

GAS CARBURIZATION OF LOW CARBON MOLYBDENUM STEELS

By

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Dhaka, Bangladesh



#92537#

DECLARATION

This is to certify that this research work has been carried out by the author under the supervision of Dr. Md. Mohar Ali, Professor, Department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology (BUET), Dhaka and it has not been submitted elsewhere for the award of any other degree or diploma.

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In the Name of
ALLAH
The Most Beneficent
The Most Merciful

ABSTRACT

Gas carburization of four 0.13 percent carbon steel samples containing about 0.48% molybdenum and about 1.26% nickel singly and in combination has been carried out in a natural Titas gas atmosphere at a temperature of 950°C and at a gas pressure of about 15 psia for four different time periods ranging from 1 hour to 4 hours followed by slow cooling in the furnace. The microstructures of these steel samples were studied by optical microscopy and the case depths and the austenite grain size of the case were determined.

Another two sets of specimens of the above steels were carburized for different periods of time under identical conditions of temperature and pressure and quenched in 10 percent brine from the carburizing temperature of 950°C after pre-cooling them to 860°C in the furnace. One set of the quenched specimens was tempered at a low temperature of 160°C. The other set of the quenched specimens was sub-zero treated in liquid nitrogen at -195°C followed by tempering at the same temperature. The microstructure of the heat treated specimens was studied by optical microscopy. The amount of the retained austenite in the hardened cases was investigated by both the optical microscopy and X-ray diffraction technique. Various mechanical properties such as surface hardness, microhardness and wear resistance were also studied systematically by Rockwell hardness testing machine, microhardness testing machine and pin-on-disc type wear testing machine.

The standard Charpy V-notch specimens made of the steel bars were heated at 950°C for 2 hours and quenched in 10 percent brine after pre-cooling to 860°C in the furnace followed by tempering at a low temperature of 160°C and tested for impact energy absorbed in order to assess the toughness of the core of the carburized and heat treated steels.

It has been found that both molybdenum and nickel increase the case depth. Nickel is more effective than molybdenum in increasing the case depth. The combined effect of molybdenum and nickel is much more greater than that of either molybdenum or nickel alone in increasing case depth. The undissolved particles of Mo_2C refine the austenite grain size of the carburized case. Nickel in solution is not found to have any effect in restricting grain growth of austenite. But the presence of nickel enhances the austenite grain size refining effect of molybdenum in the carburized case.

Under identical conditions, molybdenum and nickel enhance the formation of retained austenite in the case of carburized and hardened steels but nickel is more effective than molybdenum in enhancing the formation of retained austenite. In the presence of molybdenum this enhancing effect of nickel is increased. X-ray diffractometry has shown that sub-zero treatment substantially reduces the retained austenite content in the hardened cases of all the steels. Molybdenum and nickel both sluggish the retained austenite transformation to martensite during sub-zero treatment. Molybdenum is more effective than nickel in sluggishing the transformation of retained austenite to martensite. In the presence of nickel this sluggishing effect of molybdenum reduces.

Molybdenum improves and nickel reduces the maximum case hardness and wear resistance of the cases of low carbon steels in the carburized and hardened condition. The presence of nickel decreases the effect of molybdenum in the increment of maximum case hardness. Molybdenum and nickel both increase the core hardness but molybdenum is more effective than nickel in increasing the core hardness. Molybdenum with nickel is more effective in increasing the core hardness than molybdenum without nickel.

Molybdenum and nickel both reduce the toughness of the core of low carbon steels in the carburized and hardened condition. Nickel is more detrimental to toughness than molybdenum. The combined effect of molybdenum and nickel is greater than that of either molybdenum or nickel alone in reducing toughness.

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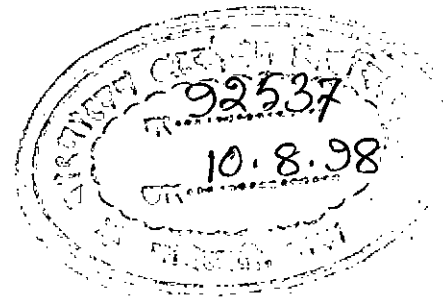
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CHAPTER - 1

INTRODUCTION



Numerous steel products such as gears, shafts, cams, steering parts, bearing and bearing races, etc. are designed for use where the surface of the part requires high hardness and wear resistance while the inside of the part needs to retain high toughness and ductility. Such a combination of properties is not usually possible from the commercial steels as well as conventional heat treatment process of the steels. A low carbon steel containing carbon less than 0.20 percent is sufficiently tough while a high carbon steel containing carbon upto about 1.00 percent possesses adequate hardness and wear resistance. By suitable heat-treatments the high carbon steel can be made very hard while the low carbon steel will develop sufficient toughness. A machine part requiring hard surface but tough core will require a combination of high carbon at the surface and low carbon for the core which on heat-treatment will develop very hard surface and tough core. This can be achieved by saturating the surface layer of a low carbon steel parts by a suitable element such as carbon keeping core composition unchanged. Then the hardness of the surface layers will be increased drastically by employing suitable heat-treatment, while a high impact strength similar to low carbon steel will be retained in the core as it changes very little in hardening.

Gas carburizing is the latest dominant industrial process for surface hardening of low carbon (usually less than 0.20 percent carbon) steel. Gaseous hydrocarbon, cracked gas, natural gas, etc. are used as carburizing medium in gas carburizing. Bangladesh has an enormous deposit of natural Titas gas with a composition of about 97.2% CH₄, 1.8% C₂H₆, 0.5% C₃H₈, 0.2% C₄H₁₀, small amount of higher hydrocarbon and 0.3% N₂.¹ It has been shown^{2,3} that the locally available natural Titas gas is a very encouraging carburizing medium for surface hardening of low carbon steel parts with a uniform case

depth and surface carbon content. From the economical consideration, Titas gas carburization is the cheapest technique for Bangladeshi industries².

Many machine parts used for heavy duty applications such as drive gears, drive shafts, cam shafts, bus and truck gears, crank-shafts, axles, etc. demand increased hardness and wear resistance in the case and better toughness in the core than those of common parts made of low carbon steel in the carburized and hardened conditions. Moreover, advanced aircraft and helicopter require high-duty transmission gear materials which must be able to retain their hardness and strength at elevated temperature upto about 315°C. At a such high operating temperature, ability of material to retain their hardness and fatigue resistance is an important design criterion for high performance gears and rolling element such as those found in helicopter transmissions. Low hardness can results in permanent surface deformation and distress during operation, which lead to a premature failure⁴. On the other hand, improved fuel efficiency for some high consumption vehicles may be achieved by reducing the weight of the vehicle components. For this, the growing trend is to couple the engine with smaller transmissions. Consequently higher stresses are imposed on the transmissions. Such parts made of plain carbon steel do not retain their strength and hardness at elevated temperature. Therefore, the parts used for heavy-duty and high temperature applications are usually made of low carbon low alloy steels containing Ni, Cr, Mo⁴⁻⁶, etc.

Small addition of alloying elements such as Nb, V, Mo, Ti, etc. can strengthen the steel tremendously and there is an additional strengthening effect due to the precipitation of the carbide or carbonitride of the added elements⁷⁻¹⁰. Such steels can retain their hardness and strength at elevated temperature. Thus, the essential prerequisite to fulfil the recent growing trend to have heavy-duty transmission parts, particularly for a voracious fuel-consuming engine operated at elevated temperatures, is to select a high-strength low - alloy steel for carburizing.

The carburizing behaviour of plain carbon steel is well established. Some studies¹¹⁻²⁰ have been already been done on the carburizing behaviour of low alloy steels containing nickel, chromium, molybdenum, vanadium and niobium. But a detailed information is not available about carburizing behaviour of low carbon steels containing Mo. The effect of nickel singly or in combination with Mo on the carburizing behaviour is also not clear. This work, therefore, has been undertaken to study the effects of the addition of about 0.48%Mo and about 1.26%Ni separately and in combination on the structure and properties of the case and core of carburized low carbon (0.13% carbon) steel using locally available natural Titas gas as a carburizing medium.

Machine parts essential for the machineries in various industries in our country are imported from abroad. Recently, Bangladesh government is patronizing the production of spares by using the locally available materials and technology. Thus, a proportion of the required spares for our industries which were imported are being manufactured in our country. Consequently, the dependence on imported spares can be minimized to some extent. Some work of low carbon steel shafts, gears, pinions, rollers etc. and very recently a few alloy steel parts supplied by out side parties has been carburized by this method and hardened in the Department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka. These attempts are undoubtedly encouraging for saving foreign exchange as well as strengthening the national economy:

Natural gas in our country is cheaper than any other carburizing medium. The attempt for gas carburizing of molybdenum steel may help to engineer the surface condition with tough matrix. Thus, gas carburization of molybdenum steel can play an important role in manufacturing of the heavy duty spare parts as per requirements.

CHAPTER - 2

CARBURIZING - A REVIEW

2.1 Carburizing Process

Carburizing is the most satisfactory and widely used method of surface hardening of low carbon steel. It is the process of adding carbon to the surface layer of steel. Since the object of surface hardening is to obtain a hard, wear-resistant surface with a tough core, the first consideration is the selection of a low carbon (usually less than 0.20 percent carbon) steel. It is then subjected to carburization to an extent of eutectoid-hypereutectoid composition and by subsequent heat-treatment to the desired hardness.

There are three general methods of carburization, each differing in technique and carburizing medium. These methods are as follows: (1) Solid or pack carburization, (2) Liquid carburization and (3) Gas carburization. The choice of the method to be followed depends upon the type of case wanted.

Solid or pack carburizing is sometimes used for relatively small parts or when a controlled atmosphere furnace is not available. A box is built to contain charcoal or other carbonaceous material and the part to be treated. Commercial compounds are usually a combination of hard wood charcoal (53%-55%), sized coke (30-32%), barium carbonate (10-12%), calcium carbonate (3-4%), sodium carbonate (2-3%). The part is put into the box and surrounded and covered by the compounds. The box is covered by a lid and is then placed into the furnace. The contents gradually increase in temperature until the furnace temperature is reached. At temperature the charcoal or coke gives off carbon monoxide which surrounds the metal part. As the chemical reactions proceed the free carbon from the carbon monoxide is absorbed by the austenitic microstructure of the steel. This process

continues as long as temperature is maintained or until the metal is saturated with carbon.

The box containing the steel part and charcoal is removed after a predetermined number of hours time has elapsed. It is very hot at austenitizing temperature and must be handled carefully. It is normally allowed to cool to ambient temperature to facilitate removing the part from the box. During the initial time at temperature, the austenite grains increase in size until they become stable. Soaking for hours at temperature also causes an additional increase in grain size. The large grains must be refined before the hardening heat-treatment.

Pack carburizing is very useful for creating small machine parts with widely different properties. It does, however, have some disadvantages that need to be considered before selecting the process. It is time consuming to pack the parts for carburizing, then heat the box, carbonaceous material and the part. It is also difficult to handle the hot box when it is necessary to remove it from the furnace. Time is required for the contents to cool to a temperature where they can be unpacked and removed from the carburizing box. The process make it difficult to quench a part directly following carburizing, if it is desired. Since some of the chemicals such as barium carbonate, hard coke, sodium carbonate, etc. required for pack carburizing are imported and are not available in the local market, pack carburization sometimes becomes a hard proposition to follow. Moreover the box containing the charge when heated gets oxidized and warrant frequent replacement.

In liquid carburization, the compound medium is liquid at carburizing temperature. Parts are immersed in a 850°C salt bath containing sodium cyanide, together with some alkaline earth salts, usually barium chloride or calcium chloride. The case of metals absorbs both carbides and nitrides²¹. The extra hardness in the case reflects the presence of the iron nitrides picked up from the cyanide. The case depth is a function of the time that the part is kept in the salt bath. The steels used with this process are usually quenched directly from the salt bath because of the short treatment time and the lower temperature. Operation of cyanide bath is complex and harmful for the personnel involved in the work due to the toxic

nature of the gas evolved. Regular checking and adjustment of the bath composition is necessary for effective carburization and uniform case depth. In this technique, some shape cannot be handled because they either float or will cause excessive drag out of salts and most of the ingredients for the bath are imported and costly. For the above unavoidable limitations of the liquid carburizing baths, limited practices are observed.

Gas carburization is the latest dominant process and most frequently used by modern industry. It involves same concepts as pack carburizing, but the parts to be carburized are placed in contact with gases rich in carbon monoxide. Hydrocarbons such as methane, propane, butane and natural gas are excellent carburizing sources. These gases are readily decomposed at the carburizing temperature and the atomic carbon thus formed diffuses within the matrix of the heated steel parts. In this method maintenance of proper composition of the carburizing medium is possible because of the continuous flow of gas within the carburizing chambers. This method of carburizing reduces the time and expense of handling and allows ready accessibility to the parts if it is desired to quench the part directly from the furnace. Complicated and large sized machine parts can also be case hardened by this technique. Some plus points such as huge reserve, easy availability of the gas in the local industries and lower cost make this process further encouraging as a carburizing medium in Bangladesh.

Currently some new methods of carburization such as ion carburizing and vacuum carburizing have been developed. A brief description of ion carburizing is given below:

Ion carburizing for case hardening is now becoming accepted in the heat treating industries. Ion carburizing differs from conventional gas carburizing in that it is done in a special furnace at subatmospheric pressure. The furnace atmosphere is natural gas eliminating the need for generated gas. The carbon source is ionized and accelerated to the work due to an electrical potential between the work piece and the surroundings. This is manifested as a glow discharge around the piece. The glow is very uniform, creating a very uniform carbon

profile over the entire surface of the work piece. Since there is no oxidant in the furnace atmosphere (like the CO_2 and H_2O in endothermic gas) there is almost no intergranular oxidation at the surface of the work piece.

Generally, ion carburizing is done at a slightly higher temperature than the atmosphere carburizing. In addition, the glow supplies carbon so effectively that the surface of the work is saturated with carbon for more of the total carburizing time. These combination shortens the cycle time without having a detrimental effect on the product quality. The work is very clean and has less distortion than conventional carburizing.

The ion carburizing glow can penetrate the surface irregularities much better resulting in a more uniform product. Because ion carburizing is not limited by the gases ability to supply carbon to surfaces, it saturates the surface with carbon very quickly. An additional time savings results from a higher effective carbon potential, therefore reducing the time to achieve saturation. Moreover, there is no intergranular oxidation with ion carburizing. There are several good applications for ion carburizing, including gears, steering components, fuel injection components and other small parts that require a rather shallow case (less than 0.05"). Ion carburizing is aimed at the high quality, precisely controlled product.

2.2 Theory of Carburization

The carburization of steel is controlled by the diffusing element in an atomic state, kinetics of the surface reaction and carbon diffusion within the matrix of the steel. The combined effects of time, temperature, atmospheric composition and the carbon concentration on the diffusion of carbon in austenite can be expressed by Fick's law of diffusion. Fick's first law describes diffusion under equilibrium condition and it states that the flux of the diffusing elements perpendicular to a plane of unit cross-sectional area is proportional to the local carbon gradient perpendicular to the plane. Mathematically, it can be expressed as,

$$J_1 = D_1 (dc_1/dx) \quad (\text{Ref. 22}) \quad (1)$$

The constant of proportionality D_1 is the diffusion co-efficient, which has the unit (distance)²/time, J_1 is the resulting flux gradient.

Fick's second law is a material balance within an elemental volume of the system and expresses the non-equilibrium condition of diffusion, where the concentration changes with time as in the given mathematical relationship:

$$dc/dt = (d/dx)D_1(dc/dx) \quad (\text{Ref. 22}) \quad (2)$$

The diffusion co-efficient which describes the diffusion of carbon in austenite varies somewhat with carbon content. The following expression, due to Tibbetts²³, is a good fit to the experimental data.

$$D = 0.47 \exp(-1.6 C) \exp\{-(37000 - 6600 C) / RT\} \text{ cm}^2 / \text{sec} \quad (3)$$

Where, C is the carbon concentration in weight percent and T is the temperature in degrees Kelvin.

Carburizing is a non-equilibrium process, i. e. the gaseous constituents of the atmosphere are not fully in equilibrium with one another and the atmosphere is not in equilibrium with the steel being carburized. Nevertheless, several important reactions for carburizing approach rapidly enough to permit the prediction of equilibrium state. The equilibrium state of the chemical reactions may be represented by numerical constant, K_p , derived from the general expression²⁴:

$$\text{Log } K_p = A/T - B \quad (4)$$

Where, K_p is a numerical factor termed as 'equilibrium constant'. The subscript p denotes the dependency of chemical reaction on pressure, T is the absolute temperature, A and B are constants derived for particular reaction.

Instead of being developed in a formal way from basic principles the mechanism of carburization may be analyzed from the view point may be divided for discussion into two

distinct groups:

- (1) Factors that control the flow of carbon in iron and
- (2) Factors that influence the transfer of carbon to the iron surface.

2.2.1 Flow of Carbon in Iron

In carburizing the carbon atoms dissolved in the austenite of the surface layer of the steel part comprising an interstitial type solid solution. From the iron and iron carbide thermal equilibrium diagram given in Fig.1, it is evident that solubility of carbon in austenite depends on temperature. At a temperature lower than about 910°C , pure iron exists as ferrite, a body centered cubic structure which transforms to austenite, a face centered cubic structure above this temperature. The packing density of ferritic and austenitic iron are 68% and 74% respectively and in both, carbon atom dissolves interstitially. Due to the presence of an atom at the center of ferritic iron, the maximum radius of the foreign atom permissible to be dissolved within it is limited to 0.385\AA . In austenite total void is less than that of ferrite but it can permit the foreign atom to enter into the void having the radius up to 0.52\AA because of the absence of the central atom. So, carbon atom having a radius of 0.77\AA can enter into the interstices of austenite creating lower distortion than that of ferrite^{5,25}. So, carbon solubility in austenite is more than ferrite. At lower temperature f.c.c. iron transforms to b.c.c. iron and its interstitial voids become narrower. Consequently, the solubility of carbon in body centered cubic iron becomes much more limited.

The maximum penetration of carbon into the matrix of steel will depend upon temperature, time and carburizing agents. Since, the solubility of carbon in steel is the highest above upper critical temperature, carburizing takes place readily above this temperature. This higher operating temperature also favors the rapidity of carburization because for every 20 degree centigrade rise in temperature, the diffusion co-efficient becomes double⁵. At higher temperature, the saturating element can transform into nascent state, which is prerequisite for case hardening operation. For the above experimental evidence, it is customary to select

a temperature about 40°C above the A_3 point of the particular steel to be carburized.

The rate of flow of carbon in austenite depends on values for the diffusion co-efficient and characteristics of the concentration gradient. The diffusion co-efficient is in turn a function of temperature and carbon concentration. The diffusion of carbon proceeds from the higher concentration developed from the supply sources to the lower concentration of the case²⁴.

The general form of the carbon gradient is influenced only by the range of concentration from the surface to the core. For a given temperature, the rate of diffusion increases with the increase in carbon concentration.

The diffusion rate increase greatly with the increase in temperature. From experiment, it is evident that the rate of carbon addition in steel at 925°C is about 20% greater than that at temperature 870°C. Due to this positive effect of temperature, the most commonly selected carburizing temperature is 925°C, which permits a reasonable carburizing rate without excessively rapid deterioration of the furnace equipment²⁶. Sometimes the carburizing temperature is raised to 955 or 980°C for the purpose requiring deep case with shorter period. Conversely the shallow case is performed at lower temperature because case depth can be controlled accurately with slow carburizing rate obtained at lower temperature. Various alloying elements such as Cr, Ni, Mo, etc. have a great influence on the activity of carbon dissolved in austenite. This influence is due to the effect of alloying elements in steel on the driving force for surface reactions²⁶. To obtain the true driving force somewhat higher temperature is selected for carburization of alloy steels.

In surface hardening practices, case depth is specified up to a depth below the surface with a definite extent of carbon saturation from which the desired mechanical properties can be achieved on subsequent heat treatment. The effective case depths are usually defined as the distance from the surface to the point where hardness drops to a given level, typically HRC 50 or a case depth having a carbon content of about 0.4 weight percent²⁶. The commonly used term 'the total case depth' is the gradual transition between case and core properties in

most carburized parts. Nevertheless, the case depth is a function of time and temperature. Harries²⁴ has developed a mathematical expression as given below showing the effect of temperature and time on the extent of penetration of carbon for normal carburizing.

$$CD = (31.6\sqrt{t})/(10^{6700/T}) \quad (\text{Ref. 24, 35}) \quad (5)$$

where, $CD = \text{case depth in inch}$

$t = \text{time in hours}$

$T = \text{absolute temperature in degrees Rankine}$

For a specific carburizing temperature, the expression becomes very simple as shown below:

$$CD = K\sqrt{t}$$

where, $CD = 0.025 \sqrt{t}$ for 1700°F

$= 0.021 \sqrt{t}$ for 1650°F

$= 0.018 \sqrt{t}$ for 1600°F

2.2.2 Flow of Carbon from the Supply Source

The primary function of carburizing media is to furnish an adequate supply of carbon to the steel surface. The transfer of carbon from the source of supply to the steel is involved with reaction occurring at this source. The carburizing agent responsible for the actual transfer of carbon is a carbon compound. The extent to which various carbon compounds may supply carbon is related to the conditions that determine the equilibrium with the surface carbon of the steel. The equilibrium composition of the reactants and products is a function of temperature and also of the carbon concentration at the surface.

Low carbon steels when heated to the carburizing temperature, will be completely transformed into austenite. At the carburizing temperature austenite iron is capable of holding in solid solution an amount of carbon approaching the saturation limit of 1.7 percent. When a source of carbon, such as carbon monoxide is brought into contact with the steel in this condition, there will be transfer of carbon from the gas to the steel. There is a

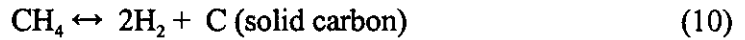
difference of opinion²⁷ as to the exact form of the carbon: some feel that there is an interaction of the carbon monoxide and the iron to form iron carbides, which are then absorbed by the steel, other feel that the carbon being brought to the surface of the metal as carbon monoxide will in the presence of austenite, break down to form atomic carbon, which will diffuse as such in the iron. For our present purpose of discussion let us assume that the action involving the formation, absorption and diffusion of carbon into iron is the essential part of the carburizing process.

In pack carburization, the carburizing compound is in reality an gas producer. Here the carbon monoxide formed is dissociated to atomic carbon and carbon dioxide where the atomic carbon is subsequently absorbed by the austenite. The carbon in the gas carburizing chamber is in the form of gaseous hydrocarbon compound or carbon monoxide. At the carburizing temperature they are dissociated to atomic carbon and the nascent carbon is then absorbed by the steel part which has been explained later in detail. In liquid carburizing, the required atomic carbon is formed by some chemical reactions between the ingredients of the fused salt which is subsequently absorbed by the steel part immersed in the bath.

In gas carburizing, the carbon of the furnace atmosphere is in the form of gaseous hydrocarbon compounds (such as methane, ethane) or carbon monoxide. The principle source of carbon in gas carburizing is methane which is present in natural gas. The reaction involving the actual transfer of carbon occurs at the steel surface (interface) and the reactants which are carbon compounds, may be considered independently. The interface reactions²⁴ for three common carbon compounds may be written :



Considering the reactions that occur for the same carbon compounds, away from the interface, the reactions for deposition of carbon²⁴ are expressed :



The excessive amount of solid carbon or free carbon deposited on the work piece and furnace, is known as soot. Sooting problem can be minimised by mixing the carrier gas with hydrocarbon gas and well judged flow rate from which a reasonable rapid carburizing can be achieved without excessive sooting. In continuous carburizing chamber, soot can be removed by introducing air into the empty chamber to burn it. In the case of batch type operation, the soot can be removed by proper brushing after operation.

If water vapour is present in the gas it will act as a decarburizing agent, removing carbon from steel by the reaction^{24,28}.



The possible gaseous species which may be present in the chamber atmosphere are CO, CO₂, H₂, H₂O, N₂, CH₄ and C₂H₆. For known equilibrium composition of the gases in the chamber, the carbon potential of the carburizing atmosphere can be calculated by the following equilibrium relationship of equation 6:

$$K_1 = (a_c \cdot P_{\text{CO}_2}) / (P_{\text{CO}})^2 \quad (\text{Ref. 26}) \quad (13)$$

where, P_{CO} and P_{CO_2} are partial pressure of CO and CO₂ respectively, a_c is the activity of carbon, K_1 is the equilibrium constant for the reaction. The carbon activity is related to the carbon content of the austenite as in the following expressions:

$$\ln a_c = \ln Y_c + (9167 Y_c + 5093)/T - 1.867 \quad (\text{Ref. 26}) \quad (14)$$

where, $Y_c = (4.65w)/(100 - w)$, T is the temperature in degrees Kelvin, w is the weight percent of carbon in austenite and Y_c is the atom ratio of carbon to iron.

2.3 Atmospheric Conditions for Gas Carburization

Usually the volume of the chamber of gas carburizing furnace is small and the chamber is sealed, which ensure small volume of gas requirement for each working shift. For a pit furnace having a working chamber 25 inches in diameter and 36 to 48 inches deep, the commonly used gas flow rate is 8 to 15 ft³ per hour²⁴. The rate at which the furnace atmosphere responds to changes in inlet gas composition depends on the mean residence time of the atmospheric gases in the furnace. The mean residence time (t_{res}) of the gas in the furnace is,

$$t_{res} = (V.T_a)/F.T_f \quad (15)$$

Where V is the furnace volume, T_f is the absolute furnace temperature and F is the flow rate of the gas measured at ambient temperature T_a . Residence times may be from 2-15 minutes²⁶. For times of this order the reaction in furnace chamber are fast enough so that equilibrium gas composition are approached closely for the usual carburizing temperatures.

For natural Titas gas as carburizing medium the possible constituents of the gas carburizing atmosphere are CO, CO₂, H₂, H₂O, N₂, CH₄ and C₂H₆ gases. For uniformity of carburizing the above gases have to be well circulated within the chamber and the parts being carburized must be well spaced to get contact of carburizing gases. A positive pressure in the furnace should be maintained for effective carburization rate and to prevent infiltration of air within the chamber. For gas carburizing a positive furnace pressure of about 12 to 37 Pa is satisfactory²⁶ and it can be controlled by adjusting the gas flow rate.

2.4 Heat Treatments for Carburized Steels

The final properties of carburized components are obtained as a result of heat-treatment performed after the carburization. It improve the structure and refine the grain of the core and the carburized case, which is inevitably coarsened during the prolonged holding time at the high carburizing temperature. It produce high hardness in the case and good mechanical

properties in the core. Finally, it inhibits the formation of cementite network in the carburized case, which is formed due to carbon addition at a level of hypereutectoid composition²⁹.

The heat treatment of carburized steel is somewhat complex because of the variation of content that occurs in one piece of steel. What is done with the surface must be done with the interior. In spite of this complication, treatments can be given that will produce the desired results without introducing any new metallurgical principles. The heat-treatment process of a carburized steel will depend upon the carburizing temperature employed, the composition of the core and the case, and the purpose for which the part is intended on the properties that must be obtained to meet specifications. Depending upon the carburizing temperature and the carbon content in the case, three heat treatment techniques can be practiced for carburized steels.

2.4.1 Direct Quench Technique

When carbon content of the case does not exceed the eutectoid composition and the carburizing temperature is just above A_3 temperature, the carburized part is quenched directly into a suitable quenchant. This is the very simple and cheapest method of heat treatment, since no heating of the steel is necessary other than tempering to reduce the brittleness of the case and to minimise the danger of cracking. But relatively coarse grained structure at the case and the core, and high proportion of retained austenite at the case is found.

Therefore, direct quenching from the furnace is only resorted to when the work is made of an inherently fine-grained steel and where best properties are not required.

2.4.2 Double Quench Technique

When the carbon content in the case is of hypereutectoid composition, the carburizing temperature is above A_{cm} temperature and the steel part is inherently coarse grained, this

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heat-treatment technique will give the best combination of hard, wear resistance, fine-grained case and a tough, refined core. In this technique, the part is slowly cooled from carburizing temperature and then reheated to above the A_3 temperature of the core followed by quenching in a suitable quenchant. This will produce excellent grain refinement, consequently better toughness for the core. The steel is then reheated to above the $A_{3,1}$ temperature, quenched and tempered which refines the grains of the case sufficient for desired hard and wear resistant surface. This technique has also some shortcomings such as complexity of the procedure, excessive tendency for distortion and cracking and the possibility of oxidation and decarburization of the case. It is the best technique for excellent properties of coarse grained steel, although it is neither the cheapest nor the easiest technique of heat treatment for a carburized part.

2.4.3 Intermediate Technique

In this technique the first reheating above the A_3 temperature in the double quench technique is avoided and the other steps are followed. This process give better properties than that can be achieved from direct quench technique and inferior to that of double quench. Thus, the part can not be used in service under high stress.

2.5 Quenchants for Carburized Steels

The carburized parts are hardened by quenching into a certain medium known as quenchant. The cooling rate for hardening is controlled by the selection of proper quenching medium. There are numerous quenchants such as brine, tap water, salts, soluble oil and water solutions, oil and air, etc. If the part is to be used as heat treated condition without any finishing operation to control dimensional accuracy marquenching²⁶ can be used. In marquenching the part is quenched into molten salts or hot oil keeping the quenchants temperature above the M_s temperature of the case, but below the M_s temperature of the core. This treatment gives a mixed structure of martensite, bainite and ferrite in the core and the case transforms to martensite while it cools in air after leaving the quenchant.

2.6 Heat Treatment of Gas Carburized Steels

In gas carburizing, quenching direct from the furnace, after pre-cooling to 840°C-860°C, is frequently practiced. The surface layer, after heat-treatment, will have a martensite structure with excess carbides. In cases of quenching direct from the carburizing furnace, the heating temperature of the core will be in the vicinity of A_3 . In low carbon steels, austenite cannot be supercooled to the martensite point in quenching from this temperature and the core will have a ferrite and sorbite structure. After heating austenite in alloy steels to a temperature close to point A_{cm} , it can easily be supercooled, without decomposition, to the martensite point. The core, in this case, will acquire a low-carbon martensite structure possessing high strength and ample toughness. After quenching, the part is tempered at a low temperature of 150°C to 180°C to relieve stress. The normal hardness at the surface of the carburized cases is usually within the range of RC 58-64. When alloy steels are subject to single hardening, a large amount of retained austenite will be found in the carburized case. This sharply reduces its hardness. Retained austenite may be eliminated by a sub-zero treatment which transforms its greater part into martensite, thereby substantially increasing the hardness.

2.7 Carburization of Low Alloy Steels

Low carbon steel is usually used for carburizing because of its excellent toughness. Sometimes alloy steels are also carburized for additional properties of both the case and the core in the case of high duty applications. In alloy steels, the alloying elements remain in the form of various carbide and nitride precipitates. To take the precipitates into solution higher temperature is required. For this reason, comparatively a higher carburization temperature is usually selected for alloy steels. Low carbon steels containing nickel, chromium, manganese, vanadium molybdenum etc. singly or in combination usually have sufficient hardenability. From these steels maximum case hardness can be attained by oil quenching and desired core properties up to a tensile

strength of 200,000 psi is obtainable with suitable toughness by proper selection of the steel composition²⁴.

Some works have already been done to investigate the carburizing behaviour of low alloy steel both in home and abroad. It has been reported^{15,16} that chromium increases the case depth whereas nickel decreases it. Both nickel and chromium enhance the formation of the retained austenite but chromium is more effective in promoting retained austenite formation in the hardened case. Chromium increases and nickel decreases the case hardness while both of them increase the core hardness. They also deteriorate the core toughness in the carburized and hardened condition.

It has also been reported²⁶ that chromium bearing steels absorb more carbon than pure iron whereas, nickel bearing steels absorb lower carbon. Thus, carbide is formed in chromium bearing steels at lower carbon potential than that of carbon steels. Carburization of chromium steels followed by heat treatment raises the wear resistance by a factor 1.5 compared to plain carbon steel due to the presence of carbides in the subsurface layer¹⁴.

Alloying element vanadium with or without nitrogen reduces the case depth but vanadium without nitrogen is more effective in reducing case depth¹⁵. Vanadium alone and in combination with nitrogen increase the case and core hardness while vanadium alone reduces the toughness and vanadium with nitrogen improves the toughness¹⁵. Vanadium in solid solution can raise the hardenability³⁰ but as undissolved carbide and nitride it may decrease the hardenability. This variable influence can also be observed for molybdenum and titanium¹¹.

Microalloying element niobium with or without nitrogen reduces the case depth of carburized steels but niobium with nitrogen is more effective than niobium in reducing the case depth^{18,19}. Niobium alone and in combination with nitrogen decreases the formation of retained austenite in the case of the carburized and hardened steels. Niobium

with nitrogen is more effective than niobium in reducing austenite retention^{18,20}.

Niobium with or without nitrogen improves the hardness and wear resistance of the cases of low carbon steels in the carburized and hardened condition. Niobium reduces the toughness of low carbon steels in the carburized and hardened condition but more detrimental to toughness than niobium with nitrogen^{18,20}.

CHAPTER - 3

EXPERIMENTAL

3.1 Materials and Specimens Preparation

Four different steels containing 0.13% carbon were used in this experiment. The composition of the steels are given in Table.1. The steels were made in an air induction furnace. All the melts were teemed at about 1600°C and produced cylindrical sound ingots.

All the ingots were then rolled down to a size of about 16 mm in diameter. Steel 1 is the base steel with which the carburizing behaviour of other steels (2-4) containing Mo and Ni alone and in combination were compared. Specimens having dimension of about 10 mm X 10 mm X 8 mm were machined from the 16 mm dia. bars of steels 1 to 4. A small hole of about 1.5 mm diameter was drilled at one corner of each specimen. The surfaces of these specimens were polished by emery papers up to grit no. 2/0. These polished specimens were used for carburizing.

3.2 The Carburizing Operation

The carburizing operation of the steel specimens was carried out in a gas fired gas carburizing furnace with automatic control system. A schematic diagram of experimental set-up shown in Figure 2. The details of the furnace control system and carburizing procedure are available elsewhere^{31,18}.

The polished specimens of steels 1 to 4 were hung by means of nichrome wire into the carburizing chamber in such a way that the tip of the thermocouple and specimens were as close to each other as possible. The top of the chamber was then closed with a lid. The gas was then made to flow into the carburizing chamber for a while to drive out the air present in it. Then the firing was commenced through two burners housed at the outer surface of the

furnace near its bottom and controller was set for the desired temperature. After commencing the firing the required temperature was achieved within half an hour. The gas flow into the carburizing chamber was maintained at a constant rate of 12 ft³/hr and a flow rator meter was used for this purpose. A signal flame was maintained at the top of a glass jet to ensure the continuous gas flow into the chamber. As long as a moderate flame is visible at the top of the glass jet , the operator can sure that the chamber is full of gas and a proper carburizing environment has been maintained.

The carburization of the specimens was carried out at a temperature of 950°C and at a pressure of 15 psia. The specimens were carburized for 1 hour, 2 hours, 3 hours and 4 hours. After the predetermined time, the firing was stopped and the specimens were allowed to cool in the chamber to room temperature. The gas flow through the carburizing chamber was maintained during the cooling period until the temperature in the chamber came down to about 450°C. This was done as a measure of precaution to prevent any possible oxidation due to infiltration of air. After cooling the specimens were taken out of the chamber for required investigations.

3.3 Optical Microscopy and Measurement of Case Depth of the Carburized and Furnace Cooled Steels

The carburized samples were cut into two pieces at right angles to the carburized surface by using a silicon carbide disc cutter with copious flow of water. One piece of each specimen was then mounted and polished by standard metallographic techniques and etched in 2% nital. The microstructures of these specimens were examined by optical microscope and photographs of the microstructure of each specimen were taken using a photomicroscope.

Attempts were made to find the extent of carburization i.e. the effective case depth. The case depth is usually specified as the depth of carbon penetration below the surface at which a definite value of some property occurs. In this work, a case depth corresponding to a carbon content of 0.4 weight percent was considered for an effective case depth following

Stickles²⁶. Then the effective case depth was measured linearly from the microstructure of these specimens by using a micrometer eye piece fitted to a Shimadzu optical microscope.

3.4. Determination of Austenite Grain Size of the Carburized Case of the Steels

The microstructure of the case of the carburized steel consists of three zones namely, hypereutectoid, eutectoid and proeutectoid zones. In the case of slowly cooled hypereutectoid steels proeutectoid cementite precipitates at the austenite grain boundaries and finally a continuous network is formed around the remaining austenite grains which subsequently transform to pearlite during cooling. The prior austenite grain size of this zone was revealed by cementite network. In proeutectoid zones the proeutectoid ferrite is expelled from the austenite grains at the grain boundaries forming discontinuous ferrite networks. These ferrite networks outline the prior austenite grains. It is very difficult to reveal the grain boundaries of the eutectoid steels. The grain size of the case was thus obtained by taking the average grain size of hypereutectoid and proeutectoid zones.

The mean linear intercept method³² was employed to measure the austenite grain size of the case of these carburized specimens. A total of about 400-500 intercepts were counted for each specimen.

3.5 Carburization and Heat treatment

3.5.1 Introduction

To study the structure and properties of the steels in the carburized and heat treated conditions the carburizing parameters such as temperature, gas flow rate, gas pressure, etc. were kept identical as in the case of the carburized and furnace cooled specimens. Three different time periods as 1 hour, 2 hours and 4 hours were selected for carburizing.

3.5.2 Carburizing and Hardening

Two sets of specimens from each of steels 1 to 4 were carburized at 950°C for 1 hour, 2 hours and 4 hours in the same process described in section 3.2. At the end of the predetermined time period, the specimens were cooled to about 860°C in the carburizing chamber. The gas flow into the chamber was then stopped and the lid of the chamber was then removed by means of a lever arm arranged on the top of the furnace. A little gas flow was maintained during cooling to the hardening temperature to avoid decarburization of the carburized specimens. The specimens were then quenched into 10% brine immediately after removing them from the chamber.

3.5.3 Sub-zero Treatment

In order to reduce the retained austenite content in the case of the carburized and hardened steels one of the two sets of the quenched specimens from each steel carburized for 1 hour, 2 hours and 4 hours was subjected to sub-zero treatment. This was done by immersing the carburized and hardened specimens into liquid nitrogen and kept them in it for an hour following lakhtin⁶.

3.5.4 Tempering

The other set of the quenched specimens without sub-zero treatment from each steel was then tempered immediately after quenching at a low temperature of 160°C for half an hour to transform the hardened martensite of the case into tempered martensite, relieving the residual stresses developed during quenching. Sub-zero treated specimens were also tempered to transform the hardened martensite into tempered martensite, relieving the residual stresses developed during quenching and sub-zero treatment in the liquid nitrogen.

3.6 Surface Hardness and Microhardness Measurement of the Carburized and Heat Treated Steels

The surface hardness of the carburized and heat treated specimens was measured by a Rockwell hardness testing machine. These heat treated specimens were then sectioned using a silicon carbide disc cutter with copious flow of water to avoid softening due to heat generated. The flat surfaces of the sectioned specimens were made parallel by a surface grinding machine and specimens were mounted and polished by standard metallographic technique. These polished specimens were then lightly etched in 2 percent nital and microhardness measurement was then carried out from the surface to the core by using Shimadzu microhardness tester. 100 gm load was applied and 5 second time was allowed to cause the indentations on the polished and lightly etched specimens. The hardness profiles were then drawn by using microhardness value (VHN) at different points from the sub-surface to the core. After taking microhardness measurements, the specimens were again polished properly and etched in 2 percent nital. Optical microscopy and photomicroscopy were then carried out.

3.7 Determination of Retained Austenite by X-ray Diffractometry

In a carbon steel, when the carbon content exceeds 0.65 percent, the transformation finished temperature (M_s) shifts to below the room temperature and all the austenite cannot be transformed to martensite during quenching.

This problem is vital for the treatment of carburized steels or steels containing austenite stabilizing elements such as manganese, nickel, etc. The effect of retained austenite on the service behaviour of the steel part is usually detrimental. Retained austenite also hampers the dimensional stability of the parts. Therefore retained austenite content in the hardened case of carburized parts must be known before their application for proper functioning under service. The retained austenite content was measured by X-ray diffraction technique using a JEOL DX-GE-2P X-ray diffractometer. In this work, X-ray diffraction study was

carried out to investigate the relative amount of retained austenite in (i) quenched and tempered (QT) specimens and (ii) quenched, sub-zero treated and tempered (QST) specimens and to study the effect of sub-zero treatment on the transformation of the retained austenite of the QT specimens. For this, the X-ray diffraction was conducted by Mo K_{α} radiation on all the specimens of steels 1 to 4 and individual structural information of the specimens was recorded on the chart papers. The chart papers were then analyzed and the possible phases with their planes responsible for the recorded peaks in the chart papers were identified first with the help of the manual available in the X-ray laboratory. Since the specimens were not in the powder form and they were composed of multi phases with nonuniform microstrain, a slight deviation of the peak position was observed for all the specimens. After detecting the phases with their planes for individual peak, the peak height or area under the peaks, the relative amount of austenite in the case was determined by using direct comparison technique explained by B. D. Cullity³³. The following mathematical relationships were used:

$$R = (1/v^2)[|F|^2 p \{(1+\cos^2 2\theta)/\sin^2 \theta \cdot \cos \theta\}] e^{-2M}$$

where, v = the volume of unit cell of the respective phase

F = structure factor

p = multiplicity factor

e^{-2M} = temperature factor

2θ = angle at the specific peak position

The values of F , p , e^{-2M} and θ were known with the help of the information provided by Cullity. By calculating the values of R_A (R for austenite) and R_M (R for martensite), the relative amount of austenite and martensite were known by using the following relationships.

$$I_A/I_M = (R_A \cdot C_A)/(R_M \cdot C_M) \quad \text{and}$$

$$C_A + C_M = 1$$

where, I_A = peak height or area under the peak of austenite phase

I_M = peak height or area under the peak of martensite phase

C_A = concentration of the austenite phase in the structure

C_M = concentration of the martensite phase in the structure

In the present work, area under the peaks of different phases was considered as the magnitude of I.

3.8 Preparation, Carburization, Heat-treatment and Testing of Wear Specimens

The cylindrical wear specimens used for wear testing were prepared with a dimension of 8mm in diameter and 6.5 mm in length from the 16 mm dia. bars of steels 1 to 4. These prepared specimens were then carburized for 1 hour maintaining the identical conditions. After carburization, the specimens were heat treated. The wear tests were carried out under a dry sliding condition using a pin on disc type apparatus as shown in Figure 3. The carburized and heat treated specimens were used as pins. Hardened steel discs with a hardness value of RC 50 having 80 mm diameter and 10 mm thickness were used as the counter body. Before the tests both the pin and counter body were degreased, cleaned thoroughly in running water and dried immediately by acetone. Each of the pins of steels 1 to 4 was weighed before the test to determine the weight loss due to wear. During the test 1500 gm dead load was used to press the pin against the disc while the disc was moving in the horizontal plane as shown in Figure 3. The moving disc was kept at 960 rpm which gave a linear speed 2.38 m/s. The tests were conducted for a duration of 1 hour which is equivalent to a sliding distance of 8600 m for each specimen. The specimens were then cleaned in water, dried in acetone and weighed. The difference between the two weights obtained before and after the tests, the weight loss due to wear was measured. From the weight loss values volume loss due to wear was obtained. The wear rate for each specimen was calculated by dividing the volume loss by the total sliding distance of 8600 m. In order to study the effect of retained austenite on the wear rate, the tests were carried out on both (i) carburized, hardened and tempered and (ii) carburized, hardened, sub-zero treated and

tempered specimens using the identical conditions.

3.9 Preparation, Heat-Treatment and Mechanical Testing of Charpy V-Notch Specimens

Two to three standard Charpy V-Notch impact test specimens from each steel as per British standard were prepared by machining the 16mm diameter bars. To assess the toughness of the core of the carburized and heat treated steels, the Charpy V-notch specimens were heated at 950°C for 2 hours in a Blue M furnace and then cooled slowly in the furnace to about 860°C and quenched in 10 percent brine and tempered at 160°C for half an hour. The quenched and tempered Charpy V-notch specimens were tested by means of a Universal impact testing machine with a range of 0-240 ft-lbs. All the specimens were tested at room temperature in the ambient air. The impact test data thus obtained were used to assess the toughness of the core of the carburized, hardened and tempered steels.

CHAPTER - 4

EXPERIMENTAL RESULTS

4.1 Optical Microscopy of Carburized and Furnace Cooled Steels

The microstructures of steels 1 to 4 carburized for 1 hour, 2 hours, 3 hours and 4 hours at 950°C and cooled slowly in the furnace were investigated by optical microscope.

4.1.1 Case

Optical microscopy revealed a variable carbon concentration in the carburized case along its depth, decreasing from the surface toward the core of all the steel specimens. Three zones were distinguished from surface to the core due to the variation of carbon concentration: (1) hypereutectoid zone, consisting of pearlite and cementite, forming a network along the former austenite grains, (2) eutectoid zone, consisting of only lamellar pearlite and (3) hypoeutectoid zone, consisting of pearlite and ferrite. After the eutectoid zone, discontinuous ferrite network was observed around pearlite. This zone is known as proeutectoid zone. The amount of ferrite in the last zone continuously increases toward the core where the original structure of the steel exists. The microstructures of all the steels were observed and the optical micrographs of steels 1 to 4 for all carburizing periods are presented in Figures 4 to 7.

The cementite networks of steel 1 were found to be the thickest and that of steel 4 the thinnest of all the steels. Steel 2 showed thinner cementite networks than steel 1 but thicker than steel 3. In all four steels, a gradual thickening tendency of the cementite networks with increasing time was observed.

In all four steels, Widmanstätten cementite plates were found to be nucleated at the grain boundary and within the grain in the hypereutectoid zone near the surface. Steel 3 showed

the highest number and steel 1 showed the lowest number of Widmanstätten cementite plates of all the steels. Steels 4 showed higher number of Widmanstätten cementite plates than steel 2. The lowest number of Widmanstätten cementite plates were found in steel 1. The amount of the Widmanstätten cementite plates was found to increase with the increase of carburizing period. A typical optical micrograph as shown in Figure 10 illustrates the Widmanstätten cementite plates nucleated at the grain boundaries and within the grains in the hypereutectoid zone in steels 1 to 4 carburized for 3 hours.

Heterogeneity in the grain structure was observed in the hypereutectoid zone of the case of steel 1 carburized for time period ranging from 1 hour to 4 hours.

4.1.2 Core

A ferrite pearlite structure was observed in the core of all the carburized steels for all the carburizing time periods (Figures 4 to 7). In steels 2 and 3 regular pearlite was not observed. Pearlitic carbide as reported by Mann and Muddle³⁴ was found in steels 2 and 3.

4.2 Case Depth

The effective case depth of the carburized and furnace cooled steels 1 to 4 under the identical conditions are shown in Table 2 and plotted in Figure 8 as a function of carburizing time. From the data presented in Table 2 and Figure 8 it is clear that for all the steels the depth of the carburized layer increases with an increasing carburizing time at the specified temperature. The graphical representation of the effective case depth values against the carburizing time for all the steels (Figure 8) indicates that at the specified carburizing temperature the growth of the carburized layer is parabolic with carburizing time.

Table 2 and Figure 8 show that under identical carburizing condition steel 2 with Mo produced higher case depth than plain carbon (base) steel 1 and lower case depth than steel 4 with Ni while steel 3 with both Mo and Ni produced the highest case depth of all the steels.

4.3 Prior Austenite Grain Size

The prior austenite grain size is important in that it controls the structures of both the case and the core of a carburized steel as well as mechanical properties. Therefore, the grain size in the case of carburized and slowly cooled specimens of steels 1 to 4 was measured for different carburizing times. The data thus obtained are listed in Table 3 and plotted in Figure 9 as a function of carburizing time.

Table 3 and Figure 9 show the prior austenite grain size of all the steels increases with increasing carburizing time. It is also clear that plain carbon steel 1 produced the coarsest grain size, whereas steel 3 produced the finest grain size of all the steels and steel 2 produced slightly coarser grain than steel 3. Steel 4 produced grain size similar to plain carbon steel 1. Table 3 and Figure 9 also show the rate of increase of prior austenite grain size of steels 1 and 4 is the highest with steel 3 has the lowest growth rate. On the other hand intermediate type grain growth is observed for steel 2.

Typical optical micrographs illustrating the grain size of the case of steels 1 to 4 carburized for 3 hours and cooled slowly in the furnace are shown in Figure 10.

4.4 Optical Microscopy of Carburized, Hardened and Tempered Steels

The microstructures of the polished and etched carburized, hardened and tempered specimens were examined by optical microscopy and photographs of the structures of each specimen were taken using a Swift Master Optical Microscope. Due to the carbon concentration gradient from the surface towards the core, a variable structure was observed in the case and core of the carburized, hardened and tempered specimens.

4.4.1 Case

The structures in the case of steels 1 to 4 carburized, hardened and tempered consist of tempered martensite and retained austenite. Steel 1 produced the minimum and steel 3 produced the maximum retained austenite along with tempered martensite while steel 2

produced higher amount of retained austenite than steel 1 and lower amount than steel 4 in the case near the surface of the hardened and tempered specimens. For all the steels percentage of retained austenite decreases towards the centre in the case and at some depth martensite becomes about 100 percent. Optical micrographs of steels 1 to 4 carburized for 1 hour and 4 hours hardened and tempered are presented in Figures 11 and 12 respectively.

The retained austenite content increases with the increase in carburizing time in all the steels. Optical microscopy revealed 40-44 percent, 45-50 percent, 65-70 percent and 53-57 percent retained austenite respectively in steels 1, 2, 3 and 4 carburized for 1 hour. For 2 hours carburization, about 50-55 percent, 60-65 percent, 80-85 percent and 70-75 percent retained austenite was observed in hardened and tempered steels 1, 2, 3 and 4 respectively. When the carburizing time is 4 hours the amount of retained austenite were found to be 60-65 percent, 65-70 percent, 88-92 percent and 80-85 percent for steels 1, 2, 3 and 4 respectively.

Martensite needles in the case near the surface of steel 4 are more or less of same size as those of steel 1 and those of steel 2 are finer than those of steels 1 and 4 while steel 3 produced the finest martensite of all the steels.

In all the steels coarse martensite was found in the case near the surface and the coarseness of the martensite decreases towards the centre in the case (Figures 11 and 12).

4. 4. 2 Core

Low carbon martensite was found in the core of the carburized specimens of steels 1 to 4 in the hardened and tempered condition. Steel 1 produced the coarsest and steel 3 produced the finest low carbon martensite while steel 2 produced coarser martensite than steel 3 and finer than steel 1. Steel 4 produced martensite of size similar to that of steel 1.

Some ferrite net was observed along with low carbon martensite in the core of steel 1. No ferrite net was found in the core of steels 2 to 4. A few ferrite grains was also observed in

the core of steels 1, 2 and 3. Steel 2 contains lesser amount of ferrite grains than steel 1 and steel 3 contains the lowest amount of ferrite grains of all these three steels. Steel 4 did not show any ferrite grain. The optical micrographs of the core of steels 1 to 4 carburized for 2 hours, hardened and tempered are presented in Figure 13 illustrating the above mentioned features.

Some globular carbides were found both in the case and core in the hardened and tempered steels 2 and 3. But the amount of the globular carbides was found much more in steel 2 than steel 3.

4.5 Optical Microscopy of Carburized, Hardened, Sub-zero Treated and Tempered Steels

The carburized, hardened and tempered (QT) specimens have a great tendency to retain austenite in the case. Sub-zero treatment can eliminate this retained austenite. The structures of the case and core of a carburized and hardened steel are different. So the effect of sub-zero treatment on the case and core structures is also different. Steels 1 to 4 carburized for 1 hour, 2 hours and 4 hours and hardened were subjected to sub-zero treatment.

4.5.1 Case

Steels 1 to 4 carburized for 1 hour in the hardened, sub-zero treated and tempered (QST) condition showed 12-14 percent, 20-23 percent, 27-30 percent and 22-25 percent retained austenite respectively. For 2 hours carburization, about 17-20 percent, 25-27 percent, 32-36 percent and 28-32 percent retained austenite were observed in hardened, sub-zero treated and tempered steels 1, 2, 3 and 4 respectively. About 22-25 percent retained austenite in steel 1, 28-30 percent in steel 2, 36-39 percent in steel 3 and 32-35 percent in steel 4 carburized for 4 hours were found in QST condition. The optical micrographs of steels 1 to 4 carburized for 1 hour and 4 hours in the QST condition are presented in Figures 14 and 15 respectively. Due to sub-zero treatment, the amount of retained

austenite decreases appreciably in all the steels for all carburizing periods used.

Some fine carbide globules were also found in both the case and core of steels 2 and 3 in the QST condition. The amount of the fine carbide globules in steel 2 was also found much more than that of steel 3.

4.5.2 Core

The microstructures observed in the core of the QST specimens of all the steels were similar to those found in the core of QT specimens.

4.6 Microhardness and Hardness Profiles

When a low carbon steel part is carburized, a variation of carbon concentration occurs from the surface to the core. The concentration of the carbon is high at the surface and decreases towards the core of the part. Due to this variation of carbon concentration a hardness gradient exists in a carburized case in the hardened condition. Because carbon content plays an important role on the desired hardness of a hardened steel. Usually the hardness profile of a carburized and hardened steel depends on (1) carburizing temperature, (2) carburizing time, (3) pressure in the carburizing atmosphere, 4) concentration of the alloying element in the metal and 5) the strength of the solute-carbon interaction. Depending upon the above mentioned factors, the hardness profile of a carburized and hardened steel may follow two extreme patterns. The profile may be steep with a somewhat sharp line of demarcation between the carburized case and the core or the profile may be very shallow i. e. the transition from the case to the core is very gradual. A profile with an intermediate nature is also very often observed. The hardness profiles from the subsurface to the core of the hardened and tempered specimens of steels 1 to 4 at various time at 950°C have been plotted in Figures 16 to 18 and those for the hardened sub-zero treated and tempered specimens of steels 1 to 4 carburized for 1 hour, 2 hours and 4 hours have been plotted in Figures 19 to 21 respectively. Figures 16 to 21 show that the hardness of the carburized case does not remain constant throughout the case. The hardness of the case is found

somewhat lower near the surface for all the steels under all the heat treatment conditions employed. With the increase in distance from the surface towards the core, the hardness gradually increases up to maximum hardness and then decreases to core hardness.

Figures 16 to 21 show the hardness profiles at the interface between the carburized case and the core of plain carbon of steel 1 are steeper than those of alloy steels 2 to 4. Among the three alloy steels, the hardness profile of steel 3 is less steeper than that of steel 2 and more steeper than that of steel 4. It is found from the hardness profiles that, in general, the case depth increases with increase in carburizing time.

From Figures 16 to 21, it is evident that the microhardness value at any point of QST specimens is found always higher than that of QT specimens at the corresponding point for the same carburizing period.

The graphical representation of the VHN values against the distance from the edge towards the center of all the steels indicates that at the selected carburizing temperature hardness of the case increases with an increase in time.

A comparison of the values in Figures 16 to 21 shows that under identical condition of carburizing and hardening, steel 2 produced maximum case hardness while steel 4 produced minimum case hardness. Steel 3 produced case hardness higher than steel 4 and lower than steel 1.

The maximum hardness of the carburized case varies with the carburizing time. From the hardness profiles (Figures 16 to 21) it is found that the maximum case hardness of all the steels increases with increase in carburizing time.

It is evident from Figures 16 to 21 and Tables 4 and 5 that the core hardness of all the steels in the QT and QST conditions does not show any change appreciably with the increase of carburizing time. No noticeable change was observed in the core hardness of all the steels in both the QT and QST conditions.

It is seen from Figures 16 to 21 that the core hardness of steel 3 is maximum and that in steel 1 is minimum while steel 2 shows lower core hardness than steel 3 and higher than steel 4.

4.7 Surface Hardness and Maximum Hardness

Since it is very difficult to avoid retained austenite although small in amount in the surface of carburized and hardened steels, it is, therefore, quite reasonable to assume that the surface hardness values obtained by microhardness tester may not be proper representation of the surface hardness of a carburized case and consequently the surface hardness of the case was measured by Rockwell hardness tester.

The surface hardness of the QT and QST specimens of carburized steels 1 to 4 as measured by Rockwell hardness testing machine are presented in Tables 4 and 5 along with the maximum hardness and core hardness obtained from microhardness tests and hardness profiles of Figures 16 to 21. The surface hardness and maximum hardness of the QT specimens of steels 1 to 4 as a function of carburizing time are plotted in Figures 22 and 23 respectively. From the experimental results of Table 4 and Figures 22 and 23 it is clear that the surface hardness decreases and maximum hardness increases with the increase of carburizing time for QT specimens of steels 1 to 4. For QST specimens of steels 1 to 4 similar trends for surface hardness and maximum hardness were observed.

Attempts were also made to observe whether there is any relationship between the surface hardness and the maximum hardness of the carburized and heat treated steels. The surface hardness was found always lower than the maximum hardness for the both QT and QST specimens for any specified carburizing time. Tables 4 and 5 show that the difference between the surface hardness and maximum hardness increases with the increase in carburizing time. This difference is more noticeable for QT specimens than that of QST specimens of all the steels.

From the data presented in Tables 4 and 5, it is also evident that for any carburizing period the surface hardness and the maximum hardness of QST specimens of all the steels are always higher than that of QT specimens.

4.8 X-ray Diffractometry Data

Since solid block specimens instead of powder specimen were used in this experiment, slight deviation is resulted between the actual position of the peak and the recorded peak position. On the other hand, a line broadening effect is also resulted for the same reason. The line broadening effect on martensite peak and austenite peak is not identical and thus relative phase amount measurement using peak height may be misleading. To avoid such a situation, area under the peak was considered as the value of I . The operational condition used in X-ray diffraction is shown in Table 6 and the X-ray diffractometry results of steels 1 to 4 carburized for 1 hour, 2 hours and 4 hours both in QT and QST conditions are presented in Table 7. Typical X-ray diffraction patterns of steel 2 carburized for 2 hours, for both the QT and QST specimens are presented in Figures 24 and 25, and the calculation of the retained austenite content from this patterns is shown in Appendix.

According to X-ray diffraction results recorded in Table 7 QST specimens of each of the carburized steels have much lower retained austenite content than QT specimens of the corresponding steel. In all the carburizing periods used, retained austenite content in steel 1 is the lowest and that of steel 3 is the highest of all the steels. Between steel 2 and steel 4, steel 4 produced higher amount of retained austenite than steel 2. According to the data given in Table 7 sub-zero treatment to recover the retained austenite is more effective for steel 1 than the alloy steels 2 to 4. Among the three alloy steels recovery of retained austenite of steel 4 is higher than those of steels 2 and 3 while the recovery of retained austenite in steel 3 is slightly higher than that of steel 2 (Table7).

4.9 Wear Test Data

The weight loss (mg) and wear rate (m^3/m) obtained from the wear test of steels 1 to 4 carburized for 1 hour in both QT and QST conditions are presented in Table 8. The wear tests were carried out under dry sliding conditions at room temperature in the ambient air and the wear rate was expressed by volume loss per unit sliding distance due to wear. It is clear from the data presented in Table 8 that the wear rate of all the steels in QST condition is always lower than those of QT condition.

It is also evident from Table 8 that wear rate of steel 4 is maximum and that of steel 2 is minimum while the wear rate of steel 1 is lower than that of steel 4 and higher than that of steel 3 at a sliding distance of 8600 m of the QT specimens.

In the QST condition, the wear rate of steel 4 is higher than that of steel 1 and lower than that of steel 3 while the wear rate of steel 2 is the lowest of all the steels (Table 8).

4.10 Impact Test Data

The impact energy absorbed at room temperature for the hardened and tempered steels is presented in Table 9. Table 9 shows that the impact energy absorbed in steels 2 to 4 are much lower than that in the base steel 1. Steel 4 shows lower impact energy absorbed than steel 2 and slightly higher than steel 3.

CHAPTER - 5

DISCUSSION

According to the composition of steels 1 to 4 shown in Table 1, the second phase particles in steels 2 and 3 are expected to be essentially molybdenum carbide (Mo_2C).

5.1 Metallography

Optical microscopy revealed thinner cementite networks surrounding pearlite near the surface in the hypereutectoid zone of steels 2 to 4 than steel 1. This indicates that the carbon concentration in the case of steels 2 to 4 are lower than that in the case of steel 1. Among steels 2, 3 and 4, steel 3 produced thinner cementite networks than steel 2 and thicker than steel 4 (Figures 4 to 7 and 10). This also indicates that the carbon concentration in the case of steel 3 is lower than that in steel 2 and higher than that in steel 4.

For all four steels, a gradual thickening tendency of the cementite networks with carburizing time was observed (Figures 4 to 7 and 10). This is because, at a certain temperature, the austenite can absorb a certain amount of carbon, making an interstitial type solid solution. Any carbon in excess of the fixed amount is expelled as cementite precipitates at the austenite grain boundaries and these cementite precipitates form cementite network. As the carburizing time increases, more and more carbon is expelled as cementite precipitates, making the cementite network thicker.

The presence of Widmanstätten cementite plates (Figure 10) nucleated at the grain boundary and within the grains in the hypereutectoid zone near the surface of all the steels is due to the supersaturation of austenite with respect to carbon. Steel 1 showed smaller number of Widmanstätten cementite plates than other three steels 2 to 4 indicating clearly

that the supersaturation of austenite with respect to carbon in plain carbon steel 1 is less than that in other steels. Among steels 2 to 4, steel 4 produced higher number of Widmanstätten cementite plates than steel 2 and lower number Widmanstätten cementite plates than steel 3. This indicates that the supersaturation of austenite with respect to carbon in steel 4 is higher than that in steel 2 and lower than that in steel 3.

Thus it is clear that both molybdenum and nickel enhance the formation of Widmanstätten cementite plates both at the grain boundary and within the grains. But Ni is more effective than Mo in the formation of Widmanstätten cementite plates. The combined effect of Mo and Ni in the formation of Widmanstätten cementite plates is greater than either Mo or Ni alone.

Retained austenite as shown in Figures 11 and 12. was found in the case of almost all the steels carburized, hardened and tempered. It has been reported that when carbon percentage in a steel exceeds 0.65 percent, the M_f temperature shifts to below room temperature²⁹ and all the austenite cannot transform to martensite. The presence of retained austenite in the case of all the steels thus clearly indicates that the carbon content in the surface of all the carburized steels was much more than 0.65 percent and M_f temperature was much below the temperature of the quenching medium (10% brine) used.

It was also found that steels 2 to 4 produced higher percentage of retained austenite than steel 1. This is due to the fact that steels 2 to 4 contain alloying elements such as molybdenum and nickel which lower the martensite transformation temperatures in such a way that the M_f temperature is sufficiently below the temperature of the quenching medium. It is also found that among steels 2, 3 and 4, steel 4 produced a larger amount of retained austenite than steel 2 and a smaller amount than steel 3. Molybdenum and nickel increase the concentration of carbon at the surface of carburized steels. The higher concentration of carbon in steel 4 due to the presence of nickel depresses the M_f temperature below that of steel 2 resulting in a greater amount of retained austenite in it.

Steel 3 which contains Mo and Ni produced the largest amount of retained austenite. Its retained austenite content is expected to be the highest and that was, in fact, observed to be the case.

The retained austenite content also increase with the increase in carburizing time in all the steels (Figures 11 and 12). The concentration of carbon in the cases of all these steels increases with the increase of carburizing time. This higher concentration of carbon depresses the M_f temperature drastically resulting in an increased amount of retained austenite.

From Figures 11 and 12 it has also been revealed that steel 1 and steel 4 produced the coarsest martensite while steel 3 produced the finest at all the carburizing periods. Steel 2 produced martensite finer than steels 1 and 4 and coarser than steel 3. The coarsest martensites in steels 1 and 4 are due to the coarsest austenite grains and the finest martensites in steel 3 are due to the finest austenite grains present in it (Figures 9 and 10, Table 3). Since steel 2 produced finer austenite grains than steels 1 and 4 and coarser than steel 3, it produced martensite finer than steels 1 and 4 and coarser than steel 3.

In all the steels coarse martensite was found near the surface in the case and the coarseness of martensite decreases towards the centre in the case. Optical microscopy of the carburized and furnace cooled steels revealed coarse austenite grains near the surface of the cases and it became finer towards the centre in the case. The coarse austenite produced the coarse martensite near the surface and the finer austenite grains produced the finer martensite towards the centre in the case.

With the increase in carburizing time, the coarseness of the martensite also increases for all the steels. This is due to the fact that with the increase in carburizing time, the prior austenite grain size was also increased (Table 3, Figures 4 to 7 and 9) from which subsequently coarse martensite was resulted.

Molybdenum in steels 2 and 3 exists as molybdenum carbide (Mo_2C) at the room temperature. At the carburizing temperature a portion of this molybdenum carbide was dissolved into austenite and some molybdenum carbide remained undissolved. During pre-cooling of steels 2 and 3 from carburizing temperature 950°C to 860°C , a little portion of Mo dissolved in austenite precipitates out as fine molybdenum carbides. During quenching molybdenum that remains in solution in austenite will remain in solution in martensite. When this martensite is tempered at 160°C , molybdenum precipitates out as fine molybdenum carbide (transition carbide).

Steels 1 to 4 produced less retained austenite in the QST condition than in the QT condition (Figures 11, 12, 14 and 15). This is because during sub-zero treatment a certain proportion of retained austenite was transformed to martensite.

Steel 1 produced the coarsest and steel 3 produced the finest low carbon martensite in the core. This is due to the fact that the prior austenite grain size of steel 1 was the coarsest and that of steel 3 was the finest of all the steels.

Steel 4 produced low carbon martensite similar to that of steel 1. This is clearly due to their similar prior austenite grain size.

Steel 2 produced low carbon martensite finer than steel 1 and coarser than steel 3. This is also clearly due to the difference of their prior austenite grain size (Figures 9, 10 and 13).

Some ferrite net was observed along with low carbon martensite in the core of steel 1. This indicates that the hardening temperature (860°C) of steel 1 was a little below the upper critical temperature. A few ferrite grains was also observed in the core of steels 1, 2 and 3 (Figure 13). The presence of ferrite grain indicates that the critical cooling rate in steels 1, 2 and 3 was faster than the actual cooling rate.

The microstructures observed in the core of the QST specimens of all the steels were similar to those found in the core of QT specimens. This is clearly due to the absence of

retained austenite which can be transformed to more stable phases at the sub-zero temperature in the core of the carburized and hardened specimens.

5.2 Case Depth

The effective case depth of steels 1 to 4 is plotted in Figure 8 as a function of carburizing time and presented in Table 2. Steel 1 is the base steel with which the carburizing behaviour of the other steels (steels 2 to 4) with Mo and Ni alone and in combination are compared. Table 2 and Figure 8 show that steels 2 to 4 produced higher case depth than the plain carbon steel 1. Among steels 2 to 4, steel 4 with Ni produced higher case depth than steel 2 with Mo. This indicates clearly that both Mo and Ni alone increases the thickness of the carburized layer and Ni is more effective than Mo in increasing the case depth.

Nickel increases the diffusion coefficient D_γ of carbon in austenite by reducing the activation energy of diffusion⁶. The higher case depth of steel 4 with Ni than the plain carbon steel 1 is thus obviously due to the higher diffusion coefficient.

The higher case depth in steel 2 than steel 1 and lower than steel 4 indicates that Mo increases the diffusion coefficient D_γ of carbon in austenite by reducing the activation energy of diffusion but not as high as that caused by Ni. The highest case depth in steel 3 containing both Ni and Mo is thus due to the combined effect of Ni and Mo.

The higher case depth in steel 3 containing both Mo and Ni than steel 2 with Mo clearly indicates that in the presence of Ni, Mo is more effective in increasing the case depth than Mo in absence of Ni. The presence of Ni is thought to enhance the effect of Mo in the increment of diffusion coefficient.

It is also evident from Figure 8 that as the time at carburizing temperature increases the case depth of the steels increases. This is due to the fact that as carburizing time increases, carbon atoms get longer time to diffuse into a greater depth and hence higher is

the case depth.

5.3 Prior Austenite Grain Size

Steel 1 is the base steel with which the austenite grain size of the other steels (steels 2 to 4) have been compared. This is a plain carbon steel with no addition of alloying elements. So it does not contain any second phase particles to inhibit austenite grain growth and thus the austenite grain size of this steel increases rapidly as shown in Figure 9 with the increase of time at the carburizing temperature.

It is also evident from Figure 9 that steel 2 produces much finer austenite grain size than steel 1 (Figure 10). Steel 2 contains molybdenum which combines with carbon forming molybdenum carbide, Mo_2C . These undissolved molybdenum carbide particles pin the prior austenite grain boundaries and inhibit grain growth and keep the grain size small. Steel 4 produces the grain size similar to that of plain carbon steel 1. This steel contains nickel. Nickel does not combine with carbon to form any second phase particle. It remains in solution in austenite. Thus there is no second phase particle to inhibit austenite grain growth.

Steel 3 produces the finest grain size of all the steels and finer grain size than steel 2 with Mo. Steel 3 contains both Mo and Ni. Nickel remains in solution in austenite and molybdenum combines with carbon forming Mo_2C particles. The presence of nickel in solution is thought to produce finer Mo_2C particles. Fine particles are more effective than coarse particles in restricting grain growth. The greater grain refining effect found in steel 3 than that found in steel 2 is thus due to the finer Mo_2C particles due to the presence of nickel in it. It is also evident from the Figure 9 that the austenite grain size of steels 2 and 4 increases with the increasing carburizing time because the carbide particles become coarsened with the increasing time at the carburizing temperature.

5.4 Hardness and Hardness Profiles

It is clear from the graphical representation of VHN values against the distance from the subsurface to the core of the carburized steels 1 to 4 that the hardness of the cases in the QT and QST conditions do not remain constant throughout the cases. Figures 16 to 21 and Tables 4 and 5 show that the hardness of the surface is much lower than the maximum hardness. The lower surface hardness in all the steels is clearly due to the presence of retained austenite along with martensite. With the increase in distance from the surface, the content of retained austenite decreases resulting in higher hardness.

It is evident from Figures 16 to 21 that hardness profiles at the interface between the carburized case and the core of plain carbon steel 1 are steeper than those of alloy steels 2 to 4. This higher steepness in the hardness profiles of plain carbon steel 1 is due to the higher concentration gradient at the interface. Among the three alloy steels, the hardness profile of steel 3 is less steep than that of steel 2 and more steep than that of steel 4. This is due to the fact that the concentration gradient from the case to the core of steel 3 is lower than that of steel 2 and higher than that of steel 4.

From Figures 16 and 21 it has been also found that the microhardness value to any point of the carburized steels 1 to 4 in QST conditions is always higher than that of QT conditions at the corresponding point for the same carburizing period. The additional hardness in the case of the carburized steels in the QST condition is due to the dual effect of sub-zero treatment. Sub-zero treatment substantially decreases the retained austenite content in the case and thus increases the hardness. On the other hand, sub-zero treated steels suffer double quenching, one from 860°C to room temperature and another from room temperature to liquid nitrogen temperature. Due to this double quenching effect these steels have much more residual stress in QST condition than in QT condition. This excess residual stress also gives additional hardness.

It is also evident from Figures 16 to 21 and Tables 4 and 5 that molybdenum containing steel 2 produced maximum case hardness and steel 4 containing nickel produced minimum case hardness and steel 3 produced case hardness higher than steel 4 and lower than steel 1.

Steel 2 contains Mo_2C precipitates. The higher hardness of the case of this steel than the base steel 1 is clearly due to the presence of these precipitates which impede the movement of dislocations and hence produces hardening effect in the steel. Molybdenum in solution in martensite also contributes to the hardening effect.

Optical microscopy of the carburized and furnace cooled steels revealed that steel 1 produced the thickest Fe_3C network and steel 4 produced the thinnest Fe_3C network of all the steels. This indicates clearly that the concentration of carbon in steel 4 is much lower than that in steel 1. This lower concentration of carbon in steel 4 is clearly due to the presence of nickel. The hardness of martensite is a function of carbon content. Hence the lower hardness of steel 4 than base steel 1 is obviously due to the lower amount of carbon in solution in martensite present in it than that of steel 1.

Steel 3 is basically identical with steel 4 with Mo added to it. The higher hardness of the case of this steel than steel 4 is certainly due to the presence of Mo_2C in it. Steel 3 is also basically steel 2 with Ni added to it. The lower hardness of this steel than steel 2 is obviously due to the lower carbon content in the martensite caused by the presence of nickel. This is also supported by the thickness of Fe_3C networks in carburized and furnace cooled steels.

It is also clear from Figures 16 to 23 and Tables 4 and 5 that the maximum hardness of the carburized steels 1 to 4 both in QT and QST conditions increases with the increase in carburizing time. This is because of the increasing carbon content of martensite. The surface hardness of all the steels decreases with the increase in carburizing time because retained austenite content increases with the increase of carburizing time.

It is found that the surface hardness of all the carburized steels in the both QT and QST conditions is lower than the maximum hardness obtained in the carburized case. The explanation of this difference in hardness lies in the fact that at the surface the steels contain certain amount of retained austenite along with tempered martensite. So, when the surface hardness is measured by Rockwell hardness testing machine, the indenter sinks into both retained austenite and martensite. As a result an average hardness of the two phases is obtained.

No appreciable change is observed in the core hardness of all the steels with the increase of carburizing time in both the QT and QST conditions (Figures 16 to 21 and Tables 4 and 5). This indicates that the residual stress developed in low carbon martensite of the core is not high enough to cause any noticeable change in the hardness.

Base steel 1 produced minimum core hardness and steel 3 produced maximum core hardness. Steel 2 produced core hardness higher than steel 4 and lower than steel 3. Plain carbon steel 1 contains some ferrite nets and ferrite grain along with low carbon martensite (Figure 13) in the core. The lower hardness in the core of steel 1 than other steels (Steels 2 to 4) is due to the presence of ferrite net and ferrite grains in it.

The higher hardness in the core of steel 4 than steel 1 is due to the presence of Ni in solution in low carbon martensite. Steel 2 with Mo produces higher core hardness than steel 4. The higher core hardness of steel 2 is due to presence of Mo in solution in low carbon martensite and Mo_2C particles in it. Steel 3 contains both Ni and Mo. The much higher hardness of this steel than steels 2 and 4 is clearly due to the combined effect of Ni and Mo in solution in low carbon martensite and Mo_2C particles. This combined effect of Ni and Mo is greater than the effect of either Mo or Ni alone.

5.5 Effect of Sub-zero Treatment on Retained Austenite

From Table 7 and Figures 11, 12 and 14, 15 it is evident that the retained austenite content in the case of steels 1 to 4 carburized for all the carburizing periods was much lower in QST condition than in QT condition.

During carburizing the carbon content in the case near the surface of steels 1 to 4 exceed 0.65 percent. Carbon content in a carbon steel higher than 0.65 percent shift the M_f temperature to below room temperature and all the austenite cannot transform to martensite when quenched in 10 percent brine. The higher percentage of retained austenite found in the case of carburized steels in QT conditions, thus indicates clearly that M_f temperature is much below the temperature of the quenching medium used. A substantial amount of the retained austenite was transformed to martensite resulting lower retained austenite content in the case when the carburized and quenched specimens were immersed in liquid nitrogen in order to cool the specimens much below the M_f temperature.

It is also clear from Table 7 that sub-zero treatment was most effective for steel 1 and least effective for steel 2 for retained austenite transformation. This is because steel 1 has no alloying element to retard the retained austenite transformation to martensite. In steel 2 molybdenum retards the austenite transformation making the sub-zero treatment least effective.

The recovery of retained austenite of steel 2 is lower than that of steel 4. This indicates that Mo in steel 2 is more effective than Ni in steel 4 to retard the austenite transformation. The recovery of retained austenite in steel 3 is slightly higher than that of steel 2. Steel 3 is basically steel 2 with Ni added to it. The slightly higher recovery of retained austenite in steel 3 than steel 2 is thus clearly due to the presence of Ni in it.

The X-ray metallographic results (Table 7) also show that the retained austenite content in the case of all the steels was not completely transformed into martensite after sub-zero

treatment. This is because for 100 percent retained austenite transformation, several cycles of sub-zero treatment followed by tempering is required. But in this experiment only one cycle was performed.

5.6 Wear Resistance

It is evident from Table 8 that the wear rate of all the steels in the QST condition is found lower than those in the QT condition. This is due to the decreasing amount of retained austenite content in the QST specimens caused by the sub-zero treatment.

It is also found from Table 8 that the wear rate of steel 4 is higher than the plain carbon steel 1. Tables 4, 5 and 8 show that the surface hardness of steel 4 is lower than that of steel 1 and the retained austenite content of steel 4 is higher than that of steel 1. The higher wear rate in steel 4 is thus clearly due to its higher retained austenite content and lower surface hardness than plain carbon steel 1.

Table 8 also shows that the wear rate of steel 2 is much lower than that of steel 1. Steel 1 being a plain carbon steel does not contain any second phase particles. Steel 2 with Mo contains Mo_2C particles. The surface hardness and retained austenite content of steel 2 are respectively lower and higher than those of steel 1. The lower surface hardness and higher retained austenite content in steel 2 increase the wear rate. But the Mo_2C particles decreases the wear rate. Therefore, the lower wear rate in steel 2 than steel 1 is clearly due to overriding effect of carbide particles.

The wear rate of steel 3 is higher than that of steel 2 and lower than that of steel 4 with Ni. Steel 3 is basically steel 2 with nickel add to it. The surface hardness and retained austenite content of steel 3 are respectively lower and higher than those of steel 2. Thus, the higher wear rate of steel 3 is clearly due to the combined effect of lower surface hardness and higher retained austenite content caused by the presence of nickel in it. In the QST condition, the wear rate of steel 4 is higher and that of steel 2 is lower than the

wear rate of plain carbon steel 1 while steel 3 produced wear rate higher than steel 4.

In QST condition the surface hardness and retained austenite content of steel 4 are respectively lower and higher than those of steel 1. The higher wear rate in steel 4 than the plain carbon steel 1 in the QST condition is clearly due to its lower surface hardness and higher retained austenite content than steel 1.

Both the surface hardness and retained austenite content of steel 2 are higher than those of steel 1 respectively. Steel 2 also contains Mo_2C particles. The lower wear rate of steel 2 than that of steel 1 is due to the overriding combined effect of surface hardness and Mo_2C particles. (Tables 4, 5, 7 and 8)

The surface hardness of steel 3 is same as that of steel 4 while the retained austenite content is greater than that of steel 4. The higher amount of retained austenite present in steel 3 than steel 4 resulted the higher wear rate in steel 3.

5.7 Toughness

It is evident from Table 9 that the impact energy absorbed in steels 2, 3 and 4 are much lower than that in the base steel 1 indicating clearly that the addition of Mo and Ni alone or in combination decrease the toughness of the core of low carbon steels in carburized, hardened and tempered condition.

Steel 2 with Mo produced finer low carbon martensite than steel 1. But it contains Mo_2C precipitates. Mo_2C precipitates impair the toughness of low carbon steel. The lower impact energy absorbed of steel 2 is clearly due to the presence of Mo_2C precipitates. Steel 2 contains much lesser amount of ferrite grains than steel 1. So, the contribution of ferrite grains is considered negligible.

It is also evident from Table 9 that steel 4 with Ni show lower impact energy absorbed than steel 2 with Mo. This indicates that Ni is more detrimental to room temperature

toughness than Mo. This may be due to the absence of ferrite grains and the presence of coarser martensite in steel 4 than steel 2. Since nickel is not a carbide former, it remains in solution. This is also reported by Saulnier³⁶.

Steel 3 with both Mo and Ni shows the lowest impact energy absorbed indicating clearly that the combined effect of Mo and Ni is much more detrimental than the effect of either Mo or Ni alone. It is thought that in presence of Ni, Mo_2C precipitates become finer. These finer Mo_2C particles may be responsible for the lowest impact energy absorbed in steel 3. In view of the core hardness the lowest impact energy absorbed in steel 3 is not unexpected.

CHAPTER - 6

CONCLUSIONS

6.1 Conclusions Drawn from the Present Work

1. Molybdenum and nickel alone and in combination decrease the thickness of the cementite network near the surface of the carburized case of the steels. But nickel is more effective than molybdenum in decreasing the thickness of cementite network.
2. Both molybdenum and nickel enhance the formation of Widmanstätten cementite plates at the grain boundary and within the grains near the surface of the carburized steel. But nickel alone is more effective than molybdenum in the formation of Widmanstätten cementite plates.
3. In presence of nickel, molybdenum is much more effective in the formation of Widmanstätten cementite plates than molybdenum in absence of nickel.
4. Both molybdenum and nickel increase the case depth. Nickel is more effective than molybdenum in increasing the case depth. The combined effect of molybdenum and nickel is much greater than that of either molybdenum or nickel alone in increasing case depth.
5. Molybdenum as molybdenum carbide (Mo_2C) particles (undissolved particles) refines the austenite grain size in the carburized case.
6. Nickel in solution does not have any effect on restricting grain growth of austenite.
7. In presence of Ni, molybdenum is more effective in retarding grain growth of austenite than Mo in absence of Ni.

8. Molybdenum and nickel both enhance the formation of retained austenite in the cases of carburized and hardened steels with or without sub-zero treatment. Nickel is more effective than molybdenum in enhancing the formation of retained austenite. In the presence of molybdenum this enhancing effect of nickel is increased.
9. Sub-zero treatment reduces substantially the amount of retained austenite in the carburized and hardened cases of low carbon steels containing molybdenum and nickel alone and in combination.
10. Molybdenum and nickel both sluggish the retained austenite transformation to martensite during sub-zero treatment. Molybdenum is more effective than nickel in sluggishing the transformation of retained austenite to martensite. In the presence of nickel this sluggishing effect of molybdenum reduces.
11. Molybdenum produces fine martensite but nickel has no effect in refining the size of martensite. Molybdenum with nickel is more effective than molybdenum without nickel in refining the size of martensite.
12. Molybdenum increases and nickel decreases the maximum case hardness of low carbon steel under all the heat treatment conditions. The presence of nickel decreases the effect of molybdenum in the increment of maximum case hardness.
13. Molybdenum and nickel both increase the core hardness but molybdenum is more effective than nickel in the increment of core hardness of low carbon steels. Molybdenum with nickel is more effective in increasing the core hardness than molybdenum without nickel.
14. Sub-zero treatment increases the surface hardness and maximum hardness of the carburized low carbon steels in the hardened and tempered condition.

15. Under all the heat-treatment conditions the surface hardness of the carburized plain carbon, molybdenum and nickel steels is always lower than the maximum hardness.
16. Molybdenum improves and nickel reduces the wear resistance of the case of low carbon steel.
17. Sub-zero treatment improves the wear resistance of the carburized, hardened and tempered cases of low carbon steels containing molybdenum and nickel alone and in combination.
18. Molybdenum and nickel both reduce the toughness of the core of low carbon steels in the carburized and hardened condition. Nickel is more detrimental to toughness than molybdenum. The combined effect of molybdenum and nickel is greater than that of either molybdenum or nickel alone in reducing toughness.

6.2 Suggestions for Further Work

1. A study on the effect of number of cycles of sub-zero treatment should be carried out on the carburized and hardened steels in order to convert 100 percent retained austenite to martensite and to have a detailed information about the subsequent effect on properties.
2. A high temperature tempering of the carburized and hardened steels may be carried out to show the variations of hardness with tempering temperature and the results thus obtained may be compared with those of low temperature tempering with shorter duration.
3. A detailed investigation on the wear resistance property of the steels is required in order to obtain a clear understanding on the wear behaviour of the carburized case of low carbon steels containing molybdenum and nickel alone and in combination.
4. A systematic study on hardenability, tensile and fatigue properties of the steels should be performed in order to have a complete understanding of the behaviour of the carburized low alloy steels.
5. Service behaviour of the carburized steels containing molybdenum and nickel alone and in combination should be investigated.
6. Transmission electron microscopic study should be carried out to obtain information on precipitate morphology.

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Table 1:

Compositions of steels 1 to 4.

Steel No	Composition, wt.%						
	C	Si	Mn	Mo	Ni	S	P
1	0.13	0.18	1.20	-	-	0.021	0.028
2	0.13	0.17	1.15	0.48	-	0.013	0.026
3	0.14	0.17	1.31	0.49	1.26	0.023	0.025
4	0.14	0.16	1.40	-	1.26	.027	0.038

Table 2 :

Effective case depth of steels 1 to 4 carburized for different time periods and cooled slowly in the furnace.

Steel No.	Case depth, μm			
	1 hr	2 hr	3 hr	4 hr
1	620	930	1175	1300
2	710	1055	1320	1460
3	940	1300	1580	1720
4	810	1170	1460	1580

Table 3 :

The prior austenite grain size of the case of steels 1 to 4 carburized for different periods of time and cooled slowly in the furnace.

Steel No.	Prior austenite grain size, μm			
	1 hr	2 hr	3 hr	4 hr
1	70	84	94	105
2	51	59	68	73
3	46	52	54	59
4	72	83	95	105

Table 4:

Relationship between surface hardness, maximum hardness and core hardness of steels 1 to 4 without sub-zero treatment

Steel No	Carburizing time,hrs	Surface Hardness		Maximum Hardness		Core Hardness	
		RC	VHN	VHN	RC	VHN	RC
Steel 1	1 hr	56	640	840	63	270	26
	2 hrs	50	540	890	65	265	25
	4 hrs	46	470	930	66	265	25
Steel 2	1 hr	54	600	880	64	365	37
	2 hrs	49	520	920	65	370	38
	4 hrs	47	480	970	67	375	38
Steel 3	1 hr	45	460	815	62	385	39
	2 hrs	37	367	845	63	390	40
	4 hrs	30	300	885	64	395	40
Steel 4	1 hr	41	410	750	61	350	35
	2 hrs	38	373	820	62	355	36
	4 hrs	35	350	860	63	350	35

Table 5:

Relationship between surface hardness, maximum hardness and core hardness of steels 1 to 4 with sub-zero treatment :

Steel No	Carburizing time,hrs	Surface Hardness		Maximum Hardness		Core Hardness	
		RC	VHN	VHN	RC	VHN	RC
Steel 1	1 hr	63	850	890	65	275	26
	2 hrs	62	800	925	66	265	25
	4 hrs	61	750	970	67	260	24
Steel 2	1 hr	64	875	940	66	375	38
	2 hrs	63	850	970	67	375	38
	4 hrs	62	800	1040	68	375	38
Steel 3	1 hr	61	750	865	64	400	40
	2 hrs	60	738	885	64	390	40
	4 hrs	59	725	940	66	395	40
Steel 4	1 hr	61	750	785	62	350	35
	2 hrs	60	738	855	63	350	35
	4 hrs	59	725	915	65	345	34

Table 6:

Operating condition of the X-ray diffractometer.

Radiation	:	MoK _α
Voltage	:	32.5 KV
Current	:	20 mA
Scan Rate	:	1°/min
Chart speed	:	20 mm/min
Scale Factor	:	2 X 10 ⁴ c.p.s.

Table 7:

X-ray diffractometry results of steels 1 to 4 carburized for 1 hour, 2 hours and 4 hours.

Steel No.	Carburizing Time, hrs	Retained austenite, pct		Retained austenite recovery, pct
		QT	QST	
1	1 hr	43 %	15 %	65 %
	2hrs	54 %	20 %	63 %
	4hrs	63 %	24 %	62 %
2	1 hr	51 %	23 %	55 %
	2hrs	63 %	28 %	56 %
	4hrs	69 %	30 %	57 %
3	1 hr	70 %	31 %	56 %
	2hrs	84 %	36 %	57 %
	4hrs	90 %	38 %	58 %
4	1 hr	56 %	24 %	57 %
	2hrs	75 %	31 %	59 %
	4hrs	84 %	34 %	60 %

92537

Table 8:

Weight loss due to wear and wear rate at a sliding distance of 8600 m of steels 1 to 4 carburized and heat treated.

Steel No.	Heat-treatment	Weight Loss, mg	Volume loss, m ³	Wear Rate m ³ /m
1	QT	11.00	1.414 x 10 ⁻⁹	1.648 x 10 ⁻¹³
	QST	7.50	0.96 x 10 ⁻⁹	1.124 x 10 ⁻¹³
2	QT	9.40	1.21 x 10 ⁻⁹	1.409 x 10 ⁻¹³
	QST	4.20	0.539 x 10 ⁻⁹	0.629 x 10 ⁻¹³
3	QT	10.50	1.349 x 10 ⁻⁹	1.573 x 10 ⁻¹³
	QST	9.5	1.22 x 10 ⁻⁹	1.424 x 10 ⁻¹³
4	QT	12.80	1.65 x 10 ⁻⁹	1.918 x 10 ⁻¹³
	QST	8.00	1.028 x 10 ⁻⁹	1.199 x 10 ⁻¹³

Table 9:

Impact energy absorbed of steels 1 to 4.

Steel No.	Impact Energy Absorbed, Ft-lbs
1	93
2	43
3	29
4	31

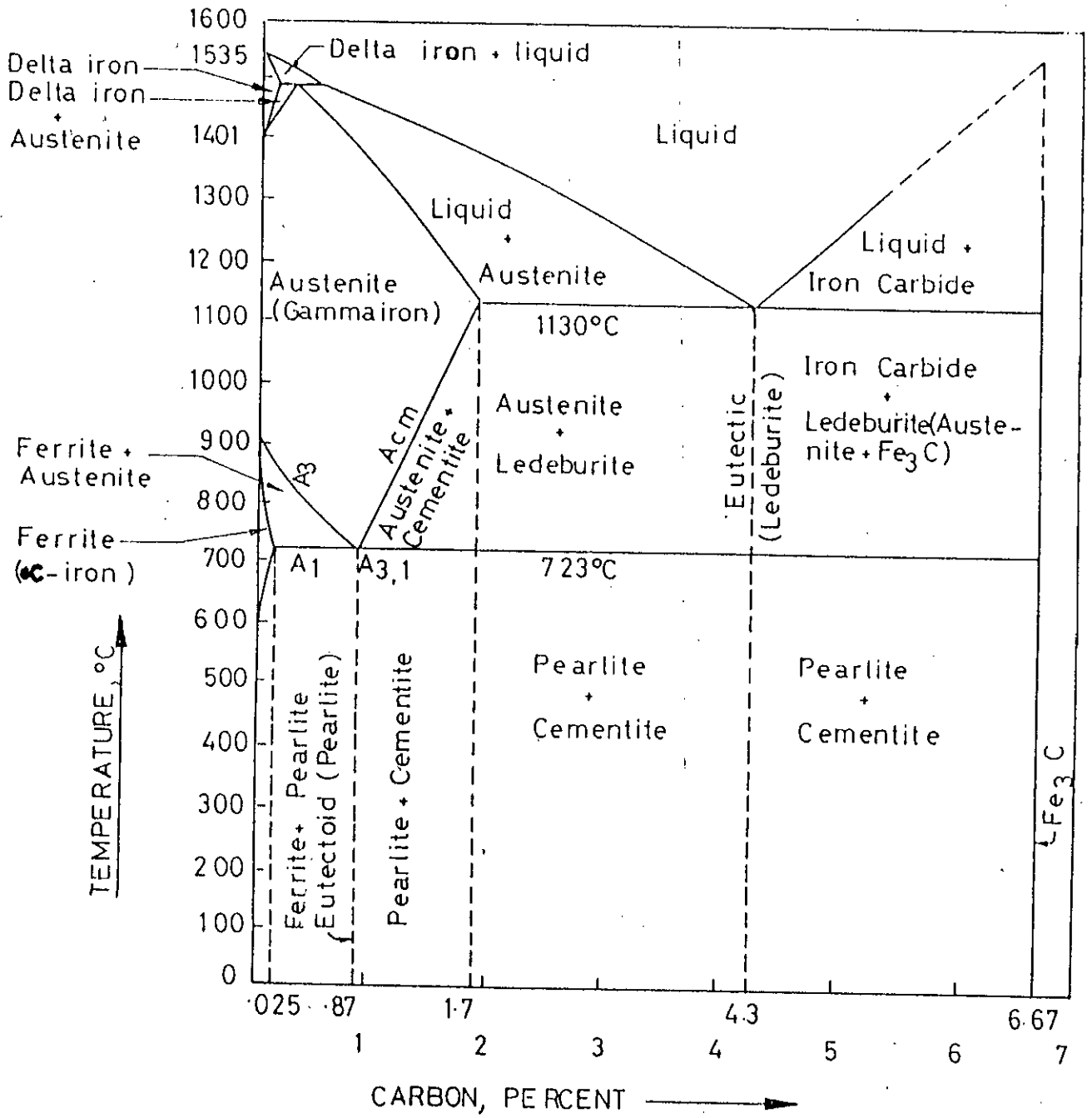
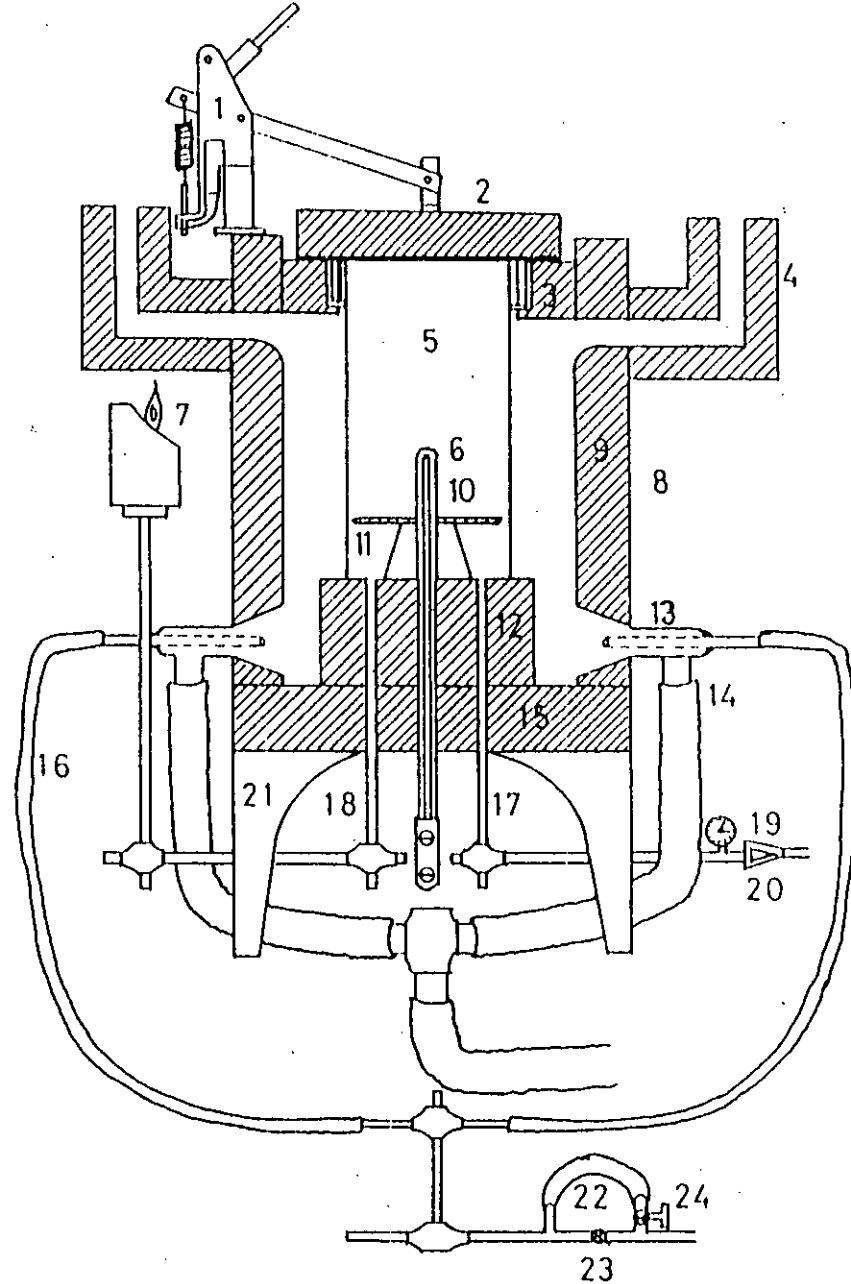


Figure 1: The iron and iron-carbide equilibrium diagram.



- | | |
|------------------------|-------------------------------------|
| 1. LID LIFTER | 2. LID AND CARBURIZING CHAMBER SEAL |
| 3. FILLER BRICK | 4. CHIMNEY |
| 5. CARBURIZING CHAMBER | 6. THERMOCOUPLE SHIELD |
| 7. SIGNAL FLAME | 8. OUTSIDE WALL |
| 9. FIRE BRICKS | 10. THERMOCOUPLE |
| 11. BOTTOM PLATE | 12. CIRCULAR PLATFORM |
| 13. GAS BURNER | 14. AIR SUPPLY |
| 15. FURNACE BOTTOM | 16. GAS SUPPLY |
| 17. GAS INLET | 18. GAS OUTLET |
| 19. PRESSURE GAUGE | 20. RATOR METER |
| 21. BOTTOM CASTING | 22. BY-PASS LINE |
| 23. SOLENOID VALVE | 24. VALVE |

Figure 2: Schematic diagram of gas fired carburizing furnace with automatic control system.

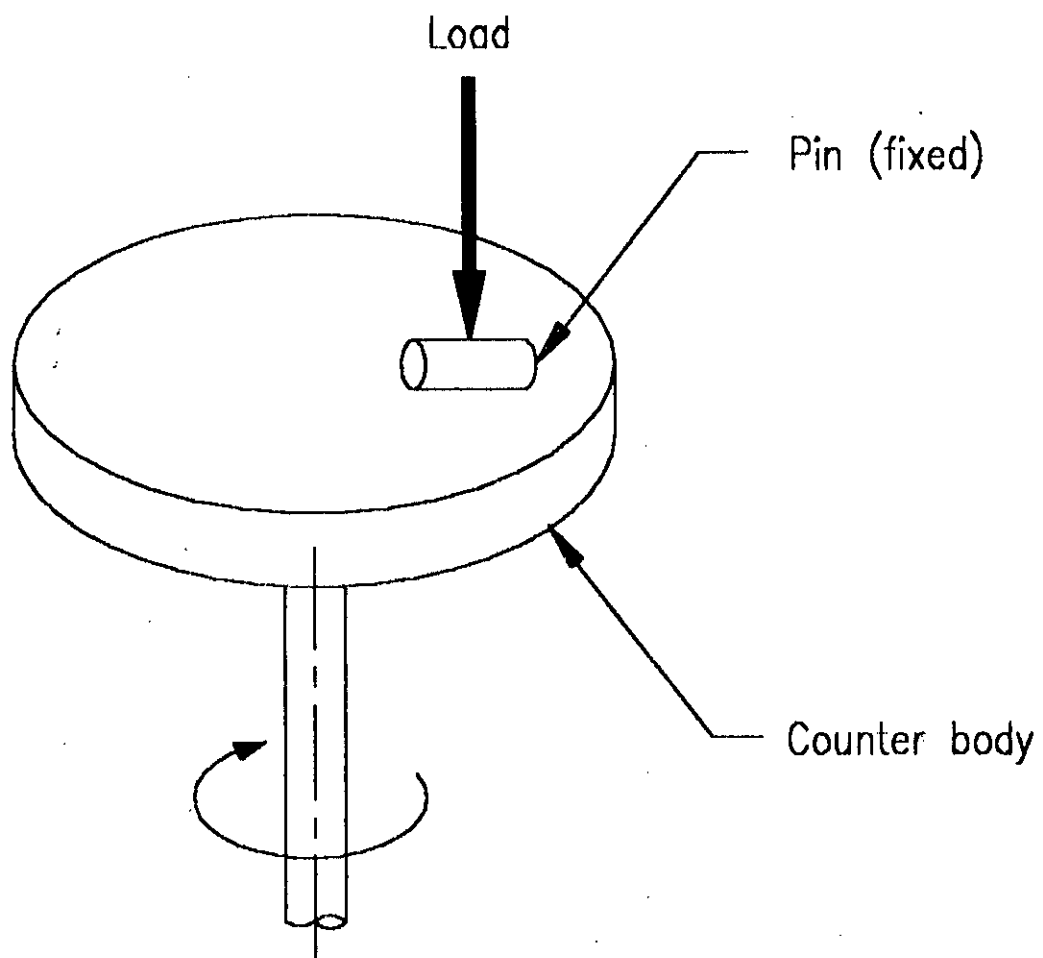
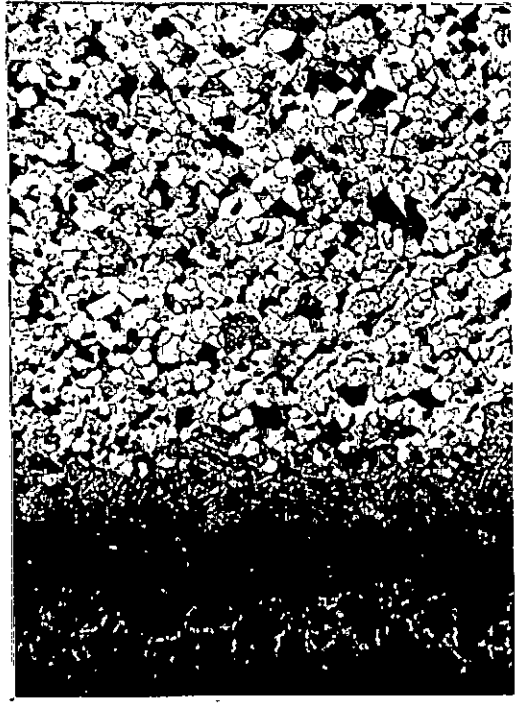


Figure 3: Schematic diagram of wear test apparatus.



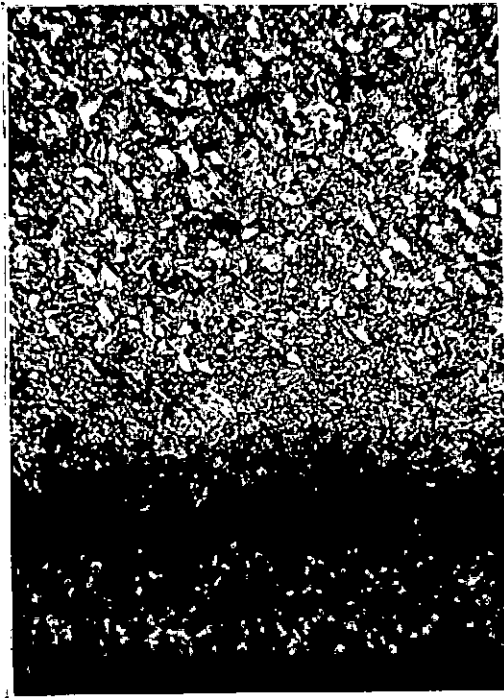
(a) Steel 1

X 150



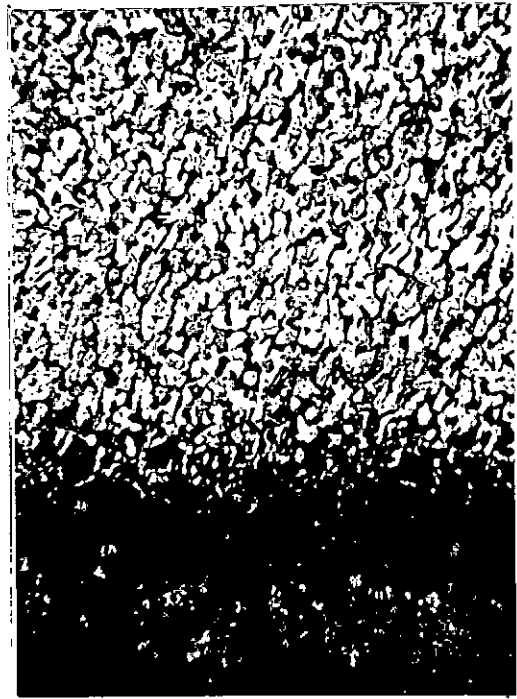
(b) Steel 2

X 150



(c) Steel 3

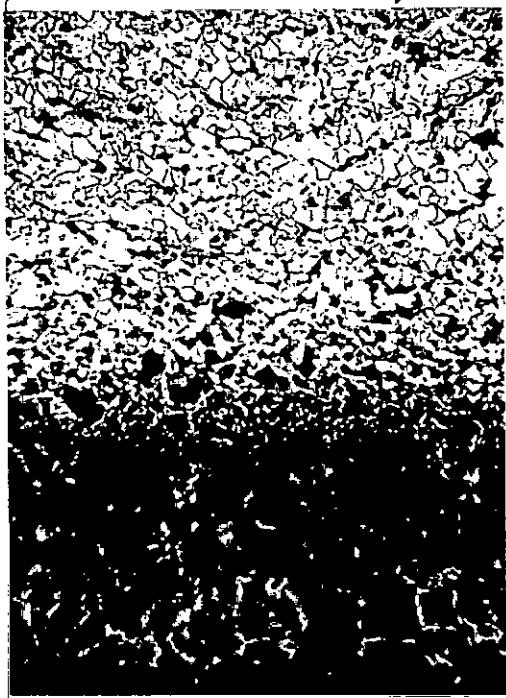
X 150



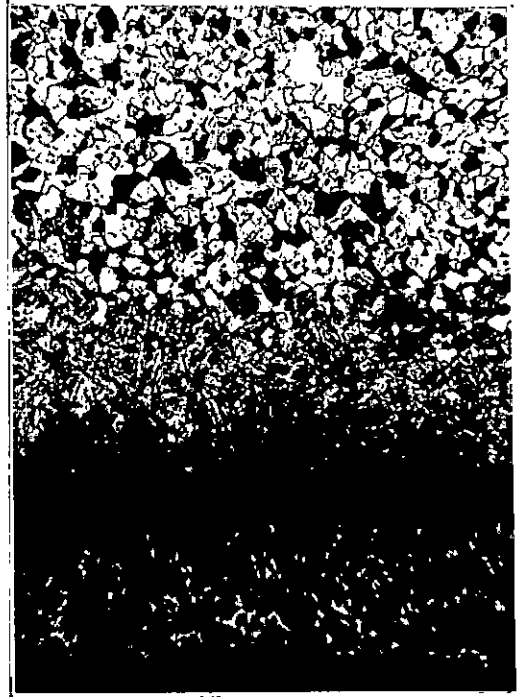
(d) Steel 4

X 150

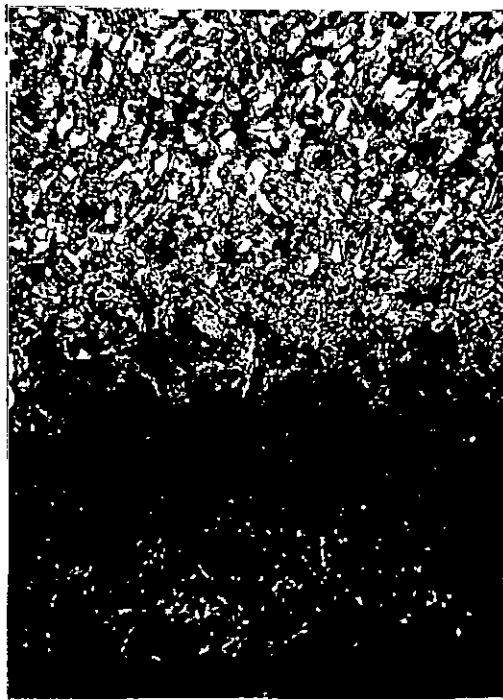
Figure 4: Optical micrographs showing microstructures and depth of case of steels 1 to 4 carburized at 950°C for 1 hour and cooled slowly in the furnace.



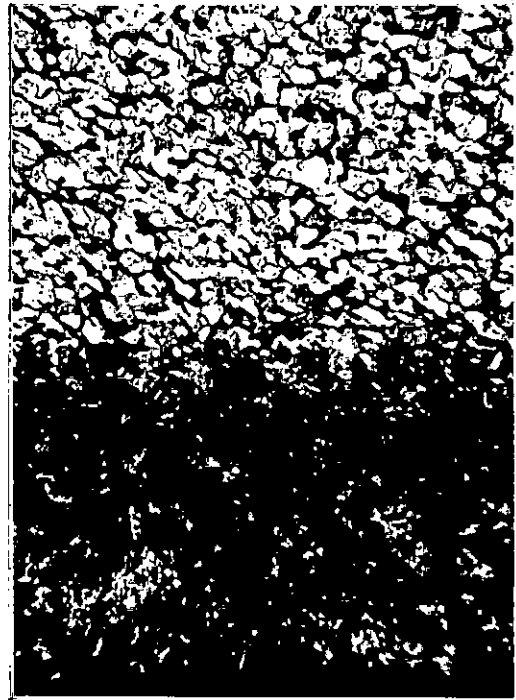
(a) Steel 1 X 150



(b) Steel 2 X 150



(c) Steel 3 X 150



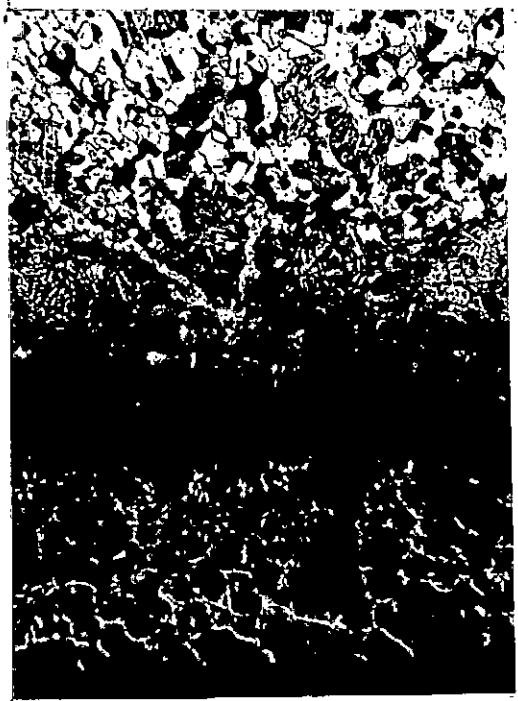
(d) Steel 4 X 150

Figure 5: Optical micrographs showing microstructures and depth of case of steels 1 to 4 carburized at 950°C for 2 hours and cooled slowly in the furnace.



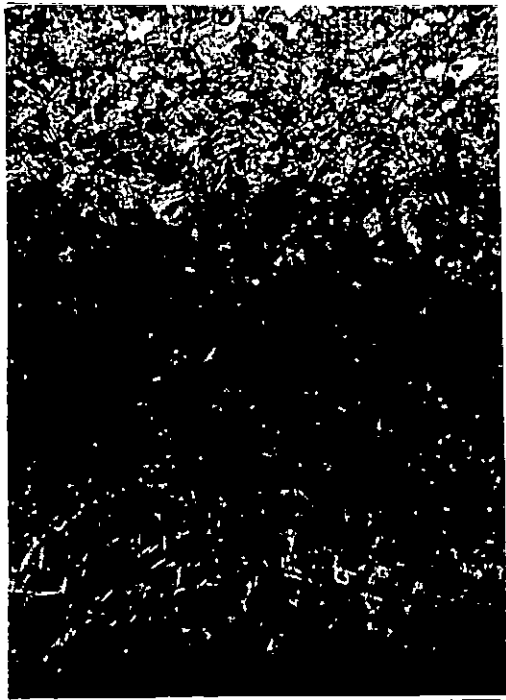
(a) Steel 1

X 150



(b) Steel 2

X 150



(c) Steel 3

X 150



(d) Steel 4

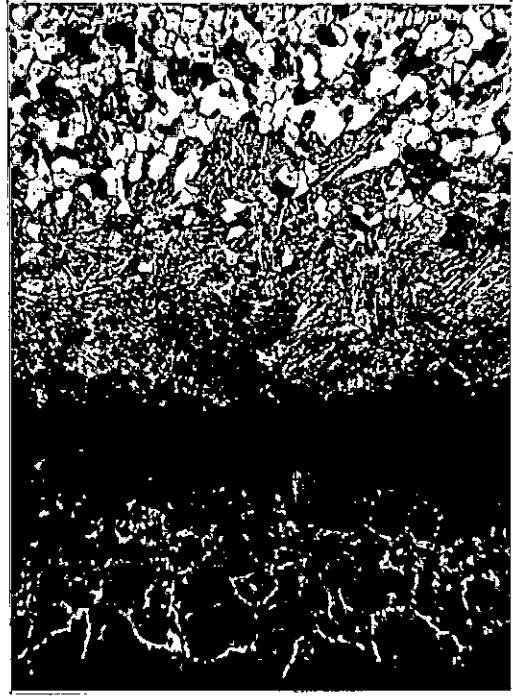
X 150

Figure 6: Optical micrographs showing microstructures and depth of case of steels 1 to 4 carburized at 950°C for 3 hours and cooled slowly in the furnace.



(a) Steel 1

X 150



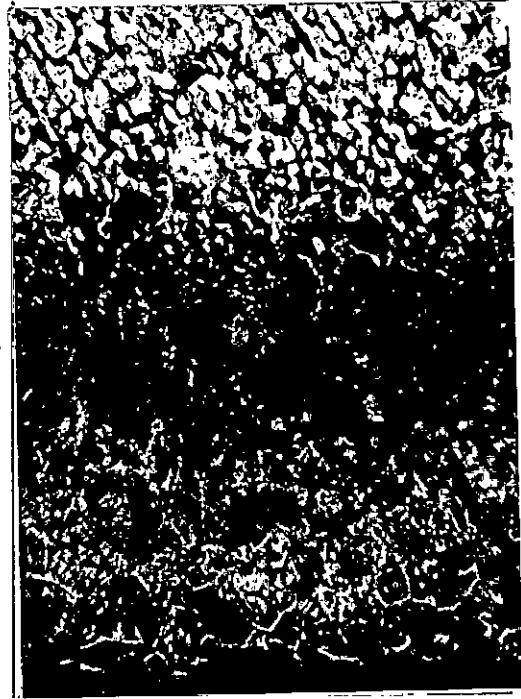
(b) Steel 2

X 150



(c) Steel 3

X 150



(d) Steel 4

X 150

Figure 7: Optical micrographs showing microstructures and depth of case of steels 1 to 4 carburized at 950°C for 4 hours and cooled slowly in the furnace.

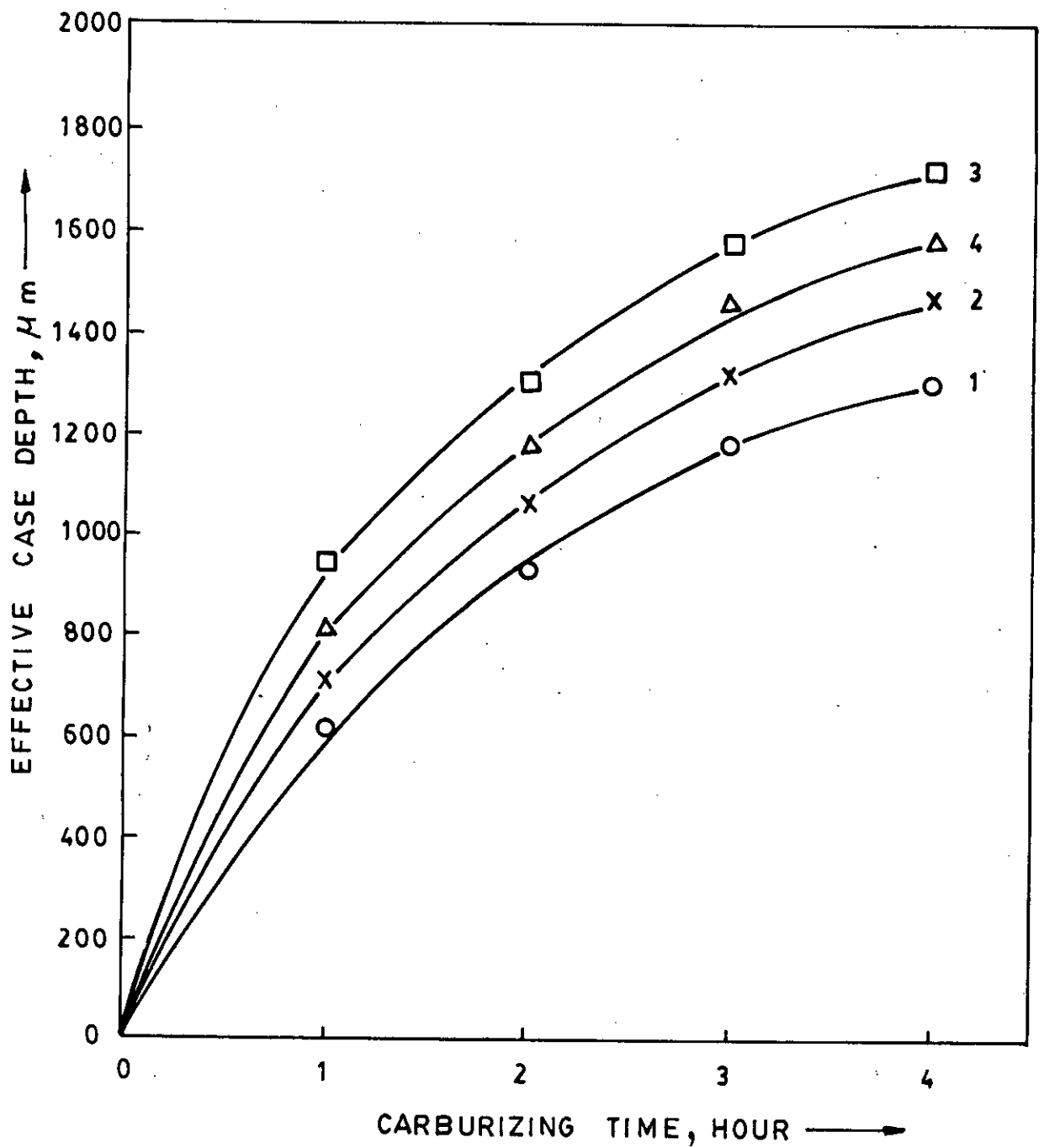


Figure 8: Variation of effective case depth with carburizing time of steels 1 to 4 carburized at 950°C.

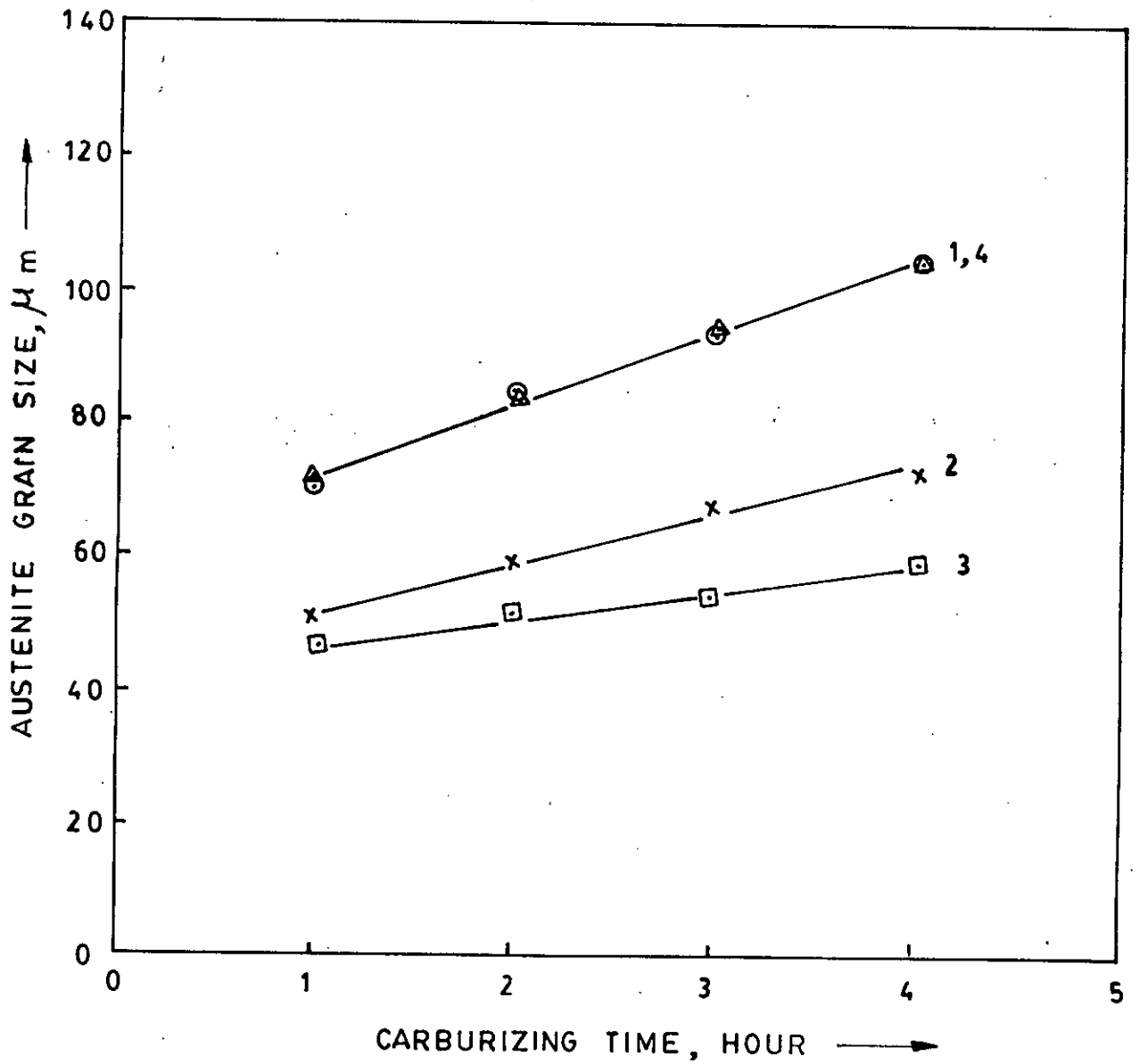
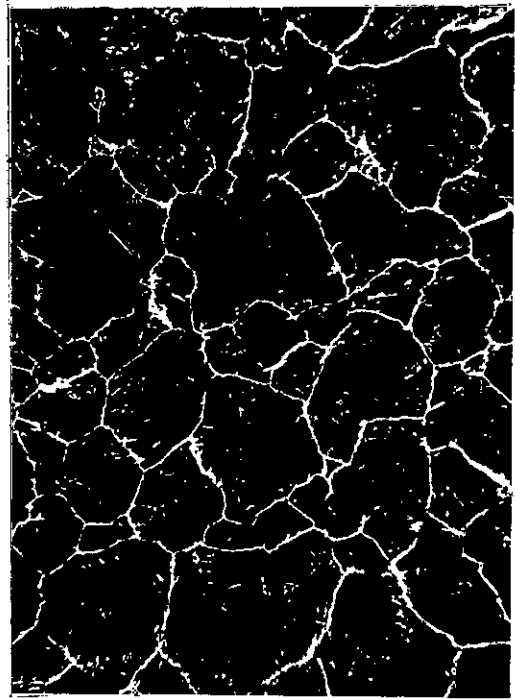


Figure 9: Variation of prior austenite grain size with carburizing time of the case of steels 1 to 4 carburized at 950°C.



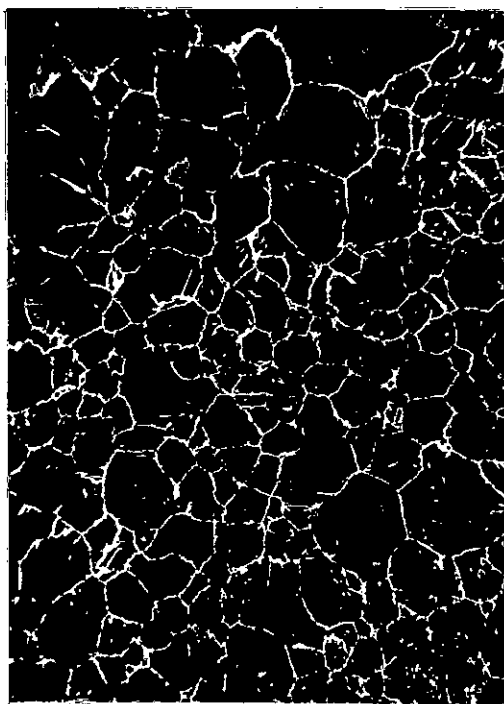
(a) Steel 1

X 300



(b) Steel 2

X 300



(c) Steel 3

X 300



(d) Steel 4

X 300

Figure 10: Optical micrographs showing prior austenite grain size in the case of steels 1. to 4 carburized at 950°C for 3 hours and cooled slowly in the furnace.



(a) Steel 1 X 300



(b) Steel 2 X 300



(c) Steel 3 X 300



(d) Steel 4 X 300

Figure 11: Optical micrographs showing the microstructures in the case of steels 1 to 4 carburized at 950°C for 1 hour, quenched in 10 percent brine and tempered at 160°C.



(a) Steel 1

X 300



(b) Steel 2

X 300



(c) Steel 3

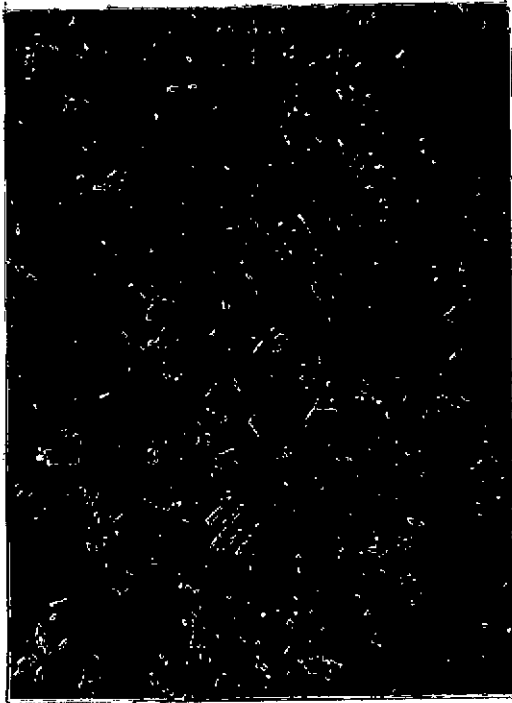
X 300



(d) Steel 4

X 300

Figure 12: Optical micrographs showing the microstructures in the case of steels 1 to 4 carburized at 950°C for 4 hours, quenched in 10 percent brine and tempered at 160°C.



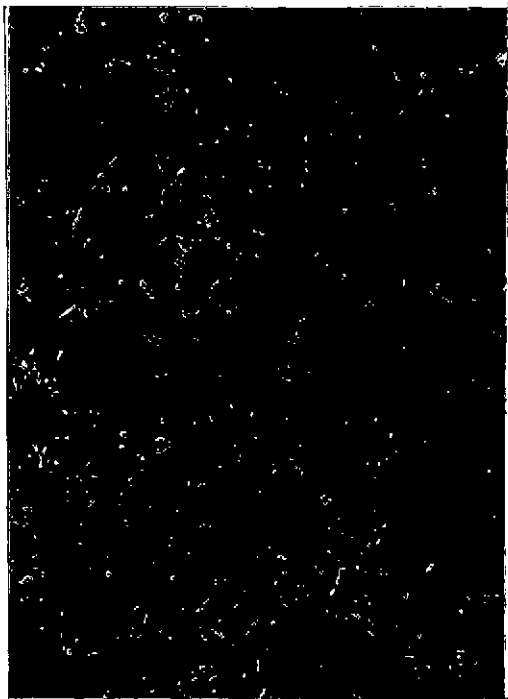
(a) Steel 1

X 300



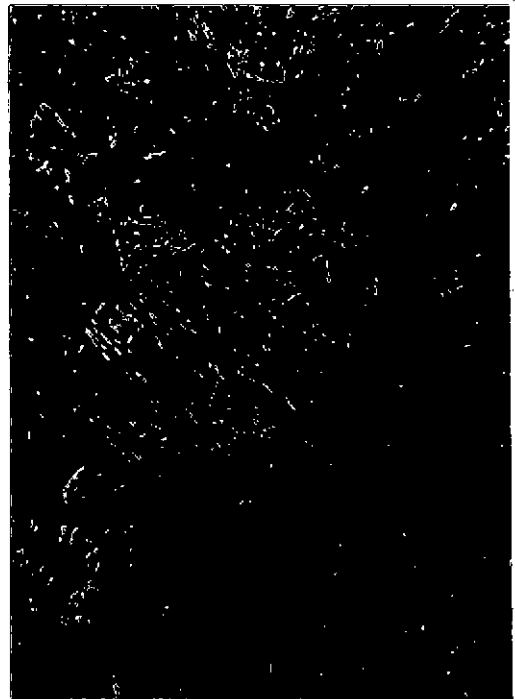
(b) Steel 2

X 300



(c) Steel 3

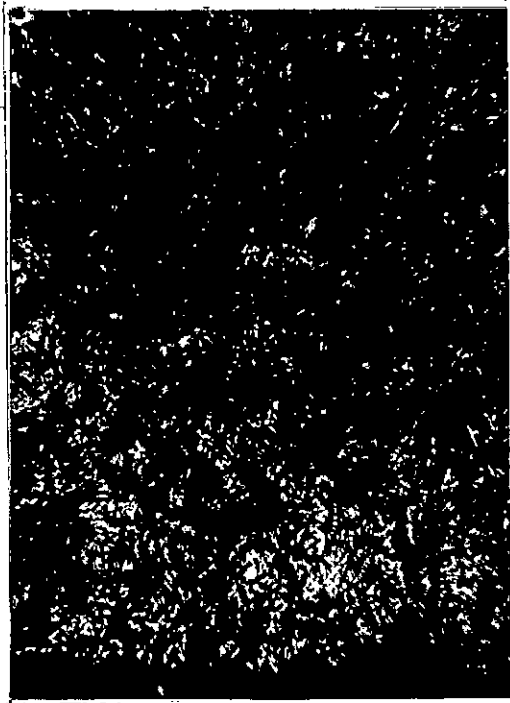
X 300



(d) Steel 4

X 300

Figure 13: Optical micrographs showing microstructures in the core of steels 1 to 4 carburized at 950°C for 2 hours, quenched in 10 percent brine and tempered at 160°C.



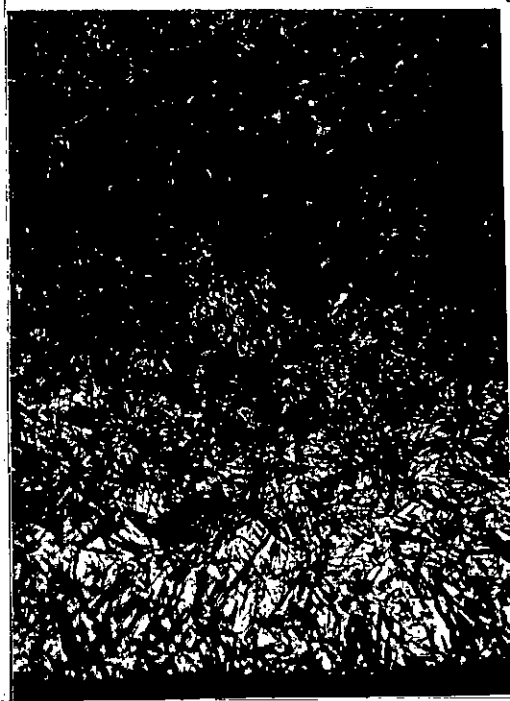
(a) Steel 1

X 300



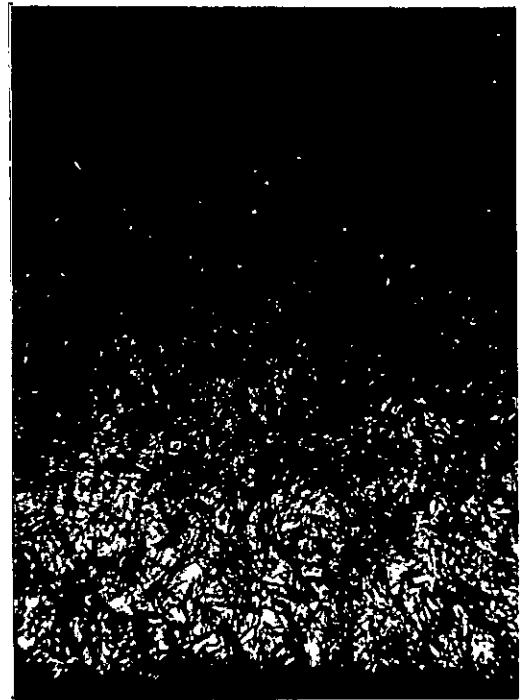
(b) Steel 2

X 300



(c) Steel 3

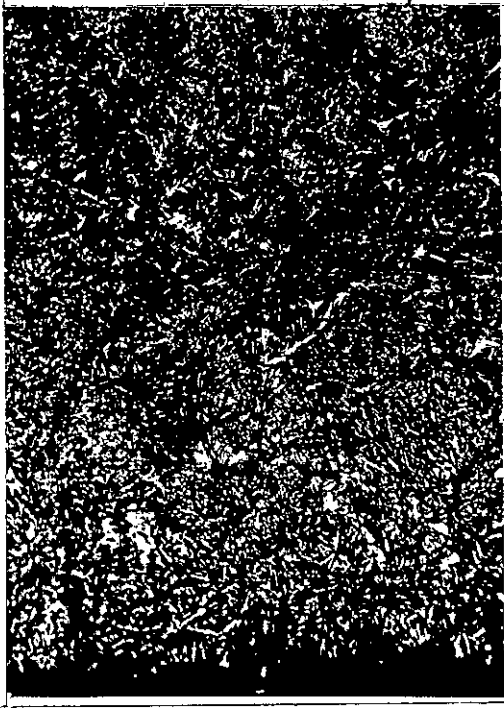
X 300



(d) Steel 4

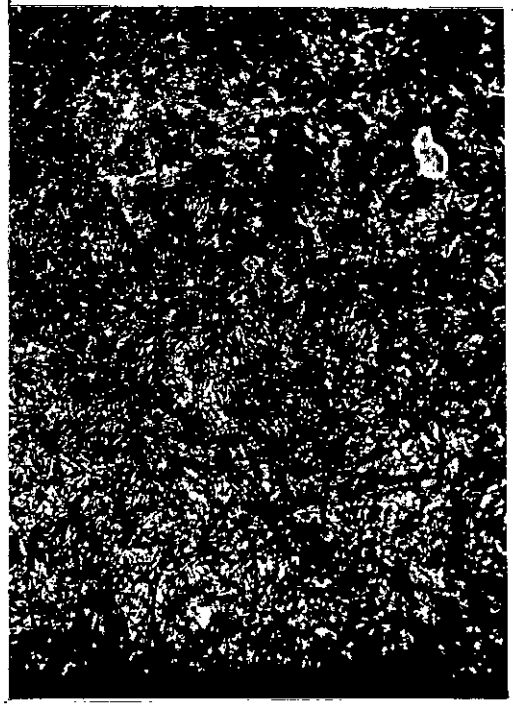
X 300

Figure 14: Optical micrographs showing microstructures in the case of steels 1 to 4 carburized at 950°C for 1 hour, quenched in 10 percent brine, sub-zero treated in liquid nitrogen and tempered at 160°C.



(a) Steel 1

X 300



(b) Steel 2

X 300



(c) Steel 3

X 300



(d) Steel 4

X 300

Figure 15: Optical micrographs showing microstructures in the case of steels 1 to 4 carburized at 950°C for 4 hours, quenched in 10 percent brine, sub-zero treated in liquid nitrogen and tempered at 160°C.

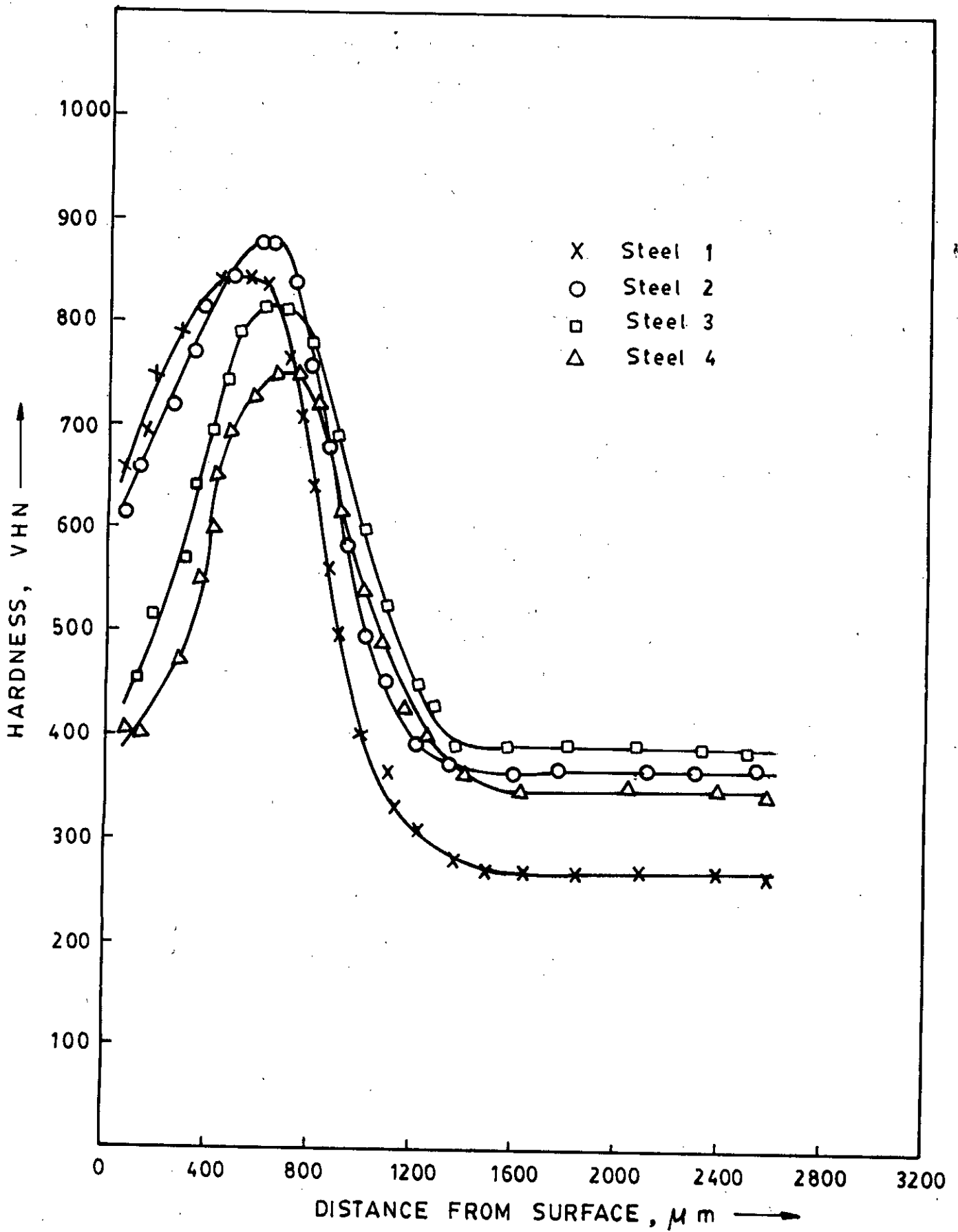


Figure 16: Microhardness profiles of steels 1 to 4 carburized at 950°C for 1 hour, quenched in 10 percent brine and tempered at 160°C.

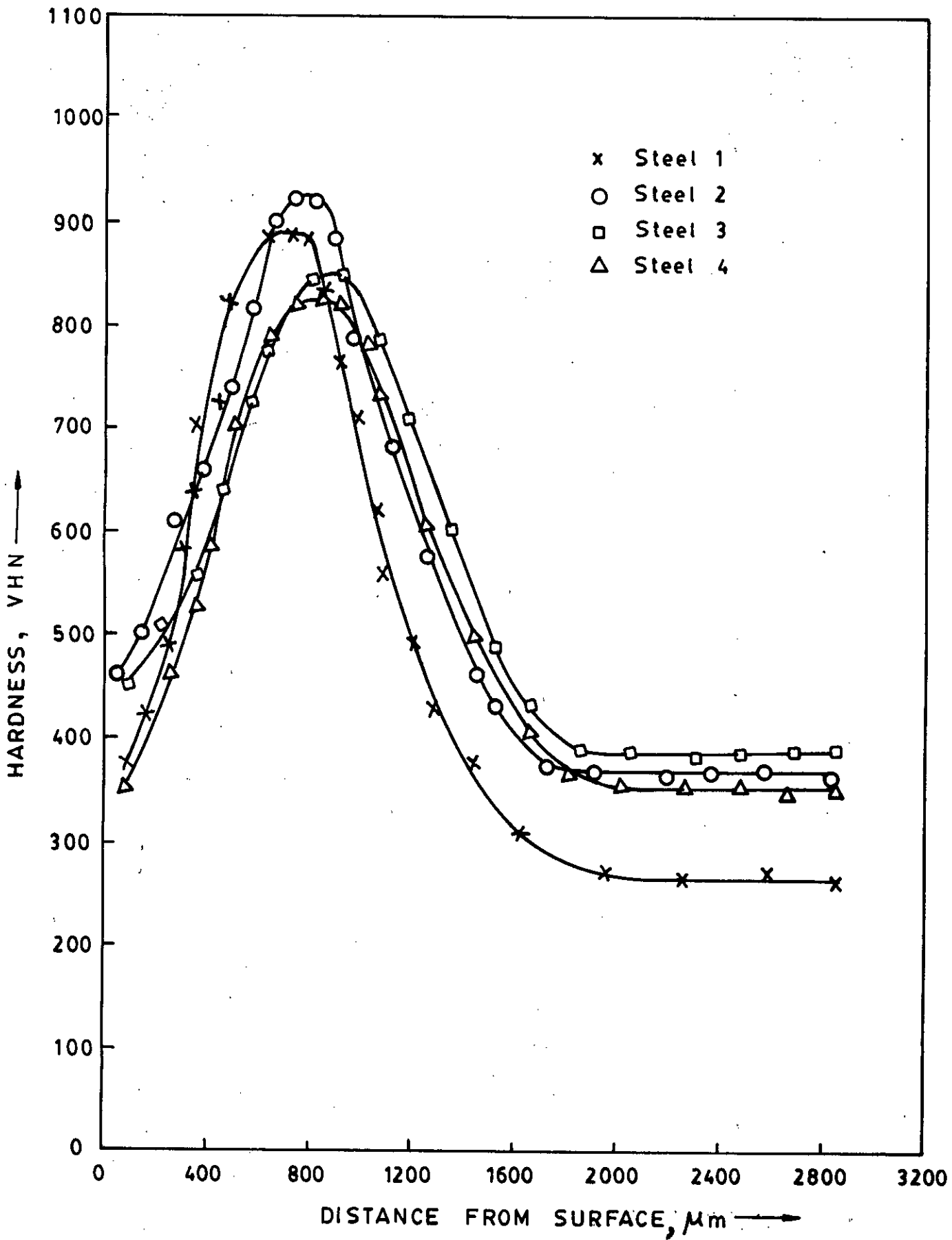


Figure 17: Microhardness profiles of steels 1 to 4 carburized at 950°C for 2 hours, quenched in 10 percent brine and tempered at 160°C.

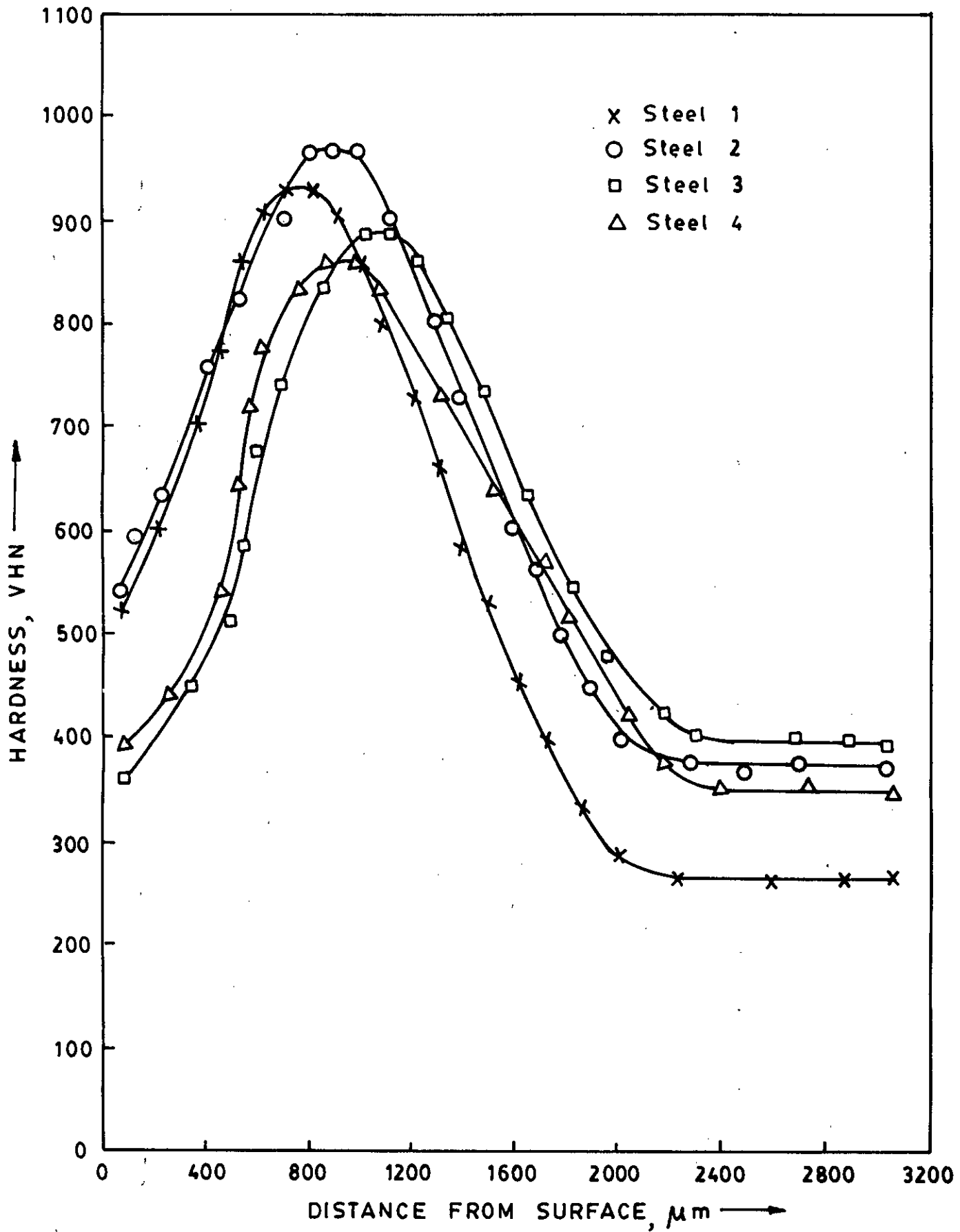


Figure 18: Microhardness profiles of steels 1 to 4 carburized at 950°C for 4 hours, quenched in 10 percent brine and tempered at 160°C.

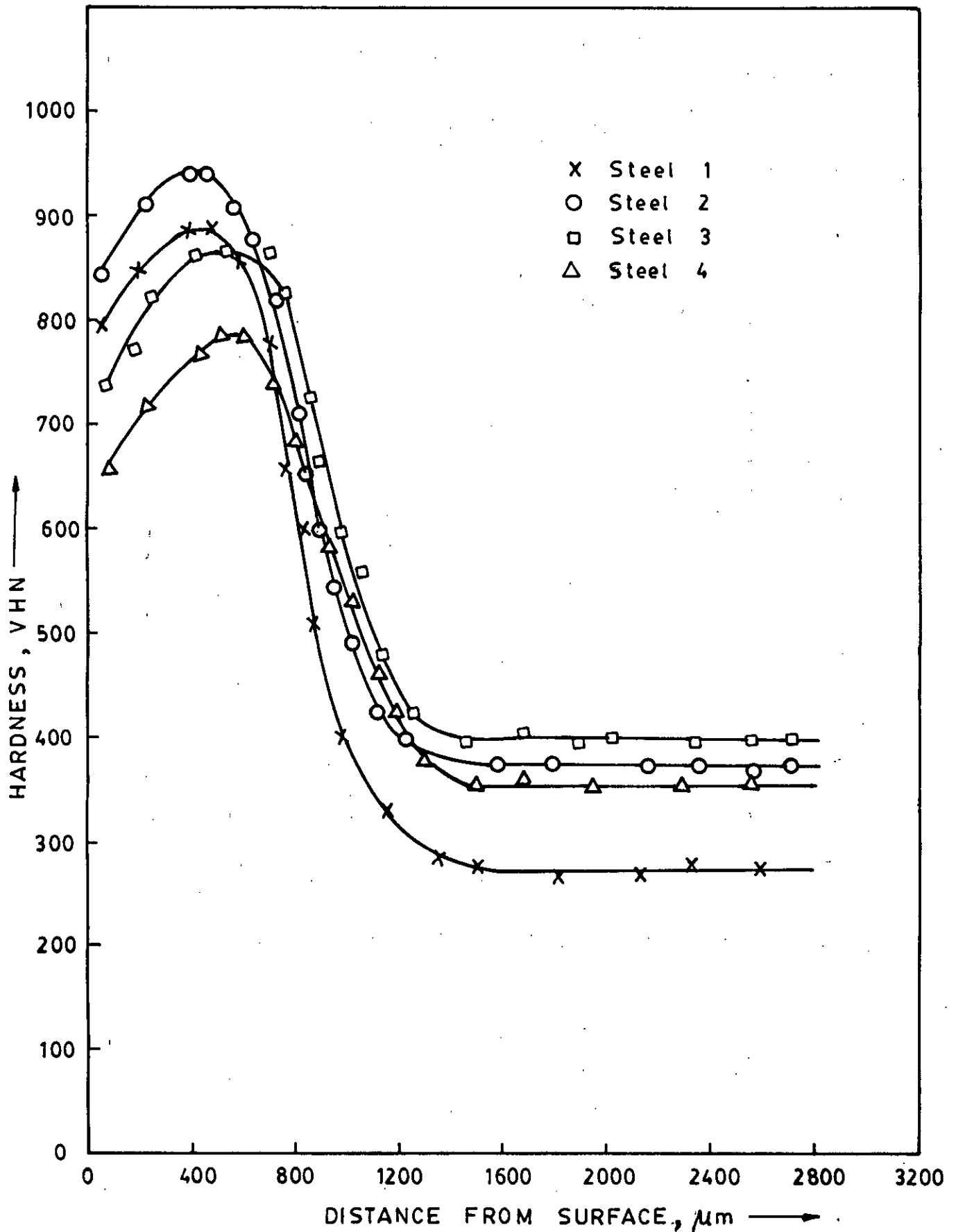


Figure 19: Microhardness profiles of steels 1 to 4 carburized at 950°C for 1 hour, quenched in 10 percent brine, sub-zero treated in liquid nitrogen and tempered at 160°C.

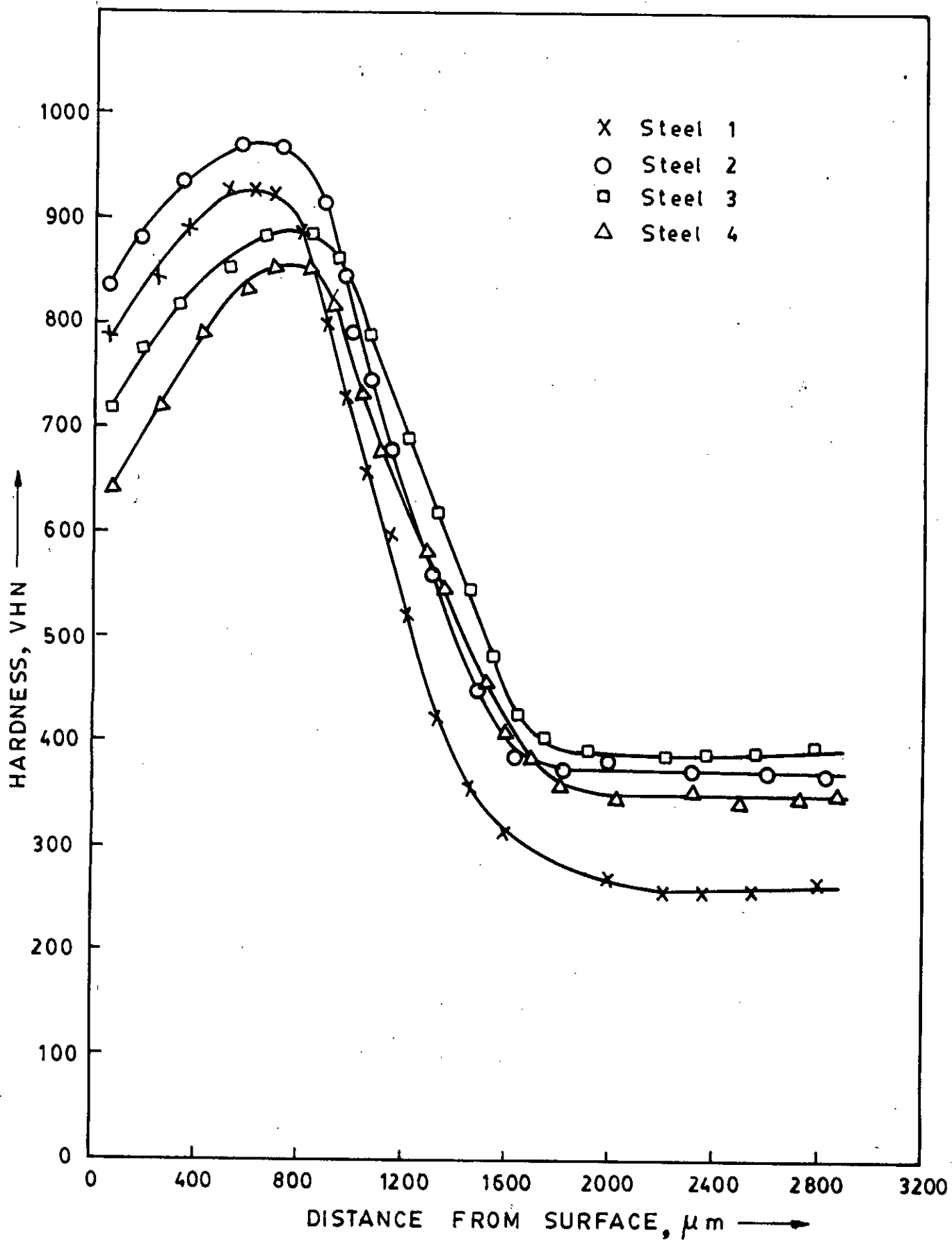


Figure 20: Microhardness profiles of steels 1 to 4 carburized at 950°C for 2 hours, quenched in 10 percent brine, sub-zero treated in liquid nitrogen and tempered at 160°C.

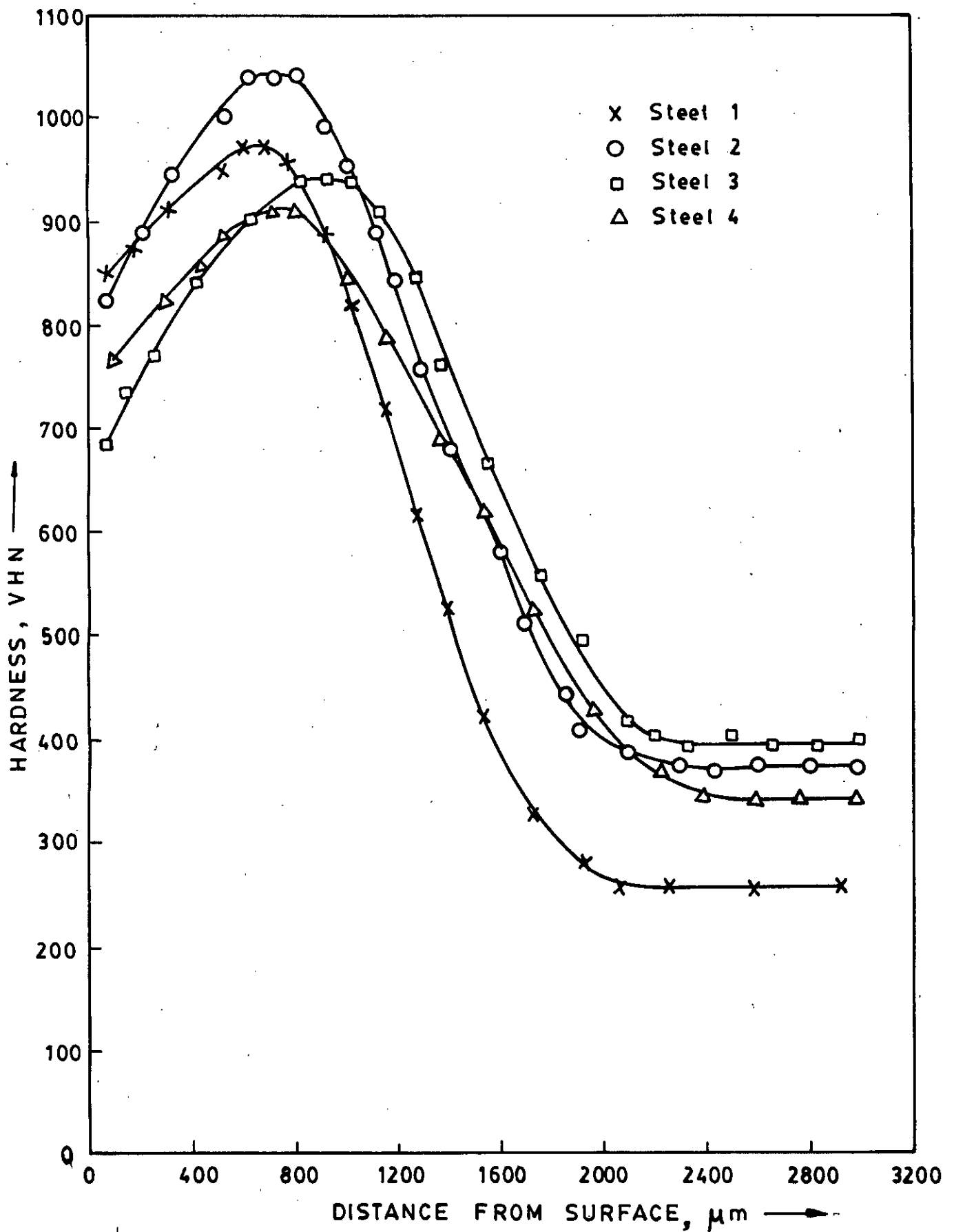


Figure 21: Microhardness profiles of steels 1 to 4 carburized at 950°C for 4 hours, quenched in 10 percent brine, sub-zero treated in liquid nitrogen and tempered at 160°C.

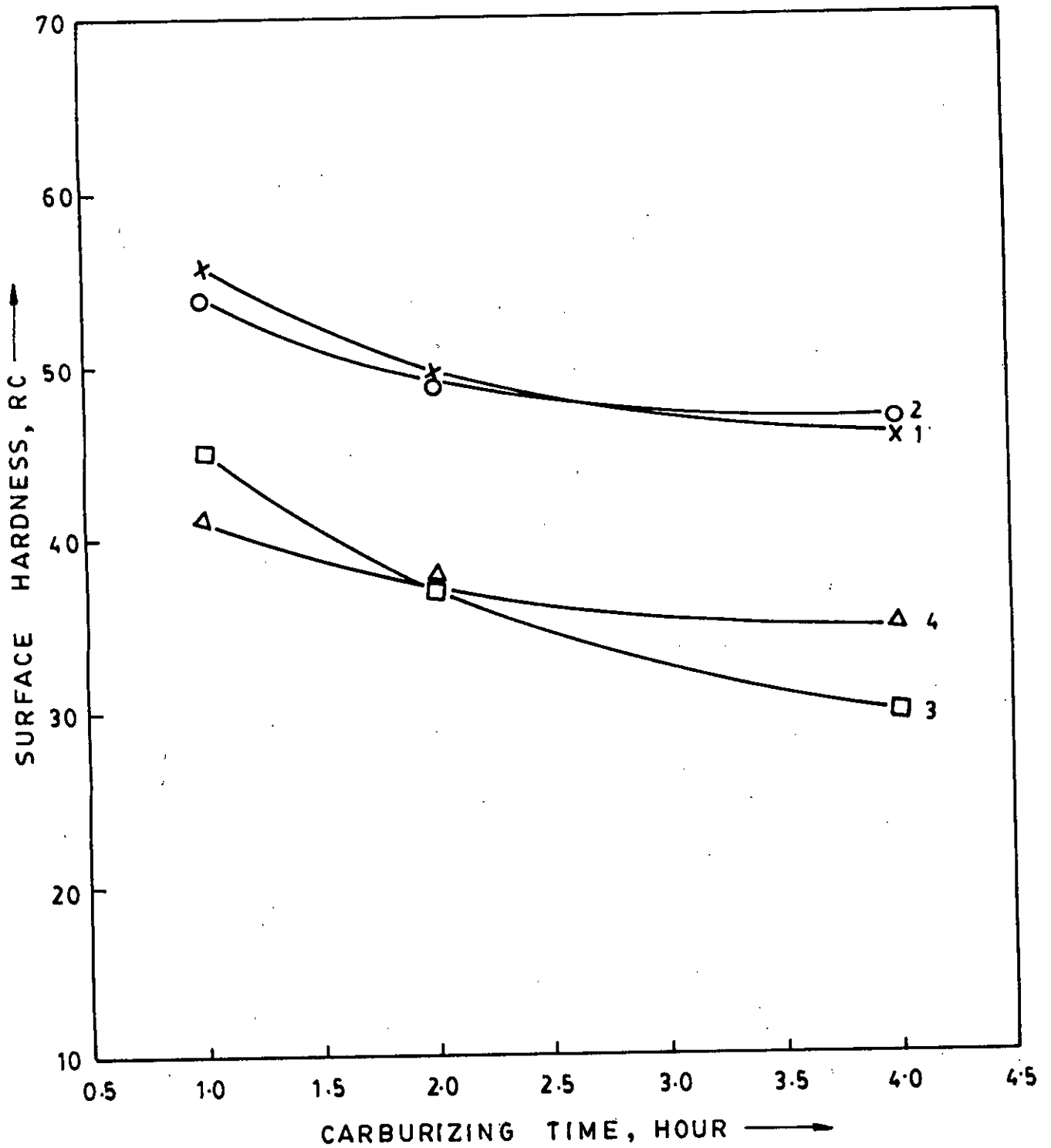


Figure 22: Variation of the surface hardness with carburizing time of steels 1 to 4 carburized, hardened and tempered.

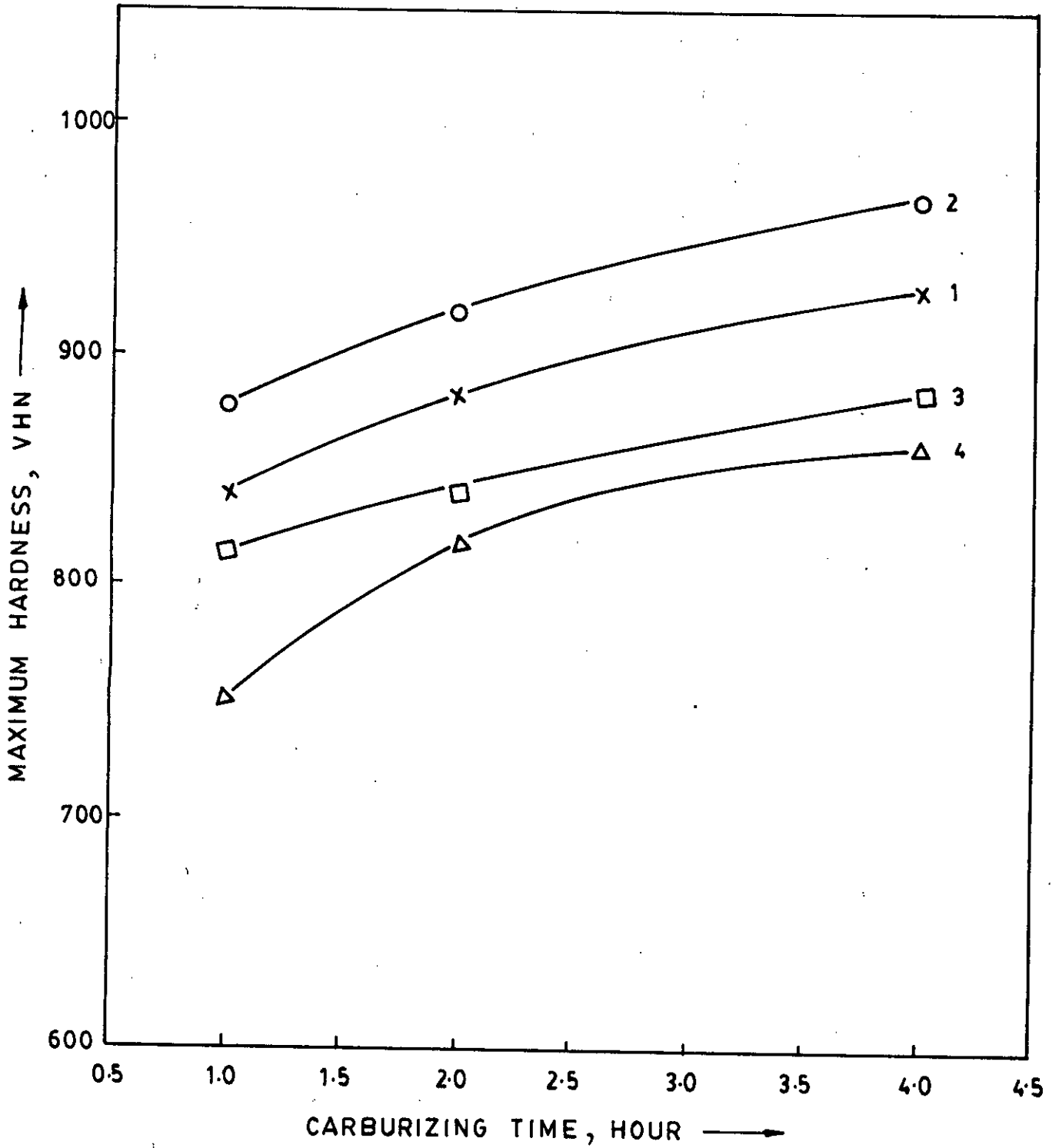


Figure 23: Variation of the maximum hardness with carburizing time of the case of steels 1 to 4 carburized, hardened and tempered.

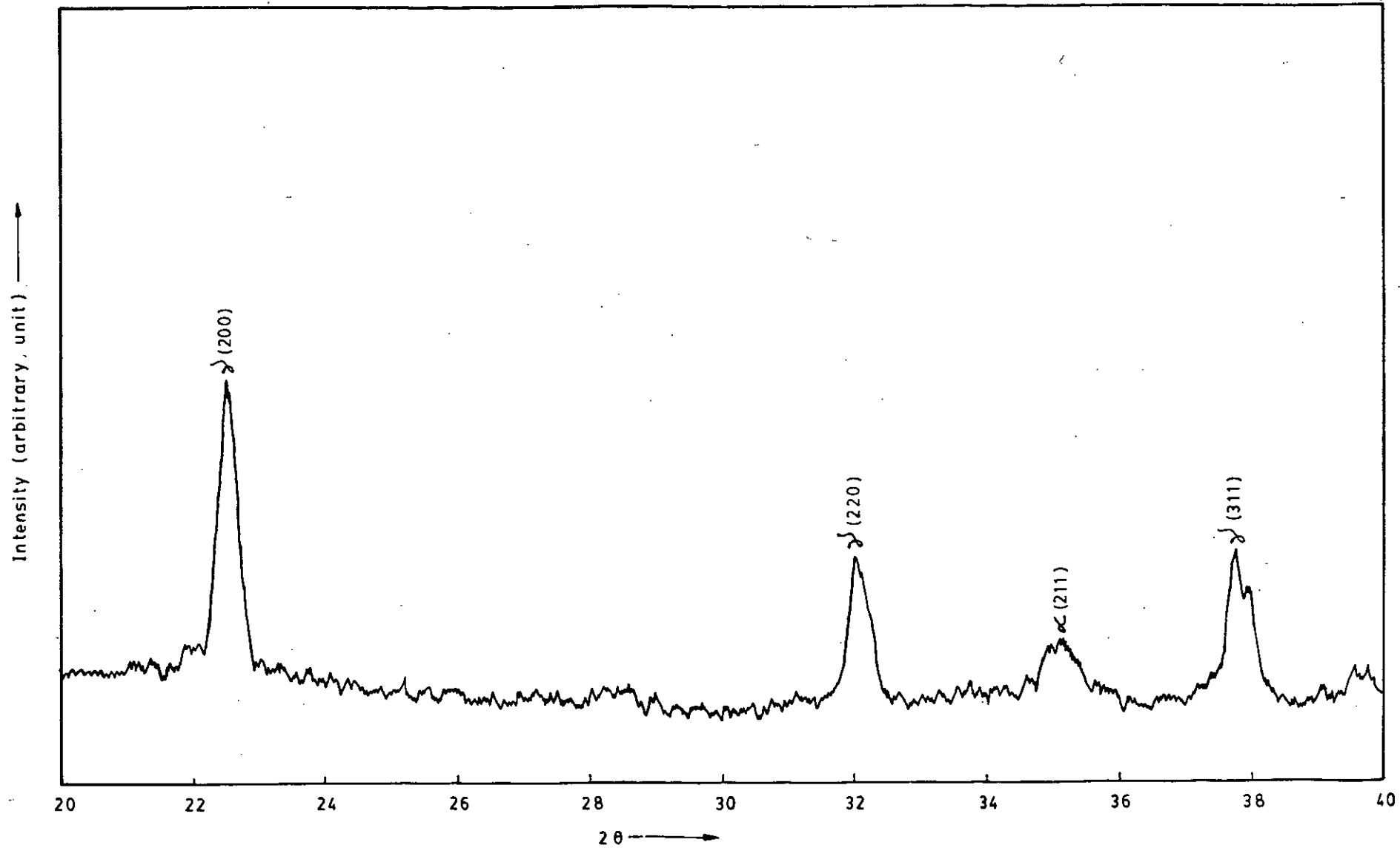


Figure 24: X-ray diffraction pattern of steel 2 carburized at 950°C for 2 hours, quenched in 10 percent brine and tempered at 160°C (QT).

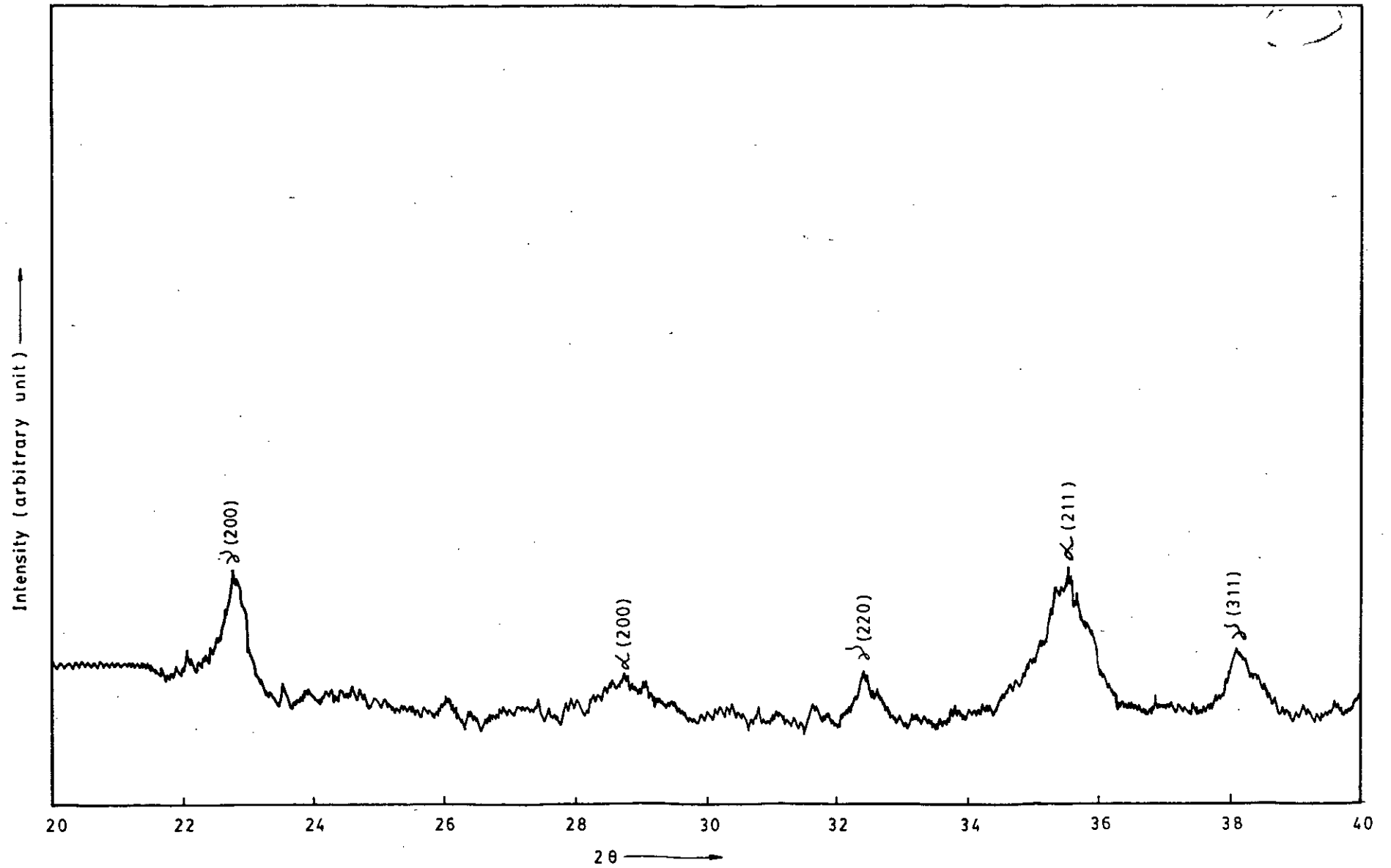


Figure 25: X-ray diffraction pattern of steel 2 carburized at 950°C for 2 hours, quenched in 10 percent brine, sub-zero treated in liquid nitrogen and tempered at 160°C (QST).

APPENDIX

DETERMINATION OF RETAINED AUSTENITE BY X-RAY DIFFRACTION

TECHNIQUE

The retained austenite content in the carburized case of steels 1 to 4 was determined by x-ray diffraction using direct comparison technique and various information explained by B. D. Cullity³³ in the Text "Elements of X-ray Diffraction".

Calculation of R for martensite

Plane for the peak = 211

Lattice parameter $a = 2.85 \times 10^{-10}$

$2\theta = 35.5^\circ$

Volume $v = (2.85)^3 \times 10^{-30} = 23.12 \times 10^{-30}$

$(1/v)^2 = 1.866 \times 10^{57}$

$\sin\theta/\lambda = 0.429$

$f = 12.73$

$|F|^2 = 4 \times f^2 = 4 \times 12.73 \times 12.73 = 648$

$p = 24$ (Appendix 12)

$e^{-2M} = 0.88$ (Table 4.20)

$(1 + \cos^2 2\theta) / \sin^2 \theta \cdot \cos \theta = 18.67$

$R_M = (1/v)^2 [F]^2 p \{(1 + \cos^2 2\theta) / \sin^2 \theta \cdot \cos \theta\} e^{-2M}$

$= 1.866 \times 10^{57} \times 648 \times 24 \times 18.67 \times 0.88$

$= 476789.5 \times 10^{57}$

Calculation of R for Austenite

Plane for the peak = 200

Lattice parameter $a = 3.60 \times 10^{-10} \text{m}$

$2\theta = 22.8^\circ$

Volume $v = (3.60)^3 \times 10^{-30} = 46.66 \times 10^{-33}$

3

$$(1/v)^2 = 4.59 \times 10^{56}$$

$$\sin\theta/\lambda = 0.279$$

$$f = 16.92$$

$$|F|^2 = 16f^2 = 16 \times 16.92 \times 16.92 = 4580.6$$

$$p = 6$$

$$e^{-2M} = 0.93$$

$$(1 + \cos^2 2\theta) / \sin^2 \theta \cdot \cos \theta = 48.3$$

$$\begin{aligned} R_A &= (1/v)^2 [|F|^2 p \{(1 + \cos^2 2\theta) / \sin^2 \theta \cdot \cos \theta\}] e^{-2M} \\ &= 4.59 \times 10^{56} \times 4580.6 \times 6 \times 48.3 \times 0.93 \\ &= 609300.83 \times 10^{57} \end{aligned}$$

Carburized, Quenched and Tempered specimens:

$$I_A = 111$$

$$I_M = 51$$

$$I_A/I_M = R_A C_A / R_M C_M$$

$$C_A = 1.703 C_M$$

$$C_M + C_A = 1$$

$$C_M = 0.3699$$

$$C_A = 0.6301$$

Martensite content = 37 percent

Austenite content = 63 percent

Carburized, Quenched and Sub-zero treated and Tempered specimens:

$$I_A = 62$$

$$I_M = 127$$

$$I_A/I_M = R_A C_A / R_M C_M$$

$$C_A = 0.382 C_M$$

$$C_M + C_A = 1$$

$$C_M = 0.7235$$

$$C_A = 0.2765$$

Martensite content = 72 percent

Austenite content = 28 percent

