GAS CARBURIZATION OF LOW ALLOY STEELS

By

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A thesis submitted to the Department of Metallurgical Engineering, BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY, DHAKA, in partial fulfilment of the requirements for the degree of Master of Science in Engineering (Metallurgical).

February, 1988

BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY,
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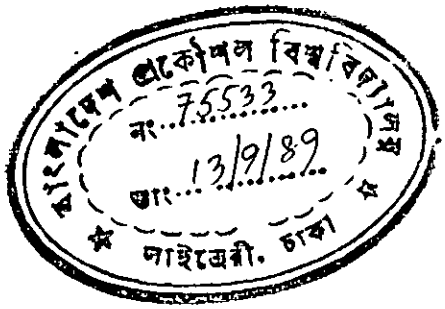
DECLARATION

This is to assert that this work has been carried out by me under the supervision of Dr. Md. Mohar Ali, Associate Professor, Department of Metallurgical Engineering, BUET, Dhaka, and it has not been submitted elsewhere for the award of any other degree or diploma.



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ACKNOWLEDGEMENTS

The author expresses his deep indebtedness and sincere gratitude to Dr. Md. Mohar Ali, Associate Professor, Department of Metallurgical Engineering, BUET for his valuable suggestions, inspiring guidance and constant encouragement in all stages of the projects as well as in preparing this thesis.

The author is very much grateful and expresses his profound gratitude to Dr. Md. Serajul Islam, Prof. and Head, Dept. of Metallurgical Engineering for his kind help at various stages of this research work particularly in the design and construction of the gas fired gas carburizing furnace with automatic control system and for the many helpful discussions he had with him.

The author is very much indepted to Prof. M. Ibrahim for his inspiration and for the many helpful discussions he had with him.

The author is also grateful to Prof. E. Haque for the helpful discussions he had with him.

The author wishs to thank Mr. F.H. Bhuiyan, Senior Foundry Instructor, Mr. L. Rahman, Senior Crafts Instructor, Babu Binoy Bhushan Shaha, Senior Laboratory Instructors and other staff members in the Department for their kind help during the experiments, preparation of specimens and in printing of the micrographs.

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ABSTRACT

Gas carburization of four 0.10% carbon steel samples containing about 0.35 to 0.85% chromium and about 1.20% nickel singly or in combination and of five 0.15% carbon steel samples containing small additions of vanadium, aluminium and nitrogen singly or in combination has been carried out in a natural Titas gas atmosphere at a temperature of 950°C and a pressure of about 15 psia for time periods ranging from 1 to 5 hours followed by cooling slowly in the furnace. The microstructures of these steels were investigated by optical microscopy, and the austenite grain size of the case and the case depths were determined.

Another batch of specimens of the above steels was carburized for different periods of time under identical conditions of temperature and pressure and quenched in 10% brine from the carburizing temperature of 950°C after pre-cooling to 860°C in the furnace followed by tempering at a low temperature of 160°C. The structure and properties such as hardness and hardenability were studied systematically by optical microscopy and microhardness measurements respectively.

The Charpy V-notch specimens made of the steels were heated at 950°C for 1 hour and quenched in 10% 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 steels.

It was found that chromium increased the case depth of the carburized steels while nickel decreased it. Microalloying elements such as vanadium with or without nitrogen and aluminium with nitrogen reduced the case depth of the carburized steels. Vanadium without nitrogen was more effective than vanadium with nitrogen in reducing the case depth while aluminium with nitrogen is the most effective of all in reducing the case depth.

Chromium as Cr_3C and nickel refined the austenite grain size of the carburized case but nickel was found more effective than chromium in inhibiting the austenite grain growth. The undissolved particles of vanadium carbide, VC, vanadium carbonitride, V(C,N) and aluminium nitride, AlN, refine the austenite grain size of the carburized case, but V(C,N) is much more effective than VC and AlN is the most

effective in inhibiting the austenite grain growth in the case of the carburized steels.

It was also observed that both chromium and nickel promote the formation of retained austenite but chromium is more effective than nickel in promoting the formation of retained austenite in the case of carburized and hardened steels. Vanadium with nitrogen and not vanadium without nitrogen is effective in promoting the formation of retained austenite in the case of carburized and hardened steels while aluminium is the most effective in promoting the formation of retained austenite in the case of the steels.

Microhardness measurements have shown that chromium in solution and as Cr_3C increases and nickel in solution decreases the hardness of the case while both of them increase the core hardness although chromium is more effective than nickel as far as core hardness is concerned. The undissolved particles of VC and V(C,N) increase the case and core hardness values while AlN reduces them; V(C,N) is more effective than VC in raising the hardness of the case and core. The hardenability is found to increase with the increase of austenite grain size and with the extent of carbon penetration in the case of carburized steels.

Impact test results have shown that nickel and chromium reduce the toughness of the core of low carbon steels in the carburized and hardened condition. VC particles reduce the toughness markedly while V(C,N) and AlN particles improve the toughness. AlN particles are much more beneficial for toughness than V(C,N) particles.

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CHAPTER-1
INTRODUCTION

The service conditions of many machine parts made of steel such as gears, shafts, cams, etc demand very hard and wear-resistant surfaces but with tough and wear-resistant cores. Such a combination of properties is not usually possible from the commercial steels. A low carbon steel containing carbon less than 0.20 per cent is sufficiently tough while a high carbon steel containing carbon upto about 1.00 per cent posseses 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. Gas carburizing is the most satisfactory and widely used method of surface hardening of low carbon (usually less than 0.20 per cent) steel. It is the process by which the carbon content of the surface of a steel is increased using hydrocarbon gases as the carburizing medium. Bangladesh has got an enormous deposit of natural gas. It has been shown[1,2] that the locally available natural Titas gas which contains 97.2% CH_4, 1.8% C_2H_6, 0.5% C_3H_8 and 0.2% C_4H_{10} and other higher hydrocarbons and 0.3% N_2,[3] can be successfully used as a carburizing medium for surface hardening of low carbon steel parts.

Many machine parts such as drive gears, drive shafts, cam shafts, ball joints, steering parts, bearing and bearing races, connecting rod bolts, studs, kingpins, bus and truck gear, crankshafts, axles, etc used for heavy duty applications require 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 carburized and hardened condition. Moreover, advanced aircraft and helicopter requirements continually demand improved transmission gear materials with increased load carrying capacity, longer life and an ability to retain their strength and hardness at elevated temperatures upto about 600°F (315°C). Ability of a material to retain hardness and fatigue resistance at elevated temperatures of about 600°F (315°C) is an important design criterion for high performance gears and rolling elements such as those found in helicopter transmissions. Low hardness can result in permanent surface deformation and distress during operation, which lead to a premature failure.[4] Those parts used for heavy duty applications and those used for high temperature applications are usually made of low carbon low alloy steels containing Ni, Cr and Mo, etc.[4,5,6] These alloying elements are effective in improving the hardenability of the steels and consequently strengthening the matrix of the carburized steel.[7] Although some work has already been done on the carburizing behaviour of these low alloy steels containing Ni, Cr and Mo, etc. in the advanced countries, a detailed information about the effect of Ni, Cr and Mo on the structure and properties of the case and core of carburized steels is not available.

Nickel when added to steel remains in solution both in austenite and ferrite, decreases the transformation temperature and improves tensile strength and toughness of the hardened steels. Chromium is soluble upto 13% in γ iron and has unlimited solubility in α iron. It also forms chromium carbide and thus increases hardness, wear resistance, hardenability and strength of hardened steels. Mo has a limited solubility in γ iron and in α iron and is a strong carbide former. It has a strong effect on hardenability and like Cr increases the high temperature hardness and strength of steels. This element is most

often used in combination with Ni or Cr or both Ni and Cr. For carburizing applications it improves the wear resistance of the case and the toughness of the core.[5,6] So it is important to study the effect of Ni and Cr separately or in combination on the effect of structure and properties of the case and core of carburized steels. This work has, therefore, been undertaken to study the effects of the addition of about 0.35-0.85% Cr and about 1.2% Ni separately or in combination on the structure and properties of the case and core of carburized 0.1%C steel.

It is now well established that a small amount vanadium (0.1 to 0.2%), aluminium (0.04 to 0.06%) and nitrogen (about 0.02%) when added singly or in combination to structural steels improves the strength and toughness of these steels tremendously.[8,9] Currently attempts are being made abroad to use these microalloyed steels in making machine parts such as crankshafts, connecting rods, connecting rod caps, gears, shafts, cams, etc.[10] But the carburizing behaviours of these microalloyed steels are very little known. The main object of this work is, therefore, also to evaluate the effect of small addition of V, Al and N singly or in combination on the structure and properties of the case and core of the carburized 0.15% carbon steels in addition to the study of the effect of Ni and Cr on the structure and properties of the case and core of the carburized 0.1% C steels.

In Bangladesh most of the spare parts essential for the machineries in various industries are being imported from abroad. Recently a tendency has been observed to produce spares by using locally available material and technology. However, most of such attempts have not turned out to be much fruitful due to the lack of development of the required properties in the materials used for their manufacture. Many spares made of low alloy steels containing Ni, Cr and Mo require

some sort of surface treatment for their proper functioning and longer service life. As has been mentioned earlier that locally available Titas gas is a very successful carburizing medium for surface hardening of low carbon steel parts. Gas carburizing (Titas gas), the most successfull commercial process of surface hardening, can therefore play an important role in this regard.

CHAPTER - 2
CARBURIZING - A REVIEW

2.1 The 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 interior, 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-hyper eutectoid composition and by subsequent heat-treatment to the desired hardness. There are three general methods of carburization, each differing in technique; they 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.

In solid or pack carburization process, parts to be carburized, are charged in a heat-resisting metallic box with carburizing mixture. The carburizing mixture is of the following proportions:[11] Hardwood charcoal - 53 to 55 parts, sized coke - 30 to 32 parts, barium carbonate 10 to 12 parts, calcium carbonate - 3 to 4 parts and sodium carbonate- 2 to 3 parts. The box is then heated to about 900-950^{o}C for a desired length of time and cooled in the furnace. The depth of carburization depends on time, temperature and the type of mixture used. For example, with a typical carburizing mixture of the above proportions, a case depth of 0.04 to 0.05 inch can be obtained at 925^{o}C in an overall carburizing time of nine hours including about five hours heating and four hours holding at the constant temperature.[11] The pack carburi-

zation is economical and advantageous for small machine parts; but it has limitations in handling larger and complicated parts and also has some limitations in heat-treatment. Since some of the chemicals such as barium carbonate, hard coke, sodium carbonate, etc. required for pack carburization 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.

The liquid carburization is used for parts requiring thin carburized cases. The complicated machine parts are also carburized by dipping them in the liquid bath. The carburizing liquid bath is a cyanide solution from where carbon is diffused from the bath to the machine parts. The peculiarity of this process that the temperature of the bath is to be maintained slightly above the Ac_1 line - about temperature 800°C. This process has the disadvantages that the material as well as the fumes coming up from the bath is poisonous.

The practice of carburization by methane as well as cracked gas is the latest development. Natural gas containing methane, ethane, etc. may also be used as a carburizing medium. Gas carburization has many advantages over pack carburizing and is, therefore, extensively applied in plants producing casehardened components in large lots. Carburization by gas will have a higher production capacity because of shorter carburizing time. Uniform case depth can be obtained by the gas compared to that by carburizing mixture. Complicated machine parts can easily be carburized by the gas. It also considerably simplifies subsequent heat-treatment since quenching can be performed directly from the carburizing furnace.

2.2 Theory of Carburization

The carburization of steel may be considered on the basis of two fundamental concepts. The first is the diffusion, influenced by the properties of the steel and concerned with the movement of carbon in the steel itself; the second deals with the source supplying the carbon and with the transfer of carbon to the steel surface. These two general concepts may be described briefly:

(1) Fick's law of diffusion: Fick's first law describes diffusion under equilibrium conditions and is stated mathematically by:

$$J_1 = D_1 \frac{dc_1}{dx} \quad \text{(Ref. 11a)} \qquad .. \qquad (1)$$

where D_1 is the diffusion co-efficient and J_1 is the resulting flux gradient.

Fick's second law expresses the non-equilibrium condition of diffusion where the concentration, at a point, changes with respect to time.

$$\frac{dc}{dt} = \frac{d}{dx}(D_1 \frac{dc}{dx}) \quad \text{(Ref. 11a)} \qquad .. \qquad (2)$$

The value of the diffusion co-efficient is critical in any calculation dealing with carburizing or any other diffusion process. The diffusion co-efficient D is defined by:

$$D = D_o \, e^{-\frac{Q}{RT}} \quad \text{(Ref. 12)} \qquad (3)$$

where D_o is the temperature independent multiplicator in cm^2/sec; Q is the heat of diffusion (activation energy, i.e. the energy required for one atom to jump to a new position in the lattice) in cal/gramme-atom; R is the gas constant (1.98); and T is the absolute temperature

in degrees Kelvin. The greater is the value of Q, the smaller is the value of D at a given temperature and hence the slower is the rate of diffusion.

(2) The equilibrium state for chemical reactions: The equilibrium state for chemical reactions may be represented by numerical constants, K_p, derived from the general expression

$$\text{Log } K_p = \frac{A}{T} - B \qquad \text{(Ref. 13)} \qquad .. \qquad (4)$$

Where K_p is a numerical factor termed the "equilibrium constant", which is derived from the values for the concentrations of the reactants and products of a chemical reaction. The subscript p denotes the dependency of the chemical reaction on pressure. T is the absolute temperature at which the reaction takes place. A and B are constants derived for the particular reaction.

Instead of being developed in a formal way from basic principles the mechanism of carburization may be analyzed from the view point of carbon flow by stating the controlling factors.[13] These factors may be divided for discussion into two distict groups:

(1) Factors that control the flow of carbon in iron and

(2) Factors that influence the transfer of carbon to the iron surface.

The mechanism may be explained as follows:

2.21 Flow of Carbon in Iron

Carburizing is concerned with the solid solution of carbon in austenite. The limits of carbon content of this phase depend on the temperature and the composition of the steel. The solid solution of carbon in gamma iron is an interstitial type of solid solution. As is evident from the iron and iron carbide thermal equilibrium diagram

Fig. 1, at temperature below about 910°C pure iron occurs as a body centered cubic structure. Above 910°C there is a temperature range in which iron has a face centered cubic structure. In face centered cubic lattice, a relatively large unoccupied "void space" exist in the unit cell.[14] Carbon being an extremly small atom can move into this void space to produce a solid solution of iron and carbon. When iron has a body-centered cubic structure at lower temperature, the interstices between the iron atoms become much smaller and consequently the solubility of carbon in body-centered cubic iron is relatively small.

The penetration of carbon into the iron forming steel will depend upon the temperature, the time at temperature, and the carburizing agent. Since the solubility of carbon in steel is greatest above the Ac_3 temperature, carburization takes place most readily above this temperature. Furthermore, the higher the temperature the greater the rate of carbon penetration, since the rate of diffusion is greater. It is customery to select a temperature about 40°C above the Ac_3 point. The curburizing temperature is an important factor and is usually maintained within 900°C to 925°C. For steels with alloy additions a some what higher temperature is required. The depth of carbon penetration depends mostly upon the holding time.

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 core.[13]

The general form of the carbon gradient is influenced only by the range of concentration from surface to the core. For a given

temperature, the rate of diffusion increases with increasing carbon concentration. For common carburising steels and practice, the surface concentration limits are from 1.0 to 1.3%C with core concentrations usually from 0.12 to 0.25%C.[13] For its attainment, the standard carbon gradient obviously requires a constant temperature of operation and a sufficient supply of carbon to the steel surface.

The case depth for a carburized machine parts may be considered to be the extent of carbon concentration which on heat-treatment provides the desired uniform mechanical properties. The case depth for a carburized steel is a function of temperature and time. It has already been mentioned that higher the temperature the greater will be the rate of carbon diffusion and thus deeper will be the case depth. Experiments shows that 40% more[15] will be the rate of diffusion by 40°C rise of temperature, say from 885°C to 925°C.

F.E. Harris[15] has developed a formula for the effect of time and temperature on case depth for normal carburizing:

$$\text{Case depth} = \frac{31.6\sqrt{t}}{10^{(6700/T)}}$$

where case depth is in inches; t is time at temperature, in hours; and T is the absolute temperature, in degrees Rankine.

For a specific carburizing temperature, the relationship becomes simply:

$$\text{Case depth} = K\sqrt{t}$$

$$= 0.025\sqrt{t} \text{ for } 1700^{\circ}F$$

$$= 0.021\sqrt{t} \text{ for } 1650^{\circ}F$$

$$= 0.018\sqrt{t} \text{ for } 1600^{\circ}F$$

When carburizing is purposely controlled to produce surface carbon concentrations somewhat less than saturated austenite, the case depth will be slightly less than the Harris equation shows. The case depth determined by the equation is total case depth, and for case depths in the range from 0.040 to 0.070 inch it will correspond to a point on the carbon gradient where the carbon concentration is about 0.07%C higher than the carbon content of the core.[15]

Recently Merlin E. Williams reported that the results in actual practice and theoretical results are in close agreement and that the metallurgist and heat-treater can apply the theoritical diffusion data to carburizing process.[16]

Diffusion co-efficients vary with the nature of the solute atoms, with the nature of the solid structure, and with changes in temperature. Higher temperatures provide higher diffusion co-efficients because the atoms has higher thermal energies and therefore greater probabilities of being activated over the energy barrier between atoms. Carbon has a higher diffusion co-efficient in iron because the carbon atom is small one. Carbon atoms have higher diffusion co-efficients in bcc iron than in fcc iron because the former has a lower atomic packing factor.[14]

The diffusion proceeds more rapidly along the grain boundaries because this is a zone of crystal imperfections.[14] Other crystal imperfections namely point defects facilitate atomic diffusion more rapidly. Interstitialcies is the most prominent point defect which favours diffusion of carbon in iron and steel.

2.22 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 occuring at this surface. The carburizing agent responsible for the actual transfer of carbon is a carbon compound. The extent to which various carbon compound may supply carbon is related to the conditions that determine the equilibrium with the surface 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 austenitic iron is capable of holding in solid solution an amount of carbon approaching the saturation limit of 1.7 per cent. 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 opinion[17] 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; others feel that the carbon being brought to the surface of the metal as carbon monoxide will, in the presence of austenite, breakdown 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 the gas producer. In other words, the carbon monoxide is the result of the reaction between the carbonaceous compound and the oxygen from the air, the air in this case being the air occulated with the compound particles. The CO thus formed will be dissociated in presence of aus-

tenitic iron to give atomic carbon which will then be absorbed in it according to the following equation:

$$2CO = (C) + CO_2 \quad .. \quad (5)$$

where (C) is the carbon dissolved in austenite at the steel surface. Without a further supply of CO this reaction between CO, CO_2 and austenite would soon reach equilibrium and carburization would stop. Herty[17] states that the $CO:CO_2$ ratio must be above 24 for this reaction (5) to proceed and also that the rate of penetration must be above 0.01 to 0.02 mm per hour.

Since, at carburizing temperature, there is always a large amount of incandescent carbon present, the CO_2 will be constantly reduced, so that there will be a continuous supply of CO according to the reaction:

$$CO_2 + C \rightleftarrows 2CO. \quad .. \quad (6)$$

This cycle continues to repeat itself, and the process of carburizing will continue as long as the proper temperatures are maintained and the $CO:CO_2$ ratio is kept high enough. In this case the carbon will be absorbed, the concentration of carbon will be built up at the surface and will then move inward according to the simple laws of diffusion of heat or dissolved substances, i.e. from region of higher concentration to one of lower concentration as stated earlier.

In gas carburizing, the carbon at the furnace atmosphere is in the form of gaseous hydrocarbon compounds (such as methane, ethane, etc.) or carbon monoxide. The principal 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 reactions[13] for two common carbon compounds, carbon monoxide and methane, may be written:

$$2CO \rightleftharpoons (C) + CO_2 \quad .. \quad (7)$$

$$CH_4 \rightleftharpoons (C) + 2H_2 \quad .. \quad (8)$$

where the symbol (C) represents the carbon dissolved in austenite at the steel surface. Considering the reactions that occur for the same carbon compounds, away from the interface, the reactions for deposition of carbon[13] are expressed:

$$2CO \rightleftharpoons CO_2 + C \text{ (Solid carbon)} \quad .. \quad (9)$$

$$CH_4 \rightleftharpoons 2H_2 + C \text{ (Solid carbon)} \quad .. \quad (10)$$

If water vapour is present in the gas it will act as a decarburizing agent, removing carbon from steel by the reaction[11,13]

$$H_2O\ (g) + Fe_3C(\text{in austenite}) \rightleftharpoons 3Fe(\text{Steel}) + H_2(g) + CO(\text{gas}).$$

In liquid carburization, the carburizing action depends primarily on the sodium cyanide, activated by the presence of alkaline earth salts usually barium (or calcium) chlorides. The salts (chloride) reacts with the cyanide to form a cyanide of alkaline earth metal, which then reacts with iron to form Fe_3C. The basic reactions[18] may be represented by the equation:

$$2NaCN + BaCl_2 \rightleftharpoons 2NaCl + Ba(CN)_2 \text{ (Barium cyanide)}$$

$$Ba(CN)_2 + 3Fe \rightarrow Fe_3C + BaCN_2 \text{ (Barium cyanamide)}$$

The carbon liberated by the shift from barium cyanide to barium cyanamide, is absorbed by the steel and the small amount of nitrogen available is absorbed producing a beneficial effect on the file hardness of the surface.

2.3 Heat Treatment of Carburized Steels

The object of carburization is to develop a hard, wear-resistant surface and a tough core in some moving machine parts. Carburization mainly provides the concentration of carbon on the surface to a case depth of about 1/16 to 1/10 inch. The case of the carburized parts is a eutectoid or hypereutectoid steel and the core is a hypo-eutectoid steel of initial composition containing from 0.1 to 0.2 per cent carbon. The mechanical properties of the case and of the core of the machine part will be obviously different due to differential concentration of carbon and in the un-heat-treated condition the properties may not be sufficient to meet the service requirements. Hence the curburized machine parts are heat-treated to get higher mechanical properties namely hardness at the surface and toughness at the core. Since the carburized machine part gets variable carbon concentration in it, a double heat-treatment is recommended-one for the core which is expected to be tough and the other for the case which is desired to be hard, wear-resistant. Highly satisfactory results are obtained by heating the carburized machine part $100^{o}F$ above the upper critical temperature of the core and then quenched in water. The machine part is again heated to a temperature $60^{o}F$ above its lower critical temperature followed by water or oil quenching and then tempering at 150^{o} to $180^{o}C$.

Double heat-treatment is the best treatment for coarse-grained steel and will give best combination of a hard wear-resistant fine-grained case and a tough and refined core. The chief disadvantages of this heat-treatment are the complexity of the procedure, excessive warping of the work, and the possibility of oxidation and decarburization. For these reasons, single hardening at 820° to 850°C is extensively used at present for inherently fine-grained carbon steels and alloy steels. Such a procedure will refine the grain in both the core and the case and, at the same time warping and decarburization will be considerably reduced.

In gas carburising, quenching direct from the furnace, after pre-cooling to 840-860°C, is frequently practised. The surface layer, after heat-treatment, will have a martensite structure with excess carbides. In cases of quenching direct from the carburising 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 Ac_3, 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.

The normal hardness at the surface of the carburised case 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 carburised 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.

CHAPTER - 3

EXPERIMENTAL SET UP

3.1 "Vapocarb" Electric Furnace

The experiments were stipulated to be carried out in an automatically - controlled electric furnace incorporating on its inside a carburizing chamber of the required size supplied with Titas gas. The details of this experimental set up are shown in Figure 2. It consists mainly of the carburizing chamber, the heating unit and the temperature controlling unit.

3.11 The Carburizing Chamber

The carburizing chamber which is made of heat- resisting steel, is a cylindrical shell, the bottom of which is closed. There are three openings at its bottom, one for the thermocouple, one for the gas inlet and the other for the gas out-let. The carburizing chamber is 17" in height and 11" in diameter. The top of the chamber is covered with a steel lid lined with fire brick. There is an arrangement at the top of the chamber to hang the specimens in it during carburization.

3.12 Heating Unit

The heating element used in this furnace comprised of the usual 80/20 nichrome wire in the form of a coil cast into a alundum shell. The coil shell was about 14" in height and about 12" in diameter and the diameter of the wire was about 3 mm. After two carburizing experiments, the coil burnt out and then another shell as shown in Figure 3 was designed for the heating. This heating unit consisted of a shell and a coil.

3.121 Shell

The shell was made into two parts such as an inner shell to hold the heating coil upon it and an outer shell to contain it.

The inner shell also called the coil shell was made of good quality fire clay with moderately high alumina content and was about 14" in height and about 12" in diameter. Eighteen grooves were made in the shell to fit two windings in parallel. The diameter of each of the grooves was about ½".

The outer shell which was designed to serve just like an outer jacket, was also made of good quality fire clay with moderately high alumina content. These shells were manufactured by the Dhaka Refractories Ltd., Mirpur, Dhaka.

3.122 Coil

Since nichrome wire of the required cross section was not available in the local market at the time of furnace construction, an iron-chromium alloy in the form of wire containing 22% chromium and a small amount of aluminium was used in making the coil. The diameters of the wire and of the coil were about 2 mm and about 12 mm respectively. A mild steel rod of ½" in diameter was fixed in the chuck of a lathe machine. One end of the wire was fixed to the end of the rod nearest to the chuck. The lathe machine was then switched on and the wire was then wound upon the rotating rod and thus a coil was made. During winding up operation, proper tension was maintained upon the wire manually. The lathe machine was then switched off and the rod was then taken out from the coil.

The coil thus prepared was connected with an ammeter in series and electric current was then passed through it from the mains. The length of the coil which recorded about 16 amperes of current was taken

as the required length of the coil. Two such coils of equal length were then connected in parallel upon the coil shell and it was then covered with the outer jacket as shown in Figure 3. The whole unit was then placed in position in the furnace and the coil was thus ready to be connected to the supply source for its preliminary heating operation.

The resistance of an individual coil was measured with a multimeter and was found to be about 15 ohms. The total current passed through the coil assembly was thus found to be about 32 amperes. The furnace was operated at a line voltage of about 240 volts.

The furnace was then ready for experiments and about 8 experiments were carried out successfully. During the 9th experiment the coil burnt out and on dismantling the furnace it was found that the outer shell and the inner shell holding the coil had cracked in many places. It was considered that the shells were not suitable to be repaired and therefore attempts to use them for further experiments were abandoned.

3.2 Design and Construction of a Gas Fired Gas Carburizing Furnace with Automatic Control System

In order to carryout the rest of the experiments a gas-fired gas- carburizing furnace with automatic control was designed and constructed. The furnace was built with fire bricks in a steel drum as shown in Figure 4. The furnace was of circular cross-section having about 5½ inches wall thickness, the external diameter being 23 inches and being 36 inches in height. The side wall was provided with two circular holes at the bottom for the introduction of the burners. A circular platform of about 8½ inches in diameter and 5 inches in height was built at the bottom of the furnace with rammed magnesite powder to support the carburizing chamber to contain the Titas natural gas and the

specimens to be carburized. The roof of the furnace was made of slabs composed of fire bricks chips and fire clay cement.

The furnace was fired with two burners housed in the side walls. A schematic diagram (Figure 4) shows the arrangement of the burners. The flame of the burners was adjusted by trial so that it might not strike back. No air was blown in and it took about 2 hours to get to the required carburizing temperature of 950°C.

The experimental carburizing chamber to contain the specimens was cylindrical steel chamber of about 6" in diameter and 12" in length. The bottom end of the chamber was closed with a steel plate with three ¼" diameter holes near the centre, one for the thermocouple, one for the gas inlet and the other for the gas out let. The top of the chamber was covered with a steel cap. An arrangement for hanging the specimens was provided at the top of the chamber. A few firebricks were placed at the top of the furnace wall and a slab made of fire clay was placed on these fire bricks in such a way that enough opening was maintained to allow the flue gases to escape to the atmosphere. The temperature of the furnace was controlled automatically by controlling the gas flow with the help of a solenoid valve which was actuated by a controlling pyrometer (Figure 5). The pyrometer controller which was connected to the solenoid valve was also connected to the cold junction of the thermocouple. When the required temperature was reached, the solenoid valve stopped the flow of gas into the burner but when the temperature in the gas chamber fall below the required temperature it again resumed the gas flow. In order to avoid the bothering sound caused by the sudden flow of gas during the resumption of combustion, a little flow of gas was continuously maintained during the entire carburizing period by using a bye

pass line with a valve as shown in Figure 4. In this way a very close control of temperature ($\pm$ 2°C) was obtained by controlling the flow of gas into the burner.

To maintain a constant flow of gas into the carburizing chamber a rator meter was connected to the gas inlet of the carburizing chamber. In between rator meter and the carburizing chamber a pressure gauge was also connected which indicated the gas pressure within the carburizing chamber.

CHAPTER-4

EXPERIMENTAL PROCEDURE

4.1 Materials and Specimen Preparation

Four 0.1% carbon steels were used. The compositions are given in Table 1. The steels were made in an air induction furnace. To minimize the formation of Cr_2O_3 and NiO, the steels were first killed with aluminium prior to the addition of nickel and chromium. All melts were teemed at about 1600°C and produced sound ingot. Piped ends of the ingots were discarded. All the ingots were rolled down to a size of about 16 mm in diameter and standard Charpy V-notch specimens were made out of these rolled bars. Steel 1 is the base steel with which the carburizing behaviour of other steels (steels 2-4) containing Ni and Cr alone or in combination was compared.

Five 0.15% carbon steels were also used in this study. The composition of these steels are given in Table 2. These steels were made in an air induction furnace in the Department of Metallurgy, University of Sheffield, England. The melts were also teemed at about 1600°C and produced sound ingots which were then extruded to 16 mm diameter bar. Steel 6 is the base steel with which the carburizing behaviour of other steels (steels 7-10) containing vanadium, aluminium or nitrogen alone or in combination was compared. Standard Charpy V-notch specimens were also made from the extruded bar of these steels.

The 16 mm diameter bars of steels 1 to 4 of Table 1 were machined to 14 mm diameter bars. Specimens of about 20 mm in length were cut from the 14 mm bars and specimens of about 10 mm in length were cut from 10 mm X 10 mm bars of steels 6 to 10. A small hole of about 1 mm diameter was drilled at one end of each specimen. The surfaces of these specimens were finished with 2/0 grade emery polishing paper.

4.2 The Carburizing Operation

The polished specimens of steels 1 to 4 and of steels 6 to 10 were hang by means of nichrome wire into the carburizing chamber in such a way that the tip of the thermocouple and the specimen 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. The power supply was switched on to heat the carburizing chamber and the controller was set for the desired temperature. It took about two hours to reach this temperature.

The gas flow into the carburizing chamber was maintained at a constant rate of 2.2 cm^3/min and a flow rator meter was used for this purpose. A signal flame was maintained at the top of a glass jet to indicate continuous flow of gas in the chamber. As long as a moderate flame is visible at the top of the glass jet, the operator can be 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^{o}C and at a pressure of 14.96 psia. The specimens were carburized for 1 hour, 2 hours, 3 hours and 5 hours at 950^{o}C. At the end of the predetermined time period, the power supply was switched off and the specimens were allowed to cool to room temperature in the carburizing chamber. The gas flow was maintained through the carburizing chamber during the cooling period until the temperature came down to about 500^{o}C. This was done to prevent any probable oxidation due to leackage of air. After cooling, the specimens were taken out of the carburizing chamber.

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

The carburized specimens 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 effective case depth includes the depth which under the microscope shows the zones of hypereutectoid, eutectoid and proeutectoid containing carbon up to 0.6 per cent, since steel specimens when quenched and tempered develop the maximum hardness from a minimum of 0.6 percent carbon content.[19] The effective case depth was then measured linearly from the microstructure of these carburized and furnace-cooled specimens by using a micrometer eye piece fitted to a Shimadzu optical microscope.

4.4 Determination of Austenite Grain Size of the Case of Carburized Specimens

The microstructure of the case of the carburized steels consists of three zones, namely, hypereutectoid, eutectoid and proeutectoid zones. The prior austenite grain size of the hyper-eutectoid zone was revealed by the cementite network and that in the proeutectoid zone was revealed by discontinuous ferrite net. Since the grain size of the eutectoid zone of the carburized steels was very difficult to determine, the grain size of the case was obtained by taking the average grain size of hypereutectoid and proeutectoid zones.

The mean linar intercept method[9] was employed to measure the austenite grain size of the case of these carburized specimens. A circle in the eye piece of a microscope was so placed on the structure that the grain boundaries are intercepting the circumference of the circle on them. The number of interecpts were counted. A total of about 200-300 intercepts were counted for each specimens. The circumference of the circle was determined by measuring its diameters with reference to a stage micrometer at the magnification used. The grain size was then determined by dividing the length of line by the number of intercepts.

4.5 Carburization, Heat-Treatment and Microhardness Measurement

The flat faces of polished two 20 mm long and 14 mm diameter specimens from each of steels 1 to 4 and two 10 mm X 10 mm X 10 mm specimens from each of steels 6 to 10 were made parallel by a surface grinding machine. These specimens were then carburized at 950^{o}C for 1 hour, 3 hours and 5 hours in the same process as described in section 4.2. At the end of a predetermined time period the specimens were cooled to about 860^{o}C in the carburizing chamber. The lid of the chamber was then removed and the specimens were quenched immediately in 10% brine. A little gas flow was maintained during cooling to the hardening temperature to avoid decarburization. The quenched specimens were then tempered quickly at a low temperature of 160^{o}C to transform the hardened martensite of the case into tempered martensite, relieving the residual stresses developed during quenching. The surface hardness of these specimens was then measured by a Rockwell hardness testing machine.

These carburized, hardened and tempered specimens were then sectioned using a silicon carbide disc cutter with copious flow of water. The flat faces were then made parallel by a surface grinding machine.

One of the pieces from each was mounted in bakellite and polished by standard metallographic technique. The specimens were etched in 2% nital and optical microscopy and photomicroscopy were carried out using an optical microscope. Microhardness measurements were then carried out by using 100 gm load on the polished and lightly etched specimens to obtain hardness profile.

4.6 Heat-Treatment and Machinical Testing of Charpy V-notch Specimens

Two to three charpy V-notch specimens from each steels were heated at 950^{o}C for 1 hour in a Blue M furnace and then cooled slowly in the furnace to about 860^{o}C. The specimens were then quenched in 10% brine and tempered at 160^{o}C for ½ hour. The specimens were immersed in chromite brick powder to minimize oxidation during heating for hardening.

The heat-treated Charpy V-notch specimens were then tested by means of Sonntag Universal Impact Testing machine with a range of 0-240 ft. lbs. All the specimens were tested at room temperature. The impact test data thus obtained were used to assess the toughness of the core of the carburized, hardened and tempered steels.

CHAPTER-5

EXPERIMENTAL RESULTS

5.1 Optical Microscopy of Carburized and Furnace Cooled Steels

The microstructures of steels 1 to 4 and steels 6 to 10 carburized at 950°C for 1 hour, 2 hours, 3 hours and 5 hours and cooled slowly in the furnace were investigated by optical microscopy. Optical microscopy revealed variable carbon concentration in the carburized case along its depth, decreasing from the surface toward the core of the all the steel specimens. Three zones were distinguished in the microstructure of the carburized layer (from the surface toward the core): (i) hypereutectoid zone, consisting of pearlite and cementite, forming a network along the former austenite grains; (ii) eutectoid zone, consisting of only lamellar pearlite; and (iii) hypoeutectoid zone, consisting of pearlite and ferrite. The amount of ferrite in the last zone continuously increase toward the core where the original structure of the steel exists. It was also found that ferrite is present as discontinous network around pearlite along the former austenite grains in the hypoeutectoid zone near the eutectoid zone in most of the steels particularly for longer carburizing periods. This zone is commonly known as proeutectoid zone. The optical micrographs of steels 1 to 4 carburized for 1 hour, 3 hours and 5 hours and cooled slowly in the furnace are presented in Figures 6 to 8 and those of steels 6 to 10 are presented in Figures 9 to 11.

The cementite networks surrounding pearlite grains near the surface in the hypereutectoid zone of steels 2 to 4 were found thinner than those of steel 1 and those of steels 7 to 10 were thinner than those of steel 6. The cementite networks of steels 8 are thicker than those of steel 7 and those of steel 9 are thinner than those of steel 7

whereas steel 10 produced the thinnest cementite networks of all. Steel 3 showed thinner cementite network than steel 2 and thicker cementite network than steel 4.

Most of the cementite networks near the surface of steel 6 carburized for 5 hours looks like feathery (Figs. 11a and 17a). Some feathery cementite networks were also found near the surface in the hypereutectoid zone in this steel carburized for 1 hour, 2 hours and 3 hours. A few feathery cementite networks were also present in steels 1 to 3, 7 and 8 carburized for 5 hours.

Many cementite plates in the form of needles were found in some pearlite near the surface in the hypereutectoid zone of steels 1, 6 and 10 carburized for 5 hours. Steels 2 to 4 and 8 carburized for 5 hours also showed a few cementite needles in some pearlite near the surface in the hypereutectoid zone. Some cementite needles were also found in some pearlite near the surface of the hypereutectoid zone of steels 1 and 6 carburized for 1 hour, 2 hours and 3 hours, the amount of the cementite needles was found to increase with the increase of carburizing period. A typical optical micrograph as shown in Figures 11a and 17a illustrates the cementite needles in some pearlite near the surface of hypereutectoid zone of steel 6 carburized for 5 hours.

Heterogeneity in the grain structure was found in the hypereutectoid zone of the case in steels 2, 3 and 10 carburized for defferent time periods ranging from 1 hour to 5 hours, in steel 9 carburized for 3 hours and in steel 4 carburized for 5 hours. The heterogeneity in the structure was severe in steel 10 carburized for 3 and 5 hours while those in steel 9 carburized for 3 hours and steel 4 carburized for 5 hours are less severe.

A visual examination of the microstructures of the core of all the carburized and slowly cooled steels revealed ferrite-pearlite structures as shown in Figures 6 to 11.

5.2 Case depth

The effective case depth of the carburized and furnace cooled steels 1 to 4 and steels 6 to 10 under the present carburizing conditions are shown in Table 3 and 4 respectively. From the data presented in these Tables it is clear that the depth of the carburized layer increases with an increase in carburizing time at the specified temperature. The effective case depths of steels 1 to 4 are plotted in Figure 12 and those of steels 6 to 10 in Figure 13 as a function of carburizing time. The graphical representation of the effective case depth values against the carburizing time for all the steels specimens (Figures 12 and 13) indicates that at the specified carburizing temperature the growth of the carburized layer is parabolic with carburizing time.

A comparison of the values in Table 3 and Figure 12 indicates that under the same carburizing condition the depth of the carburized case in steel 2 with chromium is much higher than that in the plain carbon steel 1 and that in steel 4 with nickel is much lower than steel 1 while steel 3 containing both Ni and Cr produced case depth in between steel 2 and 4. The case depth of steel 3 is slightly lower than that of steel 1.

Table 4 and Figure 13 show that under the identical carburizing condition the microalloyed steels7to 10 produced lower case depth than the plain carbon steel 6 (base steel). Among these four microalloyed steels, steel 8 with vanadium and nitrogen produced the highest case depth and steel 10 with aluminium and nitrogen produced the lowest case depth while steel 9 with vanadium, aluminium and nitrogen produced higher case depth than steel 7 with vanadium and lower case depth than steel 8.

5.3 Prior Austenite Grain Size

The prior austenite grain size of the carburized case is also important in terms of its mechanical properties. The grain size of the case of steels 1 to 4 and steels 6 to 10 carburized for different time periods and slowly cooled in the furnace, was therefore, determined, listed in Tables 5 and 6 and plotted in Figures 14 and 15 respectively as a function of carburizing time.

Tables 5 and 6 and Figures 14 and 15 show that the prior austenite grain size of steels 1 to 4 increases with the increase of carburizing time. It is evident from Table 5 and Figure 14 that the plain carbon steel 1 produced the coarsest grain size while steel 4 produced the finest grain size of all the steels and steel 3 produced finer grain size than steel 2.

Table 6 and Figure 15 show that the prior austenite grain size of steels 6 to 10 initially decreases as the carburizing time increases to 2 hours and then the grain size increases with the increase of carburizing time.

Table 6 and Figure 15 also show that the base steels 6 produced the coarsest grain size and steel 10 produced the finest grain size while steel 8 produced coarser grain size than steel 9 and finer than steel 7.

Typical optical micrographs illustrating the grain size of the case of steels 1 to 4 and 6 to 10 carburized for 5 hours and cooled slowly in the furnace are shown in Figures 16 and 17 respectively.

5.4 Optical Microscopy of Carburized, Hardened and Tempered Steels

The microstructure of the polished and etched carburized, hardened and tempered specimens were examined by optical microscopy and photographs of the structure of each specimen were taken using a Swift Master optical microscope.

5.41 Steels 1 to 4

5.411 Case

Steels 1, 3 and 4 carburized for 1 hour, hardened and tempered showed nearly cent percent tempered martensite in the case whereas steel 2 carburized for 1 hour showed tempered martensite alongwith 5 to 8% retained austenite near the surface in the case. The percentage of the retained austenite decreases towards the centre in the case and at some depth martensite becomes about 100%. The optical micrographs of steel 1 to 4 carburized for 1 hour and 3 hours, hardened and tempered are presented in Figures 18 and 19 respectively.

As the carburizing time increases retained austenite content near the surface in the case increases in all the steels (Figures 18 and 19). About 3 to 5% retained austenite in steel 1, 10 to 12% in steel 2, 5 to 10% in steel 3, and 4 to 6% in steel 4 carburized for 3 hours, hardened and tempered were found. The amounts of retained austenite in steels 1 to 4 carburized for 5 hours, hardened and tempered are observed to be 3 to 5%, 25 to 30%, 10 to 15% and 6 to 10% respectively.

The amount of retained austenite decreases towards the centre and at some depth martensite becomes about cent percent in almost all the steels. Coarse martensite was found in the case near the surface. Martensite becomes finer towards the centre in the case in all the steels (Figures 18 and 19). Martensite needles in steel 2 are of more or less same size as those of steel 1 and those of steel 3 are finer than those of steel 2 while steel 4 produced the finest martensites of all the steels.

A few cementite network was present in some places of steel 2 carburized for 5 hours and of steel 4 carburized for 1 hour, hardened and tempered.

5.412 Core

Low carbon martensite along with some ferrite-pearlite was observed in the core in all the carburized and hardened steels except steel 1 and steel 2. Steel 1 produced ferrite and fine pearlite (sorbite) and steel 2 produced low carbon martensite only in the core. The optical micrographs of steels 1 to 4 carburized for 1 hour, hardened and tempered are shown in Figure 20 .

The percentage of ferrite in steel 1 carburized for 1 hour is about 10%. This amount increase to about 15-20% when carburized for 5 hours. The percentage of ferrite-pearlite in steel 3 carburized for 1 hour is about 8% and this increases to about 10-12% when carburizing time is increased to 5 hours. The percentage of ferrite-pearlite in steel 4 carburized for 1 hour is about 15-20% and this amount increases to about 25% when carburized for 3 hours and decreases to about 10-15% when carburized for 5 hours.

A few discontinious ferrite net was observed in steels 1, 3 and 4 carburized for 1 hour, 3 hours and 5 hours whereas steels 2 and 4 carburized for 3 hours showed a very few ferrite net in the proeutectoid zone.

5.42 Steels 6 to 10

5.421 Case

Steels 6-10 when carburized for 1 hour, 3 hours and 5 hours, hardened and tempered showed retained austenite alongwith martensite in the case near the surface. The amount of retained austenite decreases towards the centre and at some depth mastensite becomes nearly cent percent(about 96-99%) in almost all the steels. The optical micrographs of these steels carburized for 1 hour and 3 hours, hardened and tempered are presented in Figures 21 and 22.

The amounts of retained austenite in steels 6-10 carburized for 1 hour, hardened and tempered are found to be about 10-15%, 5-6%, 30-40%, 50-55%, 25-35% respectively. As the carburizing time increases retained austenite centent increases near the surface in the case in all the steels. The amounts of retained austenite in steels 6-10 carburized for 3 hours, hardened and tempered are found to be about 50-55%, 30-40%, 55-60%, 70-75%, 75-80% respectively. These figures increases slightly when the steels are carburized for 5 hours, hardened and tempered.

The amount of retained austenite decreases towards the centre and at some depth martensite becomes nearly cent percent (about 96-99%) in almost all the steels. Course martensites are found near the surface in the case in all the steels. Martensite becomes finer towards the centre in the case.

Steel 6 showed the coarsest martensite and steel 10 showed the finest martensite of all the steels for all the carburizing periods (Figs. 21 and 22). Steel 8 showed finer martensite than steel 7 and coarser martensite than steel 9 (Fig. 22b-d).

A few cementite network was present in some places near the surface in the case of steels 7 to 10 carburized for 1 hour, 3 hours and 5 hours and of steel 6 carburized for 3 hours and 5 hours, hardened and tempered. A typical example of cementite network in the hardened steels is shown in Figure 23 .

Some polygonal austenite grains were found near the surface in the case in steel 7 carburized for 3 hours and 5 hours and in steels 8-10 carburized for 5 hours, hardened and tempered. These grains are very fine in steel 9 and 10. A typical example of this is shown in Figure 24 .

5.422 Core

Low carbon martensite was found in the core of all the steels. A very few ferrite net was rarely found in addition to low carbon martensite in steel 6 carburized for 3 hours and in steel 10 carburized for 1 hour and 5 hours. The low carbon martensite found in steel 6 was coarser than those in other steels while those of steels 7 and 8 were found to be crystalline i.e. fine and those of steels 9 and 10 were found to be finer than those of steels 7 and 8 for all the carburizing periods. The optical micrographs of the core of steels 6 to 10 carburized for 1 hour, hardened and tempered are presented in Figure 25 illustrating the above features.

5.5 Microhardness and Hardness Profile

When a specimen is carburized, a variation of carbon concentration occurs from the surface to the centre of the specimen. The concentration of carbon is high at the surface and decreases towards the core of the specimen. Due to this variation of carbon concentration a hardness gradient exists in a carburized case in the hardened condition. Depending upon the (i) carburizing temperature, (ii) carburizing time, (iii) pressure in the carburizing atmosphere, (iv) concentration of alloying elements in the metal and (v) the strength of solute-carbon interaction, the hardness profile of a carburized steel may follow two extreme patterns. The profile may be steep with a somewhat sharp line of demarcation between the carburized case and the matrix (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 of steels 1 to 4 at various time at 950^{o}C have been plotted in Figures 26 to 28 . Figures 26 to 28 show that

the hardness profile of steel 1 is somewhat steeper than alloy steels 2 to 4 and it becomes very steeper with increasing carburizing time. The hardness profile of steel 2 is less steeper than that of steel 3 while the hardness profiles of steels 3 and 4 show more or less the same steepness. It can be seen from these figures that change in the slope of the profiles from the carburized layer to the core is very gradual which means that the profiles are shallow. It is observed from above Figures that in general, the case depth increases with the increase of carburizing time and also confirms the case depth values measured from the carburized and slowly cooled steels.

It is also seen from Figures 26 to 28 that with increasing treatment time at the carburizing temperature, the hardness curves have a steeper slope from case to core.

Figures 29 to 31 show the variation in the hardness versus penetration distance profiles with the carburizing time at 950^{o}C for steels 6 to 10 in the hardened condition. These figures also show that the case depths of the steels increase with time at the carburizing temperature and also confirms the case depth values measured from the carburized and slowly cooled steels.

It can also be seen from Figures 29 to 31 that the hardness profiles of steels 6 to 10 are more or less shallow at all the carburizing periods.

The graphical representation of the VHN values against the distance from the edge towards the centre for steels 1 to 4 and 6 to 10 indicates that at the selected carburizing temperature hardness of the case increases with an increase in time.

A comparison of the values in Figures 26 to 28 for steels 1-4 shows that under the same condition of carburizing and hardening the hardness of steel 2 is much greater than that in the base steel 1 and

that in steel 4 is much lower than that in steel 1 while steel 3 produced hardness lower than steel 1 but higher than steel 4.

Figures 29 to 31 and Table 7 shows that under the identical condition of carburizing and hardening the microalloyed steel 8 produced maximum hardness and steel 10 produced minimum hardness. Steel 7 produced lower hardenss than steel 8 and somewhat higher than steel 9. Steel 6 produced lower hardness than steel 9 and higher than steel 10.

It is seen from Figure 26 to 31 that the hardness of the carburized case does not remain constant through out the case. The hardness of the carburized case varies with distance from the surface. The hardness of the case is found somewhat low near the surface and as the distance from the surface towards the centre increases, the hardness of the case increases and then becomes constant over a depth after which it decreases gradually.

The maximum hardness of the carburized case varies with the carburizing time. From the hardness profiles (Figs. 26 to 31 and Tables 7 and 8) it is found that all the steels show the highest maximum hardness at the longest carburizing time of 5 hours.

It is seen from Figures 26 to 28 that the core hardness of steel 2 is maximum and that in steel 1 is minimum while steel 3 shows lower core hardness than steel 2 and higher core hardness than steel 4.

It is evident from Figures 29 to 31 that steel 8 shows the maximum and steel 10 shows minimum core hardness while the core hardness of steel 7 is lower than that of steel 8 and higher than that of steel 9.

5.6 Surface Hardness and Maximum Hardness

The hardness of the surface of the carburized, hardened and tempered steels are measured by a Rockwell hardness testing machine

and presented in Tables 7 and 8 along with the maximum hardness and core hardenss obtained from the microhardenss test and hardenss profiles of Figures 26 to 31.

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 the surface hardenss values obtained by microhardness tester may not be the proper representation of the surface hardness of a carburized case and consequently the hardness of the surface was measured by Rockwell hardness tester. Attempts were made to see whether there is any relationship between the surface hardness and the maximum hardness of a carburized and hardened steel. The surface hardness is found always lower than the maximum hardness. Tables 7 and 8 show that the difference between the maximum hardness and the surface hardness increases with the increase of the carburizing time.

5.7 Impact Test Data

The impact energy absorbed at room temperature for the hardened and tempered steels are presented in Tables 9 and 10. Table 9 shows that the impact energy absorbed in steels 2, 3 and 4 are lower than that in steel 1. Steel 3 shows higher impact energy absorbed than steel 2 and lower than that of steel 4. Table 10 shows that the impact energy absorbed in steel 10 is the highest and that in steel 7 is the lowest of all. The impact energy absorbed of steel 8 is much higher and that of steel 9 a little higher than that of steel 6.

CHAPTER-6
DISCUSSION

The compositions of the steels 6 to 10 (Table 2) indicate that the second-phase particles in steels 7, 8 and 10 are expected to be essentially vanadium carbide, VC, vanadium carbonitride, V(C,N) and aluminium nitride, AlN-respectively, and those in steel 9 to be a mixture of AlN and V(C,N). These were identified and confirmed by transmission electron microscopy.[9] The compositions of the steels 1 to 4 (Table 1) also indicate that the second-phase particles in steels 2 and 3 are Cr_3C, the amount of which is much more in steel 2 than that in steel 3.

6.1 Metallography

Optical microscopy revealed thinner cementite networks surrounding pearlite near the surface in the hypereutectoid zone of steels 2 to 4 than in steel 1. Steel 3 produced thinner cementite networks than steel 2 and thicker cementite networks than steel 4. This 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.

The cementite networks of steels 7 to 10 were found thinner than those of steel 6 (Figures 9 to 11). This indicates that the concentration of carbon in the case of steels 7 to 10 are lower than that in the case of steel 6. Among steels 7,8,9 and 10, steel 8 produced thicker cementite networks than steel 7 while steel 9 produced thinner cementite networks than steel 7 and steel 10 produced the thinnest cementite networks of all these steels. This also indicates that the concentration of carbon in the case of steel 8 is more than that of steel 7 and that of steel 9 is lower than that of steel 7

while the concentration of carbon in the case of steel 10 is the lowest of all these steels.

The presence of a few feathery cementite network (Figure 17 a) near the surface in steels 1 to 3 and 6 to 8 also indicates higher concentration of carbon at their surface. Optical microscopy also revealed some cementite plates in the form of needles in some pearlite grains near the surface in the hypereutectoid zone of steels 1 to 4, 6, 8 and 10 carburized for certain periods of time. These cementite needles are formed due to the supersaturation of austenite with respect to carbon. Austenite which is supersaturated with respect to carbon develops internal stress. This stress might cause the formation of sub-grain boundaries generally in a straight line due to the alignment of dislocations of identical types. This may act as a source for the neucleation of cementite plates. These plates when sectioned for metallographic observations appear as feathers from the cementite network at the main austenite grain boundary towards the interior of the grain. Other vertical plates from the top or bottom of the grains when sectioned horizontally appears as needles within the grains.

Retained austenite as shown in Figures 18 and 19 and 21 and 22 was found in the case of almost all the steels carburized, hardened and tempered. 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 nickel and chromium which depress 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 3 produced a larger amount of retained austenite than steel 4 and a smaller amount than steel 2. Steel 2 contains chromium . Chromium increases

the concentration of carbon at the surface of carburized steels.[6] The higher concentration of carbon in steel 2 due to the presence of Cr depresses the M_f temperature much below that of steel 3 resulting in a greater amount of retained austenite in it.

Steel 4 produces smaller amount of retained austenite than steel 3. This steel contains Ni which reduces the concentration of carbon at the surface[6] and hence can not depress the M_f temperature as drastically as does Cr. Steel 3 contains both Ni and Cr. Its retained austenite content is expected to be in between steel 2 and 4. This has in fact been found to be the case.

The retained austenite contents of all these steels increase with the increase of carburizing time (Figures 18 and 19). 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.

Among steels 6 to 10, steels 8,9 and 10 produced larger amounts of retained austenite than steel 6 for all the carburizing periods (Figures 21 and 22). Steel 7 produced a somewhat lower amount of retained austenite than steel 6. This is due to the lower concentration of carbon caused by the formation of VC in steel 7. This indicates that vanadium is not effective in the formation of retained austenite in the hardened carburized case of the steels.

Steel 8 produced higher percentage of retained austenite in the carburized case than steel 7 at all the carburizing periods. Steel 8 is basically steel 7 with nitrogen added to it. The higher percentage of retained austenite in steel 8 may be due to the presence of nitrogen which is thought to increase the carbon concentration in its carburized case.

Steel 9 also produced much higher percentage of retained austenite than steel 8 for all the carburizing periods. Steel 9 is also basically the same as steel 8 with aluminium added to it. The much higher percentage of retained austenite in steel 9 is clearly due to the presence of Al which is more active than V in depressing the martensite transformation temperatures.

Steel 10 is basically steel 6 with Al and N added to it. This steel showed much higher percentages of retained austenite than steel 6 and also than steel 7 for all the carburizing periods. This indicates clearly that Al is most effective in the formation of retained austenite in the carburized case of low carbon steels.

These steels also showed increased amounts of retained austenite in their carburized cases with the increase of carburizing time (Figures 21 and 22). This is also due to the increasing carbon concentration in their carburized cases with increasing time.

It has also been found that the amount of retained austenite also decreased towards the centre and at some depth martensite became about cent percent in almost all the steels (Figures 21 and 22). The concentration of carbon is the greatest at the surface. This carbon diffuses towards the centre during carburizing periods. The concentration of carbon decreases towards the centre. As the concentration of carbon in case decreases from surface towards the centre, the retained austenite content decreases.

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

produced the coarse martensite near the surface and finer austenite grains produced finer martensite towards the centre in the case.

The martensites of steel 6 were found to be the coarsest and those of steel 10 were the finest of all the steels at all the carburizing penods (Figures 21 and 22). The coarsest martensites in steel 6 are due to its coarsest austenite grains and the finest martensites in steel 10 are due to the finest austenite grains present in it (Figure 17 and Table 6).

Steel 8 showed finer martensite than steel 7 and coarser martensite than steel 9. The differences in size of martensite are clearly due to the observed differences in the austenite grain sizes of these steels (Figure 17 and Table 6).

The differences in the size of martensite in steels 1 to 4 are also found fairly in agreement with the observed differences in their austenite grain sizes (Figures 16, 18 and 19 and Table 5).

The presence of a few cementite network (Figure 23) along with high carbon tempered martensite in the case of most of the carburized and hardened steels is not unexpected in view of the quenching temperature (860^{o}C) used.

The presence of polygonal austensite grains as shown in Figure 24 near the surface in the hardened case of steels 7 to 10 for some carburizing periods is also expected in view of the higher concentration of carbon at their surfaces.

Steel 1 contained about 10% ferrite alongwith fine pearlite (sorbite) (Figure 20a) in the core when carburized for 1 hour. Steel 1 is a plain carbon steel containing 0.1%C. It does not contain any alloying element. Because of the very low carbon content the transformation temperature rises resulting softer transformation products i.e. the critical cooling rate was faster than the actual cooling

rate in quenching and hence softer transformation products such as ferrite and fine pearlite (sorbite) were obtained.

Steel 2 did not show any ferrite-pearlite. It produced low carbon martensite (Figure 20 b). This steel contains Cr which retarded the transformation and thus moved the transformation curve to the right at or near the nose region resulting low carbon martensite.

The presence of ferrite in steels 3 and 4 along with low carbon martensite also indicates that the critical cooling rate in each of these steels was faster than the actual cooling rate.

6.2 Case Depth

6.21 Steels 1 to 4

The effective case depth of steels 1 to 4 is plotted in Figure 12 as a function of carburizing time and presented in Table 3. Steel 1 is the base steel with which the case depth of the other steels with Ni and Cr alone or in combination are compared. It is evident from Figure 12 that steel 2 with Cr produced much higher case depth than the plain carbon steel 1 and steel 4 with Ni content produced much lower case depth while steel 3 containing both Ni and Cr produced case depth in between them. This indicates clearly that Cr increases the thickness of the carburized layer and Ni reduces it. This is due to the fact that Cr reduces the diffusion co-efficient D_γ of carbon in austenite, because it raises the required activation energy Q[6] but since it increases the carbon concentration at the surface it increases the thickness of the carburized layer to some extent. Nickel on the contrary increases D_γ (by reducing the activation energy of diffusion) but reduces the carbon concentration, at the surface[6] and consequently reduces the layer thickness. Steel 3 contains both Ni and Cr. So the case depth of this steel is expected to be in between

that of steel 2 and 4 i.e. higher than that of steel 4 and lower than that of steel 2 and this has in fact been observed to be the case.

It is also evident from Figure 12 that as the time at the 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 penetrate into a greater depth and hence higher is the case depth.

6.22 Steels 6 to 10

Steel 6 is merely a plain carbon steel. This steel is used as a base to compare the carburizing behaviour of the other steels (steels 7 to 10) with small additions of V, Al and N singly or in combination. Figure 13 shows that the microalloyed steels 7 to 10 produced lower case depth than the plain carbon steel 6. This indicates that microalloying elements particularly vanadium with or without N and aluminium with N reduce the case depth.

The highest case depth in steel 6 is due to the much higher concentration of carbon at its surface which is clearly indicated by the presence of a thicker and feathery cementite network in its carburized case.

The formation of vanadium carbide reduces the solubility of carbon in austenite[20] and hence decreases the carbon concentration at the surface of the steel specimen. The much lower case depth in steel 7 with VC than the base steel 6 is clearly due to lower concentration of carbon at its surface.

Steel 8 is basically steel 7 with nitrogen added to it. This steel produces higher case depth than steel 7. The higher case depth of steel 8 is also due to the higher concentration of carbon at its

surface which is indicated from the presence of some thicker and feathery cementite network in this steel. The higher concentration of carbon causing greater case depth in steel 8 may be due to the presence of nitrogen which is thought to increase the solubility of carbon in austenite and thus favours the diffusion of carbon in austenite. The presence of nitrogen as V(C,N) is also thought to increase the diffusion coefficient by decreasing the activation energy.

Steel 10 with Al and N produced the lowest case depth. This shows that Al and N as AlN reduces the case depth drastically. The presence of the thinnest cementite network in the case clearly reveals that the concentration of carbon at the surface of this steel is very low. The much lower case depth of this steel is clearly due to the lower surface carbon concentration which is thought to be caused by the presence of Al and N as AlN.

Steel 9 which is basically steel 8 with Al added to it shows case depth lower than steel 8 and higher than steel 7. The lower case depth of steel 9 than steel 8 is due to the presence of Al which reduces the surface carbon concentration.

6.3 Prior Austenite Grain Size

6.31 Steels 1 to 4

Steel 1 is the base steel with which the austenite grain size of the other steels (steels 1 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 14 with the increase of time at the carburizing temperature.

It is also evident from Figure 14 that steel 2 produces much finer austenite grain size than steel 1 (Figure 16). Steel 2 contains chromium which combines with carbon forming chromium carbide, Cr_3C. These carbide particles pin the prior austenite grain boundaries and inhibit grain growth and keep the grain size small. Steel 4 produces the finest grain size of all the steels. This steel contains nickel. Nickel does not combine with carbon. It remains in solution in austenite. The greater effect of nickel in refining austenite grain size of the case of carburized steel than chromium is not clear.

Steel 3 contains both nickel and chromium. Its grain refining effect is expected to be greater than that of steel 2 and lower than that of steel 4 and this has infact found to be the case in the experimental work (Figure 16).

6.32 Steels 6 to 10

Figure 15 shows that the prior austenite grain size of steels 6 to 10 initially decreases as the carburizing time increases to 2 hours and then the grain size increases with the increase of carburizing time.

The base steel 6 being merely a plain carbon steel does not contain any second phase particle to inhibit grain growth. From Figure 15 it is evident that the grain size of steel 6 increases rapidly with increasing treatment time (after 2 hours) at the carburizing temperature of 950^{o}C. This is due to the free migration of grain boundaries (to attain the equilibrium grain size) when the carburizing time is increased. With the presence of second phase particles, as in other steels (steels 7 to 10), they pin the austenite grain boundaries and inhibit the grain growth causing the steels to retain a fine grain

size which is found to increase slightly during carburization with successive longer holding time (after 2 hours).

Among the precipitates VC, V(C,N) and AlN, it is accepted that the order of increasing solution temperature is VC, V(C,N) and AlN. M.A. Bepari[9,21] reported that the temperatures for the precipitates in steels 7,8,9 and 10 to dissolve completely are 1015, 1120, 1260 and 1230°C respectively. Therefore it is expected that the grain growth restriction in steel 8 will persist to a higher temperature than in steel 7 and that in steel 10 to the highest temperature. However, at the lower temperature of 950°C where the precipitates in the three steels were still effective as grain growth inhibitors, steel 8 produced finer grain size than steel 7 and coarser grain size than steel 10 suggesting that V(C,N) is much more effective than VC and AlN is the most effective in controlling the grain growth of austenite.

Steel 9 is basically steel 8 with aluminium added to it. Its grain coarsening chracteristics is expected to be in between those of steel 8 and steel 10 and this has in fact been observed to be the case.

Gladman[22] showed that fine particles are more effective than coarse particles in restricting grain growth. It has been reported[9] that the coarsening rate of V(C,N) in the presence of aluminium is considerably lower than that of VC or of V(C,N) in the absence of aluminium. The greater effect of V(C,N) in grain growth inhibition is thought to be due to the fact that its precipitates are present in a finer dispersion than those of VC. The greater effect of AlN in inhibiting grain growth is also thought to be due to their less coalescence.

The larger austenite grain size in the case of the steels carburized for 1 hour than for 2 hours may be due to the stored energy caused by previous operation (extrusion) near the surface. This stored energy which favoured the austenite grain growth was later eliminated

with increasing time from 1 hour to 2 hours at the carburizing temperature.

6.4 Hardness and Hardness Profile

6.41 General

The microhardness of the case of all the steels carburized for 1 hour, 3 hours and 5 hours, hardened and tempered, increases upto certain depth and then remains constant over a certain depth and then decreases gradually towards the core as shown in Figures 26 to 28 and 29 to 31 . Optical microscopy revealed that the steels in the hardened and tempered condition contains high carbon martensite in the case along with retained austenite, the percentage of which varied with steel composition and the carburizing time. It was also revealed that the percentage of retained austenite in the case near the surface decreases as the distance from the surface increases and at some depth martensite becomes about cent percent. Tables 7 and 8 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. As the retained austenite content decreases the hardness of the case increases with increasing distance from the surface. Because of the presence of nearly cent percent high carbon martensite the hardness becomes maximum at some depth beyond which the hardness decreases gradually towards the core. This decrease in hardness is due to the decreasing carbon content in martensite which is formed from austenite of lower carbon.

Finally the hardness reaches to a low value at the core. This lower hardness is due to lower carbon content of austenite which was subsequently transformed to a mixture of low carbon martensite, fine pearlite (sorbite) and free ferrite.

6.42 Steels 1 to 4

It is evident from Figures 26 to 28 that the hardness curves of steels 1 to 4 have a steeper slope from case to the core when the carburizing time increases to 5 hours. This is because of the increase in the concentration gradient with increasing carburizing time. The higher steepness of the hardness profile at the interface between the carburized case and the core in plain carbon steel 1 than steels 2 to 4 (Figs. 26 to 28) is due to the higher concentration gradient.

From Figures 26 to 28 it is seen that steel 2 produced higher hardness and steels 3 and 4 produced lower hardness in the case than the base steel 1 for all the carburizing periods while steel 3 produced higher hardness than steel 4.

Steel 2 contains Cr_3C precipitates. The higher hardness of the case of this steel than the base steel 1 is clearly due to the presence of these precipitates. These precipitates impede the movement of dislocations and hence produces hardnening effect in the steel.

Steel 4 although contains Ni does not have any second phase particles because Ni is not a carbide forming element. The lower hardness of the case of this steel than the base steel 1 may be due to the fine martensite needles (platelets) (Figs. 18 d and 19d) formed from fine prior austenite grains (Fig. 16 d) caused by the presence of Ni in solution in austenite.

Steel 3 is basically identical with steel 4 with Cr added to it. The higher hardness of the case of this steel than steel 4 is certainly due to the presence of Cr_3C in it. Since the amount of Cr and hence Cr_3C in this steel is much lower than that of steel 2, the hardness of steel 3 was, therefore, not increased to that extent as in steel 2.

Figures 26 to 28 and Table 7 show that the hardness of the core of steels 1 to 4 carburized for 1, 3 and 5 hours are much lower than their case hardness. This is due to the low carbon martensite in the core. The presence of some ferrite-pearlite also contributes to the lowering of hardness along with low carbon martensite in the core of steels 1, 3 and 4.

In the core, steels 2,3, and 4 showed higher hardness than the base steel 1 for all the carburizing periods (Figures 26 to 28). The lower hardness of the core of steel 1 than steels 2 to 4 is due to the presence of ferrite and fine pearlite (sorbite) in the core of this steel. Among steels 2, 3 and 4, steel 2 produced much higher and steel 4 produced lower hardness than steel 3 at all the carburizing periods. The much higher hardness in the core of steel 2 is due to the presence of larger amount of Cr_3C in it. The much lower hardness of steel 4 than steel 2 is due to the presence of ferrite along with fine low carbon martensite formed from fine austenite grains in it. The composition of the steels (Table 1) indicates that the amount of Cr_3C in steel 3 is much lower than that in steel 2. The much lower hardness of this steel than that of steel 2 is therefore due to the smaller amount of Cr_3C along with some amount of ferrite present in it.

It is evident from Figures 26 to 28 that the hardness of the case of carburized, hardened and tempered steels and also the maximum hardness increase with an increase in carburizing time. This is because of the increasing carbon content of martensite.

It is found that the surface hardness of all the carburized, hardened and tempered steels is lower than the maximum hardness obtained in the carburized case (Table 7). The explanation of this difference in hardness lies in the fact that at the surface the steels

contain some amount of retained austenite along with higher carbon martensite. So when the surface hardness is measured, by Rockwell hardness testing machine, the indentor sinks into the region containing both retained austenite and martensite and indicates the hardness contributed by both of them. The difference between the maximum hardness and the surface hardness increases with the increase of carburizing time.

In steels 1 and 4 surface hardness and maximum hardness both increase because of increasing carbon content of martensite with the increase of carburizing time. But the maximum hardness increases more rapidly than surface hardness with increase in time. Hence the difference between them increases with increasing time.

In steels 2 and 3, the surface hardness decreases due to the presence of increased amount of retained austenite with the increase in carburizing time, but the maximum hardness increases due to increasing carbon content of martensite with the increase in carburizing time. Hence the difference between surface hardness and maximum hardness increases with the increase in carburizing time.

It is also found from Table 7 that the difference in the surface hardness and maximum hardness is greater in the alloy steels (steels 2 to 4) than in the base steel 1.

6.43 Steels 6 to 10

Figures 29 to 31 show that the hardness profile of steel 6 to 10 are more or less shallow for all the carburizing periods. This is due to the lower concentration gradient. It is also evident from these Figures that steel 6 shows a somewhat steeper hardness profile than steels 7 to 10 for all the carburizing periods. The greater steepness of the hardness profile at the interface between the carbur-

ized case and the core in the plain carbon steel 6 than steels 7 to 10 is clearly due to the higher concentration gradient at the interface. The presence of alloying elements such as V and Al in the other steels decreases the concentration gradient causing a gradual change in the slope of the hardness profile from carburized case to the core and hence the hardness profile at the interface is shallow.

It is evident from Figures 29 to 31 that steels 7,8 and 9 produced higher and steel 10 produced lower hardness than the base steel 6 at all the carburizing periods. Among steels 7,8, and 9, steel 7 produced higher hardness than steel 9 and lower than steel 8.

The hardness of hardened steels is almost independent of the size of martensite needles[23] and hence independent of the size of the prior austenite grains which control the size of martensite needles (platelets). The observed difference in hardness of the carburized case is therefore not related to the difference in austenite grain size i.e. difference in martensitic platelets.

It has been mentioned earlier that the precipitates in steels 7, 8 and 10 are respectively VC, V(C,N) and AlN and that in steel 9 is a mixture of V(C,N) and AlN.

The higher hardness in the case of steels 7,8, and 9 than plain carbon steel 6 is clearly due to the presence of the precipitates in their cases. These precipitates, as mentioned earlier, impede the movement of dislocations and hence produces hardening effect in the steels.

The higher hardness of the case of steel 8 than steel 7 indicates that V(C,N) is much more effective than VC in increasing the hardness. Bepari[9] reported that precipitating V(C,N) presents a finer dispersion of particles than VC. The greater effect of V(C,N) in hardness increment is clearly due to its finer dispersion than VC.

Bepari[9] also reported that the precipitates size and amount in steel 9 are respectively somewhat coarser and smaller than those in steel 7. The lower hardness of steel 9 is due to the smaller volume fraction of the coarser particles.

Steel 10 with AlN produces lower hardness than steel 6. This indicates that AlN particles does not produce any hardening. It has been reported[8,9] that nitrogen in solution is a good strengthener in steel. The lower hardness of steel 10 than steel 6 is due to the presence of Al which removes nitrogen in solution as AlN. The volume fraction of the AlN particles in steel 10 is much smaller than those of VC and V(C,N) in steel 7 and 8 respectively.[9] It is thought that the volume fraction of AlN particles in steel 10 is so small that it does not have any effect in hardness increment.

Similar explanation is applicable to the observed difference in hardness of the core of steels 6 to 10.

It is evident from Table 8 that the surface hardness of the carburized, hardened and tempered steels 6 to 10 is also lower than the maximum hardness attained in the case. This is because the steels contain a considerable amount of retained austenite at the surface along with high carbon martensite.

It is also observed that the difference between the maximum hardness and surface hardness increases with the increase of carburizing time (Table 8). The surface hardness of all the steels is found to decrease due to the presence of increased amount of retained austenite at the surface with the increase of carburizing time while the maximum hardness increases due to increasing carbon content of martensite with the increase in carburizing time. Thus the difference between surface hardness and maximum hardness increases with the increase in carburizing time.

From the above discussion on the surface versus maximum hardness, it may be concluded that the surface hardness of carburized, hardened and tempered steel may not be the maximum value that develops through carburizing. In order to get the maximum hardness of a carburized specimen one should measure the hardness along the cross section. It should be noted that retained austenite in the surface is very soft and it will decay out in time in service leaving a harder surface beneath it.

It has been reported[6] that the desired hardness of the surface layer is from 60-64 Rc for carbon steel, and from 58 to 61 for alloy steel. The lowerhardness in the second case is due to the formation of a larger amount of retained austenite. After a single hardening operation with high alloy steels, a large amount (upto 50 to 60 per cent or even more) of retained austenite can be found in the structure of the carburized case. This sharply reduces the hardness. A sub-zero treatment is applied to such steels following hardening. This converts a large part of the retained austenite into martensite thereby substantially increasing the hardness. For example, the hardness of chromium-nickel steel after case hardening is 52 Rc. After sub-zero treatment, the hardness is increased to 60 to 62 Rc.

Because of the non availability of the facilities in the Department, the sub-zero treatment of carburized, hardened and tempered steels has not been possible. It is fairly certain that the desired hardness could be achieved if these steels (steels 1 to 4 and 6 to 10) are subjected to a sub-zero treatment.

6.5 Hardenability

An indication of hardenability of steels 1 to 4 and 6 to 10 can be obtained from the microhardness profiles of Figures 26 to 31 .

It is evident from these figures that all the steels show higher hardenability when they are carburized for longer time, hardened and tempered. It was stated earlier as the carburizing time increases, the austenite grain size increases. The coarse austenite grain size along with alloy addition increases the case hardenability.[24] The higher hardenability of each of these steels carburized for longer time is clearly due to the combined effect of coarser austenite grain size and increasing carbon content of increasing case depth with increasing time.

6.6 Toughness

6.61 Steels 1 to 4

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 Ni and Cr alone or in combination decreases the toughness of low carbon steels. Optical microscopy revealed fine pearlite (sorbite) and about 10% polygonal ferrite in the core of this carburized and hardened steel (Figure 20a). The high toughness of steel 1 is due to the presence of ferrite and sorbite in the microstructure of this steel. At the carburizing temperature there exists some undissolved chromium carbide particles in steels 2 and 3. The presence of these chromium carbide particles along with low carbon martensite is responsible for the lower toughness of these steels.

It is also evident from Table 9 that steel 3 with Ni and Cr show higher impact energy absorbed than steel 2 with Cr and lower impact energy absorbed than steel 4 with Ni. This indicates that Cr is more detrimental to toughness than Ni.

Optical microscopy also revealed much more polygonal ferrite along with low carbon martensite in steel 4 than steel 3 (Figure 20c and d). The better toughness in steel 4 than steel 3 is due to the presence of increased amount of polygonal ferrite together with fine low low carbon martensite in it.

6.62 Steels 6 to 10

It can be seen from Table 10 that steels 8 to 10 show higher impact energy absorbed whereas steel 7 shows lower impact energy absorbed than the base steel 6. This indicates that VC particles are detrimental to toughness and V(C,N) and AlN particles are benificial to toughness.

It has been reported[8,9] that nitrogen in solution impares toughness of steels. The size of martensite platelets also control the toughness of hardened steels. Toughness reduces with the coarsening of martensite.[23] Steel 6 does not contain any alloying element to combine with nitrogen already present in it. This along with the coarse low carbon martensite (Figure 25a) formed from the coarse austenite grain present in it are responsible for low impact energy absorbed. The nitrogen content in steels 7 to 10 are combined either as V(C,N) or AlN.

Steel 7 produces finer low carbon martensite than steel 6. But it contains VC precipitates. VC precipitates impare the toughness of annealed and normalized low carbon steels.[9] The much lower impact energy absorbed of steel 7 than steel 6 is clearly due to the presence of VC precipitates.

The impact energy absorbed of steel 8 is much higher than that of steel 7 and fairly higher than that of steel 6. The nitrogen

content of this steel is tied up as V(C,N) precipitates. Moreover, this steel shows finer martensites formed from the finer austenite grains produced by V(C,N) precipitates. The better toughness of this steel than that of steels 6 and 7 are clearly due to the V(C,N) particles present in it.

The impact energy absorbed of steel 10 with Al and N is the highest of all the steels (Table 10). Optical microscopy revealed very fine low carbon martensite in the core of this steel. Al refines the austenite grain size and removes the soluble nitrogen as AlN. The much higher toughness of this steel is obviously due to the fine low carbon martensite formed from very fine austenite grains produced by AlN particles.

Steel 9 shows higher impact energy absorbed than steel 6 and lower than steels 8 and 10. This steel contains V, Al and N. It is thought that in presence of Al, V(C,N) formed will be low in nitrogen i.e. nearly to be VC which reduces the impact energy absorbed. The detrimental effect of VC on impact energy absorbed appears to be greater than the benificial effect of AlN.

From the above discussion it may be concluded that VC precipitates are detrimental to toughness but V(C,N) and AlN precipitates are benificial to toughness of the core of carburized hardened and tempered steels. AlN particles are more effective than V(C,N) particles in improving the toughness of the core of carburized steels in the hardened condition.

CHAPTER-7

CONCLUSIONS

7.1 Conclusions Drawn from the Present Work

1. A very close control of temperature ($\pm 2^{o}C$) of a gas fired gas carburizing furnace can be obtained by controlling the flow of gas (Titas gas) into the burner with the help of a solenoid valve which is actuated by a controlling pyrometer.

2. Chromium increases the case depth of the carburized steels while Nickel decreases it.

3. Microalloying elements such as vanadium with or without nitrogen and aluminium with nitrogen reduce the case depth of the carburized steels. Vanadium without nitrogen is more effective than vanadium with nitrogen in reducing the case depth while aluminium with nitrogen is the most effective of all in reducing the case depth.

4. Chromium as Cr_3C and nickel refine the austenite grain size of the carburized case but nickel is more effective than chromium in inhibiting grain growth of austenite.

5. The undissolved particles of vanadium carbide, VC, vanadium carbonitride, V(C,N) and aluminium nitride, AlN all refine the austenite grain size of the carburized case, but V(C,N) is much more effective than VC and AlN is the most effective in inhibiting the grain growth of austenite in the case of carburized steels.

6. Chromium and nickel both promote the formation of retained austenite but chromium is more effective than nickel in promoting the formation of retained austenite in the case of the carburized steels.

7. Vanadium without nitrogen does not have any effect in the formation of retained austenite while vanadium with nitrogen promotes the formation of retained austenite in the case of carburized steels.

8. Aluminium is the most effective in promoting the formation of retained austenite in the case of carburized steels.

9. Chromium in solution and as Cr_3C increases and nickel in solution decreases the hardness of the case while both of them increases the core hardness although chromium is more effective than nickel as far as core hardness is concerned.

10. The undissolved particles of VC and V(C,N) increase the case and core hardness while AlN reduces the case and core hardness. V(C,N) is more effective than VC in raising the hardness of the case and core.

11. The hardenability is increased with the increase of austenite grain size and extent of carbon penetration of the case of carburized steels.

12. Nickel and chromium both reduce the toughness of the core of low carbon steels in the carburized and hardened condition. Chromium is more detrimental to toughness than nickel.

13. VC reduces the toughness markedly while V(C,N) and AlN improves the toughness. AlN is much more beneficial to toughness than V(C,N).

7.2 Suggestions for Further Work

1. In order to obtain the optimum hardness in the case, a large part of the retained austenite must be converted into martensite. A sub-zero treatment should therefore be applied to the steels following hardening.

2. A study on the wear, tensile and fattigue properties of the steels should be carried out systematically in order to have a complete understanding of the behaviour of the carburized low alloy steels.

3. A further investigation is required to have more data in order to explain the variation of the hardenability of the steels.

4. A high temperature tempering of the steels may be carried out to show the variation of hardness and the results thus obtained may be compared with those of low temperature tempering with shorter duration.

5. Service behaviour of the carburized low alloy steels should be investigated.

6. Transmission electron microscopic study should be carried out on these steels to obtain informations on precipitate morphology.

7. Effect of molybdenum on the structure and properties of the case and core of carburized low carbon steels should be systematically studied.

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TABLE 1

Composition of steels 1 to 4 in wt%

Steel No.	C	Si	Mn	Ni	Cr	S	P
1	0.10	0.18	0.37	-	-	0.028	0.025
2	0.10	0.19	0.39	-	0.85	0.030	0.028
3	0.10	0.20	0.37	1.2	0.35	0.030	0.022
4	0.10	0.20	0.39	1.19	-	0.033	0.025

TABLE 2

Composition of steels 6 to 10 in wt.%

Steel No.	C	Mn	V	N	Al	Si	S	P
6	0.15	1.42	-	0.005	-	0.28	< 0.02	< 0.02
7	0.15	1.42	0.22	0.005	-	0.26	< 0.02	< 0.02
8	0.15	1.52	0.20	0.020	-	0.30	< 0.02	< 0.02
9	0.15	1.57	0.20	0.024	0.056	0.30	< 0.02	< 0.02
10	0.15	1.42	-	0.020	0.053	0.19	< 0.02	< 0.02

TABLE 3

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

Steel No.	Case depth mm			
	1 hour	2 hours	3 hours	5 hours
1	1.13	1.56	1.95	2.84
2	1.20	1.75	2.15	2.50
3	1.06	1.50	1.83	2.43
4	0.95	1.32	1.65	2.16

TABLE 4

The effective case depth of steels 6 to 10 carburized for different periods of time and cooled slowly in the furnace.

Steel No.	Case depth, mm.			
	1 hour	2 hours	3 hours	5 hours
6	1.54	2.01	2.45	3.46
7	1.36	1.73	2.19	3.18
8	1.46	1.90	2.35	3.31
9	1.41	1.78	2.27	3.21
10	1.15	1.64	1.84	2.66

TABLE 5

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.	Austenite grain size, μm			
	1 hour	2 hours	3 hours	5 hours
1	-	236	259	349
2	109	114	120	143
3	83	90	91	99
4	76	78	87	95

TABLE 6

The prior austenite grain size of the case of steels 6 to 10 carburized for different periods of time and cooled slowly in the furnace

Steel No.	Austenite grain size, μm			
	1 hour	2 hours	3 hours	5 hours
6	193	136	178	259
7	65	53	64	73
8	58	38	43	62
9	37	27	30	29
10	33	23	21	28

TABLE 7

Relationship between surface hardness and maximum hardness of steels 1 to 4.

Steel No.	Carburizing time hour	Surface Hardness		Maximum Hardness		Core Hardness	
		Rc	VHN	VHN	Rc	VHN	Rc
1	1	56	640	661	57	166	-
	3	60	738	748	61	186	-
	5	61	750	946	66	160	-
2	1	56	640	694	58	428	43
	3	54	600	793	62	397	40
	5	47	480	989	68	381	39
3	1	53	590	642	56	211	-
	3	50	540	747	61	237	-
	5	49	520	913	65	248	22
4	1	50	540	622	55	187	-
	3	53	590	724	59	203	-
	5	54	600	894	65	173	-

TABLE 8

Relationship between surface hardness and maximum hardness of steels 6 to 10.

Steel No.	Carburizing time hour	Surface Hardness		Maximum Hardness		Core Mardness	
		Rc	VHN	VHN	Rc	VHN	Rc
6	1	57	660	748	61	392	39
	3	46	470	808	62	472	46
	5	39	380	852	63	489	47
7	1	59	725	762	61	530	49
	3	49	520	843	63	532	50
	5	46	470	882	64	542	50
8	1	54	600	797	62	570	52
	3	47	480	852	63	605	54
	5	45	460	888	65	583	52
9	1	51	560	742	60	495	47
	3	45	460	830	63	513	49
	5	42	420	870	64	510	49
10	1	54	600	724	59	373	38
	3	45	460	772	61	411	41
	5	38	373	824	62	425	43

TABLE 9

Impact energy absorbed of steels 1 to 4

Steel No.	Impact Energy Absorbed Ft-lbs
1	147
2	52
3	69
4	98

TABLE 10

Impact energy absorbed of steels 6 to 10.

Steel No.	Impact Energy Absorbed Ft-lbs.
6	48
7	28
8	64
9	50
10	107

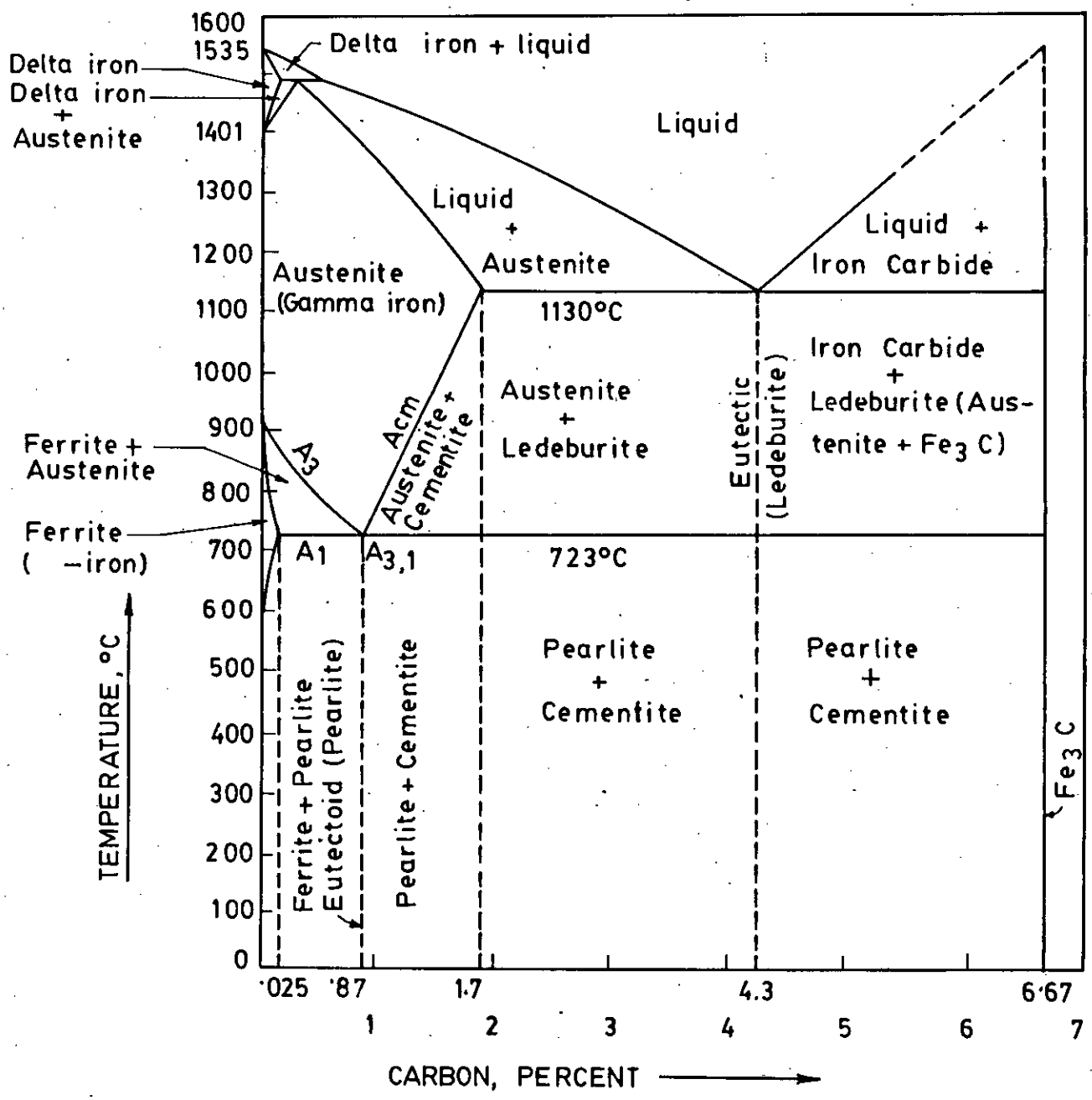


Figure 1: Iron and iron-carbide equilibrium diagram.

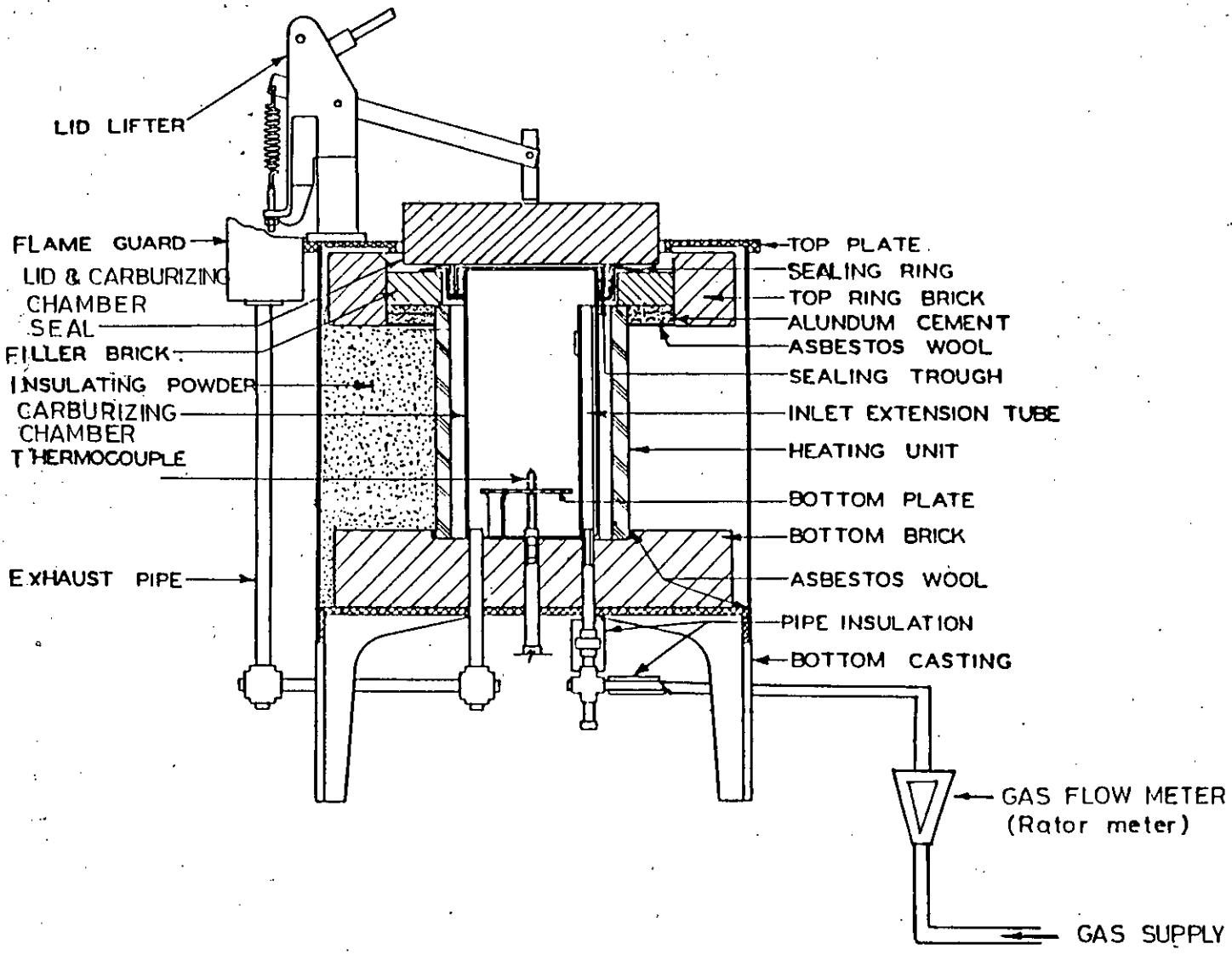


Figure 2: Schematic diagram of Vapocarb electrically heated gas carburizing furnace.

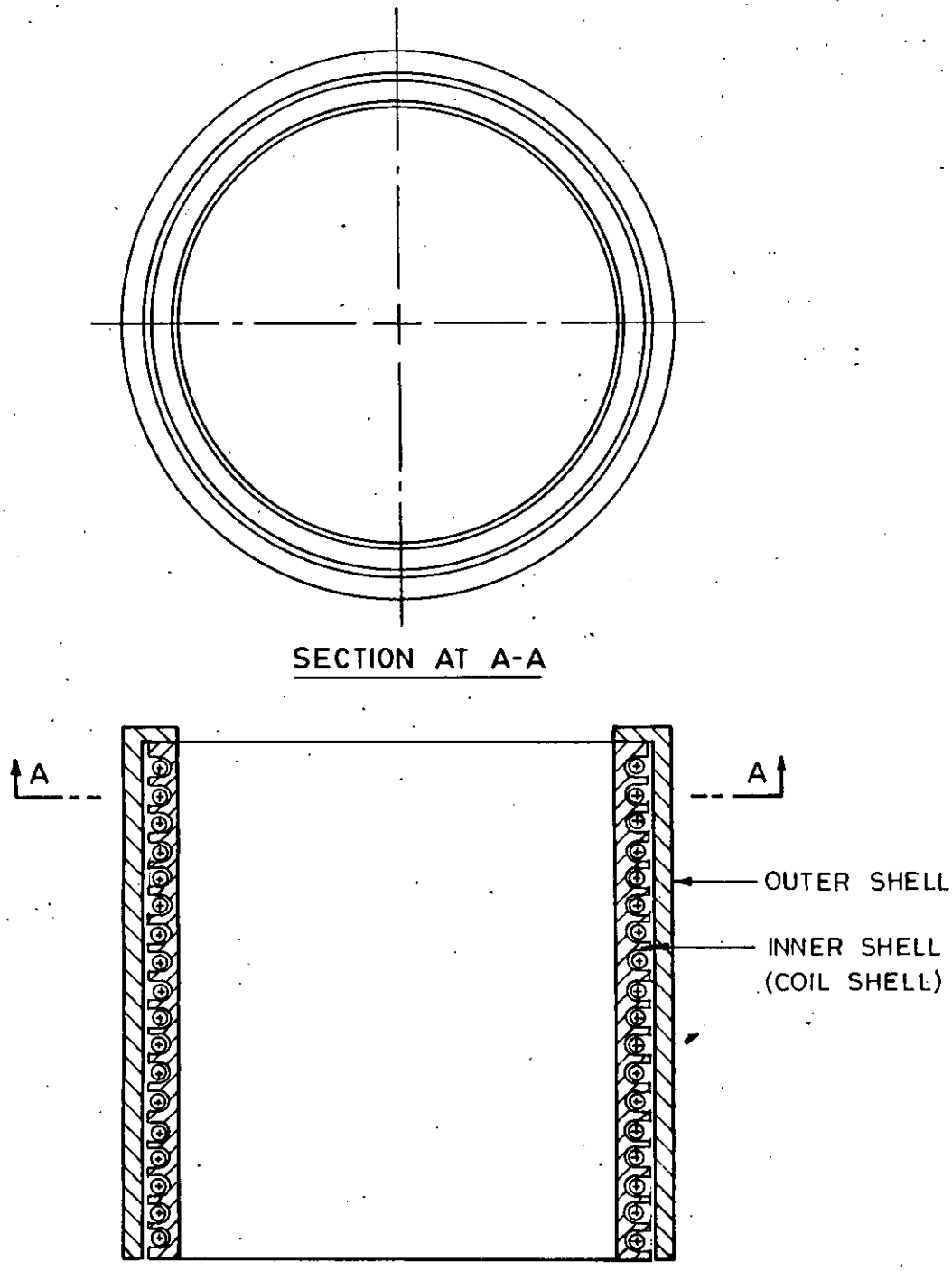
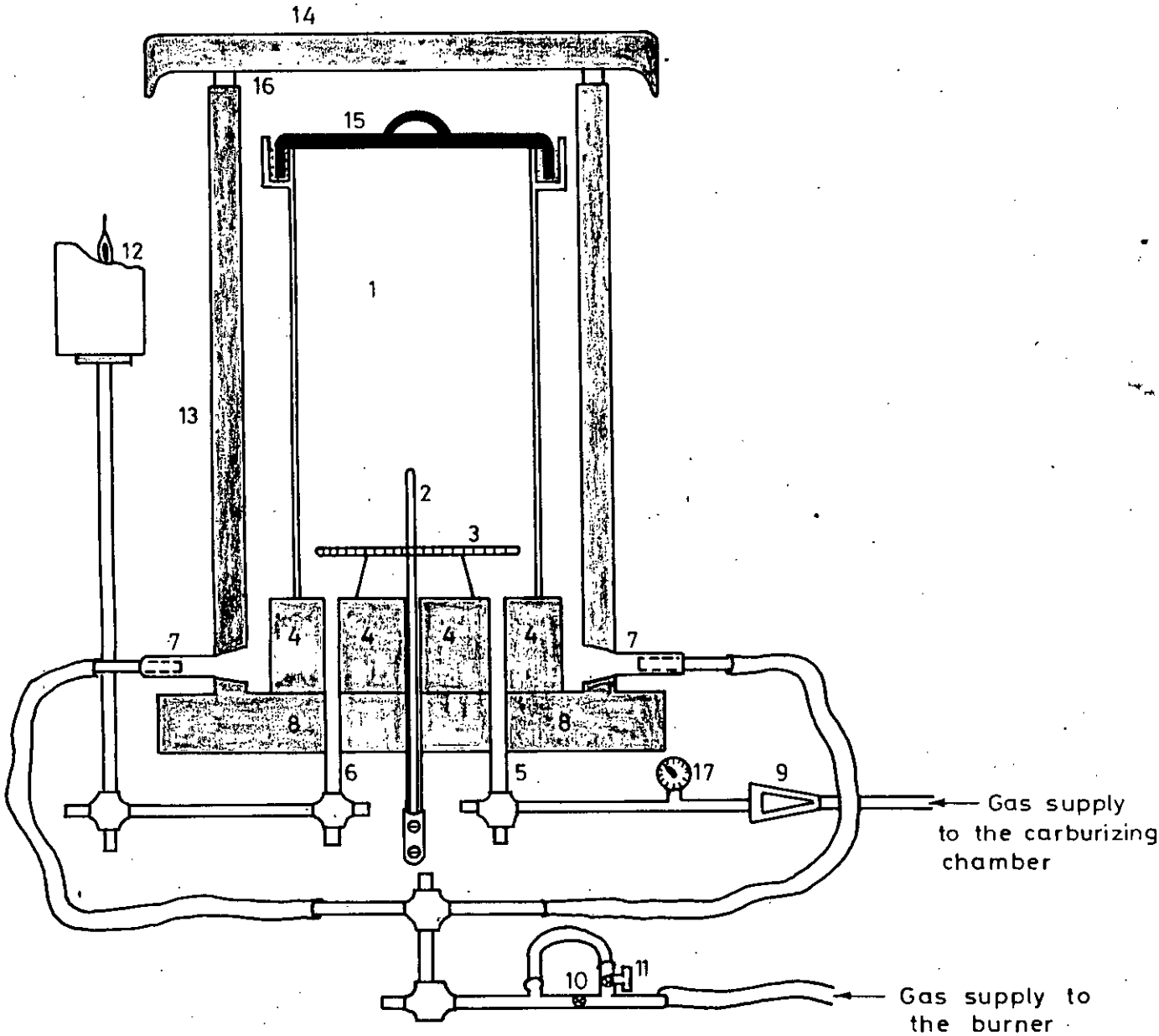


Figure 3: Heating unit showing the arrangement of inner shell (coil shell) and outer shell.



1 Carburizing chamber
2. Thermocouple
3 Bottom plate
4 Circular platform
5 Gas inlet
6 Gas outlet
7 Gas burner
8 Furnace bottom
9 Gas flow meter (rator meter)
10 Solenoid valve
11 Bye-pass line with a valve
12 Signal flame
13 Furnace outside wall
14 Lid (slab)
15 Steel cap
16 Supporting fire brick
17 Pressure gauge

Figure 4: Schematic diagram of gas fired gas carburizing furnace with automatic control system.

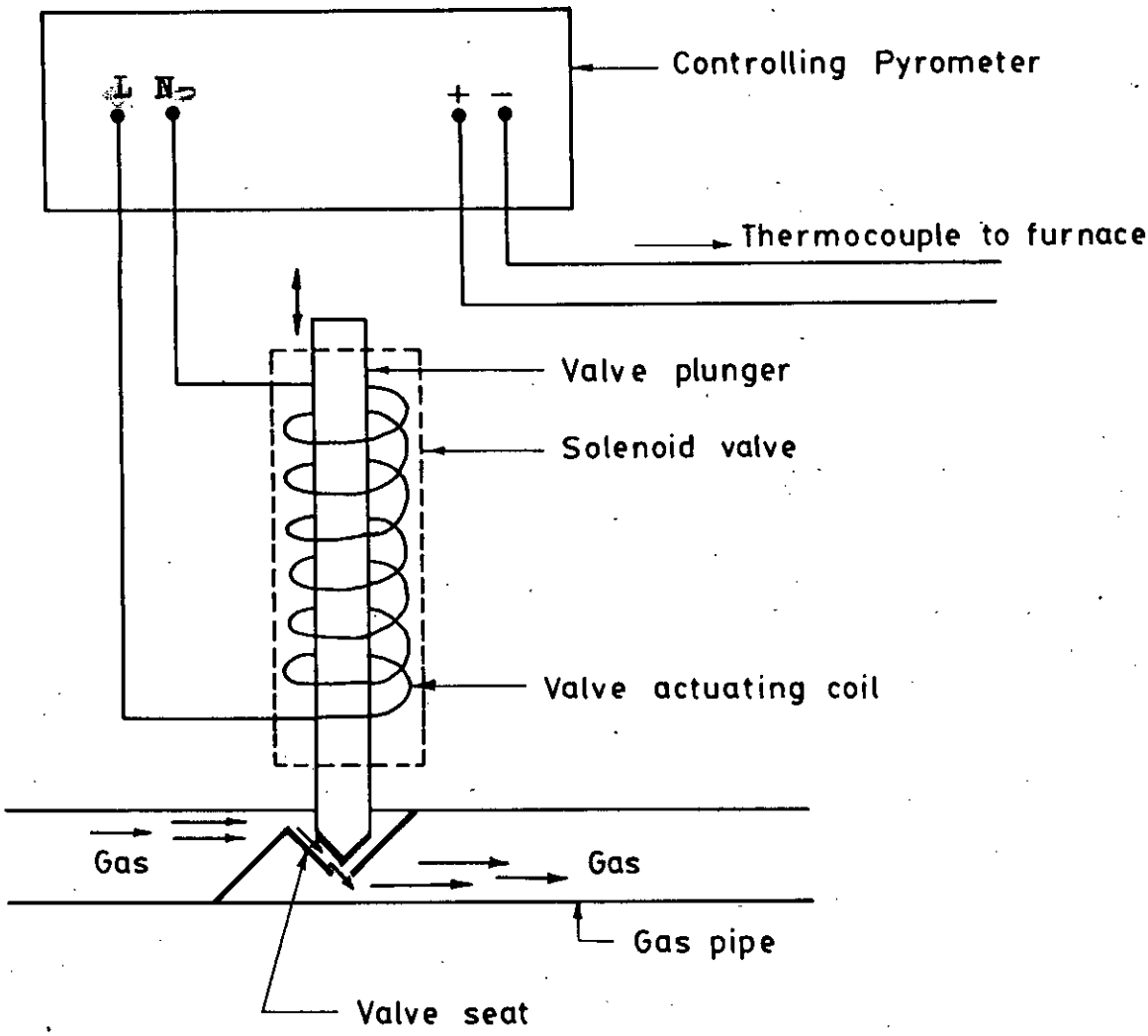
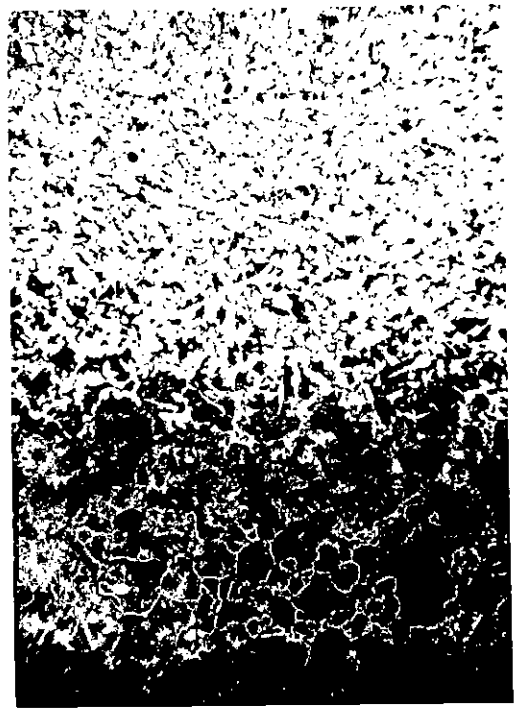


Figure 5: Schematic arrangement of Solenoid Valve.

(a) Steel 1 X 140

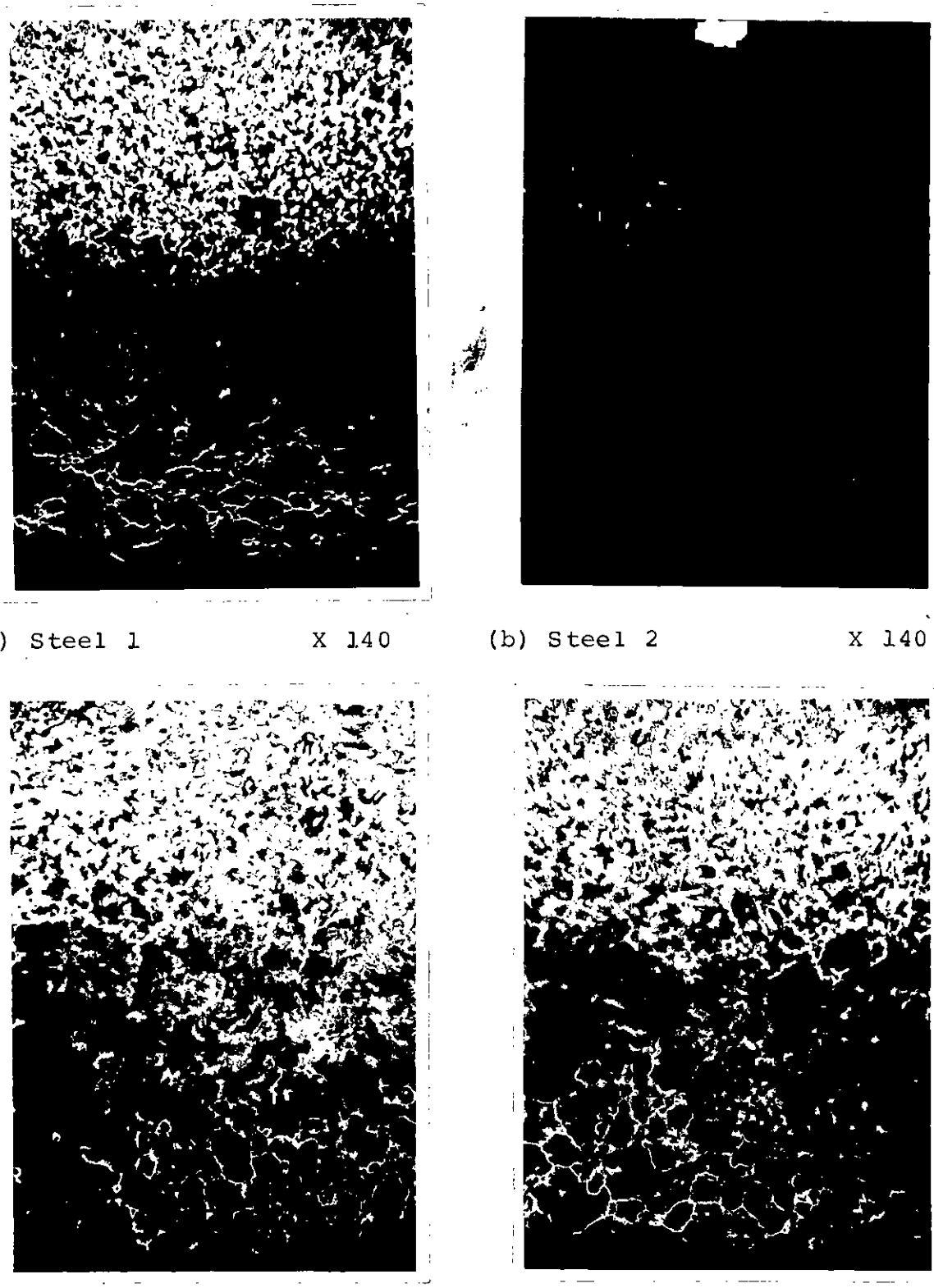
(b) Steel 2 X 140

(c) Steel 3 X 140



(d) Steel 4 X 140

Figure 6: Optical micrographs showing microstructures and depth of case of steels 1 to 4 carburized at 950^{o}C for 1 hour and slowly cooled in the furnace.

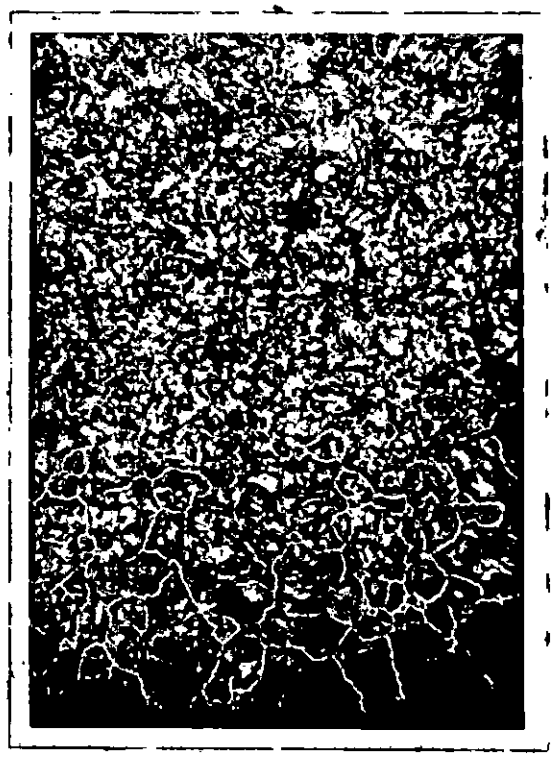


(a) Steel 1 X 140 (b) Steel 2 X 140

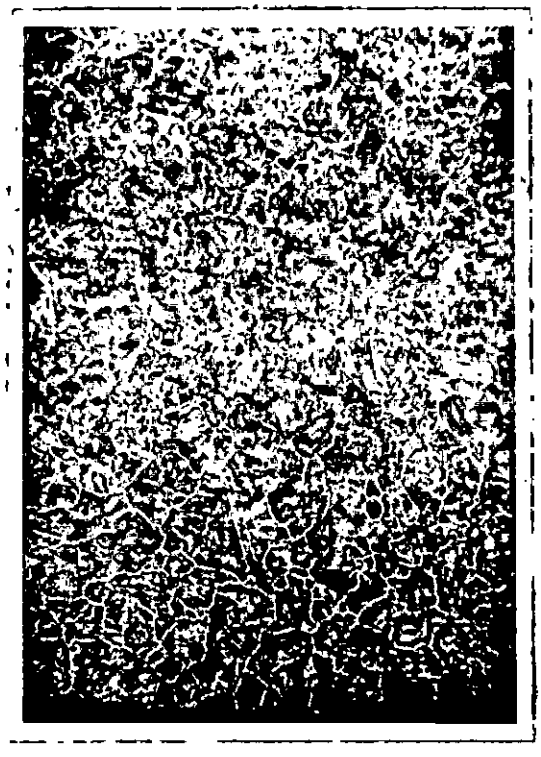
(c) Steel 3 X 140 (d) Steel 4 X 140

Figure 7: Optical micrographs showing microstructures and depth of case of steels 1 to 4 carburized at 950°C for 3 hours and slowly cooled in the furnace.

(a) Steel 1 X 140



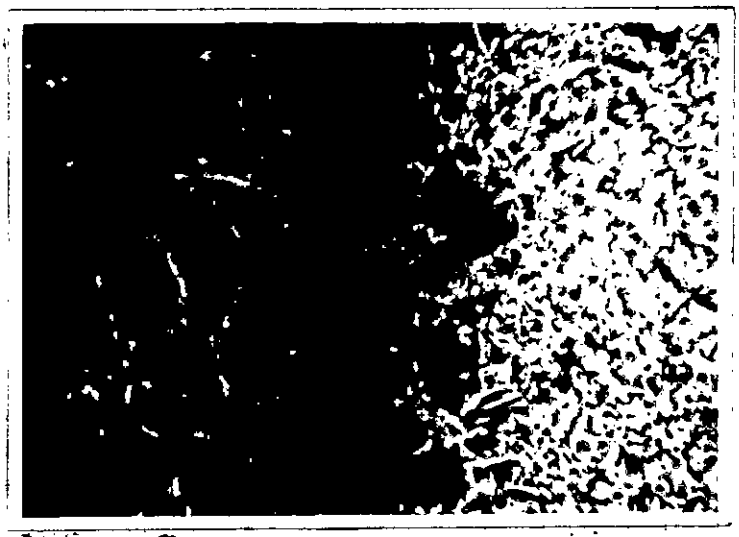
(b) Steel 2 X 140



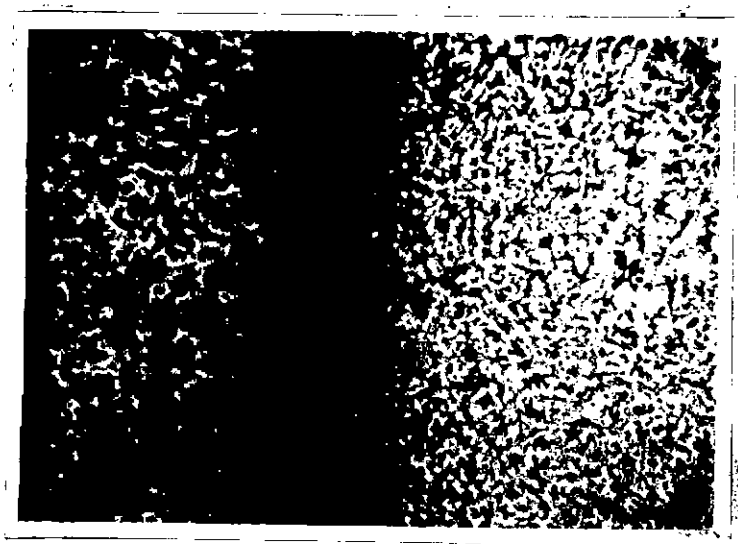
(c) Steel 3 X 140

(d) Steel 4 X 140

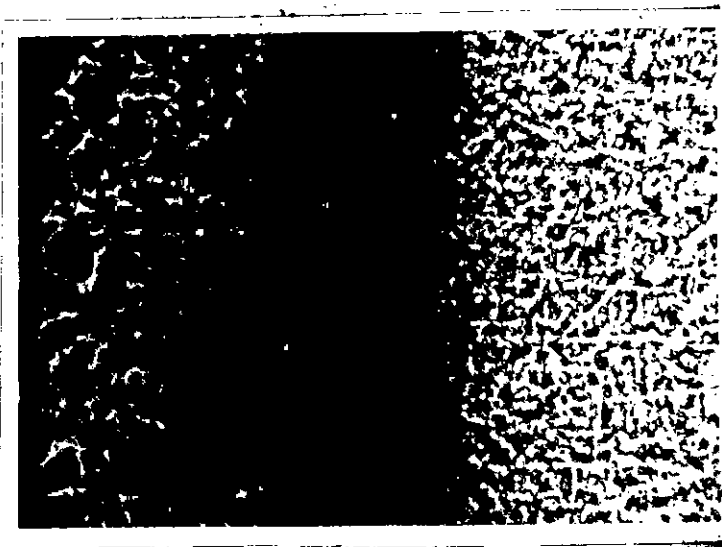
Figure 8: Optical micrographs showing microstructures and depth of case of steels 1 to 4 carburized at 950°C for 5 hours and slowly cooled in the furnace.



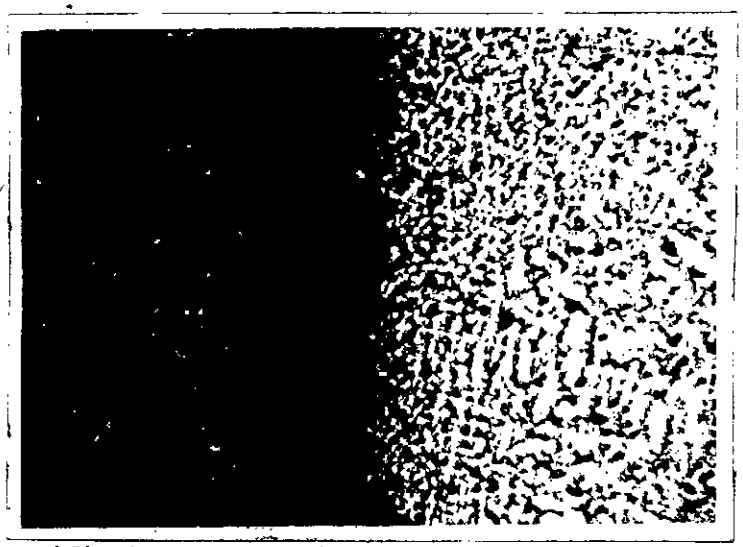
(a) Steel 6 X 140



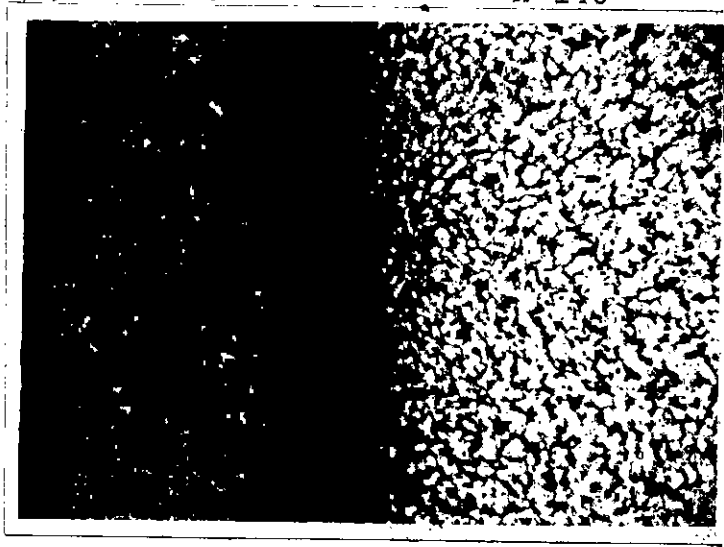
(b) Steel 7 X 140



(c) Steel 8 X 140

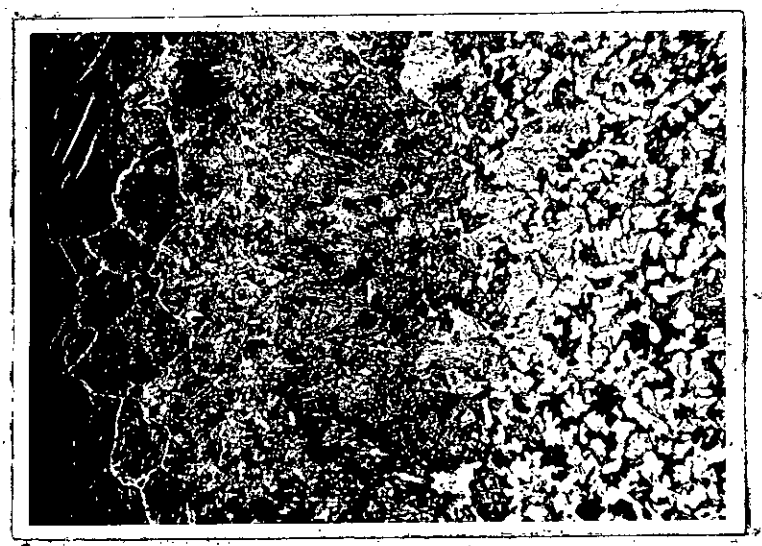


(d) Steel 9 X 140

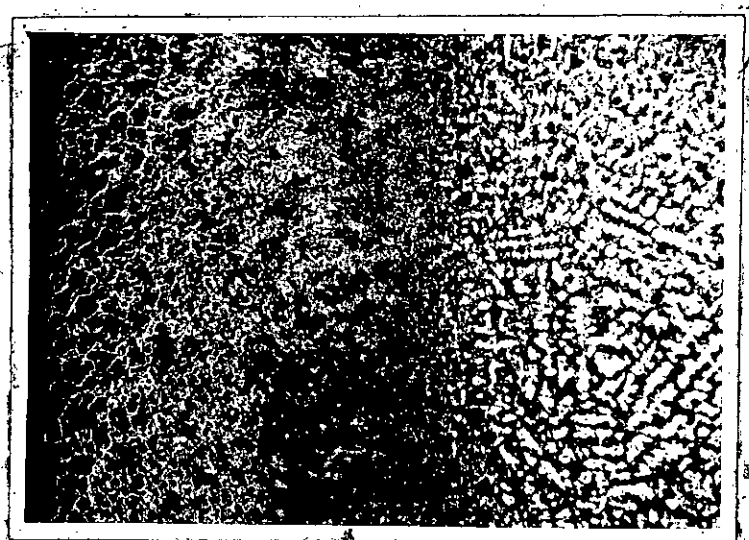


(e) Steel 10 X 140

Figure 9: Optical micrographs showing microstructures and depth of case of steels 6 to 10 carburized at 950°C for 1 hour and slowly cooled in the furnace.

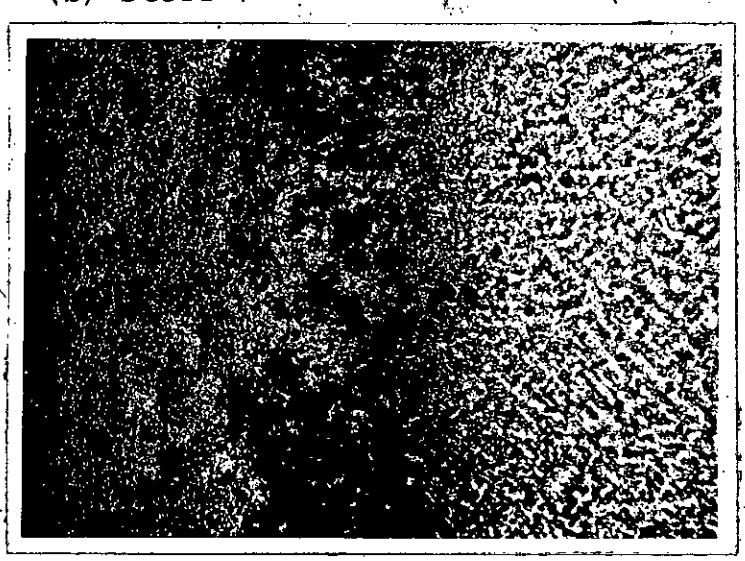


(a) Steel 6 X 140

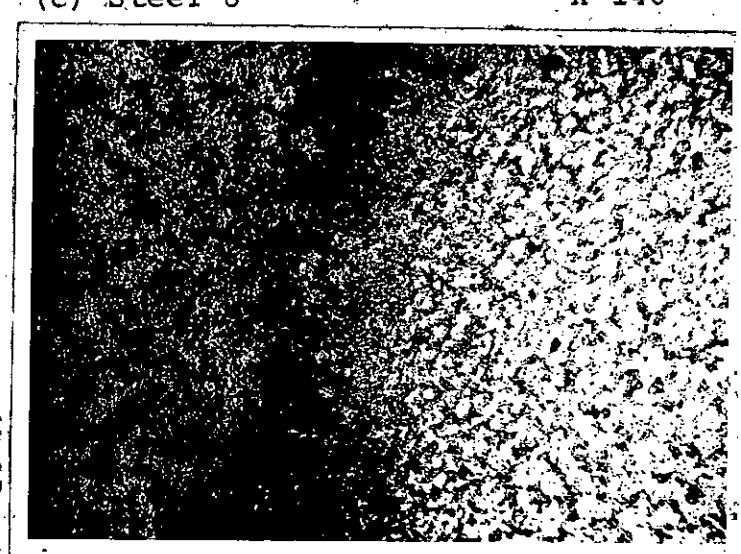


(b) Steel 7 X 140

(c) Steel 8 X 140

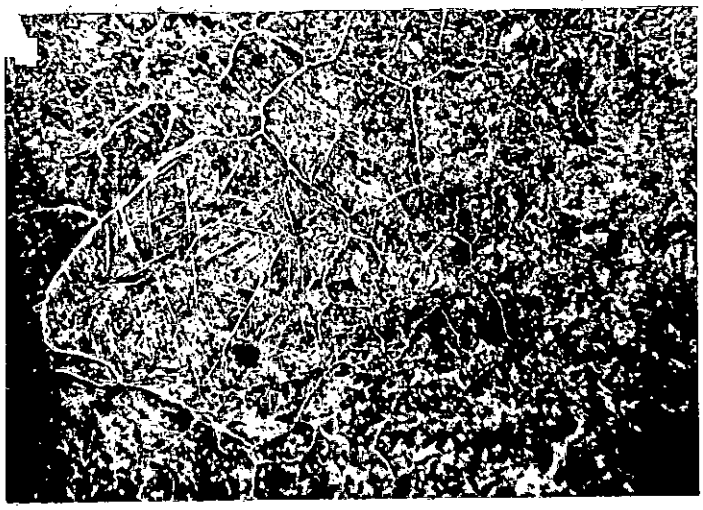


(d) Steel 9 X 140

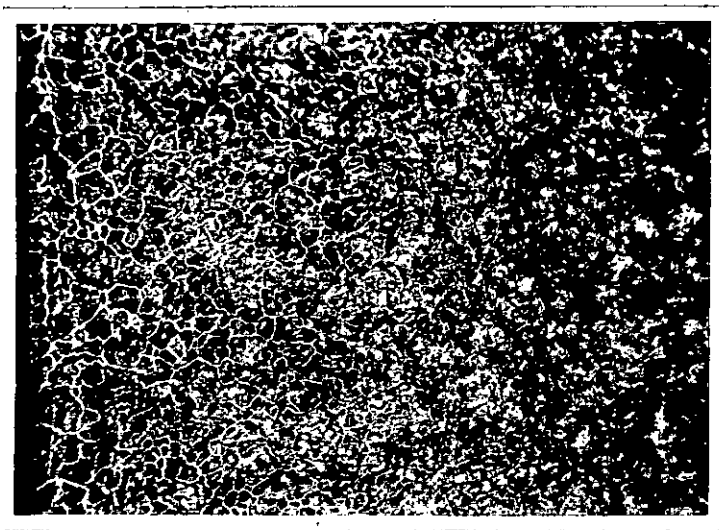


(e) Steel 10 X 140

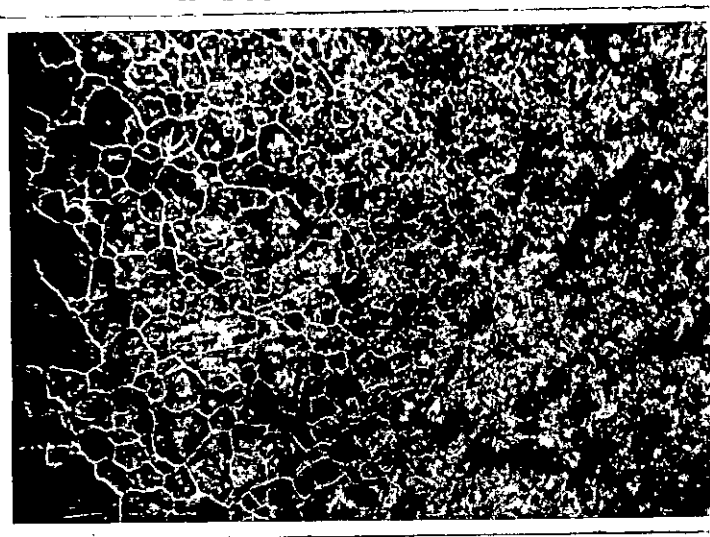
Figure 10: Optical micrographs showing microstructures and depth of case of steels 6 to 10 carburized at 950°C for 3 hours and slowly cooled in the furnace.



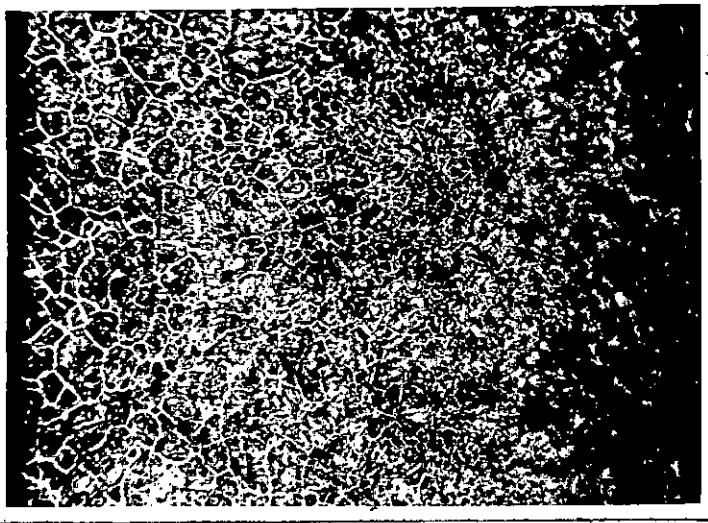
(a) Steel 6 X 140



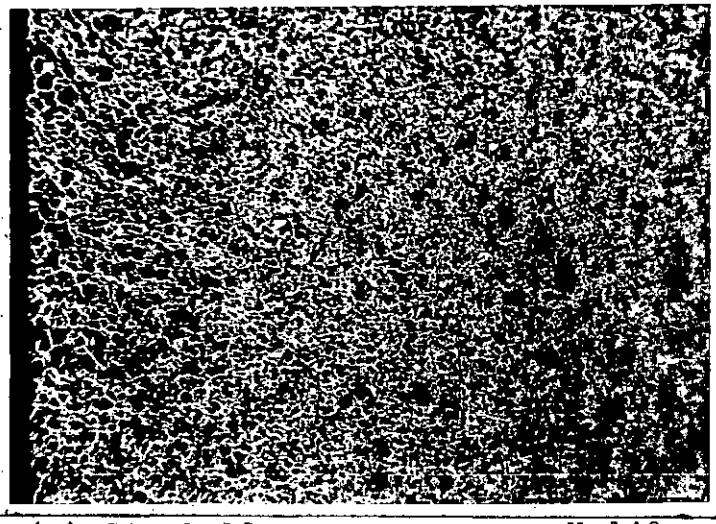
(b) Steel 7 X 140



(c) Steel 8 X 140



(d) Steel 9 X 140



(e) Steel 10 X 140

Figure 11: Optical micrographs showing microstructures and depth of case of steels 6 to 10 carburized at 950°C for 5 hours and slowly cooled in the furnace.

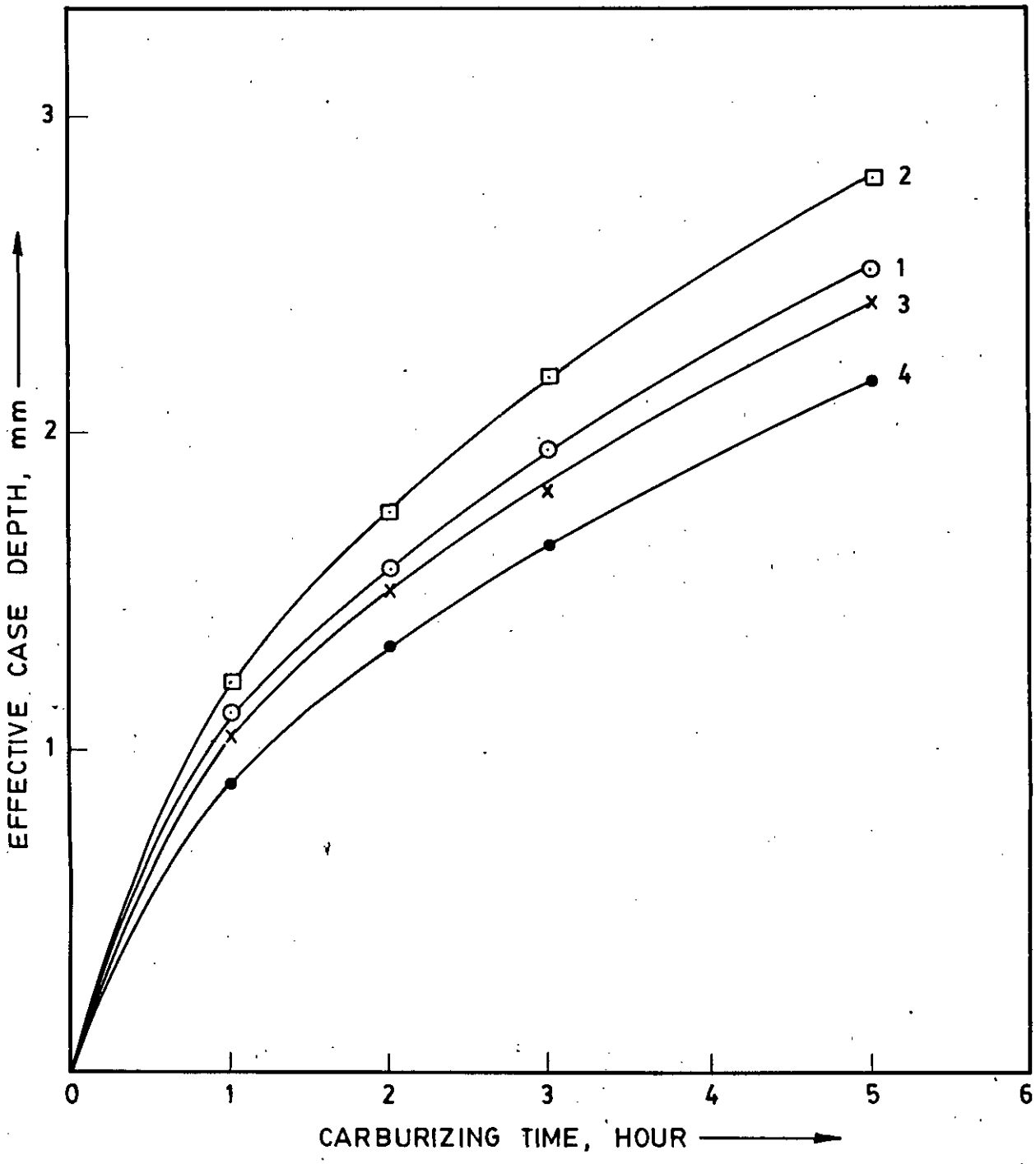


Figure 12: Variation of effective case depth with carburizing time at 950°C in steels 1 to 4.

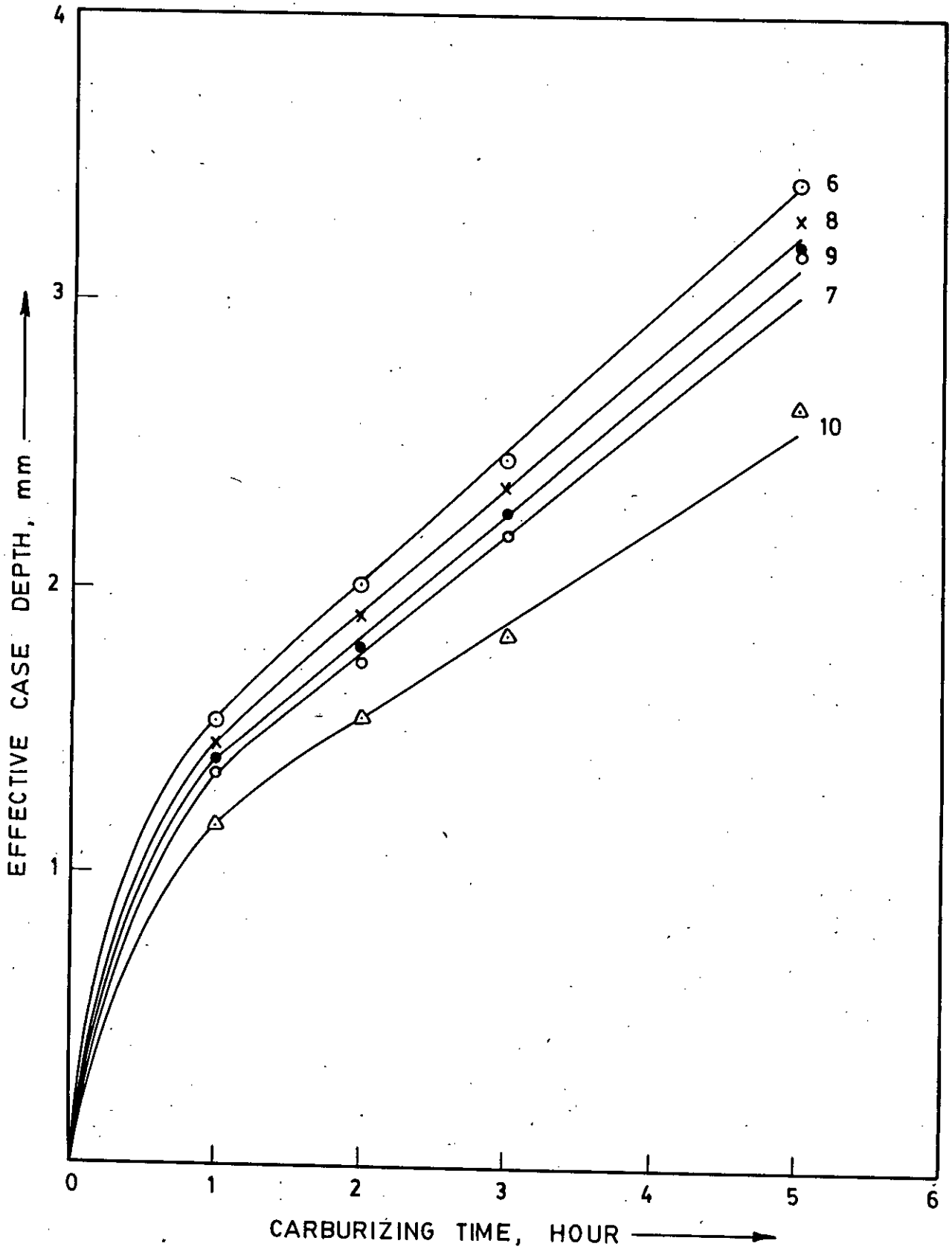


Figure 13: Variation of effective case depth with carburizing time at 950°C in steels 6 to 10.

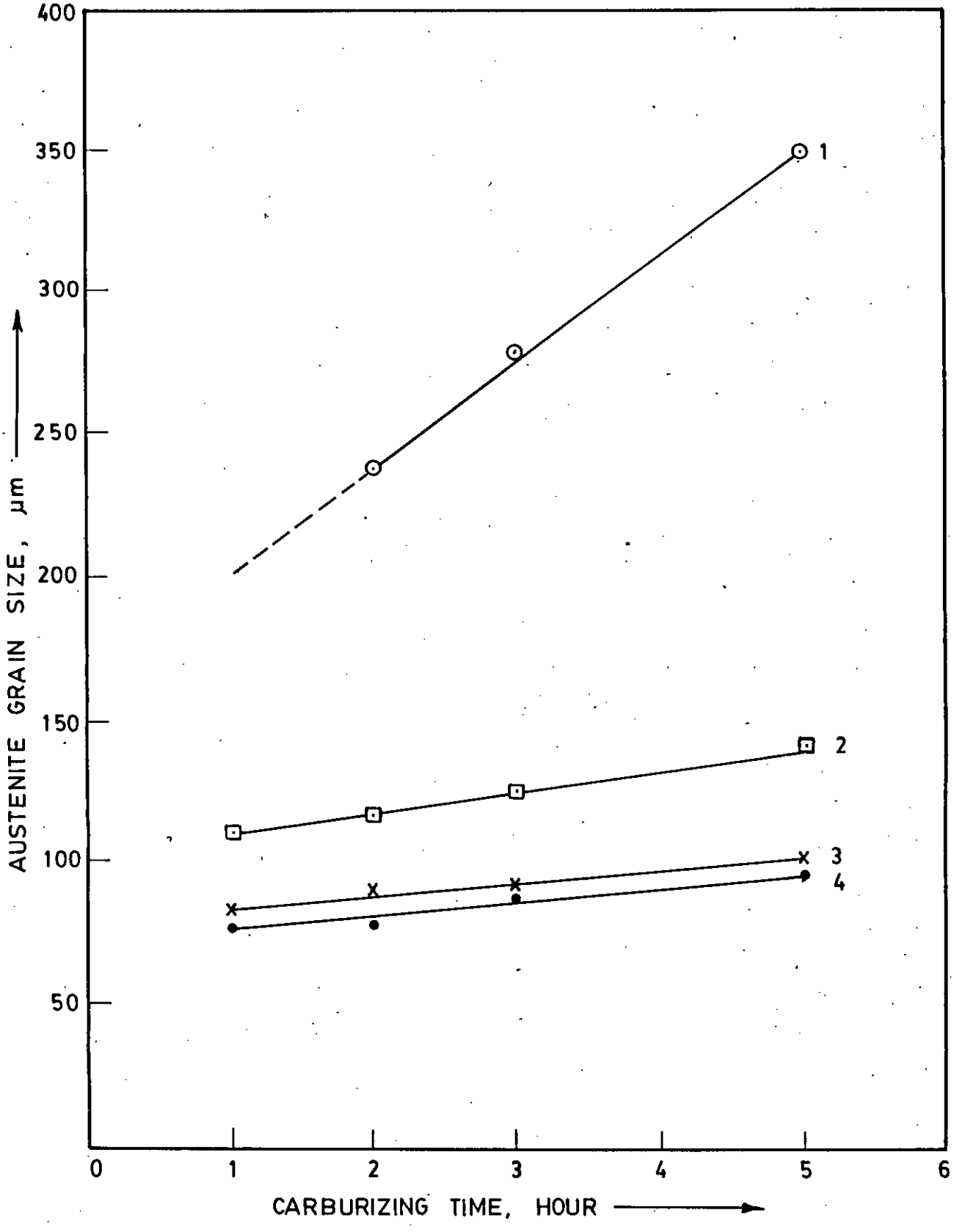


Figure 14: Variation of prior austenite grain size with carburizing time at 950°C in the case of steels 1 to 4.

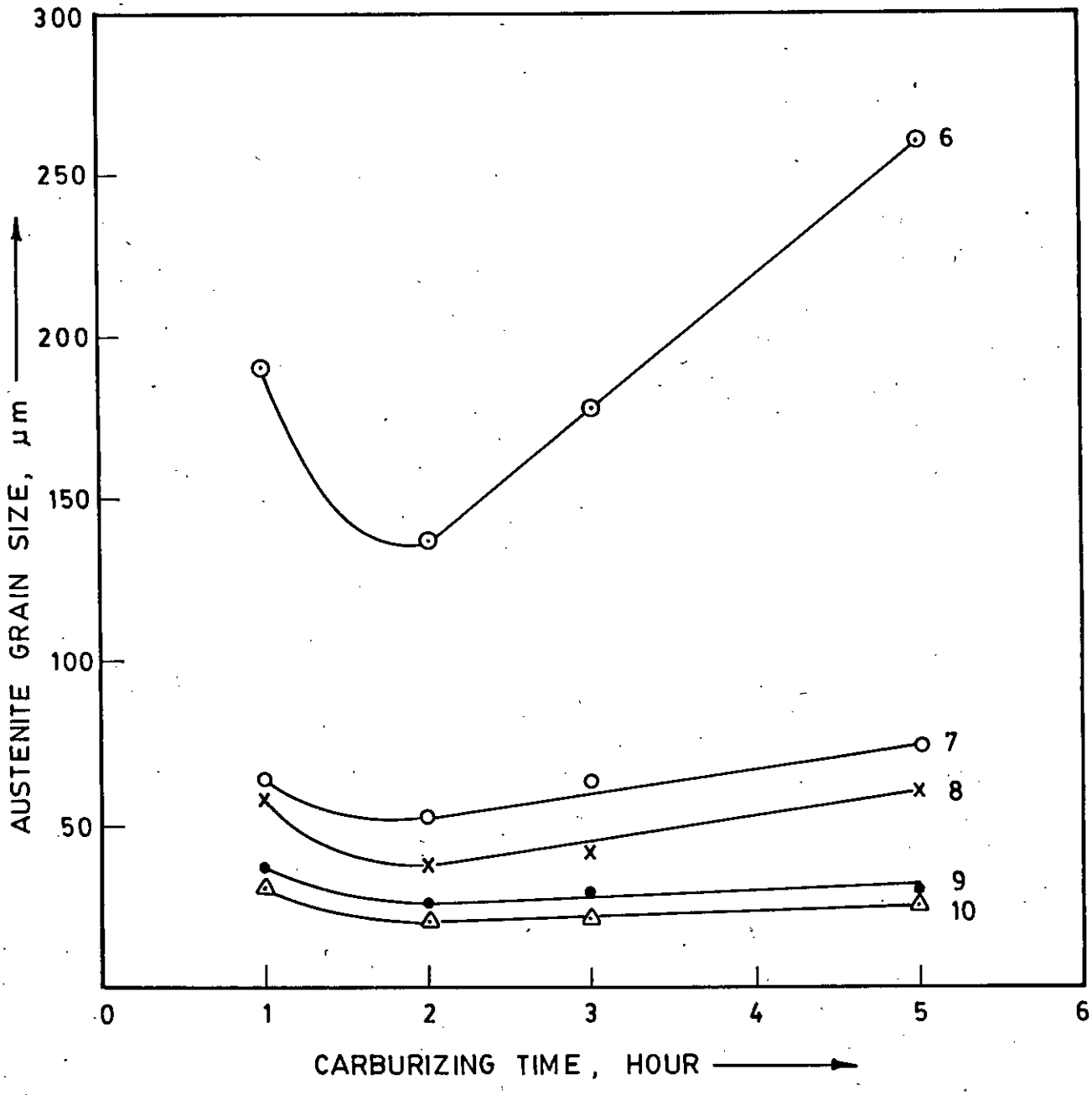


Figure 15: Variation of prior austenite grain size with carburizing time at 950°C in case of steels 6 to 10.

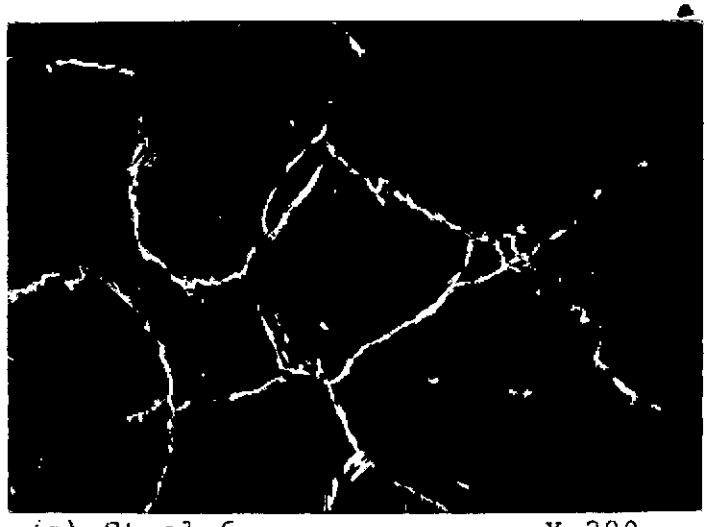
(a) Steel 1 X 280

(b) Steel 2 X 280

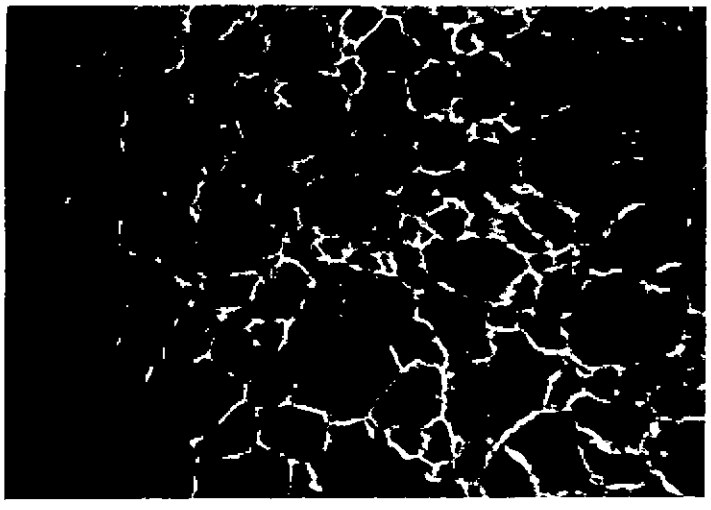
(c) Steel 3 X 280

(d) Steel 4 X 280

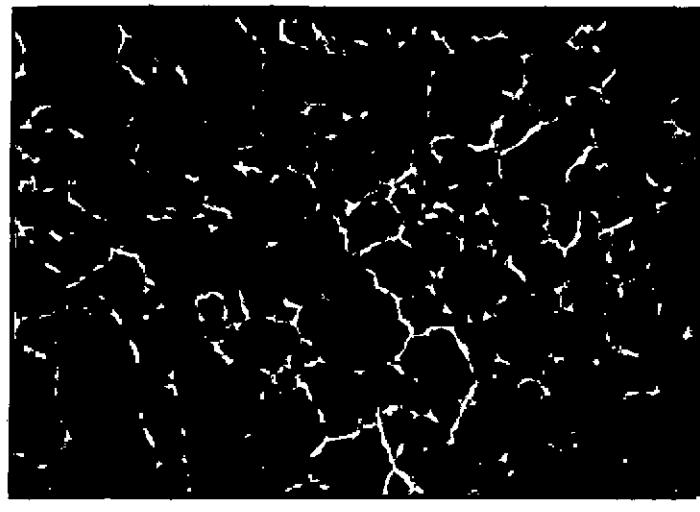
Figure 16: Optical micrographs showing the prior austenite grain size of steels 1 to 4 carburized at 950°C for 5 hours and slowly cooled in the furnace.



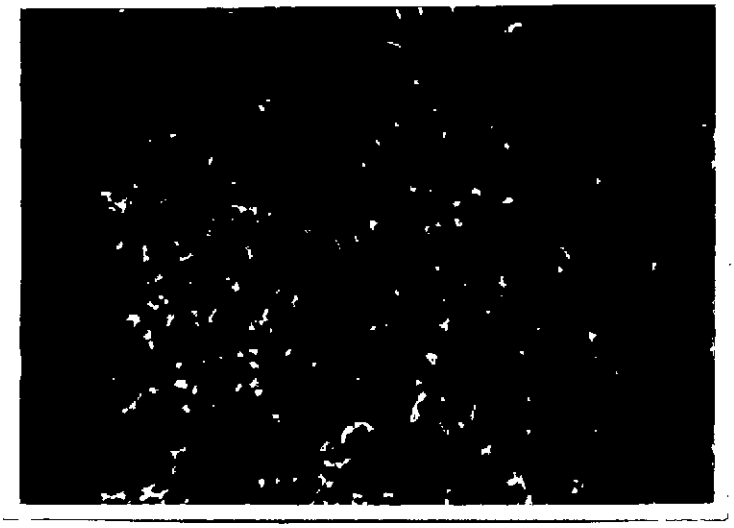
(a) Steel 6 X 280



(b) Steel 7 X 280



(c) Steel 8 X 280

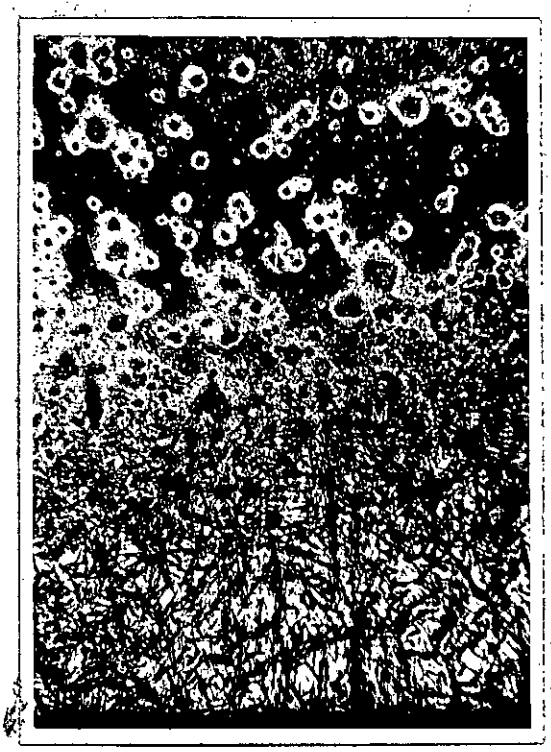


(d) Steel 9 X 280

(e) Steel 10 X 280

Figure 17: Optical micrographs showing the prior austenite grain size of steels 6-10 carburized at 950°C for 5 hours and slowly cooled in the furnace.

(a) Steel 1 X 280

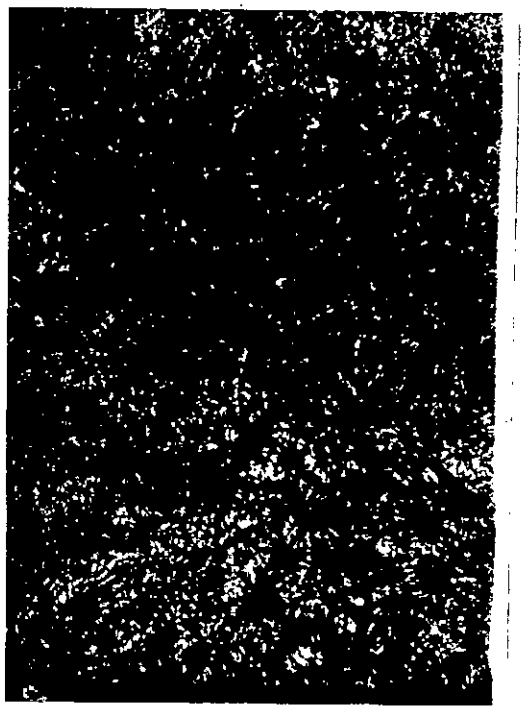


(b) Steel 2 X 280

(c) Steel 3 X 280

(d) Steel 4 X 280

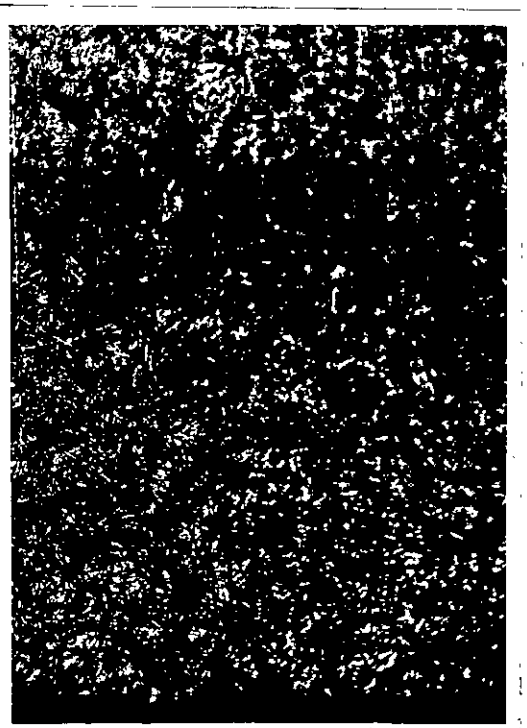
Figure 18: Optical micrographs showing microstructures of steels 1-4 carburized at 950°C for 1 hour quenched in 10% brine and tempered at 160°C.



(a) Steel 1 X 280

(b) Steel 2 X 280

(c) Steel 3 X 280



(d) Steel 4 X 280

Figure 19: Optical micrographs showing microstructures of steels 1-4 carburized at 950°C for 3 hours, quenched in 10% brine and tempered at 160°C.

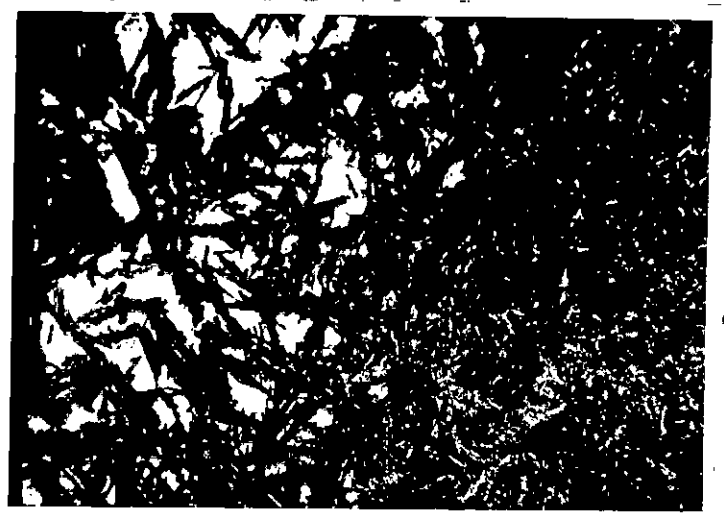
(a) Steel 1 X 280

(b) Steel 2 X 280

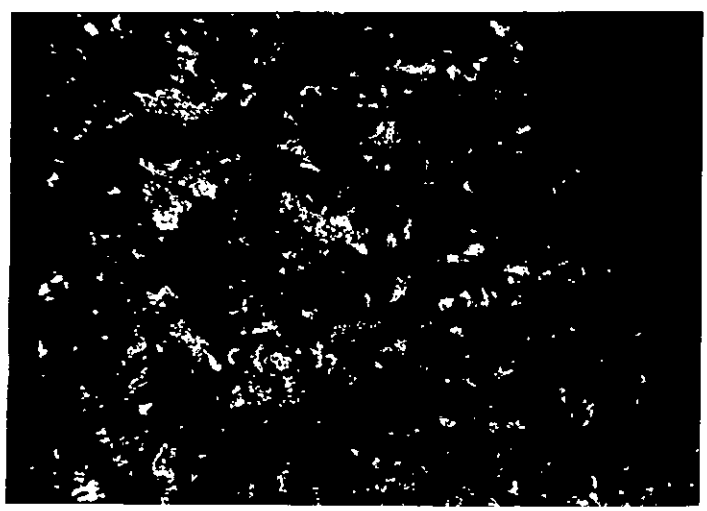
(c) Steel 3 X 280

(d) Steel 4 X 280

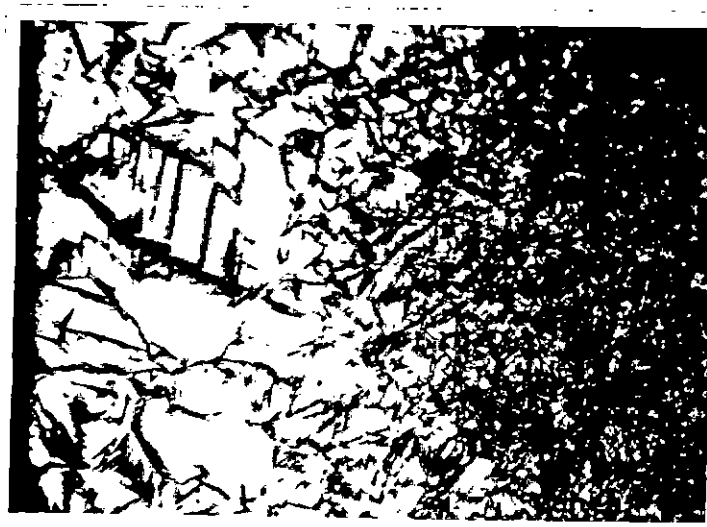
Figure 20: Optical micrographs showing the microstructures of core of steels 1-4 carburized at 950°C for 1 hour quenched in 10% brine and tempered at 160°C.



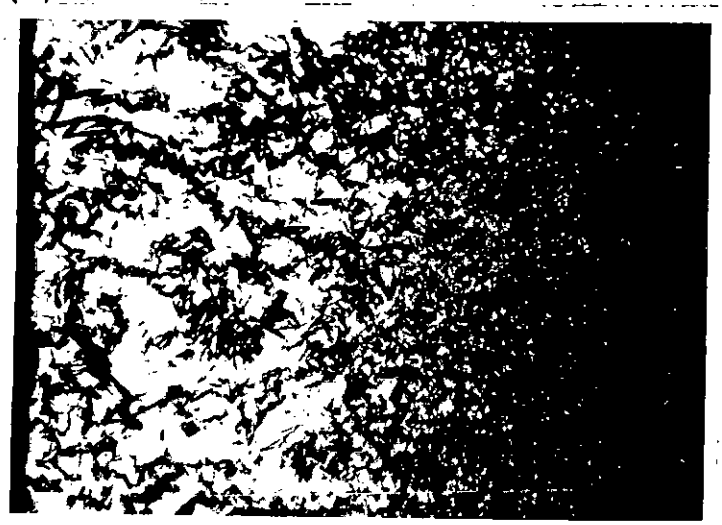
(a) Steel 6 X 280



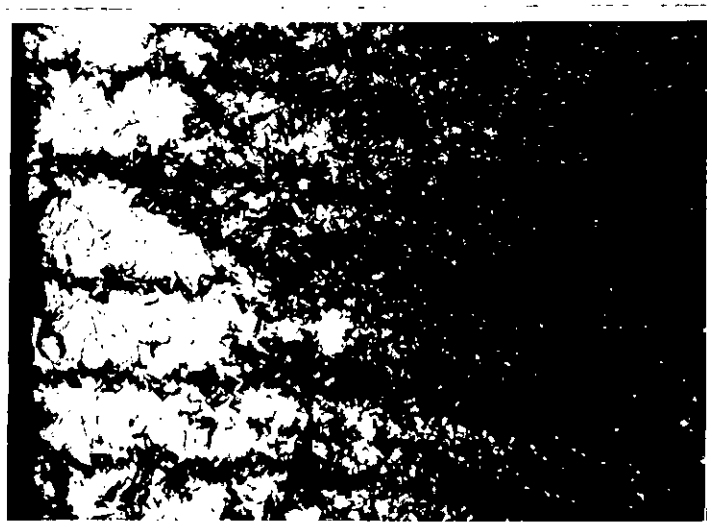
(b) Steel 7 X 280



(c) Steel 8 X 280



(d) Steel 9 X 280

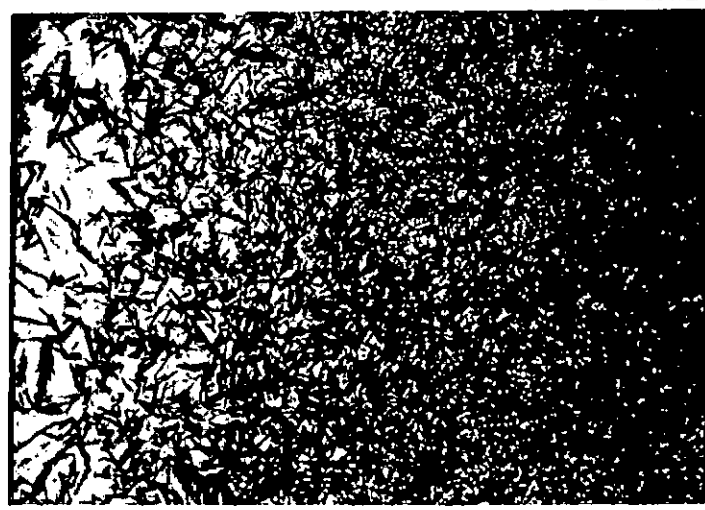


(e) Steel 10 X 280

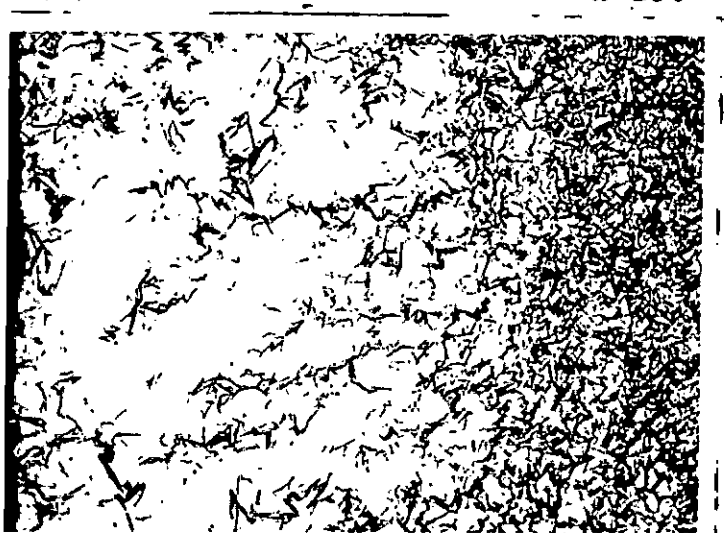
Figure 21: Optical micrographs showing the microstructures of steels 6 to 10 carburized at 950°C for 1 hour quenched in 10% brine and tempered at 160°C.

(a) Steel 6 X 280

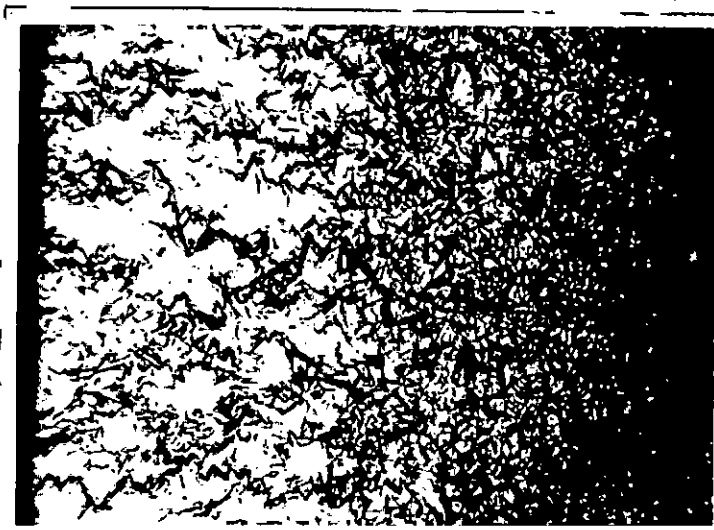
(b) Steel 7 X 280



(c) Steel 8 X 280



(d) Steel 9 X 280



(e) Steel 10 X 280

Figure 22: Optical micrographs showing the microstructures of steels 6 to 10 carburized at 950°C for 3 hours quenched in 10% brine and tempered at 160°C.

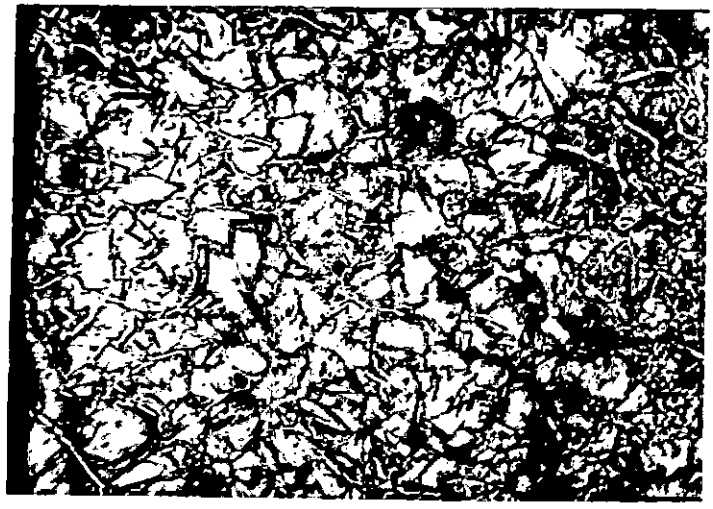


Figure 23: Optical micrograph showing the cementite networks in the case of steel 7 carburized at 950°C for 5 hours, quenched in 10% brine and tempered at 160°C, X 280.

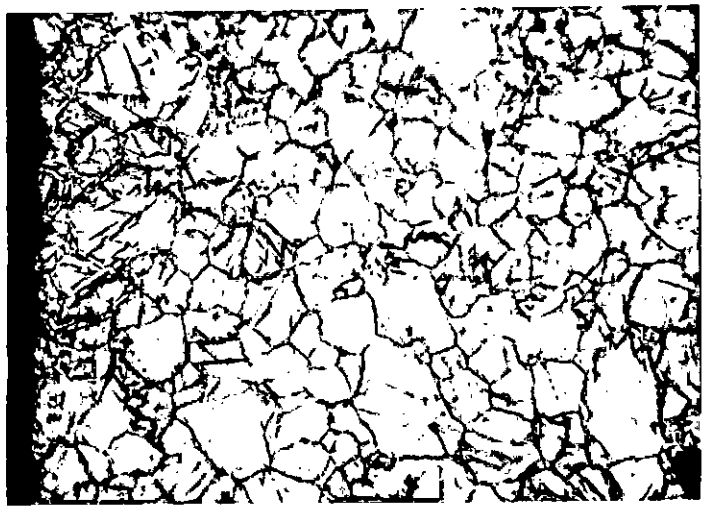
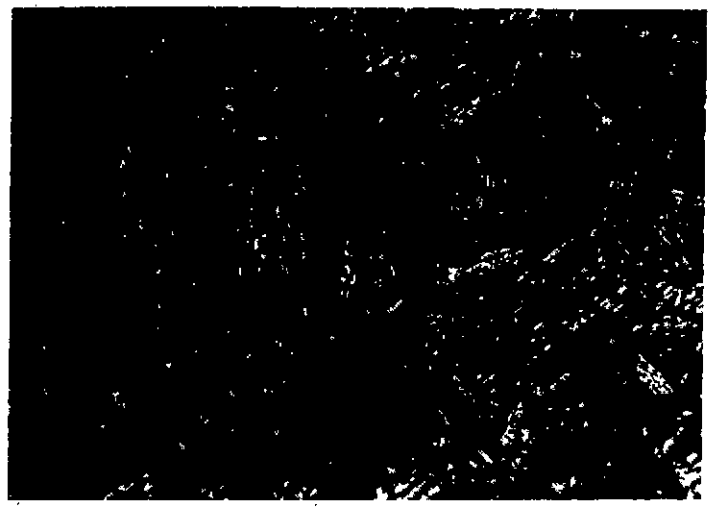


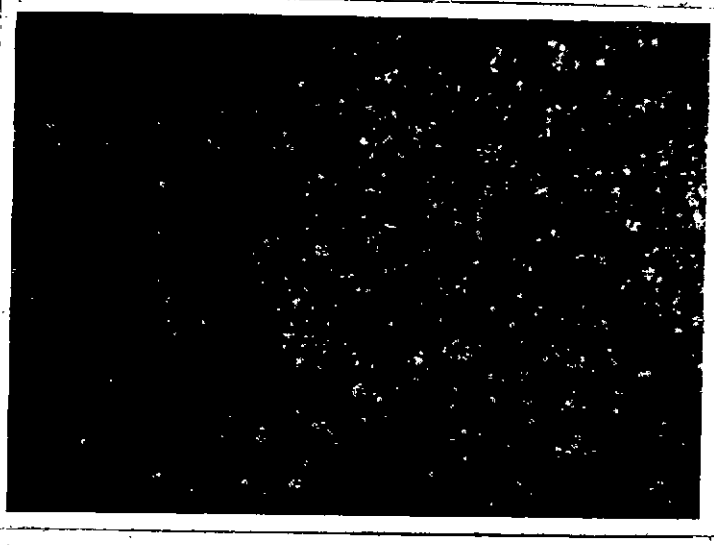
Figure 24: Optical micrograph showing the polygonal grains of austenite in the case of steel 7 carburized at 950°C for 5 hours, quenched in 10% brine and tempered at 160°C, X 280.



(a) Steel 6 X 280

(b) Steel 7 X 280

(c) Steel 8 X 280



(d) Steel 9 X 280

(e) Steel 10 X 280

Figure 25: Optical micrographs showing the microstructures of core of steels 6 to 10 carburized at 950°C for 1 hour quenched in 10% brine and tempered at 160°C.

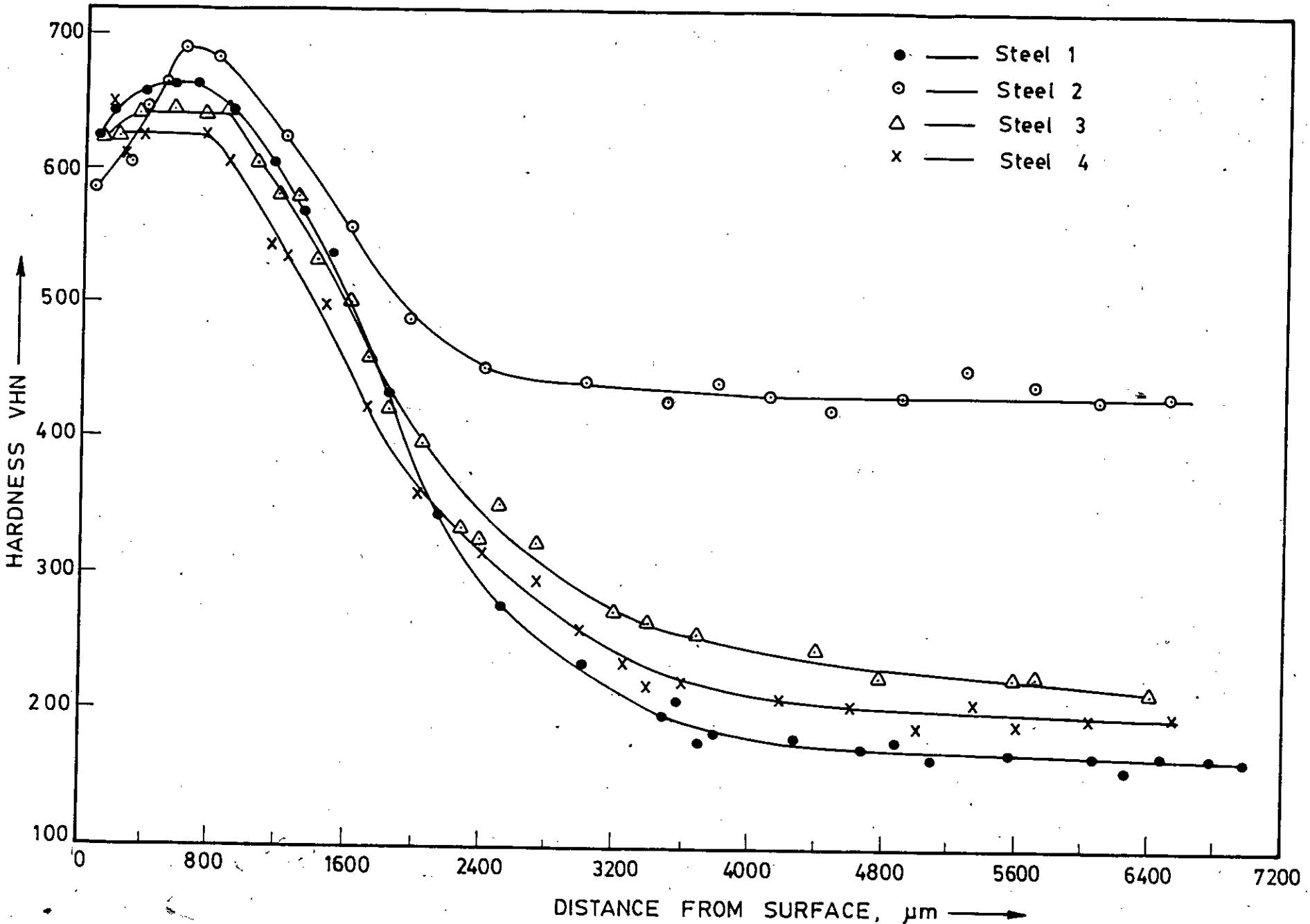


Figure 26: Microhardness profiles of steels 1 to 4 carburized at 950°C for 1 hour, quenched in 10% brine and tempered at 160°C

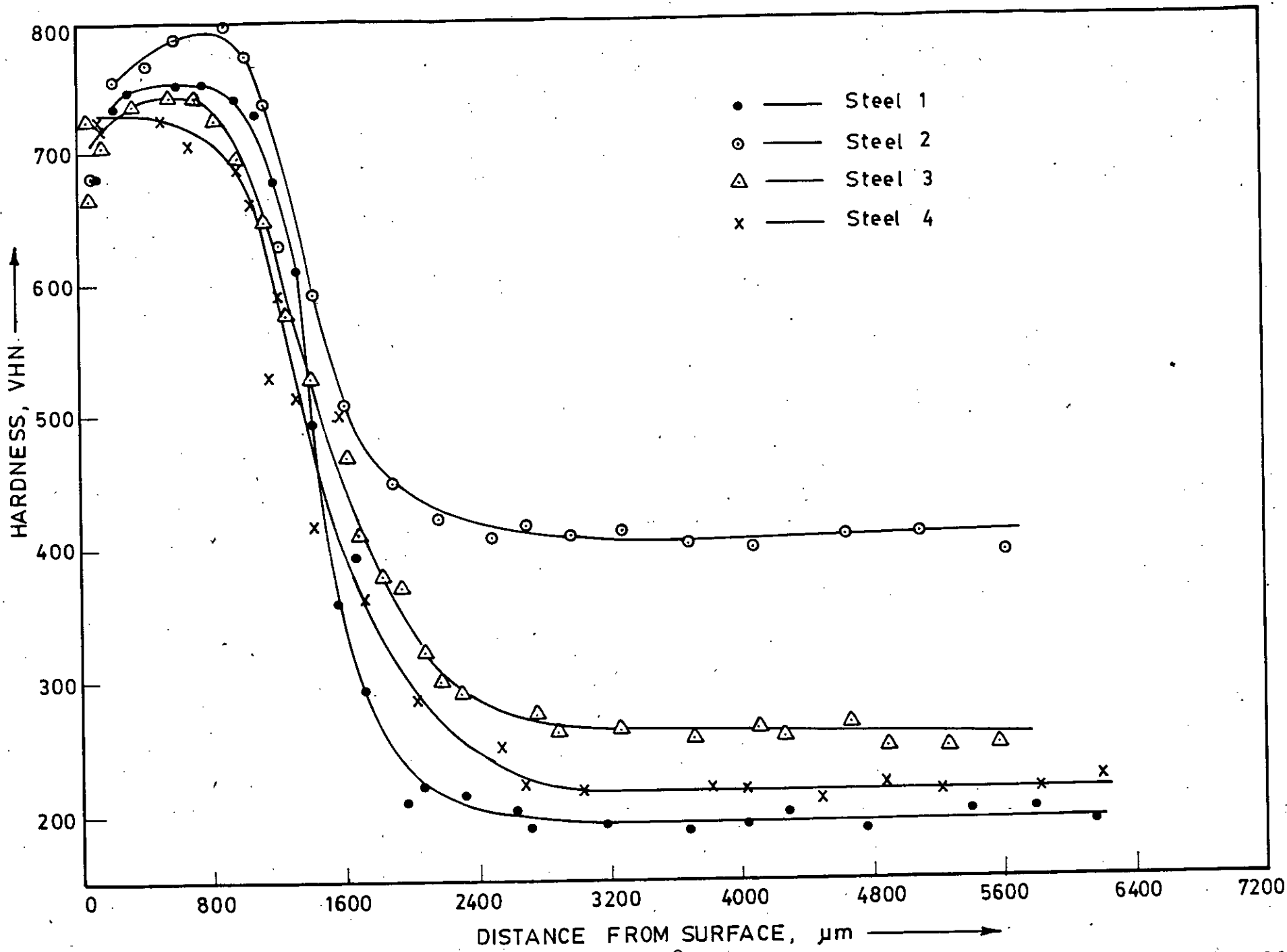


Figure 27: Microhardness profiles of steels 1-4 carburized at 950°C for 3 hours, quenched in 10% brine and tempered at 160°C.

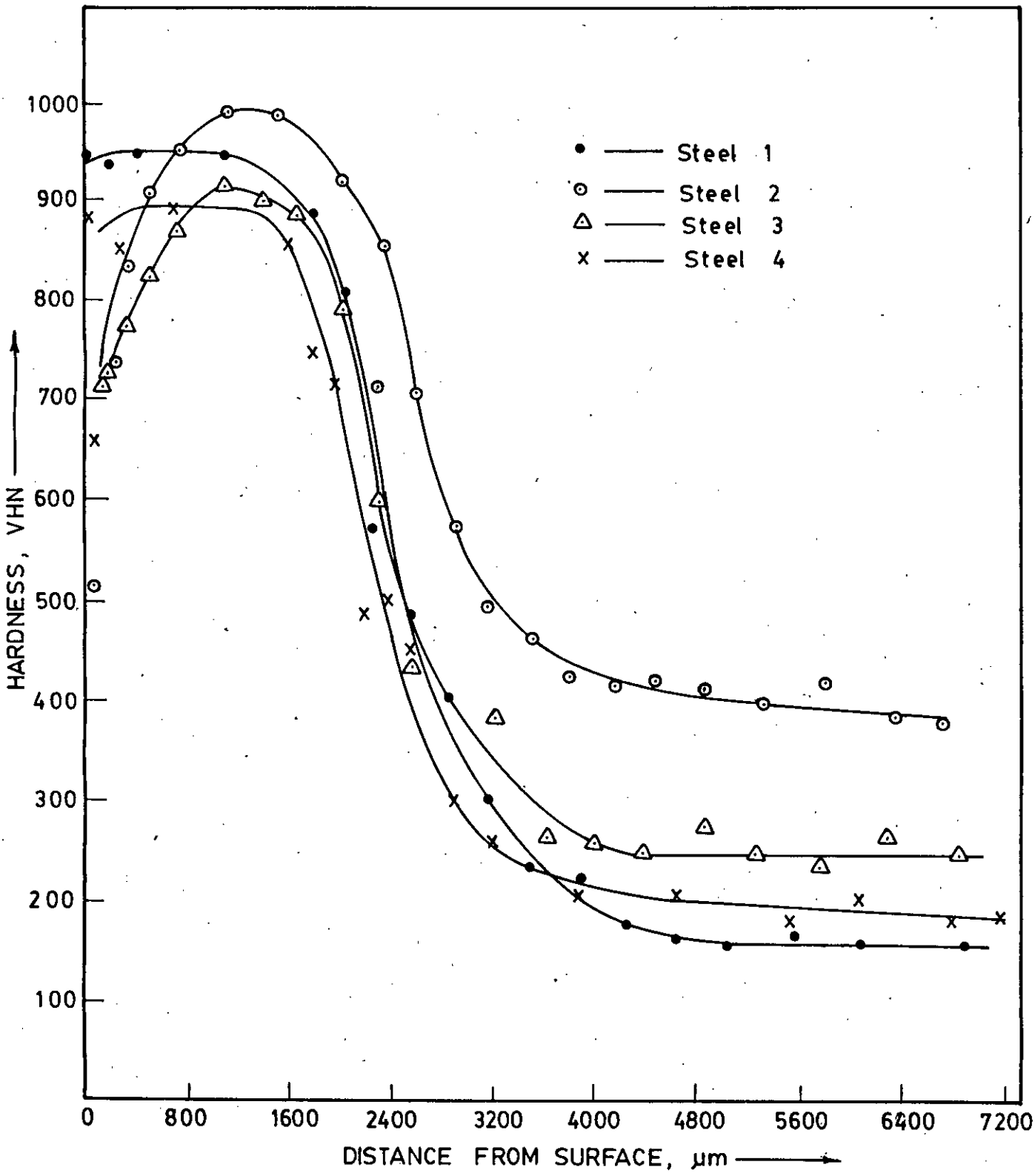


Figure 28: Microhardness profiles of steel 1-4 carburized at 950°C for 5 hours, quenched in 10% brine and tempered at 160°C.

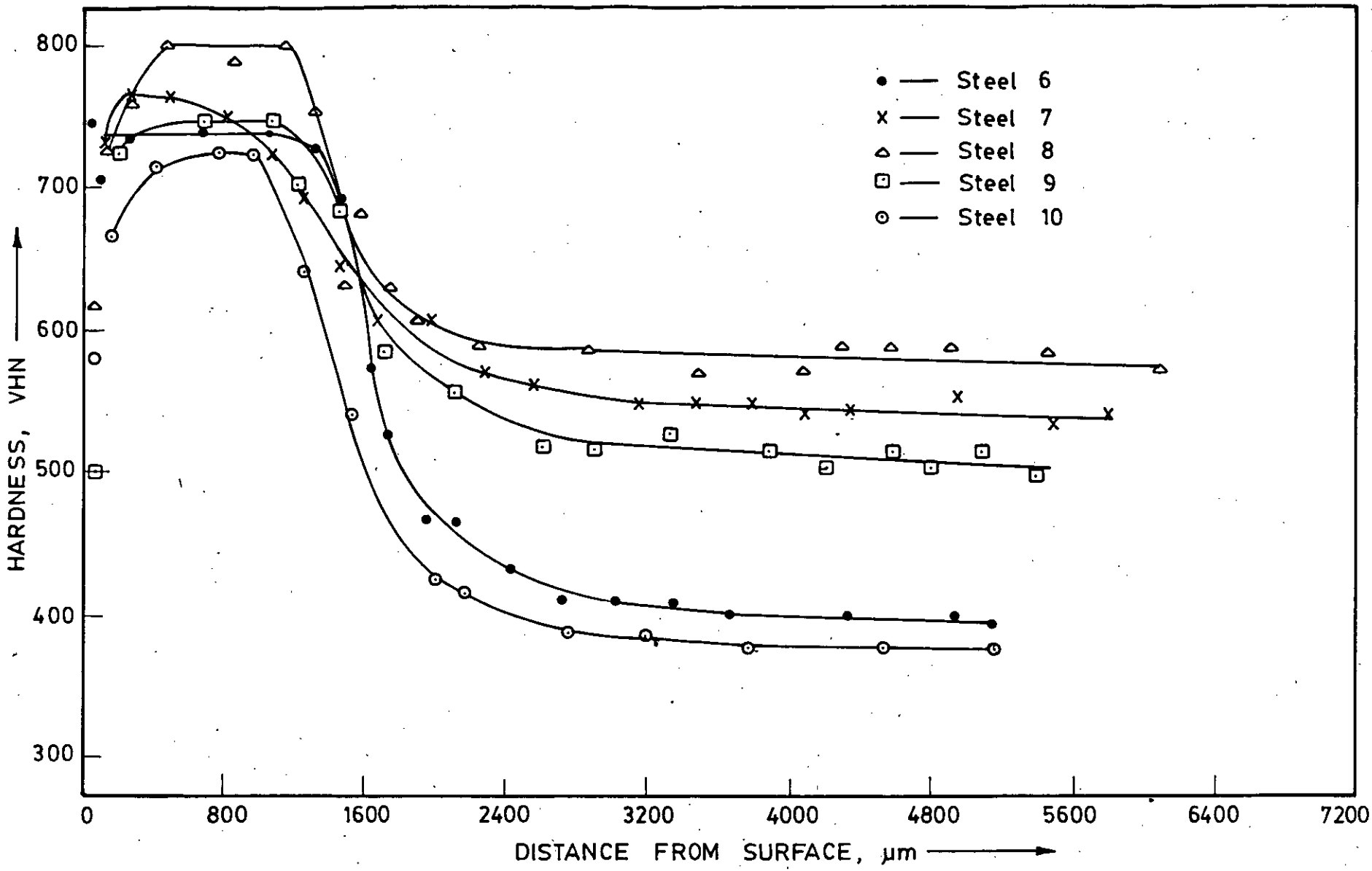


Figure 29: Microhardness profiles of steels 6 to 10 carburized at 950°C for 1 hour, quenched in 10% brine and tempered at 160 °C.

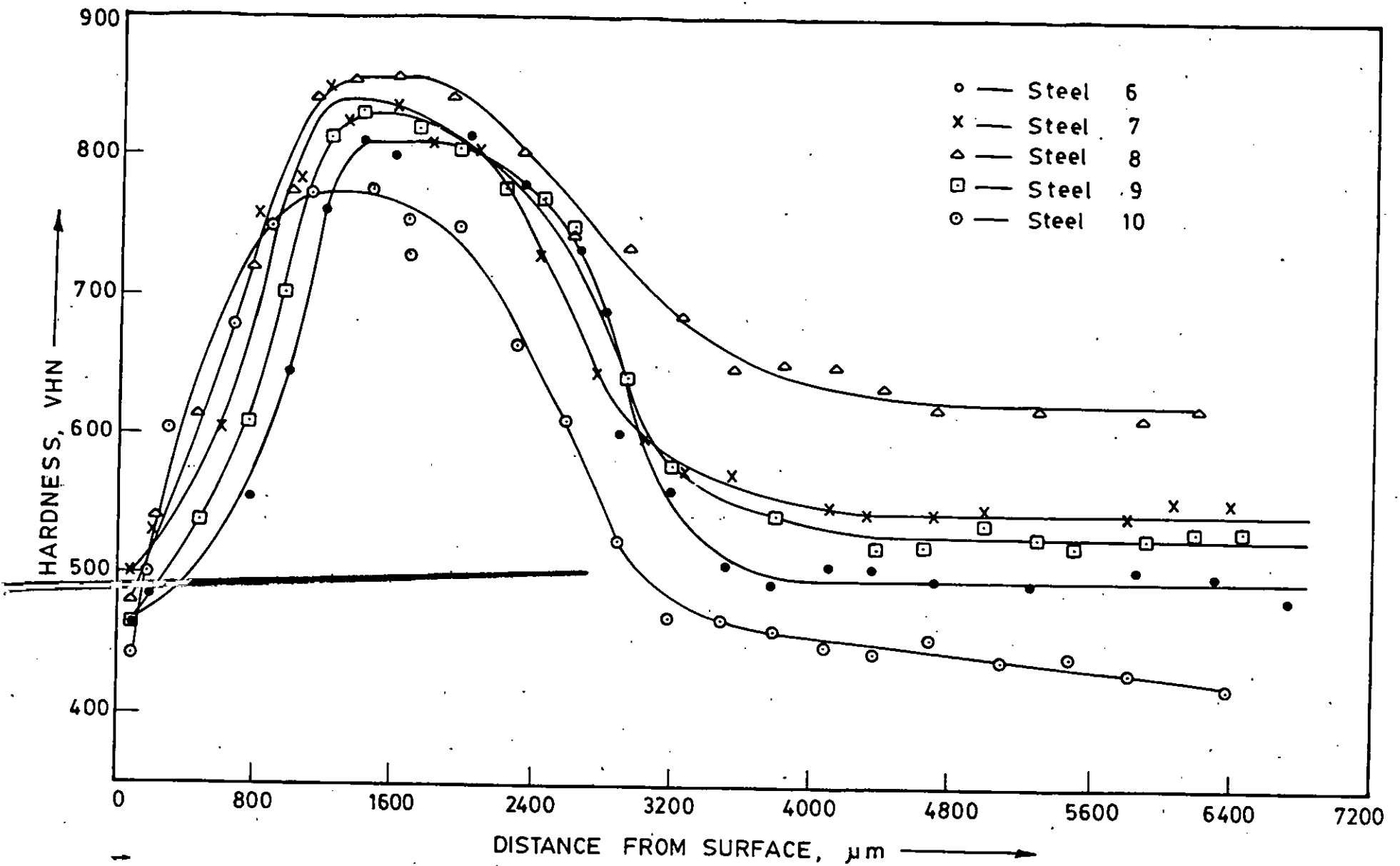


Figure 30: Microhardness profiles of steels 6 to 10 carburized at 950°C for 3 hours quenched in 10% brine and tempered at 160°C.

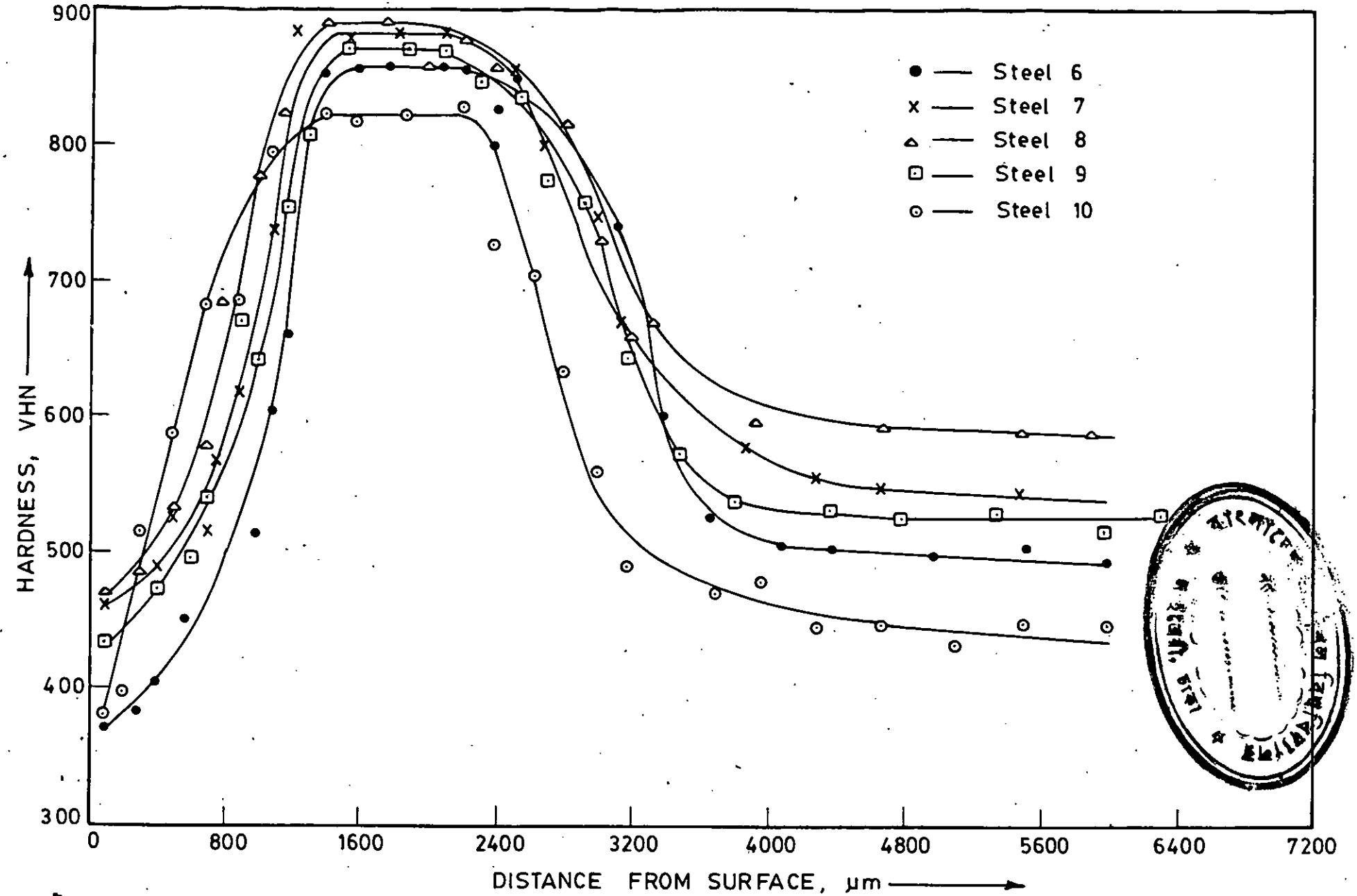


Figure 31: Microhardness profiles of steels 6 to 10 carburized at 950 °C for 5 hours quenched in 10% brine and tempered at 160 °C .