

P-2809



WEAR OF SURFACE TREATED DIE TOOL STEEL



A Project Report

Submitted by

E.K.ARUN - 71205114005
Y.BALA MURALI - 71205114009
N.PRABAKARAN - 71205114029



*in partial fulfillment for the award of the degree
of*

**Bachelor of Engineering
in
Mechanical Engineering**

**DEPARTMENT OF MECHANICAL ENGINEERING
KUMARAGURU COLLEGE OF
TECHNOLOGY
COIMBATORE – 641 006**

ANNA UNIVERSITY :: CHENNAI 600 025

APRIL– 2009

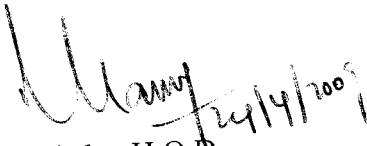
ANNA UNIVERSITY :: CHENNAI 600 025

BONAFIDE CERTIFICATE

Certified that this project report entitled "WEAR OF SURFACE TREATED DIE TOOL STEEL " is the bonafide work of

E.K.ARUN	-	71205114005
Y.BALA MURALI	-	71202114009
N.PRABAKARAN	-	71202114029

Who carried out the project work under my guidance.


Signature of the H.O.D

Dr. T. KANNAN
HEAD OF THE DEPARTMENT

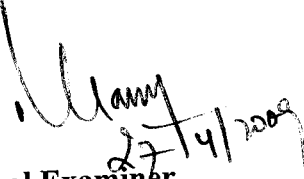
Dept. of Mechanical Engineering
Kumaraguru College of Technology
Coimbatore-641 006




Signature of the guide

Mr. P. S. SIVASAKTHIVEL
SUPERVISOR,
LECTURER

Dept. of Mechanical Engineering
Kumaraguru College of Technology
Coimbatore-641 006


Internal Examiner


External Examiner

DEPARTMENT OF MECHANICAL ENGINEERING
KUMARAGURU COLLEGE OF TECHNOLOGY
COIMBATORE 641 006

ABSTRACT

One of the surface treatments is boronizing which is technically well developed and widely used in industry to produce extremely hard and wear resistant surface layer on metallic substrate. Diffusion boronizing is a thermo-chemical treatment that permits boride layers of good performance properties to be produced on steels. The boronized steels exhibit high hardness (about 2000 HV), high wear resistance and improved corrosion resistance. Boron atoms can diffuse into ferrous alloys due to their relatively small size and very mobile nature. They can dissolve in iron interstitially, but can react with it to form FeB and Fe₂B inter metallic compounds. Depending on the potential of medium and chemical composition of base materials, single or duplex layer may be formed. During boronizing of ferrous alloys, generally, a boron-compound layer develops which consists of a surface adjacent FeB sublayer on top of a Fe₂B sublayer. Boronizing can be applied to a wide range of steel alloys including carbon steel, low alloy-steel, tool steel and stainless-steel. In addition, materials such as nickel based alloys, chromium based alloys, tungsten and niobium can be boronized to obtain very high hardness and wear resistance on their surfaces.

Austenitic stainless steels and hot work tool steels have high chromium content and are commonly used engineering materials. These are also well-suited and established for surface treatments such as nitriding and boronizing. Most studies about boronizing of steels and non-ferrous metals have done. However, a little knowledge about examination of growth kinetics of borides formed on boronized AISI H13 and AISI 316 in borax based salt bath. In this project, the boron diffusion on boride layer is evaluated by taking into account experimental data for growth kinetics of the boride layers during the molten salt bath boronizing process on AISI H13 and AISI 316 steels. The growth kinetics of the layer is analyzed by measuring the hardness as a function of the boronizing time within a temperature range of 1073–1223 K.

ACKNOWLEDGEMENT

We extend our profound gratitude to our amicable guide Lecturer **MR.P.S.SIVASAKTHIVEL** His valuable and uncountable contribution was meticulous. His relevant suggestions and appropriate corrections were helpful in the completion of this project successfully.

We place on record our sincere gratitude to our H.O.D **Dr.T.KANNAN, PhD** for his progressive outlook and valuable suggestions.

Not out of formality, but out of sheer gratitude we would like to express our thank fullness to **Prof.R.ANNAMALAI**, vice Principal for providing us with all facilities to finish the project.

We are thankful to our Lab assistants who shared their technical expertise with us for our project.

We also wish to express our special appreciation to all our friends and well-wishers for their timely help and encouragement.

Finally, we would like to thank our family members for their support in all our endeavors.

TABLE OF CONTENTS

CHAPTER NO	TITLE	PAGE NO
1	INTRODUCTION	1
2	SURFACE TREATMENT	4
	2.1. TYPES	5
	2.2. INDUCTION HARDENING	5
	2.3. TYPES	5
	2.4. SINGLE SHOT HARDENING	6
	2.5. TRAVERSE HARDENING	6
	2.6. CASE HARDENING	7
	2.7. TYPES	7
	2.8. CABURIZING	8
	2.9. NITRIDING	8
	2.10. GAS NITRIDING	9
	2.11. SALT BATH NITRIDING	9
	2.12. CYANIDING	10
	2.13. CARBONITRIDING	11
	2.14. BORONIZING	11
3	LITERATURE SURVEY	12
	3.1. MECHANISM OF THERMO-CHEMICAL GROWTH OF IRON BORIDES ON IRON	13

	3.2. INFLUENCE OF DIFFERENT SURFACE TREATMENTS ON H13 STEEL	14
	3.3. ABRASIVE WEAR RESISTANCE OF BORONIZED CARBON AND LOW ALLOY STEELS	15
	3.4. FORMATION OF BORIDE LAYERS ON STEEL SUBSTRATES	16
4	EXPERIMENTAL SETUP	17
	4.1. BORIDING METHODS	18
	4.1.1. PACK BORIDING PROCESS	18
	4.1.2. MOLTEN SALT PROCESS	18
	4.2. EXPERIMENTAL SETUP	18
	4.3. SAMPLE SPECIMEN	19
	4.4. COMPOSITION MIXTURES	19
	4.5. THERMAL TREATMENT	19
	4.5.1. CONTINUOUS PROCESS	19
	4.5.2. GRAPH	20
	4.5.3. INTERRUPTED PROCESS	20
	4.5.4. GRAPH	21
5	BORONIZING	22
	5.1. BORONIZING	23
	5.2 TYPES OF BORONIZING	23

	5.3. POWDER PACK BORONIZING	23
	5.4. PASTE BORONIZING	24
	5.5. PROCESS CHARACTERISTICS	24
	5.6. BENEFITS	25
	5.7. PROPERTIES OF BORIDE LAYERS	25
	5.8. BORONIZING AGENTS	26
	5.9. FEATURES	27
	5.10. APPLICATIONS	27
	5.11. H13 TOOL STEEL	28
	5.12. UNIQUE CHARACTERISTICS	28
	5.13. CHEMICAL COMPOSITION	29
	5.14. SS316L STEEL	29
	5.15. COMPOSITION	30
6	SPECIMEN PREPARATION	31
	6.1. SPECIMEN PREPARATION	32
	6.1.1. TESTED SPECIMEN OF H13	32
	6.2. LATHE WORK	33
	6.3. SPECIMEN CLEANING	35
	6.3.1. WITH EMERY SHEET	35
	6.3.2. WITH POLISHING MACHINE	36
7	BORONIZING TREATMENT	38
	7.1. BORONIZING TREATMENT	39
	7.1.1. BORONIZING	39

	7.2. THERMAL TREATMENT	39
	7.2.1. CONTINUOUS PROCESS	39
	7.2.2. INTERRUPTED PROCESS	41
8	RESULTS & DISCUSSIONS	42
	8.1. HARDNESS TEST	43
	8.2. RESULT	44
	8.3. EFFECT OF ROCKWELL HARDNESS ON H13 STEEL	45
	8.4. EFFECT OF ROCKWELL HARDNESS ON SS316L STEEL	46
9	FUTURE WORK	47
	9.1. MICROHARDNESS MEASUREMENTS	48
	9.2. WEAR TEST	48
	9.2.1. PI ON DISC WEAR TEST	48
	9.3. CORROSION TEST	49
	9.4. MICROSCOPIC OBSERVATIONS	49
10	REFERENCES	50

LIST OF FIGURES

FIGURE	TITLE	PAGENO
4.1	EXPERIMENTAL SETUP	18
4.2	SAMPLE SPECIMEN	19
4.3	TIME GRAPH FOR CONTINUOUS BORONIZING	20
4.4	TIME GRAPH FOR INTERRUPTED BORONIZING	21
6.1	TESTED COMPOSITION OF H13	32
6.2	TESTE SPECIMEN	32
6.3	LATHE WORK	33
6.4	SPECIMEN 1	34
6.5	SPECIMEN 2	34
6.6	WORK WITH EMERY SHEET	35
6.7	WORK WITH POLISHING MACHINE	36
6.8	POLISHING MACHINE	37
6.9	POLISHING CLOTH	37
7.1	CONTINUOUS BORONIZING	40
7.2	INTERRUPTED BORONIZING	41
8.1	HARDNESS TEST	43
8.2	HARDNESS TESTING SPECIMEN	44
8.3	GRAPH OF CONTINUOUS BORONIZING(H13)	45
8.4	GRAPH OF INTERRUPTED BORONIZING(H13)	45
8.5	GRAPH OF CONTINUOUS BORONIZING(SS316L)	46
8.6	GRAPH OF INTERRUPTED BORONIZING(SS316L)	46

LIST OF TABLES

TABLE	TITLE	PAGE NO
4.1	COMPOSITION OF BORONIZING MIXTURES	19
5.1	BORONIZING AGENTS	26
5.2	CHEMICAL COMPOSITION OF H13	29
5.3	COMPOSITION OF SS316L	30
8.1	RESULT	44

CHAPTER 1

INTRODUCTION

The surfaces of engineering components are subjected to higher stresses and greater fatigue, abrasion, and corrosive damages than the interior. Therefore, more than 90 pct of the service failures of engineering components initiate at, or near, the surface. Surface modification techniques are employed to improve the resistance to failure by producing a hard and wear-resistant case around a soft and tough core. Two major classes of treatments available for enhancing the surface properties are thermal and thermochemical. Thermal treatments, such as flame and induction hardening, modify the microstructure without modifying the surface chemistry, whereas in thermochemical methods, the surface chemistry is altered. Carburizing and nitriding are well known thermo-chemical methods. Boriding or boronizing is a recent process, which is analogous to carburizing and nitriding.

Boriding can develop surface hardness in the range of 1500 to 2000HV, as compared to a hardness in the range of 600 to 1100 HV for nitriding, 700 to 850 HV for carburizing, and 950 to 1100 HV for chromium plating. Borided layers provide a wear resistance comparable to that of sintered carbides. The wear resistance of cold-working tools is increased by about 10 times and that of hot-working tools and dies by about 3 times as a result of boriding. If the process has been performed properly and the right material and layer thickness have been chosen, boriding can extend the service life of engineering components beyond that imparted by traditional methods like carburizing or nitriding.

Various processes adopted for boriding include pack boriding, molten salt boriding, electrolytic boriding, gas boriding, vacuum boriding, etc. In pack boriding, the sample is cleaned and kept surrounded by solid mixtures consisting of boron carbide and borax mixtures. Alternatively, boron carbide or amorphous boron has been used as the boron source, along with diluents such as silicon carbide or alumina or graphite and activators like KBF_4 or NaF in the boriding mixtures.

Ferroboron can be considered as a boron source instead of boron carbide. However, it is reported that the commercial grades of ferroboron contain impurities like Si and Al; therefore, the use of this material leads to a degenerate layer. However, special-quality ferroboron can be used to get a good-quality boride case, according to the classical review book by Von Matuschka, which is a comprehensive, general, and theoretical compendium on boriding.

The molten salt boriding process is usually performed using anhydrous borax, mixed with reducing agents like boron carbide, silicon carbide, or silico-calcium. Amorphous boron is stated to be better than boron carbide, since it produces less slurry. Electrolytic boriding is carried out in inert atmospheres like argon, using borax-based melts at about 1173 K at a current density of about 0.15 to 0.25 A/cm².

Electrolytic boriding produces thicker coatings in relatively shorter times compared to molten salt or pack boriding processes. There are many other techniques for boriding, e.g., gas boriding, vacuum boriding, plasma boriding, and thermal spraying of powders. Out of all these techniques, pack and molten salt (electroless) processes are technologically simpler and more economical compared to other boriding processes.

During boriding of plain-carbon steels, needle-like FeB and Fe₂B phases are formed. When the boron potential is low, Fe₂B phase alone forms in the case. At higher potentials of boron, FeB phase also forms along with Fe₂B. The Fe₂B phase forms adjacent to the core and the FeB phase forms near the surface. It is reported that FeB₁₉ phase also forms, very rarely, when the boron potential is very high. A single-phase

structure is desirable. Fe₂B is preferred to FeB, since FeB is very hard and brittle and has a coefficient of thermal expansion that differs from the matrix by a factor of 3 and that causes spalling during cooling.

Although boriding of plain-carbon steels improves the wear, abrasion, fatigue, tensile, corrosion, corrosion-fatigue, and oxidation properties, one serious drawback is the brittleness of the case. Several methods have been attempted to solve this problem. They include partial deboriding of the saturated layer, the addition of copper to the borided layer, multicomponent diffusion impregnation of iron with elements like silicon, aluminium, etc., along with boron, superplastic boronizing, which produces a nonacicular structure with equiaxed grains of the borides, thereby reducing brittleness, and laser surface modification of borided surfaces.

The objectives of the present study are to develop low-cost boriding processes using low-cost and easily available raw materials, (2) to optimize the process parameters to get only Fe₂B phase, resulting in better toughness, and to improve the microstructural morphology and, thereby, increase the toughness and ductility of the boride layer by an interrupted thermocyclic boriding process.

CHAPTER 2

SURFACE TREATMENT

Surface treatments are the which is used to increase properties mechanical of the metals. By doing different types of surface treatment the material get different mechanical properties of it's own.

2.1. TYPES:

1. Induction hardening
2. Case hardening or surface hardening

2.2INDUCTION HARDENING:

Induction hardening is a form of heat treatment in which a metal part is heated by induction heating and then quenched. The quenched metal undergoes a martensitic transformation, increasing the hardness and brittleness of the part. Induction hardening is used to selectively harden areas of a part or assembly without affecting the properties of the part as a whole.

2.3 TYPES:

1. Single shot hardening
2. Traverse hardening

2.4 SINGLE SHOT HARDENING:

In single shot systems the component is held statically or rotated in the coil and the whole area to be treated is heated simultaneously for a pre-set time followed by either a flood quench or a drop quench system. Single shot is often used in cases where no other method will achieve the desired result for example for flat face hardening of hammers, edge hardening complex shaped tools or the production of small gears.

In the case of shaft hardening a further advantage of the single shot methodology is the production time compared with progressive traverse hardening methods. In addition the ability to use coils which can create longitudinal current flow in the component rather than diametric flow can be an advantage with certain complex geometry.

There are disadvantages with the single shot approach. The coil design can be an extremely complex and involved process. Often the use of ferrite or laminated loading materials is required to influence the magnetic field concentrations in given areas thereby to refine the heat pattern produced. Another drawback is that much more power is required due to the increased surface area being heated compared with a traverse approach.

2.5 TRAVERSE HARDENING:

In traverse hardening systems the work piece is passed through the induction coil progressively and a following quench spray or ring is utilised. Traverse hardening is used extensively in the production of shaft type components such as axle shafts, excavator bucket pins, steering components, power tool shafts and drive shafts. The component is fed through a ring type inductor which normally features a single turn.

The width of the turn is dictated by the traverse speed, the available power and frequency of the generator. This creates a moving band of heat which when quenched creates the hardened surface layer. The quench ring can be either integral a following arrangement or a combination of both subject to the requirements of the application. By varying speed power etc it is possible to create a shaft which is hardened along its whole length or just in specific areas and also to harden shafts with steps in diameter or splines. It is normal when hardening round shafts to rotate the part during the process to ensure any variations due to concentricity of the coil and the component are removed.

2.6 CASE HARDENING:

Case hardening or surface hardening is the process of hardening the surface of a metal, often a low carbon steel, by infusing elements into the material's surface, forming a thin layer of a harder alloy. Case hardening is usually done after the part in question has been formed into its final shape, but can also be done to increase the hardening element content of bars to be used in a pattern welding or similar process.

2.7 TYPES:

1. Carburizing
2. Nitriding
3. cyaniding
4. Carbonitriding
5. Boronizing

2.8 CARBURIZING:

Carburizing, also known as carburization, is a heat treatment process in which iron or steel is heated in the presence of another material (but below the metal's melting point) which liberates carbon as it decomposes. The outer surface or case will have higher carbon content than the original material. When the iron or steel is cooled rapidly by quenching, the higher carbon content on the outer surface becomes hard, while the core remains soft and tough.

2.9 NITRIDING:

Nitriding, also known as nitridization, is a process in which the nitrogen atoms are introduced into the surface of a material. By making this process the surface hardness of the material will be increased up to some level. It is one of the thermochemical processes used in the hardening method.

All nitriding processes enrich the surface of a material with nitrogen. Processes are named by the medium used to donate nitrogen. The three main methods used are:

- Gas nitriding
- Liquid or salt bath nitriding
- Ion or plasma nitriding

2.10 Gas nitriding

In gas nitriding the donor is a nitrogen rich gas usually ammonia (NH_3). When ammonia comes into contact with the heated work piece it disassociates into nitrogen and hydrogen. The nitrogen then diffuses from the surface into the core of the material. This is the oldest of the current nitriding processes though only in the last few decades has there been a concentrated effort to investigate the thermodynamics and kinetics involved. Recent developments have lead to a process that can be accurately controlled. The thickness and phase constitution of the resulting nitriding layers can be selected and the process optimized for the particular properties required. The advantages of gas nitriding over the other variants are:

- All round nitriding effect (can be a disadvantage in some cases, compare with plasma nitriding)
- Large batch sizes possible - the limiting factor being furnace size and gas flow
- With modern computer control of the atmosphere the nitriding results can be tightly controlled
- Relatively cheap equipment cost - especially compared with plasma

The disadvantages of gas nitriding are:

- Reaction kinetics heavily influenced by surface condition - an oily surface or one contaminated with cutting fluids for example will deliver poor results
- Surface activation is sometimes required to successfully treat steels with a high chromium content - compare sputtering during plasma nitriding

2.11 Salt bath nitriding

In salt bath nitriding the nitrogen donating medium is a nitrogen containing salt such as cyanide salt. The salts used also donate carbon to the workpiece surface making salt bath a nitrocarburising process. The temperature used is typical of all nitrocarburising processes; 550-590°C The advantages of salt nitriding are:

- Quick processing time - usually in the order of 4 hours or so to achieve
- Simple operation - heat the salt and workpieces to temperature and submerge until the duration has expired

The disadvantages are:

- The salts used are highly toxic - Disposal of salts are controlled by stringent environmental laws in western countries and has increased the costs involved in using salt baths. This is one of the most significant reasons the process has fallen out of favor in the last decade or so.
- Only one process possible with a particular salt type - since the nitrogen potential is set by the salt, only one type of process is possible.

2.12 CYANIDING:

This is a case hardening process that is fast and efficient; it is mainly used on low carbon steels. The part is heated to 1600-1750 °F in a bath of sodium cyanide and then is quenched and rinsed, in water or oil, to remove any residual cyanide.

This process produces a thin, hard shell (between 0.010 and 0.030 inches) that is harder than the one produced by carburizing, and can be completed in 20 to 30 minutes compared to several hours so the parts have less opportunity to become distorted. It is typically used on small parts such as bolts, nuts, screws and small gears. The major drawback of cyaniding is that cyanide salts are poisonous.

2.13 CARBONITRIDING:

Carbonitriding is a metallurgical surface modification technique that is used to increase the surface hardness of a metal, thereby reducing wear. During the process, atoms of carbon and nitrogen diffuse interstitially into the metal, creating barriers to slip, increasing the hardness and near the surface.

Carbonitriding is often applied to inexpensive, easily machined low carbon steel to impart the surface properties of more expensive and difficult to work grades of steel. Surface hardness of carbonitrided parts ranges from 55 to 62 HRC.

2.14 BORONIZING:

Boronizing is a thermo chemical surface hardening process in which boron atoms are diffused into the surface of metal work piece to form a hard layer of metal borides with the base material. When applied to the appropriate materials, boronizing provides wear and abrasion resistance comparable to sintered carbides.

The process involves heating well-cleaned material in the range of 700 to 1000 °C (1300 to 1830 °F), preferably for 1 to 12 h, in contact with a boronaceous solid powder (boronizing compound), paste, liquid, or gaseous medium.



CHAPTER 3

LITERATURE SURVEY

3.1 MECHANISM OF THERMOCHEMICAL GROWTH OF IRON BORIDES ON IRON:

Thermochemical boriding of iron alloys allows both single Fe₂B phase and FeB-base polyphase coatings to be obtained and then used mainly to improve surface hardness and wear resistance of components for tribological applications. Boride coatings, in fact, display considerable hardness and compactness except for the outermost, few micrometers thick region which, being constituted by differently oriented crystals, is friable and, consequently, should be removed from the component by means of a surface finishing procedure. Single Fe₂B phase coatings can be produced by limiting the boron potential of the boronising media, thus avoiding complications possibly arising by the formation of the much harder but even more brittle product FeB and by the high intensity stress states generally establishing at the FeB-Fe₂B interface. The best results have been obtained using pack cementation processes involving the use of powder mixtures. The fact that continuous, automated and reliable gas processes are not yet commercially available has considerably hindered a larger diffusion of boriding in surface engineering practice, as an alternative to more traditional treatments such as carbon cementation and nitriding.

The phase composition, microstructure and mechanical properties of boride coatings have been widely investigated, and the compactness of the boride layers has been related to the establishing of a preferred crystallographic orientation. The main components of the coatings (Fe₂B and FeB) generally display a strong (002) preferred orientation. It has been also reported that the compactness of iron borides is significantly influenced by the kind of the alloying elements which, diffusing from the base metal, enter the coating as a consequence of redistribution phenomena because of the differences in the alloying processes, the boride coatings display significant differences in total thickness, orientation strength and morphology at the Fe₂B/Fe and FeB/Fe₂B interfaces.

In the present work, the growth mechanism of iron borides was investigated by means of boriding treatments carried out on iron using powder mixtures with a controlled boronising potential, selected in order to allow the different stages of the process to be observed.

3.2 INFLUENCE OF DIFFERENT SURFACE TREATMENTS OF H13 HOT WORK DIE STEEL ON ITS THERMAL FATIGUE BEHAVIORS:

Thermal fatigue checking is the general failure of hot work die steels, which is relative with the structures and properties of the steels and the stress alternated during the employment. The Uddeholm test method on thermal fatigue is used to compare the behaviors of different samples, which are treated with plasma nitriding, plasma sulfur-carbon-nitriding, boronizing or not treated. The results show that the nitriding improves the thermal fatigue property of the tool steel, while the plasma sulfur-carbon-nitriding and the boronizing impair the property. The mechanisms are induced as follows. By increasing the hardness and changing the stress distribution in the surface layer, surface treatment can decrease the plastic deformation and the tensile stress during the cycling.

Therefore, the generation and growth of the cracks are restrained. On the other hand, as results of surface treating, in the surface layer the toughness declines and the expanding coefficient ascends; the latter change caused the strengthening of the tensile and compressive stress during the cycling. Thus the resistance to thermal fatigue is weakened. Whether or not the surface treatment is favor to thermal fatigue of tool steels relies on which factor is dominant.

3.3 ABRASIVE WEAR RESISTANCE OF BORONIZED CARBON AND LOW ALLOY STEELS:

Boronizing or boriding, is a thermochemical treatment that diffuses boron through the surface of metallic substrates. As boron is an element of relatively small size it diffuses into a variety of metals; including ferrous, nickel and cobalt alloys, metal-bonded carbides and most refractory alloys. Typically, boronizing is carried out in the 1113-1323k temperature range by using solid, liquid or gaseous boron-rich atmospheres.

Boronizing has been found to be an effective method for significantly increasing the surface hardness and wear resistance of metals. In the case of low carbon steel boronized by using boron carbide (B_4C) at 1223k during 1.5h, the maximum microhardness can reach 1700 and 1850 HV for 1020 and 5115 steel.

On the surface of boronized ferrous alloys, generally a boron compound layer is developed. This compound layer is normally composed of two sublayer; the outermost and the innermost are rich in FeB and Fe_2B , respectively. Underneath the compound layer the diffusion zone hardly exists because the solubility of boron in Fe is very small. Since the FeB phase is more brittle and harder, and has a higher coefficient of thermal expansion than Fe_2B one, the cracking of the double-phase boride layer is often observed.

To reduce the brittleness of the double-phase boride layer, several kinds of pre or post-boronizing treatments have been investigated; carburizing, laser treatment, quenching and gas nitriding. By boronizing a carburized steel, a reduction of the microhardness gradient across the case is obtained. In laser treatment, the coarse and columnar structure of the boride layer can be changed into a fine grain structure. In quenching, the hardness gradient between the boride layer and substrate can be reduced. In N ions implantation, part of FeB and Fe_2B is decomposed and c-BN, FeN and $Fe_{2-3}N$ compounds could be formed. In gas nitriding, the outermost sublayer can be transformed into the Fe_4N and $B_{25}N$ phases.

On the other hand, it has been shown that in a ferrous alloy, the content of some elements, such as C, Cr and Ni, has a great influence on the boride layer. With the increase of the C content, the thickness of this layer decreases and its hardness either increases, or first decreases and then increases. As carbon does not dissolve significantly in FeB and Fe₂B, C is driven ahead of the boronized layer, forming a polyphase zone rich in carbides and borocarbides and Fe₃(B,C) between the Fe₂B sublayer and the matrix. This would explain why the boride layer on high carbon steel is very brittle and bonded to the base metal. One effect of Cr is to increase the amount of the FeB phase in the boride layer. Other effects are to increase the hardness and brittleness of the surface layer and to produce a layer that is not very compact on outside. In the case of Ni, by increasing its percentage the compound layer becomes more regular and thinner. In most of the studies mentioned above, B₄C was boronizing agent. Due to the problems associated with the cracking of the boride layer, it seems advisable to study the possibility of using another boronizing agent that generates a low boronizing potential in order to form a surface layer of Fe₂B boride only. Thus, in this study, we decided to use borax as the boronizing agent and evaluated the abrasive wear resistance of carbon and low alloy steels, heat treated by using this agent.

3.4 FORMATION OF BORIDE LAYERS ON STEEL SUBSTRATES:

Boride layers on steel are known to be formed by thermal diffusion of boron into the surface of steel improving corrosion-erosion resistant properties. The possibility of formation of Fe₂B and FeB boride layers depends on the boron chemical activity and boron content as well as on the substrate composition, temperature and time of treatment. In this work efforts were made to clarify the role of the substrate composition and the temperature of treatment through the commensurability of structures of the boride layers to those of the substrate. Boronizing methods and taking into account the crystallization diagram made for the Fe-B amorphous alloy system the formation of the boride layers may proceed by an induced and controlled mechanism on low alloy ferritic steels.

CHAPTER 4

EXPERIMENTAL SETUP

4.1 BORIDING METHODS

4.1.1 PACK BORIDING PROCESS:

Initially, boron carbide-based powders were employed to develop the boride layer. Subsequently, ferroboron-based powders were also used to get a boride layer. Fine powders of the mixture were taken in a stainless steel container. The medium-carbon steel [EN8] sample of 10 mm in diameter and 10 mm in length was placed inside the box in such a way that the sample was uniformly surrounded by an approximately 15-mm-thick boriding mixture on all sides. The box was tightly closed, and the gap between the lid and the box was sealed with fireclay. The box was placed in a crucible (clay-graphite) surrounded by silica sand.

4.1.2 MOLTEN SALT (ELECTROLESS):

Anhydrous borax and boric acid were melted in a muffle and the melt was reduced by stoichiometric amounts of ferrosilicon magnesium powder; 10 pct NaF was added along with the reducer to activate the melt.

4.2 EXPERIMENTAL APPARATUS:

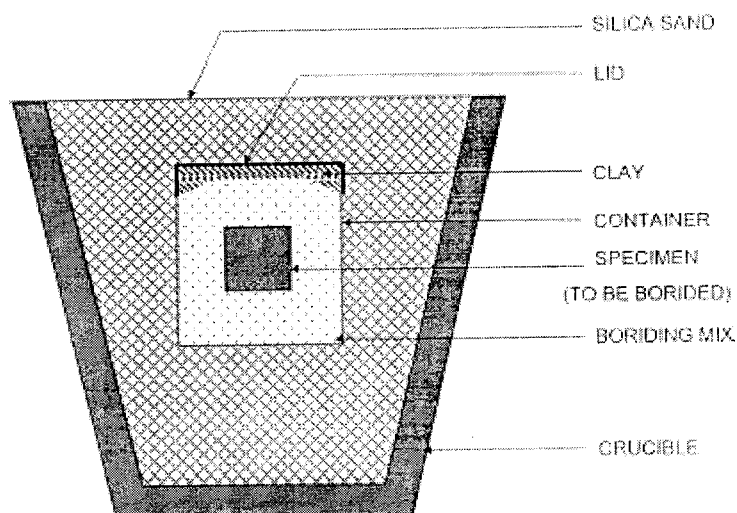


FIG 4.1 EXPERIMENTAL SETUP

4.3 SAMPLE SPECIMEN:

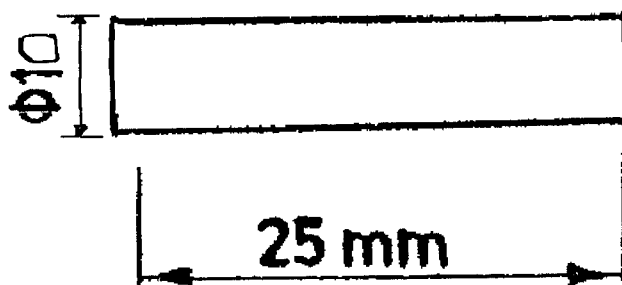


FIG 4.2 SAMPLE SPECIMEN

4.4 COMPOSITIONS (WT.%) OF BORONIZING MIXTURES:

Mixture	Borax	SiC	NH ₄ Cl	NaCl
M1	88	9	1.5	1.5
M2	73	24	1.5	1.5
M3	63	34	1.5	1.5

TABLE 4.1. COMPOSITION OF BORONIZING MIXTURES

4.5 THERMAL TREATMENT

4.5.1 CONTINUOUS PROCESS

The assembly shown in Figure 1 was loaded in the furnace at around 1073 K, and it was further heated to the boriding temperature (1223 K). When the temperature reached 1223 K, boriding was performed continuously for 4 hours. After 4 hours, the crucible was removed from the furnace and allowed to cool in still air. In the case of the molten salt process, the sample to be borided was kept in the boriding melt and boriding was carried out at 1223 K for 4 hours continuously. After 4 hours, the sample was removed from the melt and allowed to cool in air.

4.5.2 GRAPH:

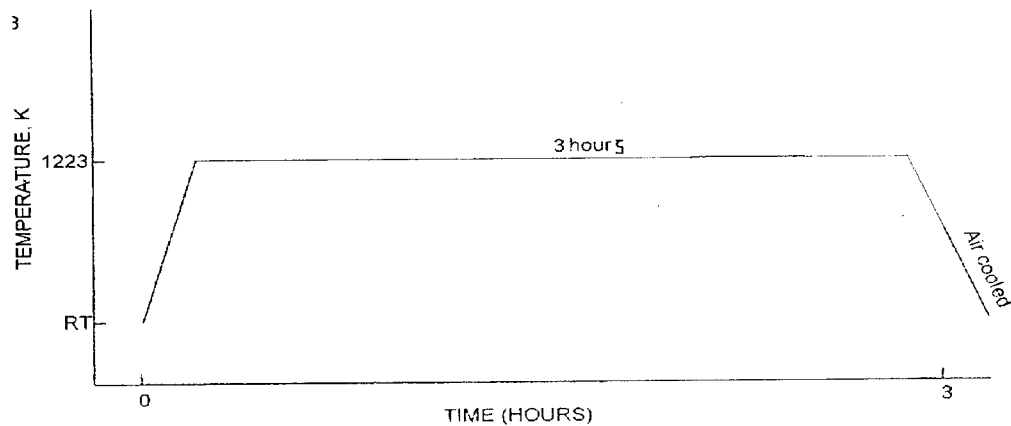


FIG 4.3 TIME GRAPH FOR CONTINUOUS BORONIZING

4.5.3 INTERRUPTED PROCESS

In interrupted boriding process, after every hour of boriding, the crucible (with the stainless steel box containing the boriding mix and sample) was removed from the furnace and allowed to cool in still air. During cooling, a thermocouple was inserted in the sand so as to touch the stainless steel box. When the temperature was about 873 K, the crucible was again loaded into the furnace at 1223 K. Boriding was done for 1 hour at 1223 K, and the sample was cooled again to about 873 K. This procedure was repeated four times. The thermal cycle is shown in Figure 2. After the interrupted boriding process (four steps of 1 hour each), the crucible was removed from the furnace and kept at 873 K for 1 hour in a separate furnace. It was then cooled in air to an room temperature.

In the case of molten salt boriding, the sample was and removed from the melt after every hour of boriding, cooled for 5 minutes in air, and again placed in the melt. The total boriding time was 4 hours. After this boriding treatment, the sample was heated for 1 hour at 873 K and then cooled Process in air.

4.5.4 GRAPH:

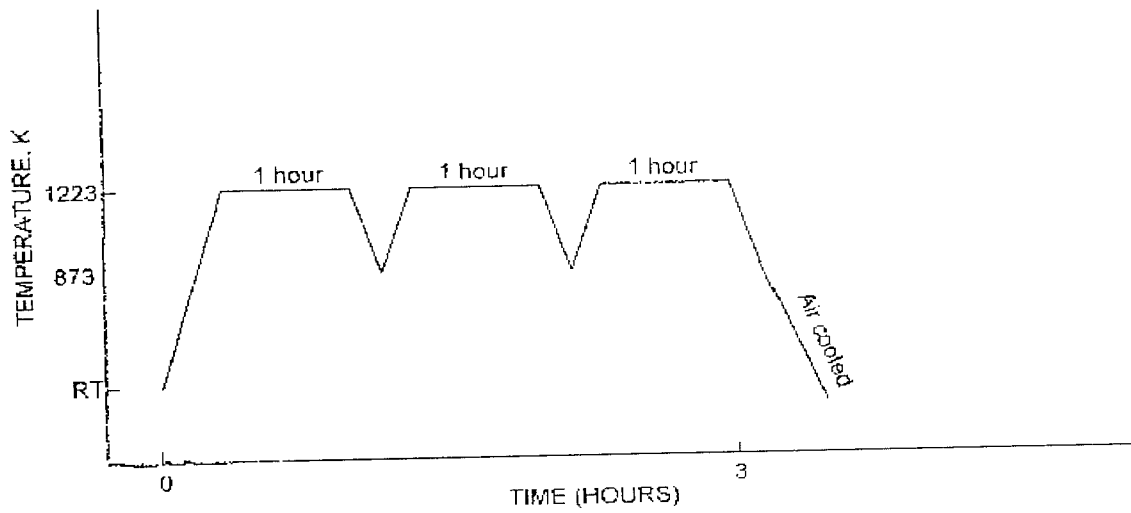


FIG 4.4. TIME GRAPH FOR INTERRUPTED BORONIZING

CHAPTER 5

BORONIZING

5.1 BORONIZING:

Boronizing is a thermo chemical surface hardening process in which boron atoms are diffused into the surface of metal work piece to form a hard layer of metal borides with the base material. When applied to the appropriate materials, boronizing provides wear and abrasion resistance comparable to sintered carbides.

The process involves heating well-cleaned material in the range of 700 to 1000 °C (1300 to 1830 °F), preferably for 1 to 12 h, in contact with a boronaceous solid powder (boronizing compound), paste, liquid, or gaseous medium.

5.2 TYPES OF BORONIZING:

1. Powder-pack boronizing
2. Paste boronizing

5.3 POWDER-PACK BORONIZING:

In powder-pack boronizing, the workpiece is placed in a suitable container and embedded in the boronizing agent. In order to minimize consumption of the latter, the container and workpiece should be of the same shape. To avoid complications, boronizing should be performed in a protective-gas atmosphere. This is accomplished either by packing the containers into a protective-gas retort and heat-treating them in a chamber furnace, or else boronizing directly in a retort furnace with the necessary protective-gas supply.

The protective gas may be pure argon, pure nitrogen, a mixture of hydrogen and either argon or nitrogen (forming gas), or, in special cases, pure hydrogen. It is important to note that oxygen-bearing compounds adversely affect boronizing. For this reason, use should not be made of gases containing CO. The workpiece to be boronized is placed in the retort, which is then flushed with the protective gas in order to expel the oxygen.

5.4 PASTE BORONIZING:

Boronizing pastes have a liquid to pasty consistency and contain an organic binder in addition to the dispersant. They are applied to the workpieces by one-time or repeated dipping, spraying or brushing. The paste must be thoroughly dry before the workpieces are heat-treated. Drying can be accelerated with the aid of a drying oven. Paste boronizing likewise necessitates the use of a protective gas. Afterwards, the paste is removed by means of blast cleaning, brushing or washing.

5.5 PROCESS CHARACTERISTICS:

Bodycote uses EKabor* powder as the boronizing media. Parts to be treated are placed in intimate contact with the EKabor powder and then placed in a furnace. The parts are heated to the range of 1500° F - 1800° F under a protective atmosphere and held for a predetermined time. The length of time the parts spend at high temperature depends on the material and the layer depth required. Times at temperature range from one to eight hours. Parts are then cooled to room temperature and the Ekabor powder removed.

Surface growth due to boronizing generally runs 15% to 30% of the layer depth per surface. This growth varies with material but is predictable and repeatable for a given part geometry and treatment cycle.

5.6 BENEFITS:

1. Increased tool and mold life
2. Good resistance to abrasive, sliding and adhesive wear
3. Reduced use of lubrication
4. Can be polished to a high finish
5. Reduced tendency to cold weld
6. Low coefficient of friction

5.7 PROPERTIES OF BORIDE LAYERS:

The hardness of boride layers produced on carbon steels exceeds that of hard chrome electroplate, hardened tool steels and tungsten carbide. The hardness of boride layers on steel ranges between 1800 and 2100 HV (Vickers Hardness). Hardness of boride layers varies with material. Boride layers also retain hardness up to subcritical temperatures (i.e. 1200° F).

Boride layer depths range from .0005" to .005", depending on material and application with the layer depth being matched to the intended application:

- Thick layers (Up to .005"): for abrasive wear.
- Thin layers (Up to .001"): for adhesive wear and friction reduction.

5.8 BORONIZING AGENTS:

The only boronizing agents of industrial importance are those based on activated boron carbide. They can be purchased as fine powders, as grains of various sizes, and as pastes. The Ekabor™ boronizing agents supplied by BorTec are listed in the following table. The main constituents are the boron donor, the activator and the filler.

Boronizing agent	Grain size	Comment
EKABOR™1	<150µm	Highest-quality surface layer; tends to bond
EKABOR™2	<850µm	Very good surface layer; the part is easy to unpack after treatment
EKABOR™3	<1400µm	Good surface layer; powder still has good flow properties after treatment
EKABOR™HM	<150µm	For hard metals, small bore sizes and thick boride layers; very good surface layers
EKABOR™Paste	.	Universal application: immersion, brushing and spraying
EKABOR™Ni	<150µm	For boronizing nickel-based materials.

TABLE 5.1. BORONIZING AGENTS

5.9 FEATURES:

1. High hardness of 1600-2800 HV can be achieved on steel alloys.
2. Hardness is retained up to subcritical temperatures (i.e. 1200° F).
3. Heat treatable materials can be fully hardened after boronizing to optimize performance.
4. Boronizing increases resistance to acids, Hydrochloric acid in particular.
5. Boronizing can be selectively applied to only the area that requires hardening.
6. A broad range of materials can be treated, including ferrous metals, some super alloys and sintered carbides.
7. Boronizing can be uniformly applied to irregular shapes.

5.10 APPLICATIONS:

1. The upper and lower dies of pressing tools used to produce ceramic crucibles. Die life of AISI O2 material increased three times by boronizing.
2. Grinding disks for commercial coffee roasting plants. Life increased five times over the nonboronized 1042.
3. Grooved drums (thread guides) for textile machinery.
4. Diesel engine oil pump gears. AISI 4140 boronized then hardened to 52-55HRC to reduce adhesive wear and friction.
5. Feed water regulating valve. AISI 410 materials boronized and heat treated

lasted nine times longer than nonboronized component.

6. Stamping dies subject to adhesive wear. AIS D2 material selectively boronized to reduce cold welding increased life of the dies eight-ten times.
7. Extrusion and injection molding augers, barrels and die components to reduce friction and increase service life.

5.11. H13 TOOL STEEL

International Mold Steel's Premium H-13 is a chromium-based, hot work die and plastic mold steel designed for increased production rates and longer tool life. It meets the restrictive standards of the North American Die Casting Association and other industrial specifications such as Chrysler's NP2080 for die casting applications. International Mold Steel's Premium H-13 is used for extended production runs that focus on reliability and consistency in the die or mold steel to minimize costly downtime.

The manufacture of this die and mold steel begins with exacting chemistry control including very low sulfur level to increase toughness properties and resistance to heat checking. It is remelted to optimize steel integrity followed by special thermal processing for the development of homogeneous microstructure with uniformly distributed carbides. This provides the die caster or molder with outstanding ductility throughout large sections, which is essential to resist cleavage cracking under more demanding operating conditions.

5.12 UNIQUE CHARACTERISTICS:

1. Thermal shock and fatigue resistant
2. Meets NADCA, Chrysler and Mold Quality specifications
3. Superior machinability and polishability

5.13 CHEMICAL COMPOSITION:

Chemical composition (% by weight) of Critical Alloying Elements and Impurities (ASTM A-681 sec. 6)

ELEMENT	MIN.	MAX.
CARBON	0.37	0.42
MANGANESE	0.20	0.50
PHOSPHORUS	0	0.025
SULFUR	0	0.005
SILICON	0.80	1.20
CHROMIUM	5.00	5.50
VANADIUM	0.80	1.20
MOLYBDENUM	1.20	1.75

TABLE 5.2 CHEMICAL COMPOSITION OF H13

5.14 SS 316 L STEEL:

Type 316 is an austenitic chromiumnickel stainless steel containing molybdenum. This addition increases general corrosion resistance, improves resistance to pitting from chloride ion solutions, and provides increased strength at elevated temperatures. Properties are similar to those of Type 304 except that this alloy is somewhat stronger at elevated temperatures. Corrosion resistance is improved, particularly against sulfuric, hydrochloric, acetic, formic and tartaric acids; acid sulfates and alkaline chlorides.

Typical uses include exhaust manifolds, furnace parts, heat exchangers, jet engine parts, pharmaceutical and photographic equipment, valve and pump trim, chemical equipment, digesters, tanks, evaporators, pulp, paper and textile processing equipment, parts exposed to marine atmospheres and tubing. Type 316L is used extensively for weldments where its immunity to carbide precipitation due to welding assures optimum corrosion resistance.

5.15 COMPOSITION:

Composition	Type 316 %	Type 316 L %
Carbon	0.08 max.	0.08 max.
Manganese	2.00 max.	2.00 max.
Phosphorus	0.045 max.	0.045 max.
Sulfur	0.030 max.	0.030 max.
Silicon	0.75 max.	0.75 max.
Chromium	16.00 - 18.00	16.00 - 18.00
Nickel	10.00 - 14.00	10.00 - 14.00
Molybdenum	2.00 - 3.00	2.00 - 3.00
Nitrogen	0.10 max.	0.10 max.
Iron	Balance	Balance

TABLE 5.3. COMPOSITION OF SS316L

CHAPTER 6

SPECIMEN PREPARATION

6.1 SPECIMEN PREPARATION:

6.1.1 TESTED SPECIMEN OF H13:

The required specimen was tested by spectro analysis test to confirm the H13 tool steel.

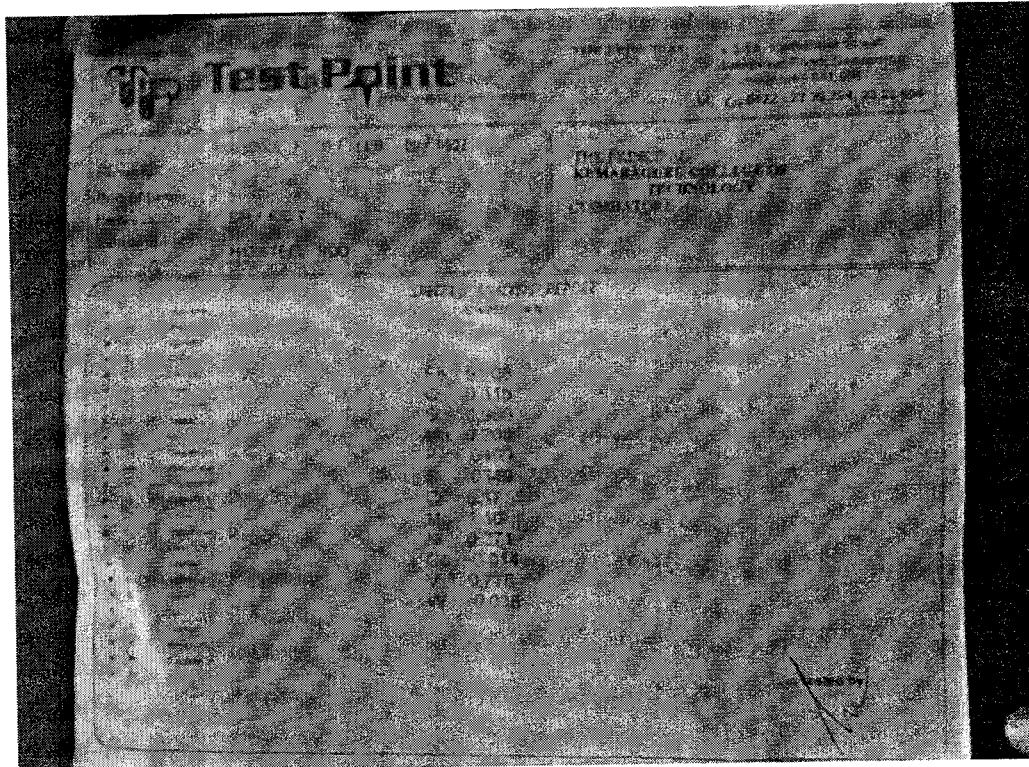


FIG 6.1 TESTED COMPOSITION OF H13

Tested specimen

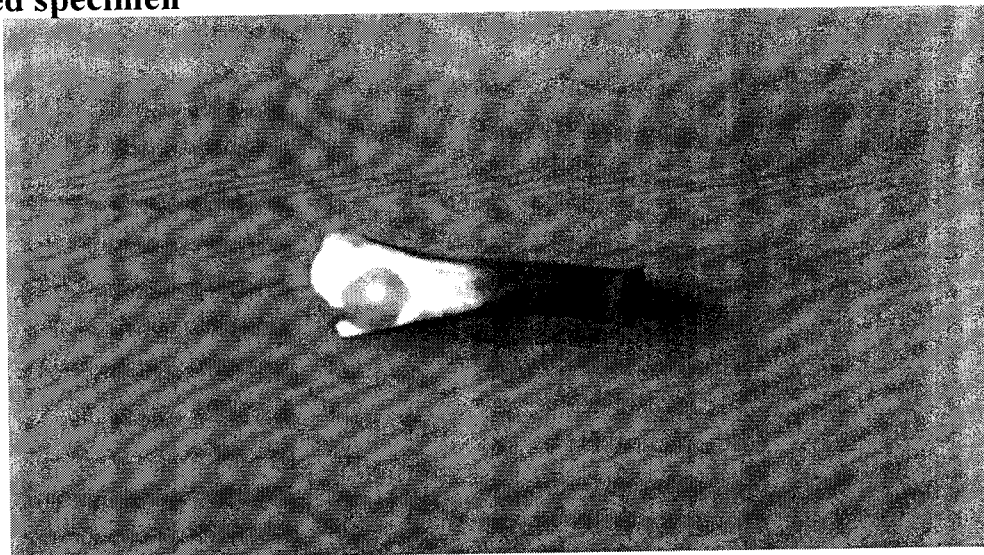


FIG 6.2 TESTED SPECIMEN

6.2 LATHE WORK:

The H13 steel rod is the main raw material of our specimen . we have taken the H13 steel rod and our specimen length is 25mm so we took the H13 steel rod to the lathe shop for making our specimen.Under the guidance of the lab assistance we made our specimen . first the h13 steel rod is cut into 25mm length . we need to make our specimen perfect so, we have done facing operation on our 25mm length specimen and then it is allowed to undergone a turning operation so we could get our required diameter and a clean surface.finally our specimen is run into a chamfering operation.so the edges of our specim would be clean and perfect.this operation is done on seven specimens.same operations are made on 15mm length specimen also.Here also seven specimens are prepared and the operations are allwed to run on the specimens.

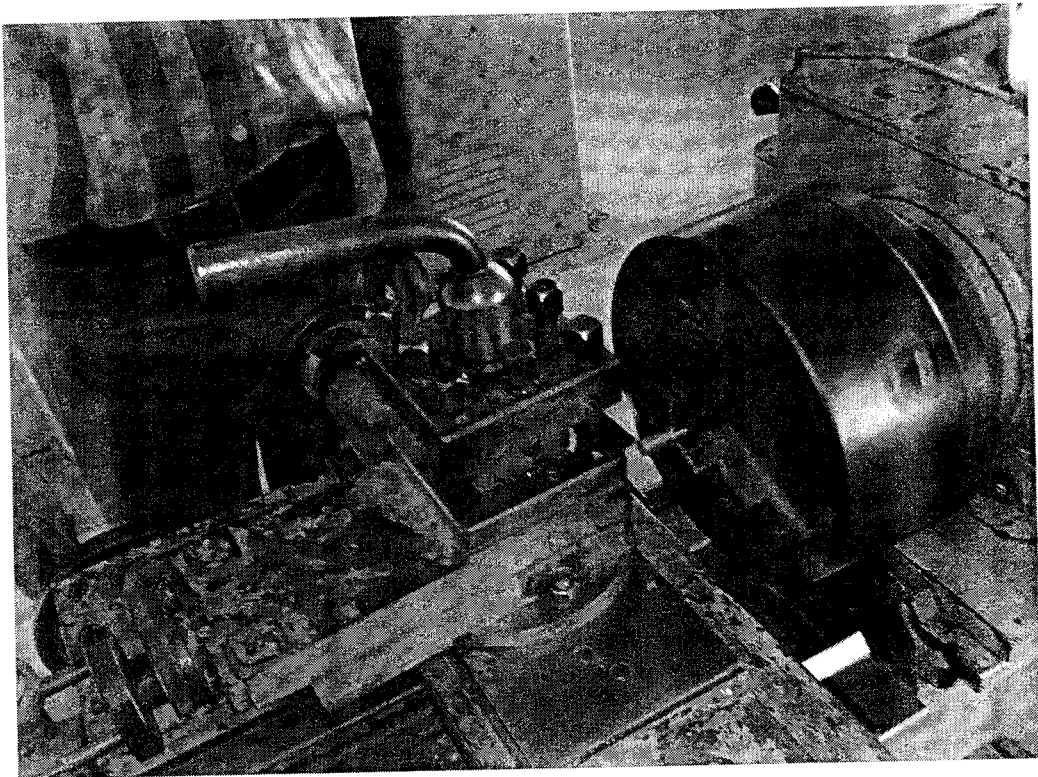


FIG 6.3 LATHE WORK

The prepared specimens are shown below.

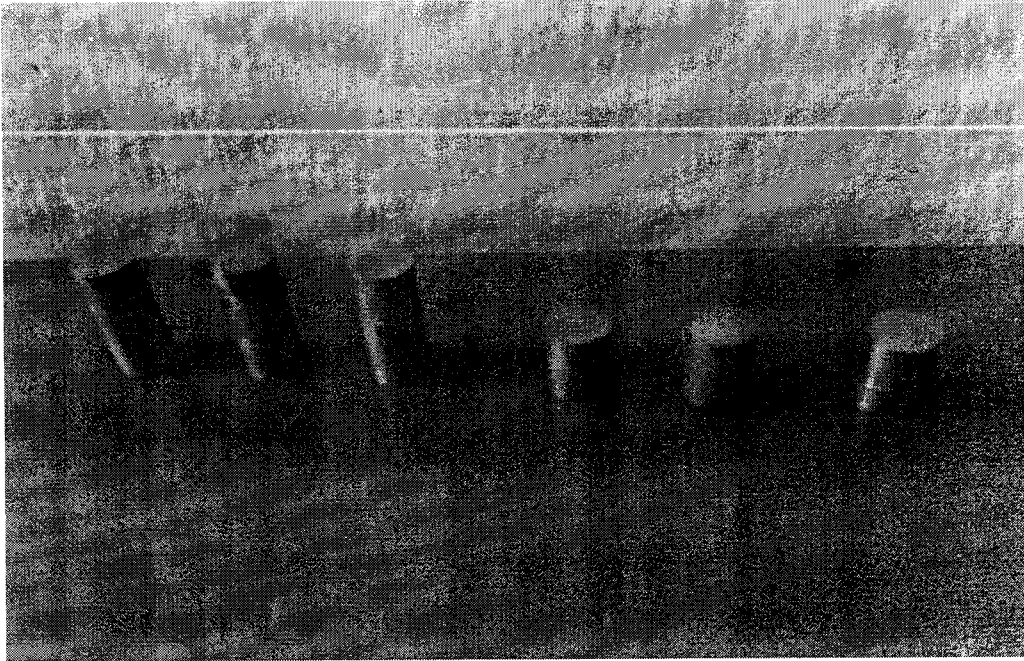


FIG 6.4 SPECIMEN 1

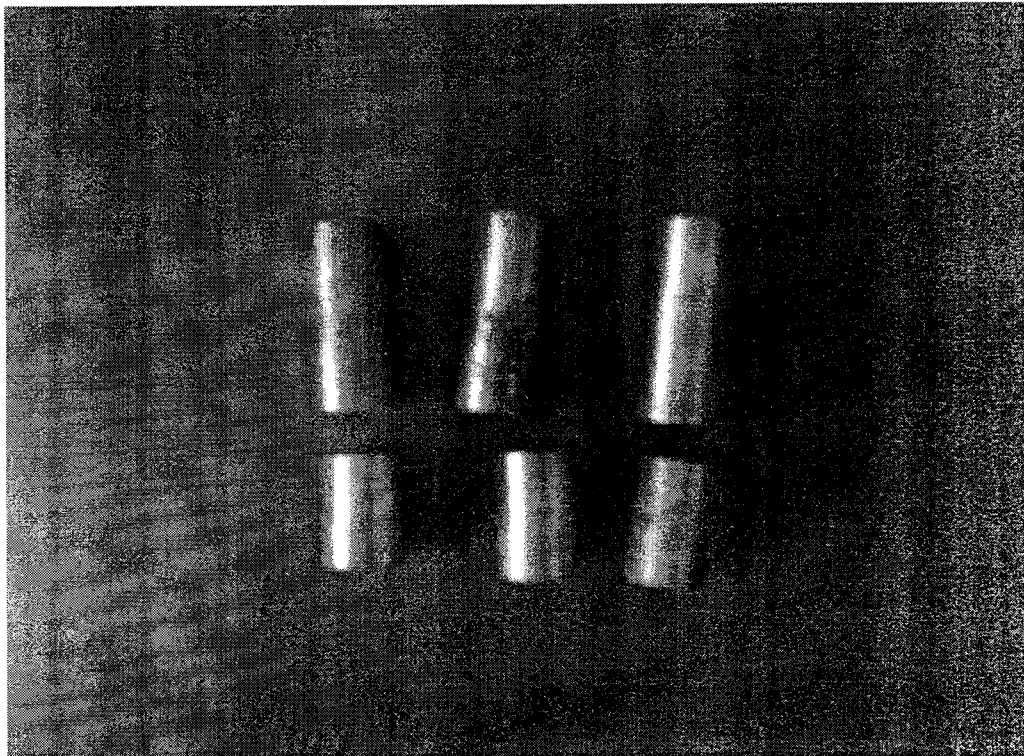


FIG 6.5 SPECIMEN 2

6.3 SPECIMEN CLEANING:

The specimen was cleaned using

1. With Emery sheet
2. With Polishing machine

6.3.1. With emery sheet:

The specimens will be rough and will not be in finishing condition. We need a clean and good surface finished specimen, so the specimens are firstly cleaned with the emery sheets. we used five grades of emery sheets. The used grades are 220, 320, 400, 600, and 800. first the specimen is thoroughly cleaned with the 220 and 320 emery sheets and at the finishing touch we used the 400, 600 and 800 grades emery sheets. This gives a good condition specimen. Thus we got a semi-polished specimen.



FIG 6.6 WORK WITH EMERY SHEET

6.3.2 WITH POLISHING MACHINE:

This is the second stage of the cleaning process. Initial stage of emery sheet was explained above and this was about the polishing machine. In the polishing machine the polishing cloth was inserted in the rotatable polishing machine. The polishing cloth was fixed and it is allowed to rotate in a normal speed. Before placing the specimen in the polishing machine ,pour some water in the polishing cloth and spray the polishing powder ,so that the powder would stick into the polishing cloth. Then slowly and carefully the specimen is placed in the rotating polishing machine .this is done on the surface of the specimen. Then the slowly rotating polishing machine is rotated in little bit of high speed .the speed should be increased and stopped. This operation should be done on all the seven specimen .after this operation we got a cleaned and a perfect conditioned specimen.

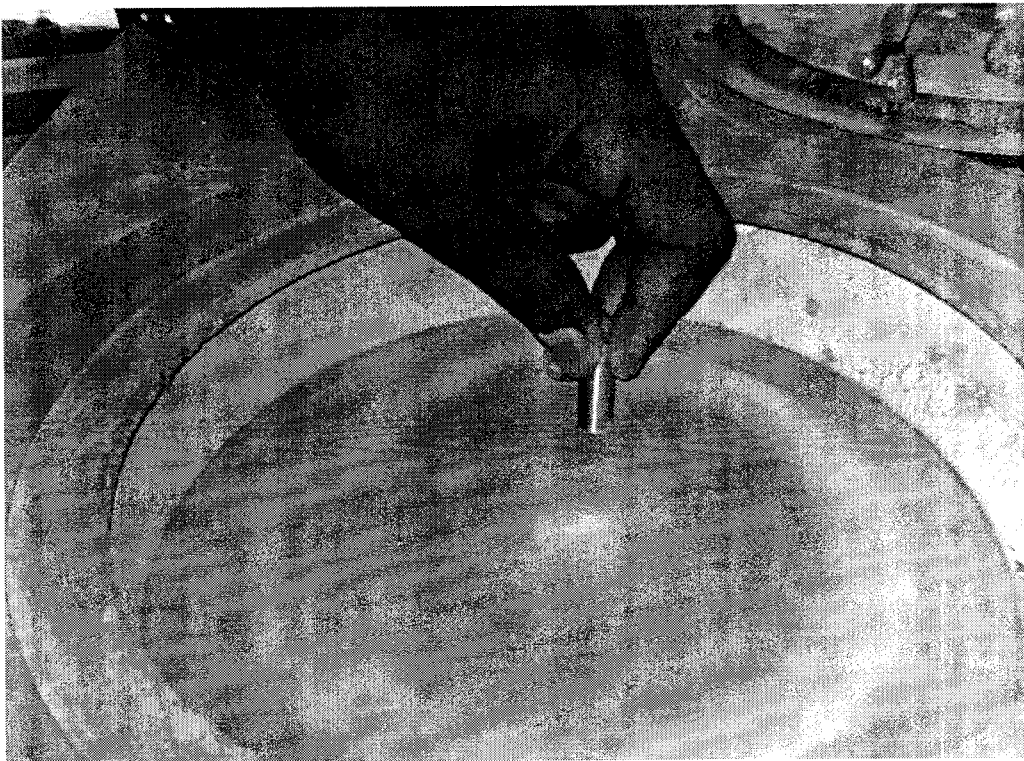


FIG 6.7 WORK WITH POLISHING MACHINE

CHAPTER 7

BORONIZING TREATMENT

7.1 BORONIZING TREATMENT:

7.1.1 BORONIZING:

Boronizing is a thermo chemical surface hardening process in which boron atoms are diffused into the surface of metal work piece to form a hard layer of metal borides with the base material. When applied to the appropriate materials, boronizing provides wear and abrasion resistance comparable to sintered carbides.

The boronizing treatment was done by two types of thermal treatment. That two types are known as,

1. Continuous boronizing
2. Interrupted boronizing

7.2 THERMAL TREATMENT

7.2.1 CONTINUOUS PROCESS

The assembly shown in Figure 1 was loaded in the furnace at around 1073 K, and it was further heated to the boriding temperature (1223 K). When the temperature reached 1223 K, boriding was performed continuously for 4 hours. After 4 hours, the crucible was removed from the furnace and allowed to cool in still air. In the case of the molten salt process, the sample to be borided was kept in the boriding melt and boriding was carried out at 1223 K for 4 hours continuously. After 4 hours, the sample was removed from the melt and allowed to cool in air.

The experiment conducted on the continuous boronizing was shown below.

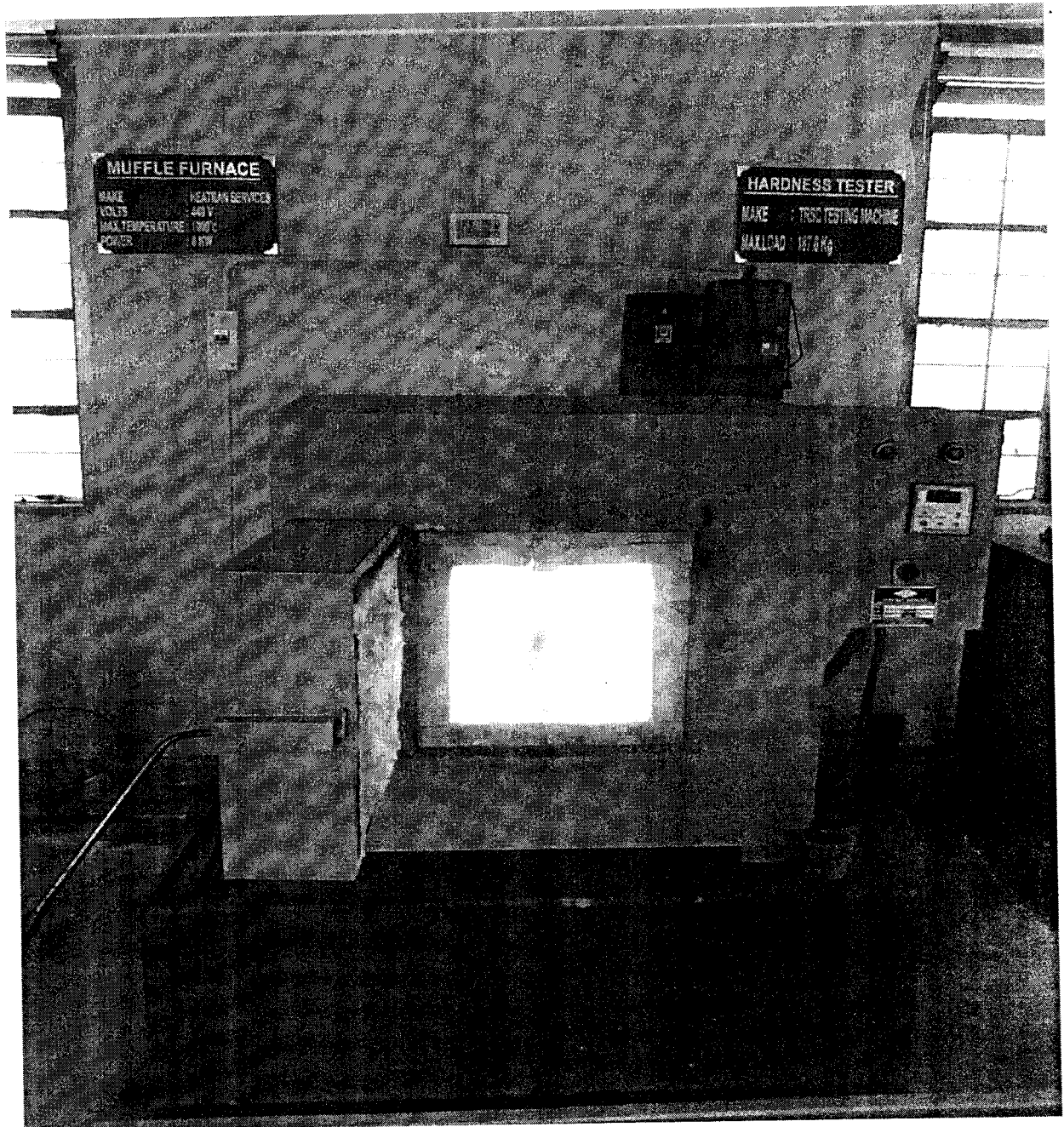


FIG 7.1 CONTINUOUS BORONIZING

7.2.2. INTERRUPTED PROCESS

In interrupted boriding process, after every hour of boriding, the crucible (with the stainless steel box containing the boriding mix and sample) was removed from the furnace and allowed to cool in still air. During cooling, a thermocouple was inserted in the sand so as to touch the stainless steel box. When the temperature was about 873 K, the crucible was again loaded into the furnace at 1223 K. Boriding was done for 1 hour at 1223 K, and the sample was cooled again to about 873 K. This procedure was repeated four times. After the interrupted boriding process (four steps of 1 hour each), the crucible was removed from the furnace and kept at 873 K for 1 hour in a separate furnace. It was then cooled in air to an room temperature.

The experiment conducted on interrupted boronizing process was shown below

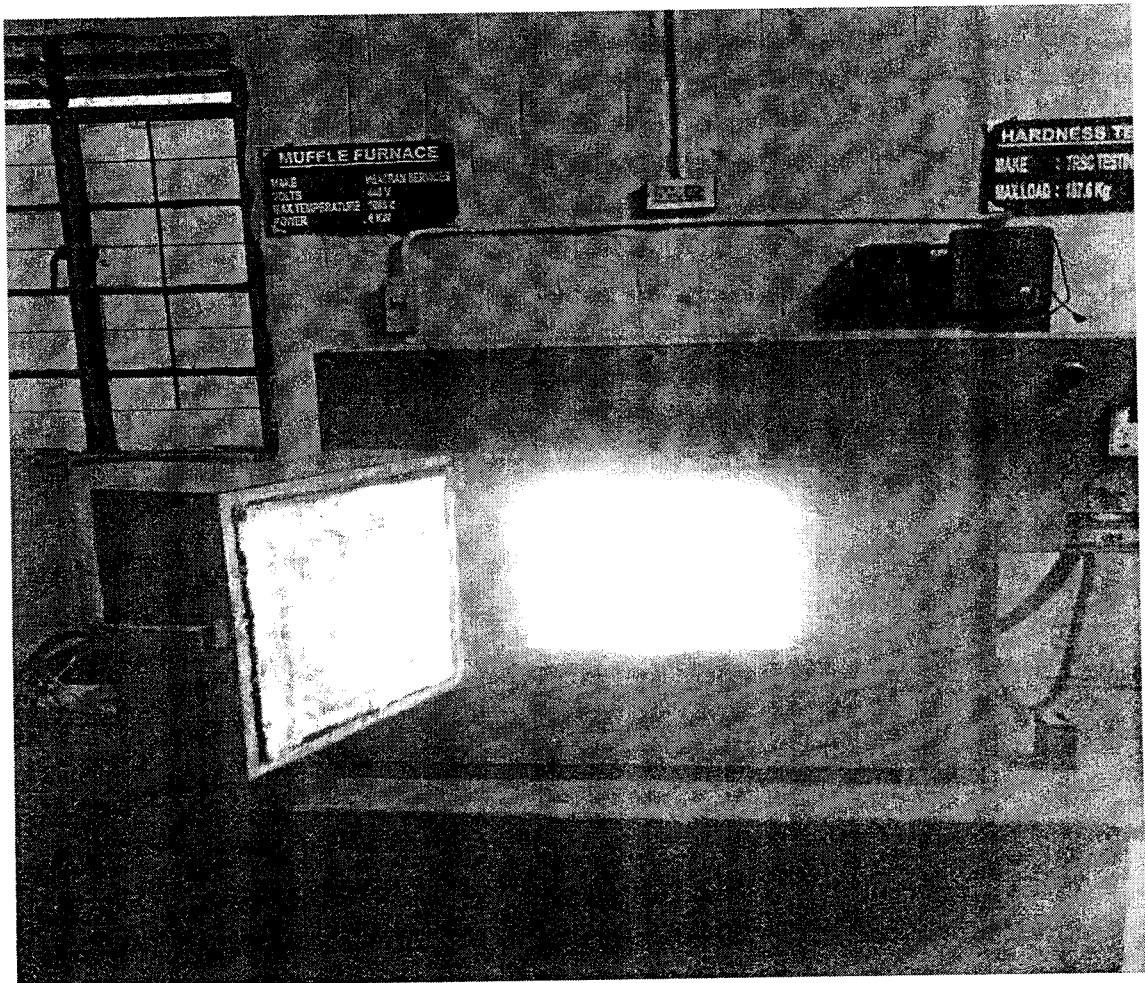


FIG 7.2 INTERRUPTED BORONIZING

CHAPTER 8

RESULTS & DISCUSSIONS

8.1 HARDNESS TEST:

The hardness test was done on the Rockwell Hardness Testing machine. By using the testing machine we could find the hardness of our specimen. Firstly the specimen is placed on the testing machine. Above there is a holder in which an indenter is placed. In the indenter a diamond tip is at the edge and this is the main thing of the testing part. A load of 150 was given in the testing machine. Initially the diamond tip is allowed to touch the surface of the specimen there will be reading thing inside it there will be a needle. The needle will start rotating when the indenter touches the specimen surface. The needle should be allowed to stop by its own. Then the 150 load is unloaded. When unloaded the needle in the reader starts to rotate and it will stop after some time. Then the load is gradually applied. During this load application the needle would rotate and at the end of the load application the needle would show the reading. This is Rockwell hardness test of the specimen.



FIG 8.1 HARDNESS TESTING

After Rockwell hardness test the specimen

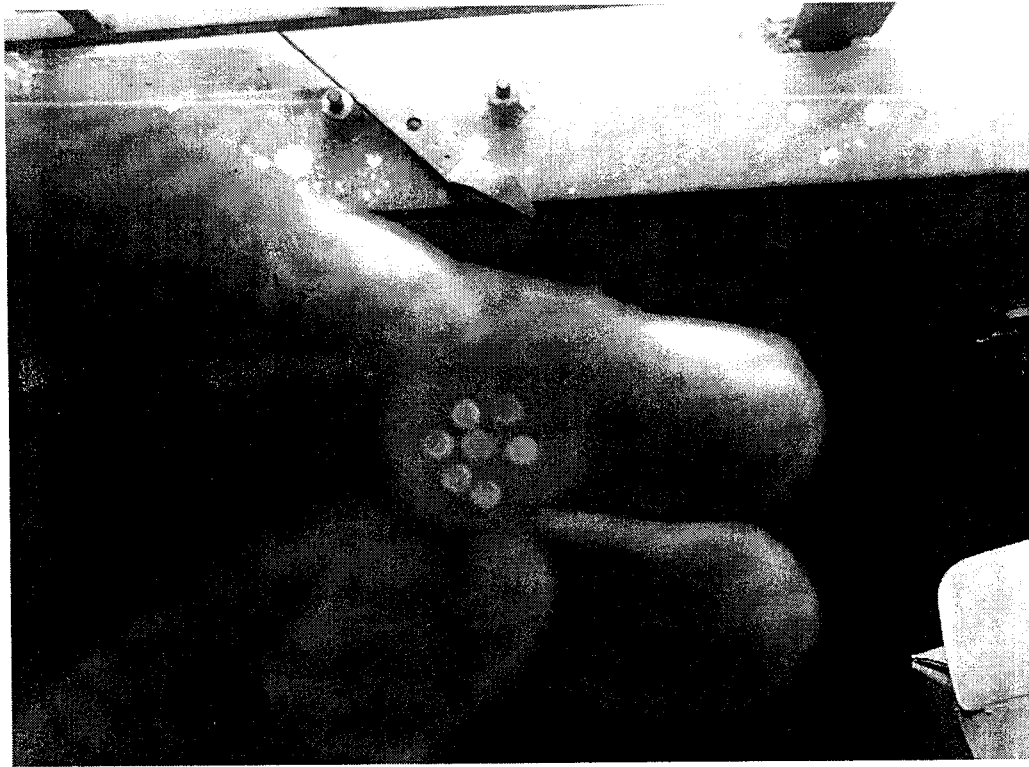


FIG 8.2 HARNESS TESTING SPECIMEN

8.2 RESULT:

PROCESS	Hardness of Untreated Specimen (H13)	Hardness of Treated Specimen (H13)	Hardness of Untreated specimen (SS316L)	Hardness of Treated specimen (SS316L)
Continuous (950 °C)	45 HRC	101 HRC	79(HRC)	170(HRC)
Interrupted (950 °C)	45 HRC	108 HRC	79(HRC)	192(HRC)

TABLE 8.1. RESULT

8.3 EFFECT OF ROCKWELL HARDNESS ON H13 STEEL:

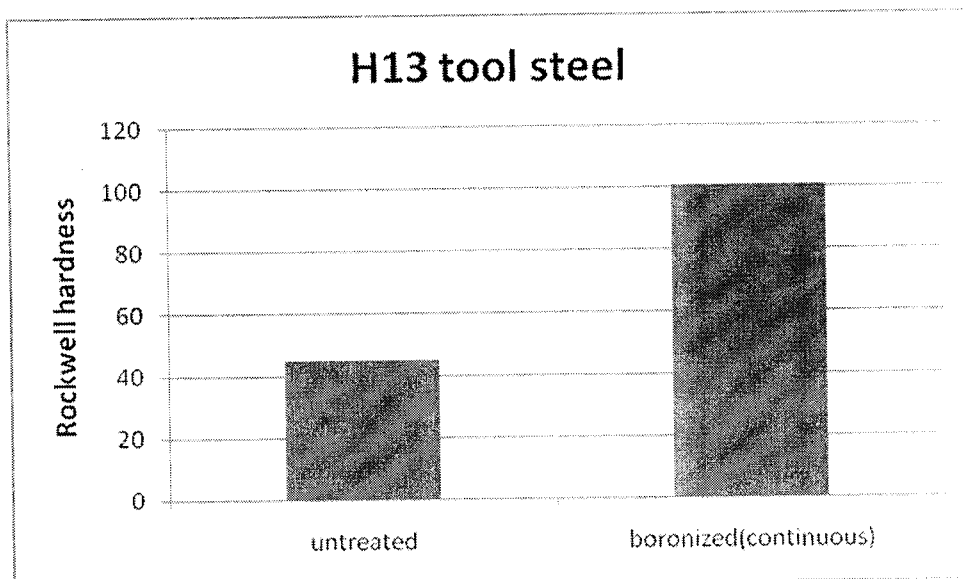


FIG 8.3 GRAPH OF CONTINUOUS BORONIZING (H13)

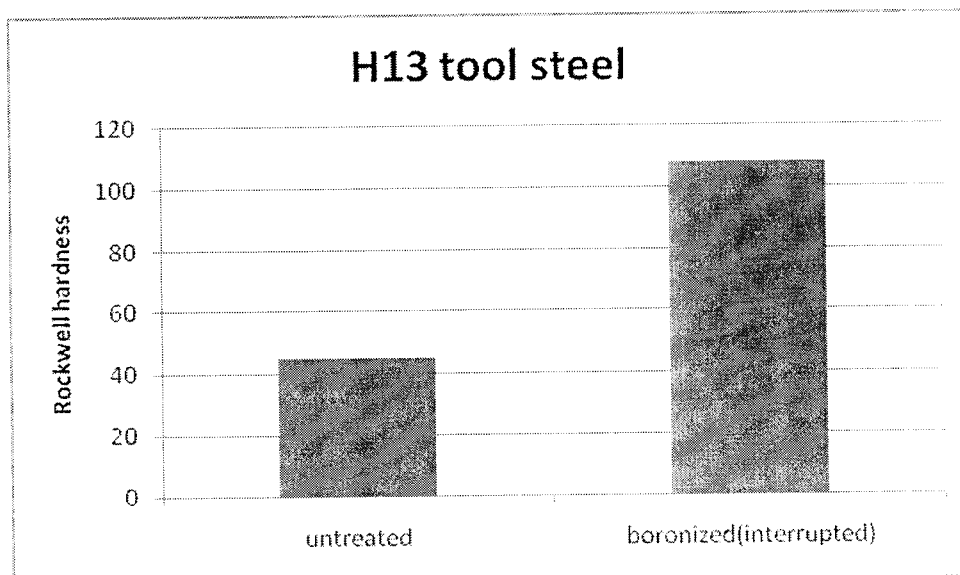


FIG 8.4 GRAPH OF INTERRUPTED BORONIZING (H13)

EFFECT OF ROCKWELL HARDNESS ON SS316L STEEL:

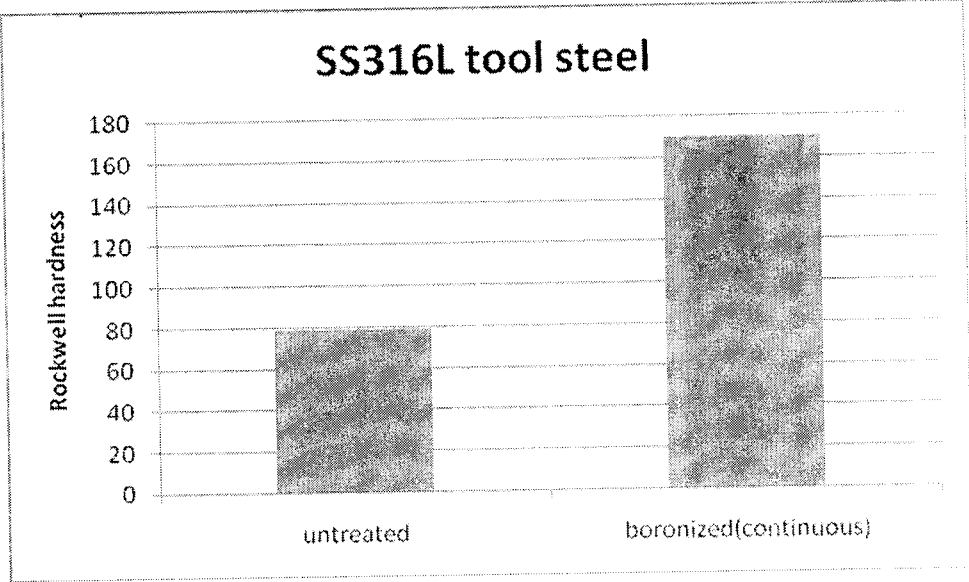


FIG 8.5 GRAPH OF CONTINUOUS BORONIZING (SS316L)

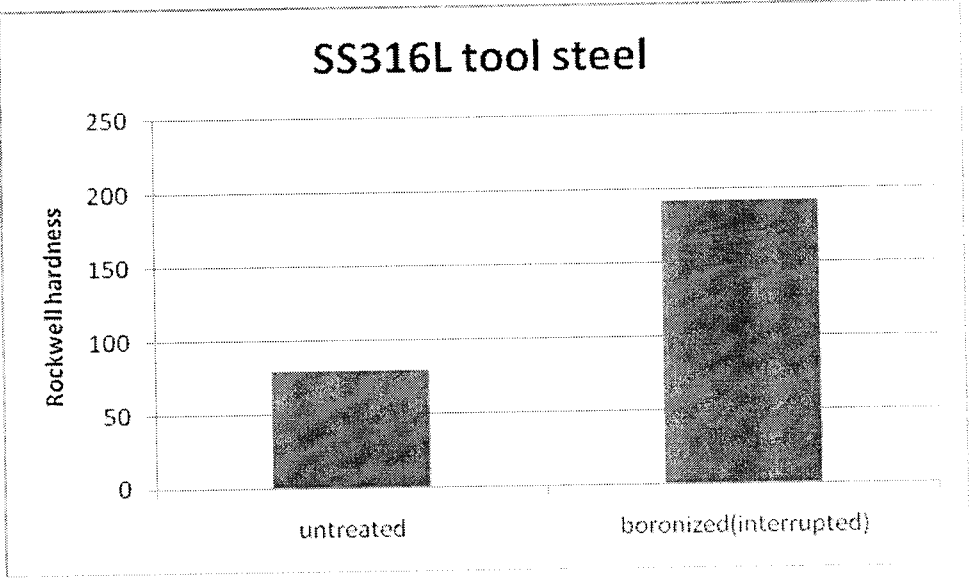


FIG 8.6 GRAPH OF INTERRUPTED BORONIZING (SS316L)

CHAPTER 9

FUTURE WORK

This is project we have done and we could continue many experiments such as

1. Micro hardness
2. Wear test
3. Corrosion test
4. Microscopic observations

9.1. MICROHARDNESS MEASUREMENTS

Microhardness measurements were carried out at 50 g load using a Mitutoyo microhardness tester. The cross section hardness profiles were measured on at least three locations along the case, and the average value was taken.

9.2. WEAR TEST

9.2.1 Pin-on-disc—adhesive wear test

Wear tests were performed using a pin-on-disc machine (Ducom TR20) under unlubricated dry sliding conditions. The disc material was EN24 steel in the hardened condition (65 HRC). The surface roughness of the disc is of fine machined quality. Both the unborided and borided specimens were tested. During the test, the ambient temperature was 303 K. The speed of the disc was 0.6 m/s. The duration of the test was 2.5 hours for each specimen. The 'Z' displacement of the pin under test was monitored using a precision linear variable differential transformer, and the wear losses of the borided and unborided specimens were compared.

9.3. CORROSION TEST

A copper chloride accelerated salt-spray corrosion test (also termed the CASS test) was chosen to study the corrosion behavior of the steels. This system should normally employ a salt-spray chamber, but an immersion technique was used as an approximation. 2.6 g of cupric chloride in one liter of glacial acetic acid was mixed thoroughly in a beaker, and the treated and untreated samples were subjected to the corrosion test for a period of 64 hours. After 64 hours, the samples were washed in running water and acetone and then dried. The loss in weight of the treated sample and the untreated sample were measured using a chemical balance, and the corrosion rate in mg/dm²/day was calculated for both the treated and untreated samples.

9.4. MICROSCOPIC OBSERVATIONS:

One flat end of the borided specimens was ground gently to remove the boride layer. Then, the samples were mounted in cold mounting material and prepared for metallographic study using various grades of emery papers. Final polishing was performed using diamond paste, and etching was carried out using equal quantities of nital and picral. The microstructure of the borided samples was studied and photographed using a Nikon-Epiphot optical metallograph. The metallographically polished specimens, as well as the fractured surfaces of the tension test specimens, were also observed using a JEOL* JSM T100 scanning electron microscope at suitable magnifications.

CHAPTER 10

REFERENCES

1. Interrupted Boriding of Medium-Carbon Steels by P. GOPALAKRISHNAN, P. SHANKAR, M. PALANIAPPA, and S.S. RAMAKRISHNAN
2. Mechanism of thermochemical growth of iron borides on iron by C. MARTINI, G. PALOMBARINI
Institute of Metallurgy, University of Bologna, Italy
E-mail: pal@bomet.fci.unibo.it
- M. CARBUCICCHIO
Department of Physics, University of Parma, and INFM, Parma, Italy
3. Abrasive wear resistance of boronized carbon and low-alloy steels by M.A. B'ejar , E. Moreno
4. Influence of Different Surface Treatments of H13 Hot Work Die Steel on Its Thermal Fatigue Behaviors by MIN Yong-an , WU Xiao-chun , XU Luo-ping , TANG Wen-jun , ZHANG Shuang-ke
School of Materials Science and Engineering, Shanghai University, Shanghai 200072, China
G. Wallner , David Liang, FENG Ying-yu
Uddeholm Tooling AB, Sweden
5. J. Subramanyam: *Met. News*. 1986, vol. 8, pp. 3-7.
6. C.H. Faulkner: *Advanced Materials and Processes*, vol. 4, 1999, p. H 43.
7. N.G. Kaidash and V.I. Pokhmurskii: *Sov. Mater. Sci.*, 1965, vol. 1, pp. 487-89.
8. V.I. Pokhmurskii, R.G. Vagula, Y.S. Gribovskii, and V.S. Zamikhovskii: *Protective Coatings on Metals*, Plenum Publishing Corp., New York, NY, 1973, vol. 5, pp. 236-39.
9. C. Badini, C. Gianoglio, and G. Pradelli: *Metall. Italiana*, 1987, vol. 79 (10), pp. 747-56.
10. D.N. Tispas and J. Rus: *J. Mater. Sci. Lett.*, 1987, vol. 6, pp. 118-20.
M.R.S. John and A.F. Sammels: *J. Mater. Sci. Lett.*, 1981, vol. 16, pp. 2327-29.
11. Y.A. Alimov: *Protective Coatings on Metals*, Oxonian Press Pvt. Ltd. New Delhi, 1986, vol. 8, pp. 83-85.
12. D.N. Tispas, J. Rus, and H. Noguerra: *Conf. Proc., Heat Treatment '87*. The Institute of Metals. London, May 11–15, 1988, pp. 203-10.