of the Establishment is to design and develop gas-turbine engines for military applications, besides carrying out advanced research work in the area of gas-turbine sub-systems. In addition, the Establishment is responsible for establishing the requisite computational, prototype manufacturing and test facilities for components and full-scale engine development. The Establishment has a strong team of over 845 personnel drawn from science and engineering which includes aeronautics, mechanical, electronics, computer science, materials science, applied mathematics, etc. and support services.

Principal achievements of Gas Turbine Research Establishment include:

- Design and development of India's "first centrifugal type 10kN thrust engine" between 1959-61.
- Design and development of a "1700K reheat system" for the Orpheus 703 engine to boost its power. The redesigned system was certified in 1973.
- Successful upgrade of the reheat system of the Orpheus 703 to 2000K.
- Improvement of the Orpheus 703 engine by replacing "the front subsonic compressor stage"

with a "transonic compressor stage" to increase the "basic dry thrust" of the engine.

- Design and development of a "demonstrator" gas turbine engine—GTX 37-14U—for fighter aircraft. Performance trials commenced in 1977 and the "demonstrator phase" was completed in 1981. The GTX 37-14U was "configured" and "optimized" to build a "low by-pass ratio jet engine" for "multirole performance aircraft". This engine was dubbed GTX 37-14U B.

1.1.3. RECENT DEVELOPMENTS

Initial sanction for the development of GTX-35-VS was given in 1989 and christened as KAVERI engine in the first meeting of Aero Engine Development Board. Three full engines and two core engines were successfully tested to prove the concept.

With redesigned compressor, six full engines and one core engine were built and successfully tested at altitude conditions and the performance evaluated. The marine version of this engine for the Navy was conceived and the first prototype was successfully integrated and tested at Naval Dockyard, Visakhapatnam.

73 hours of Altitude Testing was conducted at Central Institute of Aviation Motors (CIAM), Russia in 2009-10 and the performance and operability of the engine were verified. 57 hours of Flight Tests were completed in IL-76 aircraft at Russia covering altitude up to 12 km and Mach No. 0.7 in 2010-11. About 3000 hours of testing completed at ground and altitude conditions as on date.

1.2 ABOUT THE MATERIAL

Martensitic stainless steels grades containing 11-17% Cr (mass %), with sufficient carbon (0.12-1.0 wt % C), has established itself as a competitive class of industrial materials owing to the unique combination of strength, toughness and moderate corrosion resistance.

Martensitic stainless-steel grades are relatively cheaper when compared to the austenitic stainless steel that dominates the world market. Currently, the uses of martensitic stainless-steel grades in

several industrial applications have increased tremendously. Some of its applications include gears, valves,

pumps, shafts, bearing, turbine parts and aerospace. Many of these applications are hidden to most of us which probably explains why martensitic stainless steels do not have a prominent public profile. It is good to remind ourselves that much of our modern world rests on martensitic stainless steels as it is quietly doing their job behind the scenes.

Martensitic stainless steels are commonly used in quenched and tempered condition. The heat treatment consists of annealing to obtain austenite and dissolve the carbides, followed by quenching to transform into martensite and subsequent tempering of martensitic structure to improve toughness and ductility. According to several authors, the microstructure of martensitic stainless steel consists of martensite, undissolved carbide as well as retained austenite and the amount of carbide in the as-quenched microstructure influences the properties of this material such as hardness, strength, toughness and wears.

440 C is a 400 series stainless steel, and is the highest carbon content from 400 stainless steel series. It is a bearing steel, and used in rolling contact stainless bearing, e.g. ball and roller bearing. It is also used to make knife blades. 440C can be oil quenched to achieve maximum hardness.

440C has the highest strength, hardness, and wear resistance of all the common place 440C -series stainless alloys with high carbon content and moderate corrosion resistance. It can be heat treated to achieve a high hardness due to the formation of martensitic and presence of carbon. The hardness obtained after heat treatment will be around 58-60 HRC. It is well known that hardness is an important factor in determining the cavitation erosion resistance of a material although there are exceptions.

CHAPTER 2

2.REVIEW OF RELATED LITERATURE

2.1. LITERATURE REVIEW

The purpose of heat treatment is to cause desired changes in the metallurgical structure and thus in the properties of metal parts. Heat treatment can affect the properties of most metals and alloys, but ferrous alloys, particularly steels, undergo the most dramatic increase in properties.

Soaking time plays an important role in changing the properties and microstructure of the material. Soaking time is the time at which the material is held on to a particular temperature for a certain amount of time. It has been found that ultrafast heating with short soaking time results in finer microstructure and better mechanical properties.

The effect of soaking time during ultrafast heat treatment of a low carbon steel on microstructure has been studied. It is shown that ultrafast heating combined with short soaking times results in improved macro-mechanical properties due to finer grain size and higher fraction of non-recrystallized ferrite, that has a higher nano hardness than recrystallized ferrite. Prolonged soaking times eliminate the advantages of the ultrafast heat treatment. This occurs because, even though a long soaking time promotes a higher volume fraction of martensite than a short one, it also induces substantial grain growth and complete recrystallization of the ferritic matrix. On the micro-scale, the ferritic grains show two different types of mechanical response. The recrystallized ferritic grains are prone to show pop-in events on the nanoindentation curves that are associated to dislocation nucleation events as a consequence of their low dislocation density, while non-recrystallized ferritic grains demonstrate a continuous response.

The short soaking time has proven to be effective on a 50% cold rolled low carbon steel. A thorough qualitative and quantitative microstructural characterization of the heat-treated samples is performed using a wide range of characterization techniques. It was found that the microstructure consists of

embedded martensite and retained austenite is formed after heat treatment. It is demonstrated that ultrafast heating generally results in finer microstructure compared to the conventional heating independently on the soaking time. There is a significant effect of the soaking time on the volume fraction of martensite of the ultrafast heated material. The effect of annealing soaking time on evolution

of microstructure and mechanical properties of D2 tool was investigated. As we all know annealing is the process of increasing the ductility of the material and reducing the hardness. Thus, annealing is performed on this material to improve the ductility of the material. Tools are made ductile as ductile materials are good at withstanding both tensile and compressive loads. The increase in soaking time during annealing process causes the morphology of carbides transform from irregular shape to nearly rounded shape and distributed uniformly in a pearlite matrix. The increased amount of carbide dissolution during annealing resulted in enhancement ductility and decrement of strength of D2 tool steel. The study indicates that desirable properties of D2 Steel could be obtained by judicious selection of annealing cycle.

$CaO\text{-}Fe_2O_3$ can be used as a simulant material to study the thermal phenomena and heat transfer of a nuclear reactor accident scenario. The study of thermophysical properties plays a crucial role in the prediction of actual phenomena that occurred in a nuclear severe accident.

This work devoted to analyzing the effect of soaking time on thermophysical properties of non-prototype material $CaO\text{-}Fe_2O_3$ (21C79F; $C = CaO$, $F = Fe_2O_3$; 21:79 by wt. %). A binary mixture powder of non-eutectic material $CaO\text{-}Fe_2O_3$ (21:79 by wt. % confirmed by phase diagram) in the form of cylindrical pellets is heat-treated at 1100 °C for one, two, and three hours of soaking time and further ground to powder form and finally, the heat-treated powder were analyzed through characterization techniques to evaluate the changes in thermal properties, crystalline structure, and morphology.

The results were also compared with the initial powder sample accordingly. The properties of material revealed that the material is thermally stable and it can be used in molten form to investigate the severe accident phenomena. The properties of $CaO\text{-}Fe_2O_3$ (21C79F) is possibly near to the properties of corium (a liquid mixture of UO_2 and steel) and also environment-friendly as well as for human beings.

Some steels do not respond to change in properties with respect to varying soaking time. Now we'll look at one such material that is, 15-5 PH steel which was manufactured using Selective Laser Melting process. With recent advancements in additive manufacturing, Selective Laser Melting (SLM) is emerging as an unconventional manufacturing process of exceptional flexibility capable to fabricating any complex part without requiring expensive fixtures, tooling, mould or any other additional auxiliary with a very short lead time from design conceptualization to fabrication.

Herein, micro-structural evolution and its correlation with various mechanical and corrosion properties of heat treated SLM 15-5 PH stainless steel are investigated to widen the application of SLM parts for industrial usages.

Results show, standard aging condition (H900) increases yield strength, hardness and corrosion resistance through the formation of fine spherical ε-Cu-rich precipitates (~15 nm), but makes the specimens brittle resulting in an increase in wear rate and a decrease in impact energy. Combined effects of coarsening of elliptical ε-Cu-rich precipitates and increase in reverted austenite phase in over ageing (H1150) make the specimen ductile having relatively low yield strength, hardness, wear rate but high impact energy. Solution annealing was found to reduce anisotropy in mechanical properties through the homogenization of microstructure.

Higher aging temperature and longer soaking time doesn't have significant impact on different mechanical properties but deteriorates the corrosion properties. Solution annealing before aging is recommended for the homogeneity in microstructure. These results can be used as a guideline to select proper post heat treatment for SLM 15-5 PH stainless steel to impart application oriented mechanical and corrosion properties.

Now discussing about the processes involved in complete transformation of retained austenite to martensite. AISI 440C stainless steel possesses a low M_f (martensite finish) temperature, which is far below room temperature. After sub-zero treatment in liquid nitrogen, the steel forms plate martensite with significant amounts of retained austenite.

Dilatometric experiments with microstructural observation were performed to investigate the tempered martensite and the decomposed retained austenite during multiple tempering treatments. The results indicate that a complete transformation of retained austenite can be more easily achieved by multiple tempering cycles than by a single long-time cycle.

65Si2MnWE steel shows variations in its microstructure and mechanical properties with varying soaking and tempering temperatures. The microstructure such as martensite and precipitations were carefully examined, and the tensile, impact and hardness tests were carried out to evaluate the mechanical properties.

The results show that the coarsening of martensitic occurs at high soaking temperature due to the facts of precipitation dissolution, dislocation reduction, and also the reduction of reserved austenite. Large amount of alloyed carbide precipitations distribute along grain boundaries after tempering, and these precipitations tried to dissolve into matrix at high tempering temperature. Moreover, with the increase of tempering temperature, the transformation and growth of carbide, and the recovery of martensite simultaneously take place. According to the analysis on mechanical properties, the optimal temperature range for soaking is determined as 850–900 °C, and the optimal tempering temperature should be in the range of 350–400 °C.

Now looking into the effect of austenitizing on the microstructure and hardness of AISI 420 with addition of 0.13% vanadium and 0.62% molybdenum were studied. Steel samples were austenitized at temperatures between 1000 and 1200°C, followed by oil quenching. The as-quenched microstructures were found to range from almost fully martensitic structures to martensite with up to 35% retained austenite after quenching, with varying amounts of carbides. Optical and scanning electron microscopy was used to characterize the microstructures, and X-ray diffraction was employed to identify the carbide present in the as-quenched structures and to quantify the retained austenite contents. Hardness tests were performed to determine the effect of heat treatment on mechanical properties. As-quenched hardness values ranged from 700 to 270 HV, depending on the amount of retained austenite.

2.2 PROBLEM STATEMENT

To study the effect of soaking temperature on mechanical properties (Hardness) of AISI 440C martensitic stainless-steel alloy.

CHAPTER 3

METHODOLOGY

Our study of effect of soaking time on hardness of AISI 440C were carried out in the following sequence: -

1. Heat treatment of samples was carried at 1020°C, 1050ºC, 1100ºC, 1150ºC and 1200ºC for constant soaking time of 1 hr for each sample. (Hardening)
2. The macro hardness of the sample was checked using Rockwell Hardness Tester.
3. Then the samples were polished to obtain a good surface finish which is a pre requisite for micro hardness testing.
4. At the end the hardness of samples from case to core was observed using micro hardness tester.

3.1. COMPOSITION ANALYSIS

The elemental composition of the material was analyzed pre and post treatment to observe change in composition of elements. In our experiment the elemental composition of the material was carried out

with energy dispersive x-ray spectroscopy.

3.1.1. ENERGY DISPERSIVE X-RAY FLUORESCENCE

Energy Dispersive X-Ray Spectrum (EDS or EDX) is a chemical microanalysis technique used in conjunction with scanning electron microscopy (SEM). The XRF detects x-rays emitted from the sample during bombardment by an electron beam to characterize the elemental composition of the analysed volume. When the sample is bombarded by the SEM's electron beam, electrons are ejected from the atoms comprising the sample's surface. The resulting electron vacancies are filled by electrons from a higher state, and an x-ray is emitted to balance the energy difference between the two electrons' states. The x-ray energy is characteristic of the element from which it was emitted.

Fig:3.1 X-Ray Fluorescence

The EDS x-ray detector measures the relative abundance of emitted x-rays versus their energy. The detector is typically lithium-drifted silicon, solid-state device. When an incident x-ray strikes the detector, it creates a charge pulse that is proportional to the energy of the x-ray. The charge pulse is converted to a voltage pulse (which remains proportional to the x-ray energy) by a charge-sensitive preamplifier. The signal is then sent to a multichannel analyser where the pulses are sorted by voltage. The energy, as determined from the voltage measurement for each incident x-ray is sent to a computer for display and further data evaluation. The spectrum of x-ray energy versus counts is evaluated to determine the elemental composition of the sampled volume.

Semi quantitative compositional analysis was carried out using this facility. The obtained composition value along with standard composition values is mentioned in table given below. The obtained composition values from Energy Dispersive X-Ray Spectrum analysis is in complete agreement with that of standard composition.

Standard composition of AISI440C

Element	C	Si	Mn	Cr	Mo	P	S	Fe

Composition (%)	0.95-1.2	1	1	16-18	0.75	0.02	0.01	Balance

Table 1: Standard Composition of AISI 440C

3.2. HEAT TREATMI

Heat treating is defined by the IFHTSE (International Federation for Heat Treating and Surface Engineering) as: 'a process in which the entire object, or a portion thereof, is intentionally submitted to thermal cycles and, if required, to chemical and additional physical actions, in order to achieve desired (change in the) structures and properties'.

Heat treatments are also used in the manufacture of many other materials, such as glass. Heat treatment involves the use of heating or chilling, normally to extreme temperatures, to achieve the desired result such as hardening or softening of a material. Heat treatment provides an efficient way to manipulate the properties of the metal by controlling the rate of diffusion and rate of cooling within the microstructure. Heat treating is often used to alter the mechanical properties of a metallic alloy, manipulating properties such as hardness, strength, toughness, ductility and elasticity.

Heat treatment produces a microstructure that also comprises an aggregate of ferrite and carbon-enriched stabilized austenite, from austenite partially quenched to martensite rather than transformed to carbide free bainite. Using martensite, rather than bainitic ferrite, has the immediate attraction of attaining potentially higher strength levels.

The heat treatment sequence involves quenching to a temperature between martensitic-start(M_s) and martensite-finish(M_f) temperatures, followed by a portioning treatment either at, or above the initial quench temperature, designed to enrich the remaining untransformed austenite with carbon, escaping from the supersaturated martensite phase, thereby stabilizing retained austenite phase to room temperature.

The heat treatment of the samples is carried out in muffle furnace, at 1020°C, 1050ºC, 1100ºC, 1150ºC and 1200ºC for constant soaking time of 1 hour and then they are quenched in quench probe tank at 45°C.

3.2.1. MUFFLE FURNACE

Fig :3.3.1. Muffle Furnace

A muffle furnace is a furnace with an externally heated chamber. The walls of which radiantly heat the contents of the chamber, so that the material being heated has no contact with the flame.

Muffle furnace are most often utilized in laboratories as a compact means of creating extremely high-temperature atmospheres. They are employed to test the characteristics of materials at extremely high and accurate temperatures. A muffle furnace is also known as retort furnace.

A muffle furnace is a piece of oven-type equipment that can reach high temperatures. It usually works by putting a high-temperature heating coil in an insulated material. The insulating material effectively acts as a muffle, preventing heat from escaping.

Furnace is usually heated to desired temperatures by:

- Conduction
- Convection
- Blackbody radiation from electrical-resistance heating elements

A precious metal thermocouple senses the temperature in the chamber and transmits this information to the temperature control in millivolts. The control section consists of a temperature controller, a current controller, a transformer, a contractor (relay), a circuit breaker. The temperature controller senses the furnace temperature (by means of thermocouple) and adjusts electricity to the heating elements by means of the current controller. The current controller controls electricity to the heating elements by adjusting the magnitude of the electrical current (rather than turning the electricity completely on or off). This is preferred method of controlling electricity to molybdenum disilicate heating elements. When all the necessary work is done and the current is controlled then sample gets heated to the desired temperature and then removed.

There is usually no combustion involved in the temperature control of the system, which allows for much greater control of temperature uniformity and assures isolation of material being heated from the by-products of fuel combustion.

Applications:

- Fusing glass
- Creating enamel coatings
- Ceramics
- Soldering
- Brazing

3.3. HARDNESS ANALYSIS

Hardness is defined as resistance to bending scratching, abrasion, cutting or any permanent indentation. Hardness is not a material property. Hardness test involves the use of a specifically shaped indenter, significantly harder than the test sample that is pressed into the surface of the sample using a specific force. Either the depth or size of the indent is measured to determine a hardness value. After the proposed heat treatment cycle, hardness analysis was done on macro level. Rockwell hardness testing machine has been used for hardness measurement.

3.3.1 ROCKWELL HARDNESS TEST

Rockwell hardness testing is an indentation testing method. The indenter is either a conical diamond (Brale) or a hard steel ball. Different indenter ball diameters from 1/16 to 1/2 in. are used depending on the test scale.

To start the test, the indenter is "set" into the sample at a prescribed minor load. A major load is then applied and held for a set time period. The force on the indenter is then decreased back to the minor load. The Rockwell hardness number is calculated from the depth of permanent deformation of the indenter into the sample,

Fig. 3.3.1 Rockwell Hardness Tester

i.e. the difference in indenter position before and after application of the major load. The minor and major loads can be applied using dead weights or springs. The indenter position is measured using an analogue dial indicator or an electronic device with digital readout.

The various indenter types combined with a range of test loads form a matrix of Rockwell hardness scales that are applicable to a wide variety of materials. Each Rockwell hardness scale is identified by a letter designation indicative of the indenter type and the major and minor loads used for the test. The Rockwell hardness number is expressed as a combination of the measured numerical hardness value and the scale letter preceded by the letters, HR. For example, a hardness value of 80 on the Rockwell C scale is reported as 80 HRC. Common Rockwell hardness scales include A, B and C which are listed below.

Scale	Load	Applications
A	60	Cemented carbides thin sheets, Shallow case-hardened steels. Only scale that is continuous over a wide range of hardness.
B	100	Aluminum, Copper, Soft steels and malleable Iron.
C	150	Hardened steels, Hard irons, Deep case-hardened steels, Titanium.

Table 2: Rockwell Hardness Scale

3.3.2 VICKERS HARDNESS TESTER

Fig: 3.3.2. Vickers Hardness Tester

In Vickers hardness test, the hardness of the material is determined by indentation of square based diamond pyramid having an angle of 136˚ between opposite slant faces. Instead of changing the indenters as well as the load, depending upon the nature of the material tested, only the load is changed in the Vickers hardness test. The load may be varied from 1kgf to 120kgf. The load is selected in accordance with the size and hardness of the specimen. The size of the indentation obtained in this test is small. The specimen is placed over the anvil and the load is slowly applied to the indenter and then released by means of lever. After the anvil is lowered a microscope is swung over the specimen and the diagonal of the square indentation is measured. In some types of machines, the indentation can be focused on to a graduated ground glass screen and measured.

Vickers and Brinell's hardness numbers are expressed in the same units of kgf/mm^2. The Vickers and Brinell's hardness numbers both coincide for hardness value of about 400. At the higher values of hardness, the Vickers number is larger than Brinell's number.

3.3.3 MICROHARDNESS TESTER

Microhardness is a hardness test employed to check light-gauge metals and alloys. This test uses very light loads for making an impression in the specimen. Also called Micro indentation testing. Microhardness tester in our project has been used to measure the variation of hardness from case to core.

In microhardness testing, an indentation is made on the specimen by a diamond indenter through the application of a load P. The size d of the resultant indentation is measured with the help of a calibrated optical microscope, and the hardness is evaluated as the mean stress applied underneath the indenter. The measurement of hardness with a microscope attachment, comprising the indenter and means for applying small loads, dates back more than 50 years. Initially used for small components (watch gears, thin wire, foils), microhardness testing was extended to research studies of individual phases, orientation effects in single_crystals, diffusion gradients, ageing phenomena, etc. in metallic and ceramic materials. Nowadays, testing at temperatures up to 1000°C is possible. In Europe, the pyramidal Vickers-type (interfacial angle 136°) indenter, which produces a square impression, is generally favoured. Its counterpart in general engineering employs test loads of 5–100 kgf: in microhardness testing, typical test loads are in the range 1–100 gf (1 gf=1 pound=1 p=9.81 Nm). The load applied on the sample during microhardness testing is 25gf. A rhombic-based Knoop indenter of American origin has been

Fig :3.3.3 Microhardness Tester

recommended for brittle and/or anisotropic material (e.g. carbides, oxides and glass) and for thin foils and coatings where a shallow depth of impression is desired. The kite-shaped Knoop impression is elongated, with a 7:1 axial ratio.

Microhardness tests need to be very carefully controlled and replicated, using as large a load as possible. The surface of the specimen should be strain free (e.g. electropolished), plane and perpendicular to the indenter axis. The indenter is lowered slowly at a rate of <1 mm min^{-1} under vibration-free conditions, eventually deforming the test surface in a manner analogous to steady-state creep.

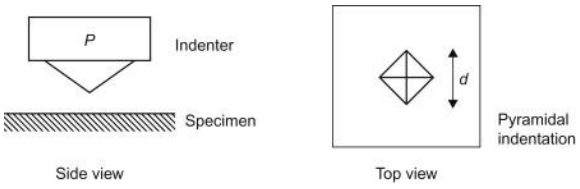

Fig: 3.5 Side view during indentation and top view after indentation

3.4. SAMPLE PREPERATION FOR MICROHARDNESS ANALYSIS

After the proposed heat treatment cycle, microstructural analysis of all the samples was carried out. It includes cutting, mounting, grinding, polishing followed by etching. After that microstructures of all the samples were studied. The details of all the processes involved and the machines used are given below.

3.4.1. CUTTING

Cutting is defined as the sectioning a small area of interest of the given material for its Microstructural study. Cutting is also done for easier handling. Depending upon the material, the

sectioning operation can be obtained by abrasive cutting (metals and metal matrix composites), diamond wafer cutting (ceramics, electronics, biomaterials, minerals), or thin sectioning with a microtome (plastics).

PROCEDURE

The specimens from the heat treatment samples were sectioned according to the field of interest using table top abrasive cutter and high-speed precision saw. The feed rate of table top abrasive cutter was 0.2mm/sec while that of high-speed precision saw was 2mm/min.

METALLOGRAPHIC PREPARATION DETAILS

Cutting is defined as the sectioning a small area of interest of the given material for its Microstructural study. Cutting is also done for easier handling. Depending upon the material, the sectioning operation can be obtained by abrasive cutting (metals and metal matrix composites), diamond wafer cutting (ceramics, electronics, biomaterials, minerals), or thin sectioning with a microtome (plastics). Proper sectioning is required to minimize damage, which may alter the microstructure and produce false metallographic characterization. Proper cutting requires the correct selection of abrasive type, bonding, and size; as well as proper cutting speed, load and coolant.

When cutting a specimen from a larger piece of material, care must be taken to ensure that it is representative of the features found in the larger sample, or that it contains all the information required to investigate a feature of interest. Cutting with abrasives may cause a high amount of damage, while the use of a low-speed diamond saw can lessen the problems. There are many different cutting methods, although some are used only for specific specimen types.

MACHINE DETAILS

1. Abrasi Matic™ 300 Abrasive Cutter

The AbrasiMatic 300 Abrasive Cutter is a bench-top cutter featuring manual cutting action in 3 directions or automated cutting in 1 direction. This gives the user the maximum versatility to section a wide variety of sample materials, sizes and geometries. It is engineered with innovative capabilities to be used in both

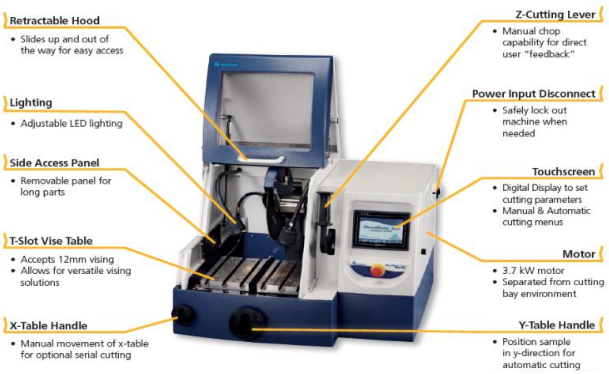

Fig: 3.4.1 Abrasi Matic™ 300 Abrasive Cutter

production support and laboratory environments. The AbrasiMatic 300 now includes an accompanying digital display to track X-axis travel improving precision, consistency and repeatability in serial sectioning applications.

2. IsoMet™ 4000 & 5000 Precision Saws

The IsoMet 4000 & 5000 Precision Saws cut materials with minimal specimen deformation and low kerf loss. The IsoMet 4000 & 5000 saws feature a manual blade positioning knob that accelerates set-up while clamping a specimen in a large unrestricted workspace. A wide selection of vices allows the user to precisely section virtually any material including metals, ceramics, composites, cements, laminates, plastics, electronic components, and biomaterials.

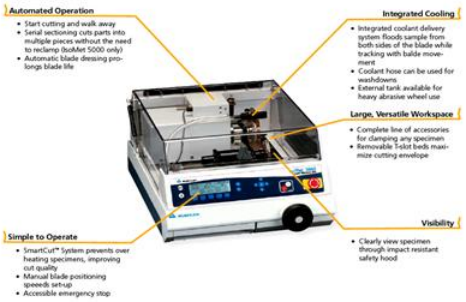

Fig: 3.4.1 IsoMet™ 4000 & 5000 Precision Saws

3.4.2 MOUNTING

Mounting of specimens is usually necessary to allow them to be handled easily. It also minimizes the amount of damage likely to be caused to the specimen itself. The mounting operation accomplishes three important functions:

- It protects the specimen edge and maintains the integrity of materials surface features
- Fills voids in porous materials
- Improves handling of irregular shaped samples, especially for automated specimen preparation.

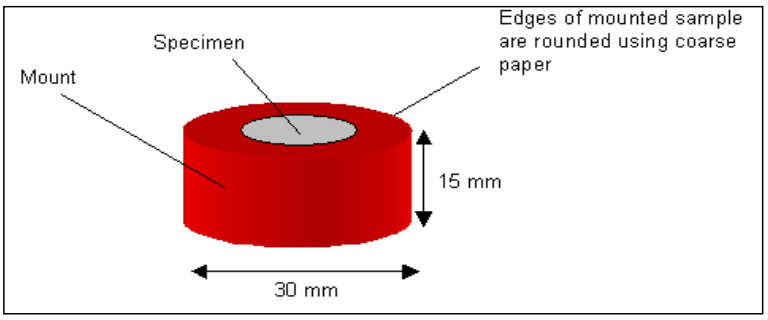

Fig.3.4.2 Pictorial representation of mounting specimen

MACHINE DETAILS

SimpliMet™ 3000 Automatic Mounting Press

The SimpliMet 3000 mounting press simplifies mounting in any lab with fully automatic operation and a variety of mold sizes. The electrohydraulic press with touch pad controls allows operators to attend to other tasks during the mounting cycle.

Fig: 3.4.2 SimpliMet™ 3000 Automatic Mounting Press

PROCEDURE

After the cutting was over, the specimens were mounted using phenolic powder as base material in compression mounting machine. Phenolic powder used was 1.0-micron alpha alumina of brand Buehler micro polish phenolic powder of Hardness 88 (Shore D). Pressure attained by the mounting press was 295bar, heating time was 110 seconds and cooling time was 240 seconds. The temperature attained during mounting is generally 180° C-200° C. The mount is left in the press chamber for manual cooling to room temperature for better hardness of the mount. It includes six steps which are listed below.

1.	Pressurizing	In this stage the pressure needed for mounting in the mounting chamber is reached.
2.	Pre-Heating	In this stage the compression mounting compound is heated to attain its melting temperature under the established pressure.
3.	Heating	In this stage the compression mounting compound is melted to under the established pressure and temperature.
4.	Cooling	In this stage the compression mounting compound is solidified by decreasing the temperature under the established pressure by passing water in the outer shell of the press chamber.
5.	De-Pressurizing	In this stage the pressure built in the press chamber is decreased to

		atmospheric pressure.
6.	Manual cool off	This is the Cooling stage of the mount in the press chamber to room temperature after depressurizing. It is an optional step. The mount is left in the press chamber for manual cooling to better hardness of the mount.

PROCESS DETAILS

Mounting of specimens is usually necessary to allow them to be handled easily. It also minimizes the amount of damage likely to be caused to the specimen itself. The mounting operation accomplishes three important functions:

(1) It protects the specimen edge and maintains the integrity of materials surface features

(2) Fills voids in p

Table 2: Steps involved in mounting

(3) Improves handling of irregular shaped samples, especially for automated specimen preparation.

The majority of metallographic specimen mounting is done by encapsulating the specimen into a compression mounting compound (thermosets -phenolics, epoxies, diallyl phthalates or thermoplastics

- acrylics), casting into ambient castable mounting resins (acrylic resins, epoxy resins, and polyester resins), and gluing with a thermoplastic glue. For metals, compression mounting is widely used. Phenolics are popular because they are low cost, whereas the diallyl phthalates and epoxy resins find applications where edge retention and harder mounts are required. The acrylic compression mounting compounds are used because they have excellent clarity.

3.4.3. GRINDING

PROCEDURE

Surface layers damaged by cutting must be removed by grinding. Mounted specimens are ground with rotating discs of abrasive paper, for example wet silicon carbide paper. The coarseness of the paper is indicated by a number: the number of grains of silicon carbide per square inch. The grinding procedure involves several stages, using a finer paper each time followed by 60, 250, 400, 600 and

800.

PROCESS DETAILS

Surface layers damaged by cutting must be removed by grinding. Mounted specimens are ground with rotating discs of abrasive paper, for example wet silicon carbide paper. The coarseness of the paper is indicated by a number: the number of grains of silicon carbide per square inch. The grinding procedure involves several stages, using a finer paper each time followed by 250, 400, 600 and 800. Each grinding stage removes the scratches from the previous coarser paper. This can be easily achieved by orienting the specimen perpendicular to the previous scratches. Between each grade the specimen is washed thoroughly with water to prevent contamination from coarser grit present on the specimen surface.

MACHINE DETAILS

1. DuoMet™ 2 Belt Surfacer

High speed motors provide rapid material removal, generating required initial flatness, even with large and rough specimens. High speed manual coarse grinder ideal for metallographic, spectrographic and macro hardness test specimen

Fig: 3.4.3 DuoMet™ 2 Belt Surfacer

preparation. It has convenient sink and faucet for rinsing specimens.

2. MetaServ 3000 twin grinder-polishers

The MetaServ 250 twin grinder-polishers offer a combination of performance, economy and reliability for most microstructural analysis applications. Designed for manual or semi-automatic grinding and polishing, the base can be combined with one of two power heads.

The molded protective housing provides long lasting corrosion and impact resistance. Each bowl includes a removable splash guard reduces overspray and allows easy access to the platen. A built-in drain and bowl wash flush out particles and minimize build-up of grinding-polishing debris. The adjustable water flow nozzle can be positioned anywhere over the platen,

providing cooling precisely where needed. All base models include an easy-to-use control panel with quick access to power on/off, plate run/pause, digital up/down timer and reset, water on/off run-active control, variable plate speed control from 50-500 rpm and an emergency stop. In addition to the emergency stop button, other safety features include built-in circuit protection and low voltage controls.

3.4.4 POLISHING

Polishing discs are covered with soft cloth impregnated with abrasive diamond particles and an oily lubricant or water lubricant. Particles of two different grades are used: a coarser polish – alumina powder of particle size 1 microns in diameter which should remove the scratches produced from the finest grinding stage, and a finer polish – alumina powder of particle size particles 0.05 micron in diameter, to produce a smooth surface.

PROCEDURE

Polishing was done to remove the lines generated during grinding and also to get mirror like surface finishing. Polishing was done on a disc covered with soft cloth using solution of alumina powder of size 1 micron followed by 0.05 micron.

PROCESS DETAILS

Polishing discs are covered with soft cloth impregnated with abrasive diamond particles and an oily lubricant or water lubricant. Particles of two different grades are used, a coarser polish – alumina powder of particle size 1 microns in diameter which should remove the scratches produced from the finest grinding stage, and a finer polish – alumina powder of particle size particles 0.05 micron in diameter, to produce a smooth surface. Before using a finer polishing wheel, the specimen should be washed thoroughly with water to avoid contamination. The drying can be made quicker using a hot air drier.

MACHINE DETAILS

Buehler SuperMet Two Spindle Polisher

It is a heavy-duty fine polisher having two Wheels each of 12" Diameter. This machine also consists of a Sink with two faucets, Mounted in Tech-Met Cabinet.

Fig 3.4.4 Buelher SuperMet Two Spindle

3.4.5 ETCHING

Procedure

The polished samples have been then etched with Nital (2ml HNO3+ 98ml Ethylalcohol). It includes immersion of specimen into the freshly prepared etchant for 10 to 20 seconds at room temperature.

Process Details

Etching is used to reveal the microstructure of the metal through selective chemical attack. In alloys with more than one phase, etching creates contrast between different regions through differences in topography or the reflectivity of the different phases. The rate of etching is affected by crystallographic orientation, so contrast is formed between grains, for example in pure metals. The reagent will also preferentially etch high energy sites such as grain boundaries. This results in a surface relief that enables different crystal orientations, grain boundaries, phases and precipitates to be easily distinguished.

The specimen is etched using a reagent. This is applied using a cotton bud wiped over the surface a few of times (Care should be taken not to over-etch - this is a difficult point to determine). The specimen should then immediately be washed in and dried.

Following the etching process there may be numerous small pits present on the surface. These are etch pits caused by localized chemical attack, and in most cases, they do not represent features of the microstructure. They may occur preferentially in regions of high local disorder, for example where there is a high concentration of dislocations.

If the specimen is over etched, these pits tend to grow, and obscure the main features to be observed.

3.5 OPTICAL MICROSCOPY

Microstructural analysis of the samples has been done under optical microscope. The prepared samples were taken and microstructure has been seen and captured at different magnifications ranging from 50X to 600X. Microstructural study has been done on these micrographs.

3.5.1. MICROSCOPE OPTICAL SYSTEM CONFIGURATION

An optical microscope consists of the following two major basic functions.

- Creating a Magnified Image of a Specimen
- Illuminating a Specimen

The function to create a magnified image of a specimen consists of three basic functions of "obtaining a clear, sharp image", "changing a magnification", and "bringing into focus". An optical system for implementing these functions is referred to as an observation optical system.

Meanwhile, the function to illuminate a specimen consists of three basic functions of "supplying light", "collecting light", and "changing light intensity". An optical system for implementing these functions is referred to as an illumination optical system. In other words, the observation optical system projects a sample (specimen) through an optical system and moreover leads a projection image to eyes or a pickup device such as CCD.

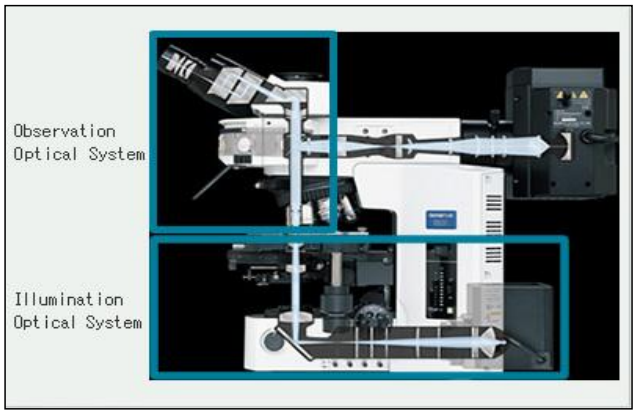

Fig. 11: Microscopic system

On the other hand, the illumination optical system effectively collects light emitted from the light source and leads the light to a specimen to illuminate it. The layout of observation and illumination optical systems in an optical microscope is as in the figure below for an upright microscope.

Fig 3.5.1 Optical Microscope

Meanwhile, for an inverted microscope the layout relation between those optical systems is upside down at the center of a specimen with respect to an upright microscope.

3.5.2. PRINCIPLE OF OPTICAL MICROSCOPE

An optical microscope creates a magnified image of an object specimen with an objective lens and magnifies the image further more with an eyepiece to allow the user to observe it by the naked eye. Assuming a specimen as AB in the following figure, primary image (magnified image) A'B' of inverted real image is created with an objective lens (ob). Next, arrange the eyepiece (oc) so that primary image A'B' is located closer to the eyepiece than the anterior focal point, then more enlarged erect virtual image A"B" is created. Put your naked eye in the eye (pupil) position on the eyepiece barrel to observe the enlarged image. In short, the last image to be observed is an inverted

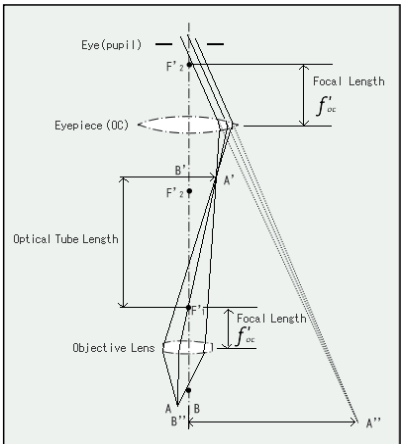

Fig. 3.5.2 Principle of optical Microscopic

Comment [A1]: Why this header?

As described above, this type of microscope which creates a magnified image by combining an objective lens making an inverted real image and an eyepiece making an erect virtual image is called a compound microscope. The observation optical system in an optical microscope is commonly standardized on this compound microscope. Meanwhile, such type of microscope that directly observes an inverted real image magnified with an objective lens is called a single microscope. A microscopic observation on a TV monitor, recently popularized increasingly, uses the way of directly capturing this inverted real image with a CCD camera, thereby being comprised of a simple microscope optical system.

MACHINE DETAILS

The machine used is a NIKON microscope. The magnification value of objective lens ranges from 5X to 100X and magnification of eye piece is 10X. The NIKON microscope is attached with a computer system so that the microstructure of the specimen can be captured.

Fig.3.5.3 NIKON Microscope

Sl no.	Test specimen	Temperature (°C)	Soak time (hrs)	Quenchant used	Hardness before heat treatment (HRC)	Hardness after heat treatment (HRC)

CHAPTER 4

RESULTS

4.1 TABULATION

1.	**HP-89**	**1020**	**1**	**Oil**	**20**	**57-58**	**Yes**
2.	**HP-90**	**1050**	**1**	**Oil**	**20**	**58-59**	**Yes**
3.	**HP-91**	**1100**	**1**	**Oil**	**20**	**53-54**	**Yes**
4.	**HP-92**	**1150**	**1**	**Oil**	**20**	**42-43**	**Yes**
5.	**HP-93**	**1200**	**1**	**Oil**	**20**	**30-32**	**Yes**

Table 4.1 Tabulation of Results after macro hardness

4.2 HARDNESS DEPENDENCE ON SOAKING TEMPERATURE DURING HARDENING

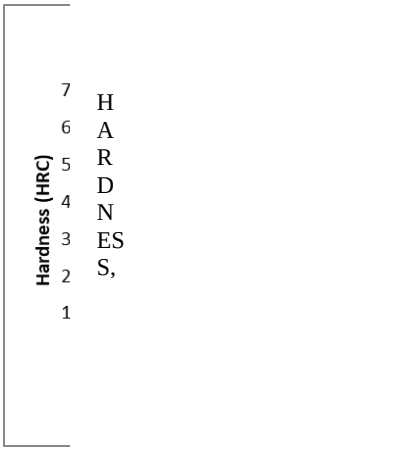

Fig 4.1: Graph of hardness dependence on soaking time during hardening

4.3. MICROHARDNESS SURVEY

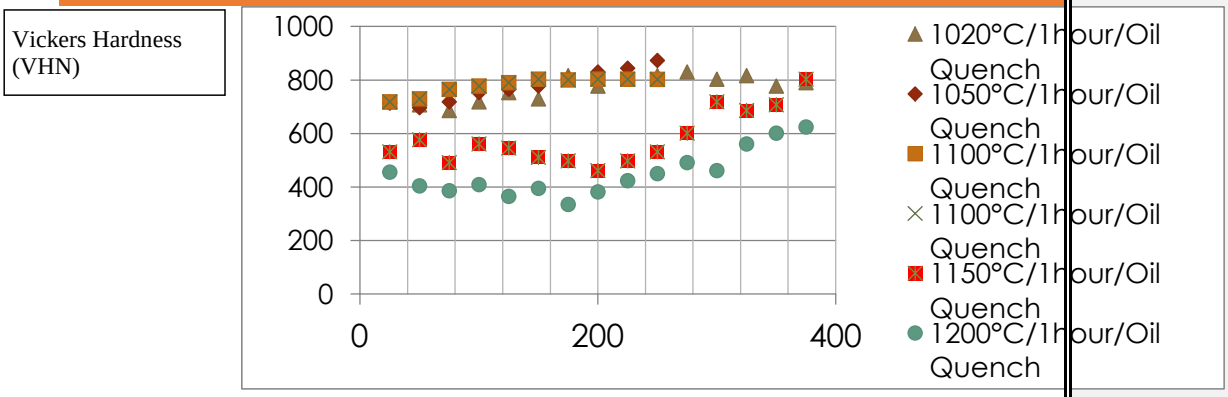

Fig 4.2: Graph of varying hardness from case to core

CHAPTER 5

CONCLUSION AND FUTURE SCOPE OF WORK

5.1. CONCLUSION

- After conducting Macro hardness and micro hardness tests on several specimens, we came to a conclusion that.
- The Hardness of the test specimen reduced drastically as the temperature increased from 1020º C to 1200º C. This was due to oxidation of carbon from the test specimen which resulted in the reduction of hardness.
- The micro hardness tests revealed that there was a uniform increase in the hardness value of the test specimen from case to core for 1020º C, 1050º C and 1100º C.
- Whereas, at 1150º C and 1200º C it was observed that the hardness value was varying from case to core, this was due to excessive oxidation of carbon from the surface which resulted in the production of carbon dioxide which escaped from the surface and hence a drastic reduction in the hardness value.
- From the micro hardness graph, we can clearly see that the hardness value is highest at 1050º C which is justified from the macro hardness graph. Hence, the value of soaking temperature should not exceed more than 1100º C.

5.2. FUTURE SCOPE OF WORK

Surface oxidation studies-reason for lower hardness at surface when soaking temperature is more.

CHAPTER 6

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