DEPARTMENT OF MATERIALS AND METALLURGICAL ENGINEERING
BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY
DHAKA-1000
BANGLADESH

EFFECTS OF INCLUSIONS ON THE QUALITY OF STEEL

A thesis submitted in partial fulfillment of the requirement for the degree of Master of Science in Materials and Metallurgical Engineering at Bangladesh University of Engineering and Technology.

ABUL HOSSAIN

DECLARATION

This is to certify that this work has been carried out by the author under the supervision of Professor A.S.W.Kurny, Department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka, Bangladesh and it has not been submitted elsewhere for the award of any degree or diploma.

Countersigned

Supervisor

Author

This thesis titled "EFFECTS OF INCLUSIONS ON THE QUALITY OF STEEL", submitted by ABUL HOSSAIN Roll No. 100611001F, Session: October 2006, has been accepted as satisfactory in partial fulfillment of the requirement for the degree of Master of Science in Engineering (Materials and Metallurgical Engineering) on 8 June, 2009.

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ACKNOWLEDGEMENT

The author expresses his wholehearted indebtness and gratitude to his respected thesis supervisor Professor A.S.W. Kurny, Department of Materials and Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka, who has inspired him to initiate this study. He is extending his full co-operation by guidance, constant advice, cordial encouragement and patronage in carrying out the research work as well as in preparing this thesis.

He expresses his cordial gratitude to all his respected teachers of Department of Materials and Metallurgical Engineering. He is particularly indebted to Prof. A. A. M. Rezaul Haque and Prof. Qumrul Ahsan to their useful suggestions and also for their help in the preparation of this thesis. He also wishes to thank the laboratory instructors and other assistants of this department.

The author wishes to record on his sincere thanks to **BASHUNDHARA GROUP** and **BASHUNDHARA STEEL COMPLEX LIMITED (BSCL)** for permitting him to undertake this study and to the honorable Executive Director of BSCL Engr.M.A.H. Serajee for his encouragement and support.

Finally he is grateful to all the members of his family particularly to his mother and wife for their understanding and constant encouragement throughout the period of his study.

Department of Materials and Metallurgical Engineering
BUET, Dhaka.

ABSTRACT

Mini steel of mills or plants in Bangladesh melt scraps in Induction Furnaces. Very little refining the liquid steels is possible in these induction furnaces. In many plants the molten steels are cast into what are usually known as pencil ingots. These ingots are cast in vertical moulds resulting in the formation of pipe at the top. Such steel plants that produce pencil ingots do not have refining (ladle refining) facilities. In absence of proper refining and quality control, these ingots usually contain slags, non-metallic inclusions and inhomogeneities and are usually of poor quality. As a result deformed bars produced from pencil ingot contain significant amount of slags and inclusions and show severe segregation effect and inhomogeneity in the microstructure. These defects affect the properties of the reinforcing bars.

Since these pencil ingots are produced in small lots and with little or no metallurgical control, the products obtained from different heats using apparently similar raw materials usually give different amount of inclusions and these give uncertain physical and mechanical properties. As a result the finished products of these pencil ingots are in general of inferior quality and give substandard reinforcing bars.

Many of the steel plants do not use ladle refining furnace (LRF) for refining the liquid steel or do not do the ladle refining of the melt properly. The billets as well as the reinforcing bars produced from such unrefined or improperly refined melts contain slags, inclusions and inhomogeneity in the microstructure. The deformed bars thus produced show inferior properties.

Proper refining in LRF of induction melted assorted scrap can give fairly clean and refined liquid steels. The billets and the reinforcing bars produced from such refined melts are generally free from inclusions and slags. Still better quality is obtained if such refined steel can be cast continuously in billet form.

Metallographic study of the reinforcing bars produced from properly refined continuously cast billet show uniform grain size, no heterogeneity in the microstructure, little or no slag and little inclusions and thus have better properties.

The following is summary of the results obtained through this investigation.

- ❖ Pencil ingots contain higher and larger sized inclusions. During hot rolling some of the inclusions (>40μm) are plastically deformed or fractured and act as a critical flaw size to cause failure.
- ❖ Reinforcing bars produced from such pencil ingots showed low yield strength, tensile strength, percentage of elongation and reduction in area and in general fail in bend tests.
- ❖ Billets cast without any refining give poor physical properties but sometimes satisfactory results.
- ❖ Billet processing through ladle refining furnace ensures smaller sized (<20 μm) and lower number of inclusions .The reinforcing bars from such properly refined continuously cast billets give superior mechanical properties such as yield strength, tensile strength, ductility, formability and micro-structural requirements.

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CHAPTER 1

Introduction

An Induction furnace is a melting unit. Very little, if any, refining takes place in an induction furnace. Thus the quality of steel produced in an induction furnace is directly related to the quality of scrap and other raw material. Any inconsistency in the input is directly reflected in the characteristic of the output. In spite of this, the main reason for adopting induction furnace for producing steel in India and consequently in Bangladesh is that the power requirement as well as production cost is lower in induction furnaces.

In recent years LRF are being used .Such furnaces allow limited removal of inclusions and have helped improve the quality of steel produced. In Bangladesh only limited number of steelmaking units use ladle refining furnaces for enhancing the quality of steel produced.

Except in steel foundry molten steel is poured into moulds to produce pencil ingots. The moulds are themselves massive castings of more uniform shapes with a cross-section like square, rectangular, round, polygonal, etc which are used according to the subsequent mechanical working operations that are to be carried out on the pencil ingots.

The growth and development of steel plants has coincided with the introduction and acceptance of continuous casting .Continuous casting has now become a reliable route for billets production with resultant high yields from charged materials to good billets. Liquid steel to billet yield is approximately 95% compared to 83% from the pencil ingot.

Ladle metallurgy is also introduced recently in some plants. Few of them have gone for inert (argon or nitrogen) gas purging in order to reduce inclusion content, gas content and make homogeneous composition and temperature in the ladle.

This research work is aimed at determining the extent of variation of inclusion contents with the liquid metal processing routes and the effects of inclusions on the properties of structural steel.

CHAPTER 2

Literature Review

2.1. Steelmaking in Global and Local Sector

The demand for steel goes hand in hand with economic growth. In the early stages of development of an economy, construction of buildings, bridges, roads and other infrastructures constitutes a key development activity. All these construction works require an enormous amount of steel.

World steel production has been on the rise. For the last few years, China, the fastest growing economy, has been the largest consumer as well as producer. Our neighbor India, the ninth largest steel producer, is also a dominant player in the steel sector. Except for India and Japan, Asia is the net importer of steel and so is our country (Bangladesh). It is found that semi finished products and hot rolled sheets and coil dominate among the export steel items.

2.1.1. Steelmaking- Global practice

The structure of iron and steel industry is rather complex involving many different sequential as well as parallel processing and fabrication routes. The broad spectrum of the iron and steel industry can be summarized as Fig.2.1.

Steelmaking can be defined as a process in which steel is produced in a liquid state and subsequently solidified into semi finished shapes. Commercial steelmaking plants can be broadly classified into two groups

i) Integrated steel plants and

ii) Mini steel plants.

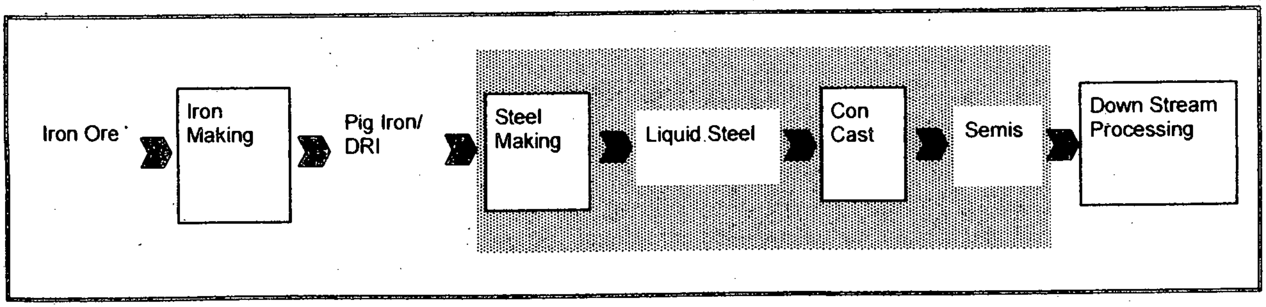


FIGURE. 2.1: The broad spectrum of the iron and steel industry.

In integrated steel plants iron ore is first possessed in a blast furnace to get what is known as pig iron (Fig.2.2). Pig iron is an unrefined form of iron containing a large amount of impurities like carbon, silicon, manganese, sulphur and phosphorous; total amount of these elements being about 8%. Liquid pig iron from the blast furnace is then refined to produce steel by reducing the impurity contents within acceptable limits in basic oxygen furnace (BOF).

In modern practice, liquid steel from BOF is further refined in a ladle furnace (LF). Liquid steel is then solidified into desired shapes in a continuous casting (Con Cast) machine into billets or slabs. Alternatively steel is cast in separate moulds known as ingots. Solid steel is then rolled into desired shapes and products, such as reinforcing rods, sheets etc. Integrated steel mills may sale the semi finished products to other downstream processors.

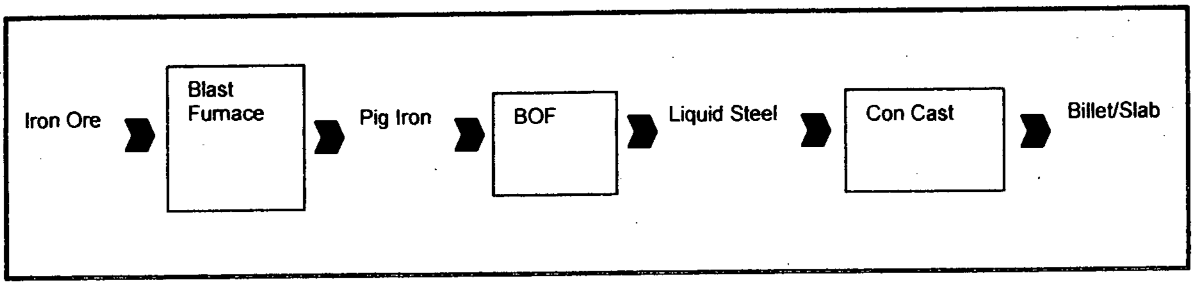


FIGURE. 2.2: Integrated steel mill practice.

In mini steel plants, steel scrap is used as main raw material (Fig 2.3). Steel scrap is melted and refined in electric arc furnace (EAF). Further refining of liquid steel from the ladle furnace steel goes to continuous casting machine. Nowadays an alternate raw material, direct reduced iron (DRI), is used in EAF that has replaced some of the scraps. DRI is produced by reducing iron ore in the solid state; on the other hand, pig iron is produced in a liquid state in a blast furnace. Earlier, mini steel plants used to produce steel in open hearth furnaces (Chittagong Steel Mills Ltd. used to have this type of furnaces). But open hearth furnace is nowadays almost obsolete because of longer production time, lower productivity and higher cost.

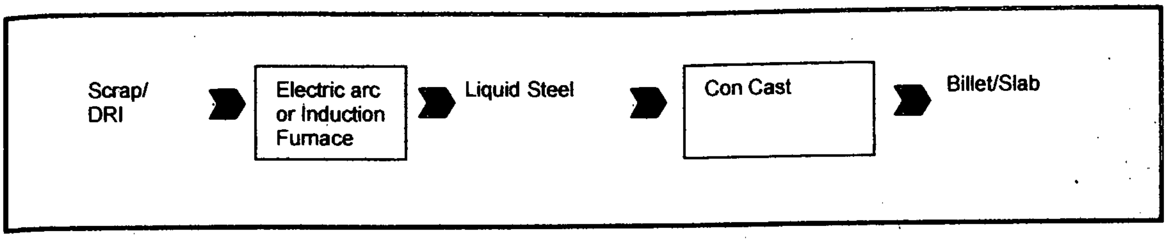


FIGURE 2.3: Mini steelmaking practice.

Integrated steel mills adopting BOF is still the dominant route of steelmaking. However electric steelmaking, requiring lower capital investment, is having increased share of steel production. In 2004, BOF and electric steelmaking respectively accounted for 63.0% and 33.8% of world steel production. By 2020, it is predicted that, electric steelmaking will account for about 50% of world steel production (Fig. 2.4).

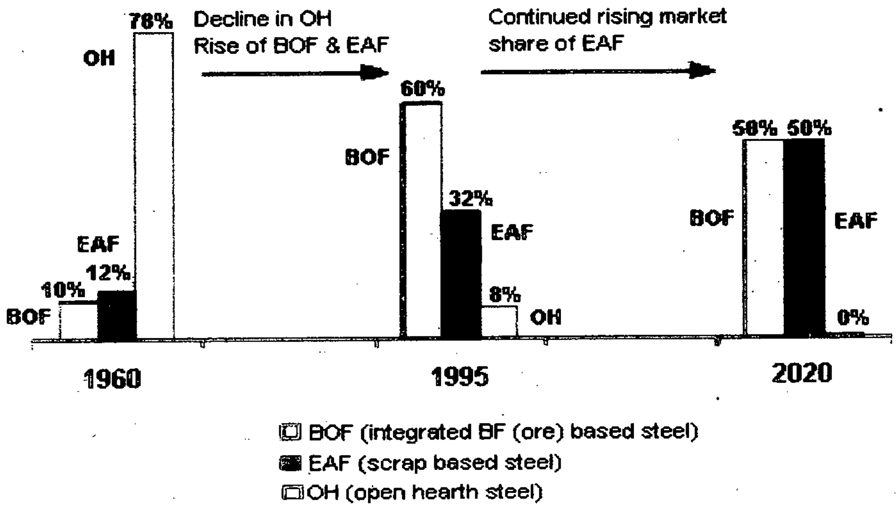


FIGURE 2.4: EAF is progressively having increased share in steelmaking.

2.1.2. Steelmaking in Bangladesh

The history of steelmaking in Bangladesh is nearly as old as the age of this country. Towards the end of 1960's, Chittagong Steel Mills Ltd., the only steelmaking plant in the area which now forms Bangladesh, started production using open hearth furnace. The mill was closed down in 1987. Around that time very small steelmaking units started to grow in the private sector. Initially, technology based on small Electric Arc Furnace (EAF) has been mainly imported from

India. These tiny EAFs could not survive long, and Induction Furnace (IF), also imported mainly from India and Italy, replaced them. New units were also established based on induction furnace. At present almost all steelmaking plants in Bangladesh use induction furnaces.

The steelmaking practice in Bangladesh is very peculiar and unique. This has to be viewed in the context of our proximity to India. India, possessing huge amount of iron ore, has a long history of steelmaking using integrated steel mill practice. In the last few decades, however, mini steelmaking practice has also been growing in India. Small sized steelmaking plants mainly based on induction furnace are operating. These induction furnaces based plants mainly cater to the lower segment of the rebar market.

As for casting, most of the local producers solidify their molten steel in channel or pencil ingots. Such kind of solidification practice is liable to cause defects and is not allowed in any standard specification for rebar. Acceptable casting processes for billets include continuous casting which is practiced by only a handful of local producers.

One of the most important products of steelmaking in Bangladesh is reinforcing bar or rebar. Flow sheets (Fig.2.5) to involve steel making where steel is obtained in a molten state which is subsequently solidified and rolled into rebar. As has been mentioned earlier none of these four processes can conform to ASTM or BS since IF is used for melting. However process 1 is expected to yield better quality products than that produced by the other processes. Some producers directly roll rebar from ship braking scrap without any melting. This process also does not conform to any standard specification.

Proper steel-making practice is yet to be adopted in the country. In spite of the present limitations of the local manufactures, one has to appreciate their dynamism in taking over the steel-making sector from the public sector. The private sector has shown its willingness to face the challenges of developing the country's steel sector. But its efforts need to be facilitated, guided and, wherever necessary, regulated in order to create a healthy steel sector for the benefit of the country. There have to be regulations as well as incentives for them to bring in modern and correct technology to be able to produce steel as per international standard specifications.

Ensuring the quality of steel as per international standard is very important for long-term performance and reliability of structures and installations. Failure of such structures in a natural calamity such as earthquake can lead to huge loss in terms of not only money but also of human lives. Consumers, both individuals and organizations, should also be aware of the implications of steel quality in construction work and should be willing to pay premium prices for quality.

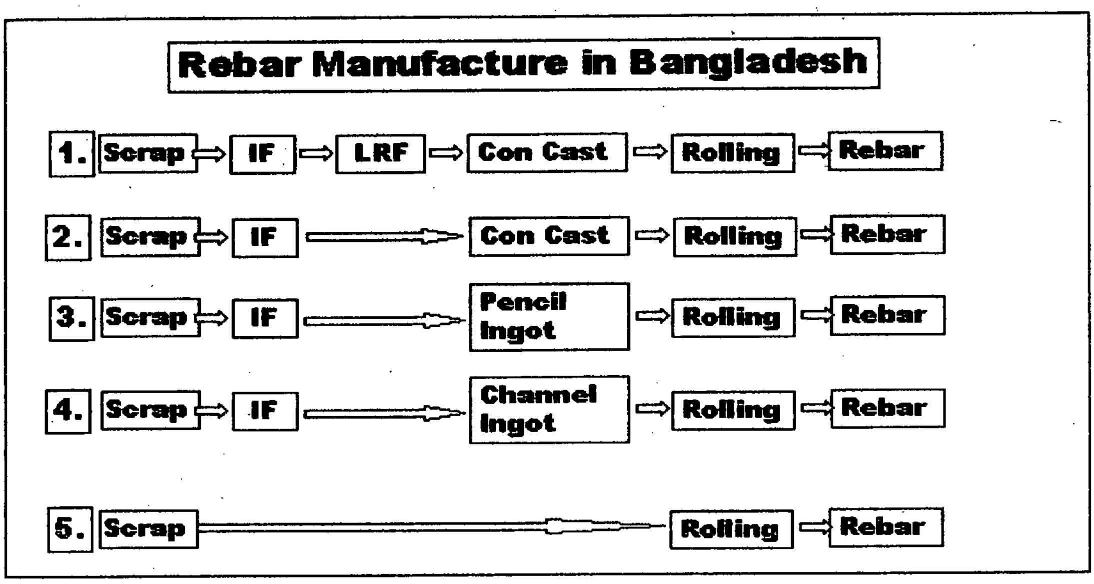


FIGURE 2.5: **Flow sheet of rebar (reinforcing bar) manufacture currently practices in Bangladesh.**

Training in steel making is hardly available in the country and this forces them to bring in foreign professionals and workers. In order to solve the problem, the government, industries and the universities have to sit together and devise a scheme to train local engineers for the steel sector on an urgent basis. Other important aspects of this sector are that it is highly energy-intensive and is based basically on imported raw materials. [3]

2.2. Raw materials and charging

Induction furnaces use the following input materials for the production of steel. The technology of melting these input materials varies according to the availability of raw materials and location of the plant. Quality of products made by the induction furnaces depends on the quality of input materials.

- Shredded steel melting scrap
- Heavy steel melting scrap -mill cut ends or old steel components
- Light steel melting scrap such as turnings/brooding or the steel sheets cutting
- Old cans and coated steel sheet cuttings
- Railway scrap
- Pig iron or cast Iron
- Direct reduced iron (both coal and gas based)
- Ship-breaking steel scrap

The induction furnaces use virtually 100 per cent steel scrap as raw material to produce new steel products. The most important implication of scrap recycling is the conservation of natural resources. A ton of scrap is at least equal, if not superior to a ton of pig iron. To make a ton of pig iron requires two tons of iron ore, coal, limestone and other raw materials. In addition, more transportation is involved in making a ton of pig iron than in generating a ton of scrap.

2.3. Steelmaking in the induction furnace

An induction furnace (Fig.2.6) is a refractory lined vessel that is surrounded by a water cooled copper coil. The charge to be melted is placed in the centre of the coil, which melts due to induced current. In order to insulate the power coil, from the melt charge, it is necessary to insulate the coil with refractory. The refractory used in induction furnace is monolithic rammed and sintered prior to melting heat. When alternating current is applied to the induction coil, it produces a magnetic field that in turn generates a current flow through the charge material, heating and melting it. The amount of energy absorbed depends on the power applied, electrical resistivity of the charge material, and the operating frequency.

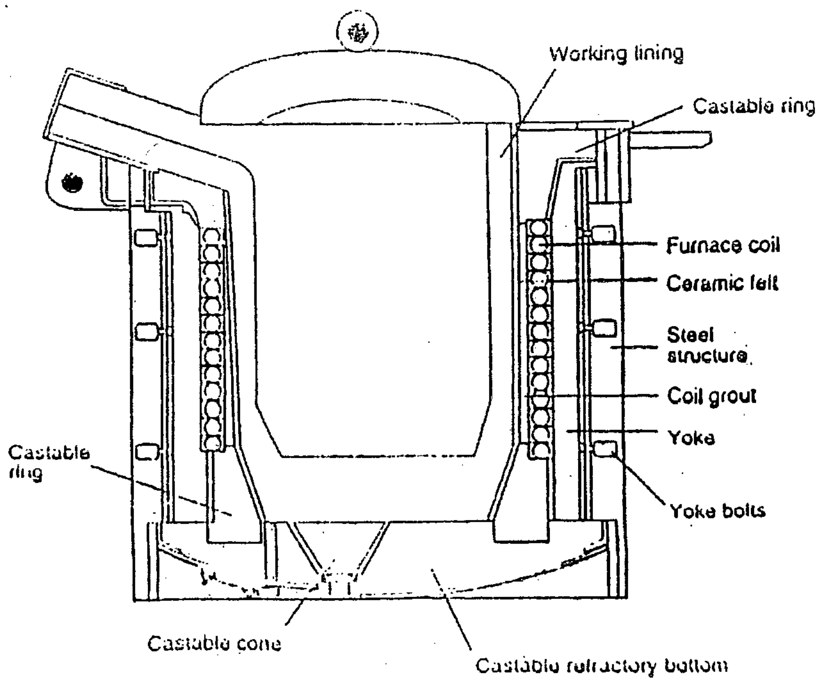


FIGURE.2.6: Cross-section of a Typical Induction Furnace.

Although the technology for melting steel inductively has been available for many years, the refractory systems capable of withstanding the high melting temperature requirements and the aggressive stirring action of the line frequency (60Hz) furnaces were not sufficiently developed. The advent of the solid-state, medium-frequency power supply and superior grades of refractory materials, have made the induction furnace a good option for the small scale steel producers.

There are three types of induction furnaces. [3]

Table.2.1.Types of induction furnaces:

Types of induction furnaces	Frequency range, Hz
A. Mains frequency	50-60
B. Medium frequency	200-500
C. High frequency	500-1000

2.4. Slags in steel making processes

2.4.1. Sources of slag

With an oxidizing atmosphere existing in a steel making plant, the process of steelmaking will inevitably involve the oxidation of iron and impurities. Further, the refractory lining of the plant is gradually eroded during the operation of the furnace. Certain process operations in the furnace are based on introducing various additions (fluxes). Most compounds formed in these processes are lighter than iron so that they float up to the metal surface and form a slag layer.

The main sources of slag in a furnace are as follows:

1. The products of oxidation of impurities introduced into the furnace together with pig iron and scrap: silicon, manganese, phosphorus, chromium, etc., i.e. oxides SiO_2, MnO, P_2O_5, Cr_2O_3, etc.
2. The products of erosion of the furnace lining. A silica lining is the source of SiO_2 in the slag.
3. Impurities (sand, dirt, mixer slag, etc.) brought in with the charge. Sand and clay consist mainly of SiO_2 and Al_2O_3, while mixer slag often contains much of sulphur (as MnS).
4. Rust of scrap, especially when light-weight scrap (trimmings, roof iron, etc.) is used, i.e. the oxides FeO, Fe_2O_3, $Fe(OH)_2$.
5. Additional materials and oxidizers (limestone, lime, bauxite, fluorspar, iron and manganese ores, etc.), which may contain $CaCO_3$, CaO, Al_2O_3, CaF_2, Fe_2O_3, Fe_3O_4, MnO_2, etc.

2.4.2. The role of slag

During melting and tapping, the molten metal is in continuous contact and interaction with the slag. The composition, temperature, fluidity and other parameters of the slag have a decisive effect on the course of a heat and the quality of final metal. The prime object of a steel maker is to remove harmful impurities (usually sulphur and phosphorus) from the metal. The problem is solved by removing these impurities to the slag and by forming conditions which prevent their passage back to metal. By varying the composition, temperature and certain properties of the slag, it is also possible to control the concentrations of Mn, Cr, Si and other impurities in the metal. For that reason, the steel maker has to form slag of the desired composition and proper thickness if he is aimed at producing high quality steel.

The slag should meet the following requirements:

(1) It should ensure the desired degree of refinement of the metal from harmful impurities

(2) During the oxidizing periods of a heat, slag must ensure intensive passage of oxygen from furnace atmosphere to metal

(3) During other periods of a heat and in the ladle upon tapping, slag should prevent the passage of oxygen from atmosphere to metal

(4) Slag must prevent the passage of gases (nitrogen and hydrogen) from atmosphere to metal

(5) The slag being skimmed off from the furnace should not contain much of iron so as not to Lower the degree of utilization of iron in the charge

(6) During melting in open-hearth or twin-bath furnaces, slag must conduct well the heat of flames to the metal

(7) During teeming, slag must prevent the cooling of the metal in the ladle

(8) In many cases, the composition of slag should meet certain special requirements, for instance, when the slag will be used as a building or road-construction material or as a fertilizer.

As may be seen, the requirements to be met by the slag are quite divers. Slag of the desired composition is formed by introducing a calculated quantity of additions (slag formers). When needed, a slagging-off procedure is carried out, i.e. a definite amount of slag (sometimes almost 100%) is removed from the furnace by skimming-off and a new slag of the desired composition and properties is formed by giving measured quantities of particular additions. In some cases, skimming off and slag removal may be carried out twice or even three times.

2.4.3. Slag forming process

Proper slag has a great bearing on the quality of the steel produced and the performance of the furnace. The claim that 'under a good slag the steel is good' is absolutely valid. Slag is formed in the melting down of the charging materials during which much of the silicon, manganese, phosphorus, chromium, and some iron of the metallic portion of the charge are oxidised. Scorification of the lining during melting and additives for refining also contributed to the formation of slag.

Slag is composed of basic, acid, and amphoteric oxides (an amphoteric oxide is one capable of reacting either as an acid or as a base) as well as of sulphides CaS, MnS, FeS. The basic oxides include CaO, MnO, MgO, FeO; the acid oxides are SiO_2, P_2O_5, TiO_2, V_2O_5, the amphoteric oxides are Al_2O_3, Cr_2O_3, Fe_2O_3.

The basic oxides of the slag- CaO, MnO, MgO, FeO, etc. combine with the acid and amphoteric oxides- SiO_2, P_2O_5, Al_2O_3, Fe_2O_3, etc. forming the corresponding chemical compounds: silicates, phosphates, aluminates, and ferrites. Thus, the acid oxides SiO_2 and P_2O_5 combine with the basic oxides CaO and FeO, forming the silicates Ca_2SiO_4 and $FeSiO_3$ and the phosphates $(CaO)_4P_2O_5$ and $(FeO)_3P_2O_5$. Consequently, slag is formed in the process of neutralizing the acid oxides with the basic ones, resulting in the formation of corresponding salts. At the high temperatures of the process silicates, phosphates, ferrites, and other chemical compounds of the slag tend to dissociate to some extent, i.e., decompose into the original oxides.

Basic Slag may also contain the basic oxides CaO, MnO, MgO, and FeO in free state. Oxides and other chemical compounds variously affect the melting temperature of basic slag. Such oxides as CaO, MnO, Cr_2O_3, and MgO raise, while SiO_2, P_2O_5 Fe_2O_3, and Al_2O_3 lower, the melting temperature of slag.

The main oxides in basic slag are CaO, SiO_2, FeO, and MnO. The basicity of slag is defined by the ratio $\frac{\%CaO}{\%SiO_2}$. This definition of basicity is valid only for slags having a small content of P_2O_5. If a pig iron high in phosphorus is charged into the furnace, the basicity of slag is determined from the ratio

$$\frac{\%CaO}{\%SiO_2 + \%P_2O_5}.$$

Viscosity, or fluidity, is one of the most important physical properties of molten slag. This is explained by the fact that the diffusion processes are liable to slow down markedly as the viscosity of slag increases. The slag must be sufficiently fluid to perform its functions satisfactorily.

2.5. Ladle refining

Secondary refining can be carried out in a simple ladle like furnace provided with bottom plug for argon purging and lid with electrodes to become an arc furnace for heating the bath. Another lid may be provided to connect it to vacuum line, if required. Chutes are provided for additions and an opening even for injection. In short it is capable of carrying out stirring, vacuum treatment, synthetic slag refining, plunging, injection etc. all in one unit without restraint of temperature loss, since it is capable of being heated independently. Every ladle furnace need not be equipped with all these arrangements.

As per the requirements of refining the ladle furnace may be provided with the necessary facilities. For example if gas content is no consideration, vacuum attachment may be eliminated. The principal component of the facilities is shown in Fig.2.7 schematically.

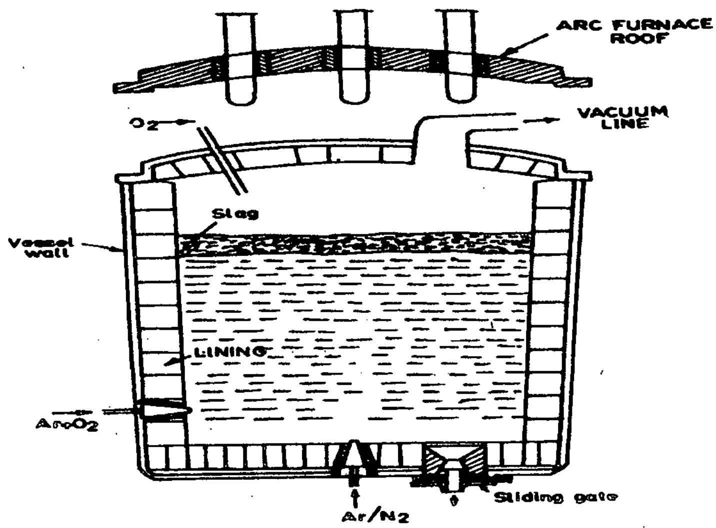


FIG.2.7. Principle components of a ladle furnace facility. [3]

In this chapter secondary refining of liquid steel by specially prepared slags in a ladle furnace will be discussed.

Stirring of the metal together with a specially prepared slag makes it possible to intensify the passage to slag of certain harmful impurities which should be removed to the slags phase (sulphur, phosphorus, or oxygen). The treatment of steel with the slags as a method for metal cleaning from unwanted impurities.

2.5.1. Inert gas blowing

When the metal is blown with inert gases, thousands of gas bubbles pierce it. Each bubble is essentially a small vacuum chamber, since the partial pressure of hydrogen and nitrogen in it are zero. During inert gas blowing, the metal is stirred intensely, and its composition is equalized all over the volume. When a good slag is formed on the metal surface, stirring promotes assimilation of non metallic inclusions by the slag. When steels especially low in carbon are to be made, the oxygen for bath blowing is diluted with an inert gas, which shifts the equilibrium of the reaction; $O_2 + 2C = 2CO_g$ to the right, as the gaseous phase of the reaction products will contain the inert gas in addition to carbon monoxide, and the partial pressure P_{CO} will be lower. Numerous bubbles of an inert gas facilitate the processes of gas evolution, since they are essentially cavities with extended interfacial area, which is important for the formation of a new phase. Since inert gas blowing lowers the temperature of the metal, it is often used for controlling metal temperature in the ladle. From the engineering standpoint, blowing of a large mass of metal by inert gas in the ladle is simpler and less expensive than vacuum treatment, so that the method is used instead of vacuum treatment where possible.

In the general case, inert gas blowing produces the following effects:

(1) Decreases the gas content of the metal;

(2) Agitates the melt vigorously, which facilitates the processes of removal of non metallic inclusions to slag and equalizes the metal composition;

(3) Facilitates the occurrence of the carbon oxidation reaction;

(4) Lowers the temperature of the metal.

Blowing with nitrogen is often employed when the metal contains no nitride forming elements (chromium, titanium, vanadium, etc.). In the temperature range from 1500^0C to 1600^0C, nitrogen in molten iron dissolves poorly.

Metal in the ladle can be blown with inert gas through a false stopper, through a tuyere built in the ladle wall, through porous refractory inserts arranged in the ladle bottom shown in Fig.2.8.

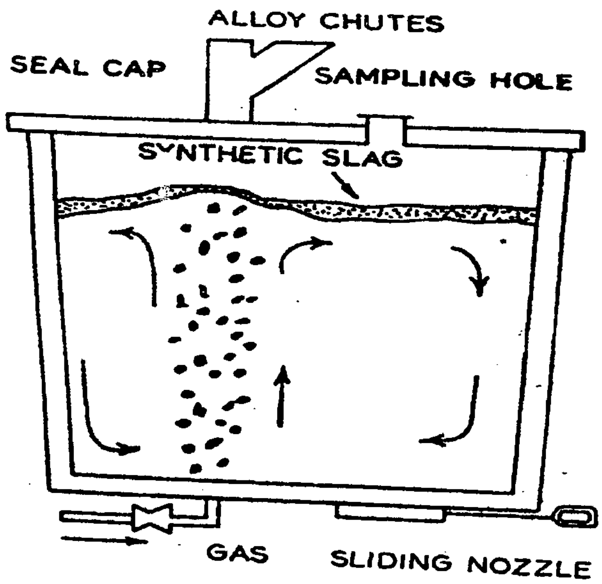


Fig: 2.8. Nitrogen blowing in ladle refining furnace. [27]

The consumption of inert gas is usually 0.5-2.5 m^3/ton steel. With the flow rate of gas (argon) within this range, the rate of temperature reduction in the ladle is 2.5-4.5%^{0}C/min, depending on the mass of molten steel (without blowing, the metal in the ladle is cooled at a rate of 0.5-1.0^{0}C/min). During blowing heat is additionally spent for heating the inert gas and radiation from the intensively mixed surfaces of metal and slag. The major portion of heat losses is associated exactly with the increased thermal radiation. These losses can be decreased noticeably by closing the ladle with a lid during blowing, which at the same time diminishes the degree of oxidation of the metal which is exposed during blowing.

A simple and reliable method of gas blowing is by using what is called a false stopper.

Blowing arrangements of the type of false stopper are safe in operation, since no changes are needed in the ladle lining. They have, however, an essential drawback: false stoppers (as common ones) withstand only one heat. The refractories of a stopper are quickly eroded by the metal gas suspension that moves along the stopper.

Another method is also popular: The gas is blown through porous refractory plugs provided in the ladle bottom. Blowing is done through a number of plugs simultaneously, so that the gas produces a stronger effect on the metal. Porous refractory plugs can withstand several blow operations. Along with a high gas permeability they must have a high refractoriness to withstand temperatures of 1550-1650°C, an ample thermal stability, and chemical resistance to metal and slag.

The method of gas blowing through the porous bottom of the ladle (Fig.2.9) has also become popular. Blowing with the gas flow rate up to 0.5 m^3/t steel is quite sufficient for equalizing the composition and temperature of the metal; at rates of up to 1.0 m^3/t, blowing can have a positive effect on purification of the metal from non-metallic inclusions; for sensible results in degassing, the flow rate of an inert gas should be not less than 2-3 m^3/t metal. Inert gas blowing is usually done with properly deoxidized metal. If the metal is not completely deoxidized before blowing, carbon oxidation and reduction of non-metallic oxide inclusions can occur in addition to the processes described above.

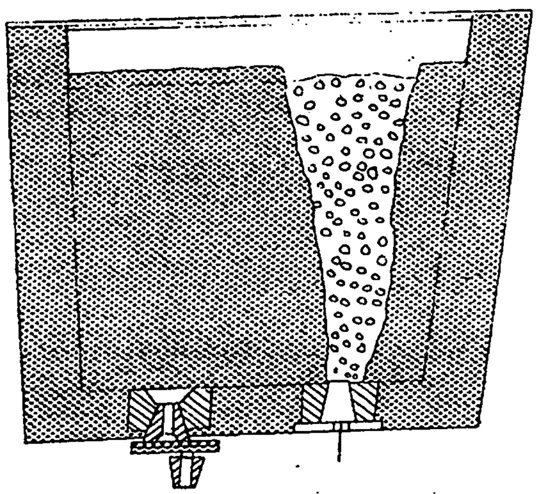


Fig: 2.9. Nitrogen purging through a porous plug.

Sometimes, inert gas blowing is combined with vacuum treatment of the metal. In that case, the consumption of inert gas can be reduced substantially. By combining inert gas blowing with synthetic slag treatment it is possible to increase substantially the effectiveness of application of slag mixtures, since strong stirring of the metal by blown gas increases the time of contact between the metal and slag and the contact surface area. If the ladle in which the combined treatment is carried out is closed by a lid, the inert gas atmosphere in the space between the lid and slag surface will prevent oxidation of the metal, while the lid decreases the heat losses and thus makes it possible to increase the time of contact of the metal and slag.

When the metal has to be stirred under the slag for a long time, the bath is heated up by means of electrodes introduced through holes in the lid. Thus a new plant is obtained, which resembles both a ladle and a furnace. In such cases, common fireclay cannot be used in the lining of the ladle, since it is destroyed quickly due to contact with free running high basic slag. Common stoppers are also of no use, since fireclays rings of which they are assembled are eroded quickly by the slag. The ladle must be lined with high refractory materials. The effect of inert gas blowing of oxidation has been made use of in developing the process of argon oxygen decarburization

2.5.2. Nitrogen in steelmaking

Nitrogen present in steel products has two main sources: the nitrogen that is present in the feed materials, such as scrap; and the nitrogen that is absorbed from the atmosphere during the steelmaking process. Table: 2.2 shows the amount of nitrogen present in each of the feed materials. [6].

Table: 2.2 Nitrogen content of feed materials [6-11]

Feed Material	**Nitrogen Content**
Scrap	30-120 ppm
HBI/DRI	20-30 ppm
Cold pig iron (CPI)	20-30 ppm
Coke	5000-10000 ppm
Oxygen	30-200 ppm
Carbon Carrier Gas (Air)	78%
Bottom stirring gas (N_2)	>99.9%
Bottom stirring gas (Ar)	<30 ppm
CaO	400 ppm

While steel is liquid the nitrogen present exists in solution. However, solidification of steel may result in three nitrogen-related phenomena: formation of blowholes; precipitation of one or more nitride compounds; and/or the solidification of nitrogen in interstitial solid solution.

The maximum solubility of nitrogen in liquid iron is approximately 450 ppm, and less than 10 ppm at ambient temperature [12]. Further, the presence of significant quantities of other elements in steelmaking to avoid liquid iron affects the solubility of nitrogen. More importantly, the presence of dissolved sulphur and oxygen limit the absorption of nitrogen because they are surface-active elements. This is exploited during excessive nitrogen pickup, particularly during tapping.
The soluble component of nitrogen is commonly referred to as 'free nitrogen'. Like carbon, nitrogen atoms are significantly smaller than iron atoms and exist within the interstices in the atomic structure. This atomic arrangement is known as an interstitial solid solution [13].

The effect of nitrogen on steel properties can be either detrimental or beneficial, depending on the other alloying elements present, the form and quantity of nitrogen present, and the required behaviour of the particular steel product. In general, however, most steel products require that nitrogen be kept to a minimum. High nitrogen content may result in inconsistent mechanical properties in hot-rolled products, embrittlement of the heat affected zone (HAZ) of welded steels, and poor cold formability. In particular, nitrogen can result in strain ageing and reduced ductility of cold-rolled and annealed LCAK steels. [14]

2.6. Non- metallic inclusions in steel

2.6.1. Introduction

Non-metallic inclusions, which are undesirable components of all steels, play an important role with respect to their effect on the steel properties. Controlling inclusions in steel is closely connected with the concept of "clean steel". The improvement in steel properties by control of non-metallic inclusions plays an important part in defending the applications of steel against newer competitive materials.

Generally, non-metallic inclusions in steel normally have a negative contribution to the mechanical properties of steel, since they can initiate ductile and brittle facture. Among various types of nonmetallic inclusions, oxide and sulphide inclusions have been thought to be harmful for common steels.

All steels contain non-metallic inclusions to a greater or lesser extent. The type and appearance of these non-metallic inclusions depends on factors such as grade of steel, melting process, secondary metallurgy treatments and casting of steel. Because of this, it is of particular significance to determine how pure the steel is. The term steel cleanness is relative one, since even steel with only 1 ppm each of oxygen and sulfide will still contains 10^9 -10^{12} non-metallic inclusions per ton. From the viewpoint of "cleanness" all steels are "dirty".

Non metallic inclusions in steel are the cause for dangerous and serious material defects such as brittleness and a vide variety of crack formations. However, some of these inclusions can also have a beneficial effect on steels properties by nucleating acicular ferrite during the austenite to ferrite phase transformation especially in low carbon steels. According to definition, the non-metallic inclusions are chemical compounds of metal with nonmetal which are present in steel and alloys like separated parts. [28]

2.6.2. Classification of non-metallic inclusions

Compounds of metals (iron, manganese, silicon) with non-metals (oxygen, sulphur, nitrogen, hydrogen, phosphorus), which may be present in steel, ate termed non-metallic inclusions. According to their composition, non-metallic inclusions are usually classified as follows:

(1) Sulphides (FeS, Al_2S_3 , ZrS_2, etc.);
(2) Oxides (FeO, MnO, SiO_2 , Al_2O_3, (FeO)2 SiO_2, etc.);
(3) Phosphates (Fe_3P, Fe_2P, etc.);
(4) Nitrides (NiN, ZrN, VN, BN, Si_3N_4, etc.):
(5) Carbides (Fe_3C, Mn_3C, Co_3C_2, W_3C, WC, etc.) and
(6) Complex inclusions: silicates, oxysulphides, carbonitride, etc.; the bulk of inclusions in steel very often relate to this particular group.

In practice, the term non-metallic inclusion usually implies oxides, sulphides and oxysulphides. An analysis of other types of inclusions is done in particular cases, such as in the manufacture of special steels. Since gaseous inclusions in steel also belong to non-metallic ones, inclusions of the type of oxides or sulphide are sometimes called slag ones. According to their size, inclusions are divided into two groups:

(1) Micro inclusions which can be detected in micro sections only under the microscope at magnifications not less than x 50 (magnifications of x 100 to x 600 are common in use, while magnifications up to x 1000 or even x 1500 are used in special cases.)
(2) Macro inclusions which can be seen in a fracture or on the surface of metal (polished surface is preferable) by the naked eye or at a low magnification e.g. magnifying glass.

In general, inclusions are subdivided into macro- (>20 μm), micro- (1-20 μm), and submicro-inclusions (<1 μm). As a result, multiple analysis methods would have been conducted to examine the different inclusion size ranges. Figure 2.10 illustrates general methods currently in use for examining steel inclusions based on their applicable size ranges overlaid with a typical inclusion frequency distribution. [29]

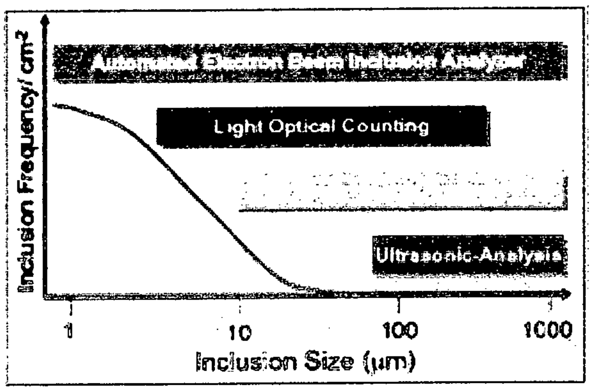
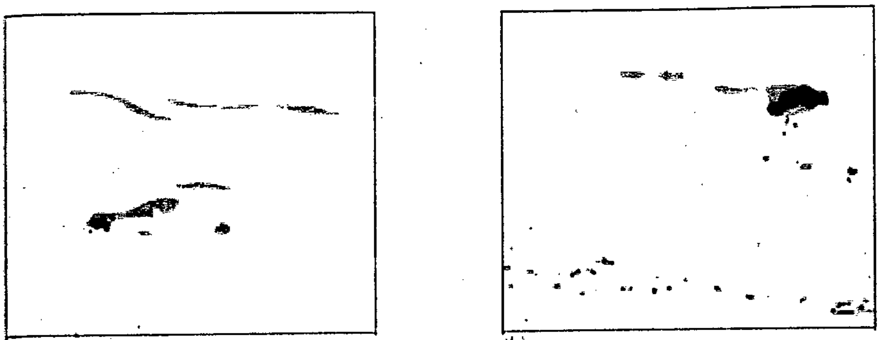


Fig.2.10.Comparison of applicable size ranges for various inclusion analysis tools.

The majority of inclusions in steels are oxides and sulphides. Among various types of nonmetallic inclusions, oxide and sulphide inclusions have been thought harmful for common steels. Usually, nitrides are present in special steels (stainless steels, tool steels) which have elements with a strong affinity for nitrogen (e.g. chrome, vanadium), which create nitrides.

Figure 2.11 shows sulfides and oxides of non metallic inclusion in steel.



(a) (b)

Fig2.11: Non-metallic inclusion in steel: oxides-dark gray and sulfides-light gray. [28]

By mineralogical content oxygen inclusions are divided into the following groups:

- Free oxides – FeO, MnO, Cr_2O_3, SiO_2 (quartz), Al_2O_3 (corundum) etc.
- Spinels-compound oxides which are formed by bi- and tri-valent elements as a ferrites, chromites and aluminates.
- Silicates which are presented in steel like a glass formed with pure SiO_2 or SiO_2 with admixture of iron, manganese, chromium, aluminum and tungsten oxides and also crystalline silicates.

Depending on the melting temperature, in liquid steel non-metallic inclusions are in solid or liquid condition.

As mentioned above the majority of inclusions in steels are oxides and sulfides. Sulfides in steel have been paid much attention because their treatment is an important problem in the steelmaking process. They affect on the properties of the final products by their deformation during the steel working process; especially their morphology has a significant effect on the steel properties. According to analysis based on the steel ingots containing 0.01-0.15% S, the morphology of MnS can be classified into three types:

1) Type I is a globular.MnS with a wide range of sizes, and is often duplex with oxides.
2) Type II has a dendritic structure and is often called grain-boundary sulfide because it is distributed as chain-like formation or thin precipitates in primary ingot grain boundaries.
3) Type III is angular sulfide and always forms as mono phase inclusion.

Most of the above mentioned sulfides are formed both during the process of secondary metallurgy or the solidification process. Recently, with the development of steelmaking technology, the sulfur concentration in steel was lowered drastically. Also, the continuous casting technology of steels with higher cooling rate than the ingot casting almost replaced the ingot casting.

So, the sulfides in the modern commercial steel are usually formed on solidification process or in solid steel during the subsequent cooling process. For example, the Widmanstätten plate-like MnS_2, is formed in solid steel and Figure 2.12 shows the common morphology of MnS in conventional continuously casting steel, including the globular duplex oxide–sulfide (particle A, B and C) and the Widmanstätten plate-like MnS (particle D). [28]

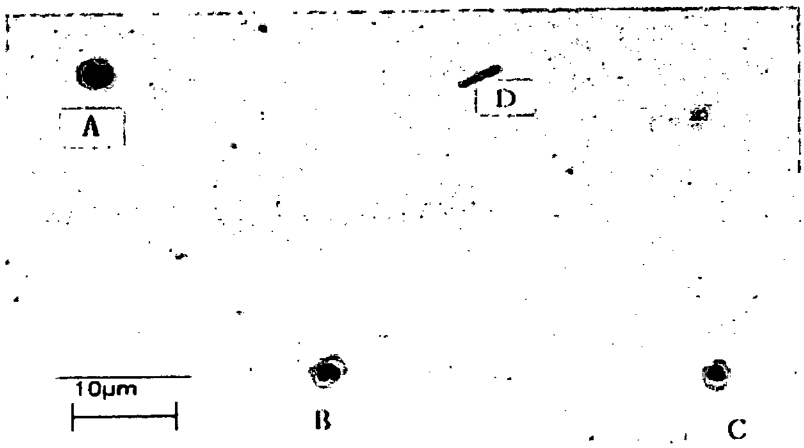


Figure 2.12: Typical duplex oxide–sulfide inclusion (particle A, B and C) and plate-like MnS (particle D) in conventional continuous casting silicon steel. [28]

2.6.3. The principal sources of non-metallic inclusions

The principal sources of non-metallic inclusions in steel are as follows:

(1) Inclusions present in the charge materials : pig iron and scrap;

(2) Inclusions passed to the metal from the lining of the furnace, launder, ladles, etc. due to the washing off of the lining by molten metal; and

(3) Inclusions formed during the steelmaking process: by interaction between metal and slag (for instance, due to passage of oxygen or sulphur from slag to metal); on addition of deoxidants or alloying elements; by precipitation from solidifying steel (due to a decrease in solubility or temperature).

Experience shows that the last group is the principal source of inclusions in steel. Inclusions of the second group occur in steel in noticeable quantities only in the rare cases of a poor state or improper maintenance of the lining of the furnace, launder, ladles, etc.; they can be easily distinguished under the microscope by their appearance and relatively large size. Inclusions of the first group (brought in with the charge) are usually present in small quantity and removed easily at early stages of the heat.

Non-metallic inclusions which form by reactions occurring in the liquid or solidifying metal are called natural or endogenous inclusions; occasional or foreign inclusions are termed exogenous. Strict observance of the specified steelmaking technology and proper maintenance and control of the lining are essential for making the metal with the least concentration of inclusions, controlling the composition of inclusions to a desired level and the removal of most inclusions to slag. Steels usually contain 0.01 – 0.02% (by mass) of inclusions, their concentration and composition depending on the type of steelmaking process, the quality of charge materials, and process technology.

2.6.4. Formation of inclusions (Thermodynamic principle)

The processes of formation of inclusions by reactions between the dissolved components of steel and the precipitation of inclusions due to a decrease in the solubility of these elements in iron on decreasing temperature (during steel solidification in moulds) involve the formation of a new phase in the original liquid phase.

As is known, the formation of a new phase is determined by thermodynamics (the principal possibility for a process to occur) and kinetics (the intensity and rate of the process). The latter depends mainly on the intensity with which the nuclei of the new phase form. The process still has not been analyzed thoroughly, since it is not clear how the micro inhomogeneities present in the melt can influence the nucleation of a new phase. It is an established fact, however, that the intensity of nucleation of a new phase is higher at a lower interfacial tension on metal-inclusion boundaries, $\sigma_{Me\text{-}inc}$ and a higher degree of super saturation. Oxide inclusions of the type of FeO & MnO have a very low value of $\sigma_{Me\text{-}inc}$ (around 0.18 j/m^2) and can precipitate even at a very low super saturation. Precipitation of inclusion is facilitated substantially in cases when interfaces are already present in the melt. In many cases, some non-metallic inclusions precipitate on the surface of other, less fusible inclusions existing in the melt.

Non-metallic inclusions (either formed in the melt or entered there to by another way) can collide with one another. The number of collisions depends, in the first place, on the intensity of metal stirring (natural or forced). Very fine inclusions (less than 10^{-4} cm in size) move randomly in the melt (Brownian motion). Upon collision, non-metallic inclusions either combines completely (coalesce) or form larger conglomerates (coagulate).

2.6.5. Removal of inclusions (Thermodynamic principle)

The driving forces of coalescence of inclusions are interfacial tension forces. The interfacial tension at metal inclusion boundaries, $\sigma_{Me\text{-}inc}$ is substantially higher than that at boundaries of colliding inclusions,

$\sigma_{inc\text{-}inc}$ wetting between inclusions is much better than that between an inclusion and the metal. The forces of cohesion between inclusions are higher than the force of adhesion of inclusions to the metal. The magnitude of $\sigma_{Me\text{-}inc}$ is different for various inclusions (and various compositions of molten metal). At a higher $\sigma_{Me\text{-}inc}$ inclusions coalesce more readily. On the other hand, coalescence of inclusions can occur more easily at a lower $\sigma_{inc\text{-}inc}$.

At the moment of an occasional contact between two inclusions, a thin metallic interlayer remains for a while between them; the thickness of the interlayer may be so small that it will hold both inclusions together by adhesion forces. Complete coalescence of the two inclusions may occur later.

Table.2.3.Melting points and densities of non metallic inclusions:

Compositions of inclusions	Melting point °C	Density at 20 °C, g/cm^3
Ferrous oxide FeO	1369	5.8
Manganous oxide MnO	1785	5.5
Silica (quartz, quartz glass)SiO_2	1710	2.2-2.6
Alumina(corundum) Al_2O_3	2050	4.0
Crome oxide Cr_2O_3	2280	5.0
Titanium oxide TiO_2	1825	4.2
Zirconium oxide ZrO_2	2700	5.75
Iron silicate (fayalite)$(FeO)_2SiO_2$	1205	4.35
Iron silicates with less than 40% SiO_2	1180-1380	4.0-5.8
Iron silicates with more than 40% SiO_2	1380-1580	2.3-4.0
Manganese silicates containing 15-50% SiO_2	1210-1600	4.0-5.0
Aluminu silicate with less than 10% Al_2O_3	1545	3.05
Iron sulphide FeS	1193	4.6
Manganese sulphide MnS	1620	4.04
Magnesia MgO	2800	3.58

The processes of contact and coalescence of inclusions depend substantially on the viscosity of the melt and the intensity of stirring. It may be taken approximately that the rate of coalescence of inclusions is directly proportional to the interfacial tension $\sigma_{Me\text{-}inc}$ and inversely proportional to viscosity η. With a very high intensity of stirring and accordingly high rates of motion of metal layers, bonds between coalesced inclusions may be broken and these will break off from one another. Experience shows that the desired coalescence of inclusions is obtained at a particular optimal intensity of metal stirring.

Non-metallic inclusions are lighter than the metal (Table. 2.3) and should float up under the action of gravitational forces. Assuming that their shape is spherical and neglecting the motion of the liquid proper, the rate of rise of inclusions, v, can be found by the well-known Stokes formula;

$V = (2/9)\, g r^2 (p_{Me} - p_{inc}) / \eta$

Where p_{Me} and p_{inc} is the density of the metal and inclusions, r is the radius of inclusion; and η is the viscosity of the metal.

Stokes formula has been derived on the assumption that the lifting force, which is determined by the density difference and the size (radius) of inclusions, is opposed only by the viscosity of the liquid. Therefore, the rising speed of inclusions should be higher at a lower viscosity and larger size of inclusions. In practice, however, cases are frequent when the metal is stirred quite intensely. With small sizes of inclusions, adhesion forces are of importance. Fine inclusions can be entrained by metal currents and thus will soar for a long time in the melt.

With a lower wettability (higher value of $\sigma_{Me\text{-}inc}$), an inclusion can be retained in contact with the metal by lower forces and therefore, can break off more easily and float up in the metal. On the contrary, inclusions which are wetted readily by the metal cannot break off from it as easily.

Alumina inclusion ($\sigma_{Me\text{-}inc}$= 1 J/m^2) are an example of poorly wettable inclusions; on the other hand, iron silicates ($\sigma_{Me\text{-}inc}$= 0.4J/m^2) are an example of readily wettable inclusions which stick firmly to the metal. The former are sometimes called ferrophobic and the latter ferrophilic.

Gas bubbles passing through the bath (in bath boil, during inert-gas blowing. etc.) are favorable for flotation of inclusions. The surface tension on the inclusion, gas interfaces, $\sigma_{inc\text{-}g}$ is lower than the force of adhesion of inclusions to the metal, $\sigma_{Me\text{-}inc}$ and therefore, inclusions stick to gas bubbles and are thus carried off to slag.

Too vigorous stirring of the metal can cause disintegration or destruction of earlier formed inclusion conglomerates and thus impair the process of their removal. Besides, stirred metal can entrain slag particles, and therefore, the concentration of exogenous inclusions in the metal. In practice, an optimal intensity of metal stirring can accelerate erosion of refractories and thus increase the concentration of exogenous inclusions in the metal. In practice, an optimal intensity of metal stirring would be found in each particular case to ensure floating up of inclusions in the metal.

It is essential to carry out the process so that rising inclusions could be assimilated by the slag, but not entrained back by descending metal currents. The rate of capture of inclusions by slag depends on many factors; in particular on the interfacial tension at slag-inclusion boundaries $\sigma_{sl\text{-}inc}$. The process of assimilation of inclusions by slag occurs more easily at a lower interfacial tension, i.e. with a better wettability of inclusions by slag. Thus, with higher $\sigma_{Me\text{-}inc}$ inclusions can break off more easily from the metal and with lower $\sigma_{sl\text{-}inc}$, they can be more easily assimilated by slag. The rate of removal of inclusions from metal to slag depends additionally on the area of contact surface (the ratio of slag-metal interfaces to the mass of metal), intensity of bath stirring, physical properties of slag, etc.

Thus, the factors that determine the rate of removal of inclusions from the metal include:

(1) The size, composition (melting point) and density of inclusions;

(2) The coalescing capacity of inclusions;

(3) The interfacial tension on metal-inclusion and slag-inclusion boundaries;

(4) The intensity of bath stirring; and

(5) The physical characteristics of metal and slag (temperature, viscosity, etc.).

The general principles outlined above can be used in particular practical cases to determine the ways for decreasing the content of inclusions in metal and methods for transferring them into a state where their harmful effects on metal properties will be minimized.

2.6.6. Inclusions effect on mechanical properties

The effect of non-metallic inclusions on steel properties show the importance of the behavior of the inclusions as well as of surrounding metal matrix during plastic working of steels. The aims of the metallurgist are to eliminate undesirable inclusions and control the nature and distribution of the remainder to optimize the properties of the final product.

An attempt by using program ABACUS was performed to model the behavior of slag inclusions and their surrounding matrix material during hot rolling and hot forging of hardenable steels. It is shown that it can be helpful for studying the behavior of inclusions, which is difficult or even impossible to obtain from a conventional experiment.

Figure 2.13 shows the effective strain contour during plastic deformation. Three regions of strain concentration (red) can be seen and a trihedral void (white region) close to the round inclusion is formed. The strain concentrations arise at the inner surface of the matrix. Another interested thing is that two edges of the pore tend to emerge and a bonding is formed. The difference in mechanical properties between the matrix and the inclusion is found to be the primary reason to create a void. The weak bonding at the interface between the matrix and the inclusion seems to facilitate to open the void. [28]

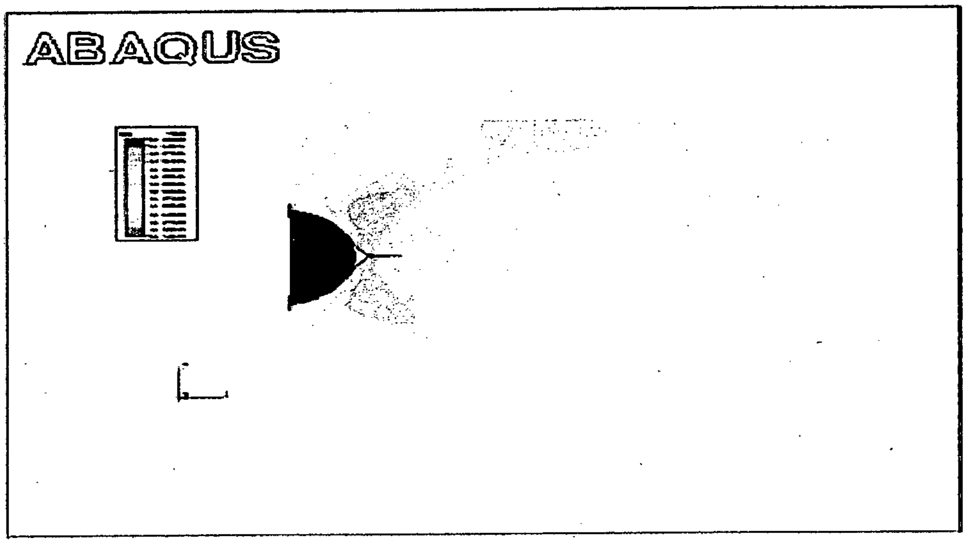


Figure 2.13: Void formation close to the inclusion. [28]

The effects of inclusions on ductility and toughness are well documented, and it is clear that the inclusions play a vital role in the mechanism of ductile fracture. The involvement of inclusions and other second phase particles in the ductile fracture mechanism extends the range of importance of inclusions to properties other than ductility, as both C.O.D. Testing and, in certain cases, plane strain fracture toughness testing, can involve the ductile fracture mechanism. The importance of inclusions in these respects has been demonstrated in previous papers.

A further aspect of inclusions is the effects they may have on fatigue properties, particularly on the fatigue life in rolling contact, where it has been shown that the effects of the various inclusions can be correlated with the tessellated stresses developed due to differential contraction during cooling. Also, the nature of the oxide inclusions in free-cutting steels has been shown to have important effects on the abrasive wear of the cutting tools in addition to the effects of volume fraction of sulphides. The effects of inclusions on fatigue and on machinability are modified by control of the nature of the inclusions, but the evidence to date on inclusion effects on ductility has not revealed such differences between inclusions other than those attributable to differences in shape. Thus any assessment of inclusions for fatigue or machinability would involve identification of the inclusions, whereas for ductility studies, detailed descriptions of the particle distribution and shape are required. It is of interest to consider the important morphological features of inclusion distributions in relation to ductility and toughness. [15]

2.6.7. Effects of inclusions (second phase particles) on tensile strength

Second phase particles have important effects on strength, toughness and ductility. The effects of finely dispersed particles on strength have been studied intensively over a long period of time and alloys relying on these strengthening mechanisms are widely used. The dispersion – strengthened alloys commonly suffer some embitterment because of the increased strength. Second phase particles can have other effects on toughness however. The presence of large filamental carbides can rise the impact transition temperature and the presence of non- metallic inclusions can influence the fully ductile impact energy. The presence of second phase particles can also cause major changes in ductility and although the mechanistic effects are well established, the quantitive relationships between ductility and microstructure are elusive.

Deformable non-deformable particle (inclusion) transition is fundamental to the whole study of the effect of second phase particles on the strength of alloys. For deformable particle there is, as yet, no single equation which envelopes all contributions to the strength, so that for accurate work, it is first necessary to established the mechanism of strengthening. Most mechanisms in which the major strengthening effect can be attributed to some irreversible energy change, ΔE, occurring as the particles are cut by a dislocation e.g. 'chemical' or 'order' strengthening[16,17] and 'Stacking fault strengthening'[18] show a variation of incremental flaw stress, σ_c over the flaw stress of the solid solution given by

$\sigma_c = K_1 r^{afb} (\Delta E)^c$

Where,

K = material constant

r = particle size (inclusion diameter)

f = volume fraction

a, b and c are numbers

Typically $0<a<1/2$, $0<b<2/3$ and $1<c<3/2$. Thus the strength of this type of alloy is weakly dependent on the volume fraction of second phase but strongly influenced by the detailed interaction between the dislocation and the particle (inclusion). [16]

The influence of the inclusions on ductile fracture is greatest for large inclusions and at low carbon contents. It is unfortunate that so many detailed investigations have been confined to the rolling direction. Curiously, for rolled bars of pearlitic steels, Gladman et al. [19] found that the effects of sulphides, oxides and cementite on ductility were similar when judged on a volume fraction basis. This arises because pearlite, like oxide and sulphide inclusions, can give rise to significant cavity formation at an early stage of straining. [20]

2.6.8. Inclusions and Ductility

Earlier studies on ductility have shown that the volume fraction and shape of second phase particles are important in controlling ductility whilst particle size (and therefore spacing) appears to have little effect. In a derivation based on strain concentration at the sides of inclusions resulting from the notch effect of the inclusion (frontal radius of curvature) and on a criterion of void coalescence based on a critical ratio of inclusion spacing to inclusion size, it was shown that

$\varepsilon_T = \varepsilon_0 + \frac{1}{2} \ln((x^2/f_0{}^2 + k/r^2)/(1 + k/r^2))$

where,

ε_T is the strain to fracture

ε_0 is the void initiation strain.

f_0 is the initial volume fraction of inclusions, r is the aspect ratio of the inclusions.

k is a strain concentration factor.

X is the critical void length: spacing ratio.

In the case of inclusions in a polygonal ferrite matrix, void initiation was equated with the onset of plastic straining) X was approx. 0.04 and a value of k = 2 gave a reasonable fit with the experimentally observed void growth. Attention was drawn to the shortcomings of the theoretical derivation which took no account of non-uniform inclusion distributions, and also no attention was given to the operative stress system. Nevertheless, the ductility results could be rationalized in terms of volume fraction and shape only. [15]

2.6.9. Inclusions and C.O.D.

Inclusions in the plastic zone of a C.O.D. test behave in a qualitatively similar manner to inclusions in the tensile test, although quantitatively the extent of cavitation is difficult to assess due to uncertainties in the strain distribution in the plastic zone. Baker and Charles [30] have definitely established a significant effect of particle shape, and have further suggested that the total of the effects of shape, volume fraction and size (i.e. all of the independent variables) are characterized by a single parameter, i.e. the projected length. However, it is suggested that a much wider range of inclusion contents, shapes, and sizes will be required to confirm this contention.

It is interesting to note, that the total projected length (projected in the z direction) is equal to the reciprocal of the number of particles per unit length of an intercept in the Z direction as pointed out as earlier .Thus the projected length could be evaluated very simply by manual methods. [15]

2.6.10. Inclusion and K_{IC}

The effects of inclusion shape by the relatively simple expedient of examining the anisotropy of K_{IC} in rolled bar and plate. Further, the results suggest that the K_{IC} value is independent of particle size and/or spacing of the inclusions. It is important to acknowledge that the K_{IC} values were obtained on specimens which showed a ductile failure mode. It is quite probable that the effects of inclusions would be significantly modified if failure were to occur by some other mechanism such as cleavage. There appears to be little information available at the present time concerning" effects of inclusions on fracture toughness levels associated with the cleavage failure mechanism. [15]

2.6.11. General observation

The effects of inclusions on properties such as ductility and toughness have commonly related the observed effects to a specific feature of the inclusion distribution such as projected length or inclusion spacing. Most studies, however, have suffered simultaneous variations in a number of the parameters which characterise the inclusion distribution. A number of workers were able to identify an effect of inclusion shape at constant volume fraction and constant particle volume i.e. varied inclusion shapes were produced in a given steel. However, in comparing steels with a variation in the volume fraction of particles, then the number of particles per unit volume or the particle size would change for a given particle shape. Thus, the effects of particle size and particle spacing have not been identified as independent effects. In effect, the important quantitative features of the inclusion distribution are not known precisely, although it is established that inclusion shape is an important feature. The volume fraction of inclusions is known to be important, though whether this is an effect of inclusion size, spacing, or projected length is not clear. It is for these reasons that attempts to improve ductility and toughness have been aimed at a reduction in the volume fraction of inclusions and/or a modification of the shape of inclusions. Attempts to change the size distribution of inclusions have not shown any conclusive effect on toughness or ductility as yet. Inclusion shape control can be attempted by chemical means, control of rolling temperature, and geometric aspects of processing. [15]

2.6.12. Deformation behavior of inclusions in structural steel during hot rolling

The structural steel quality is affected by the shape and the distribution of inclusions. The investigation of inclusion deformation in rolling process is significant for understanding the deformation mechanism of inclusion and guiding production. The behavior of spherical non-metallic inclusions in structural steel reinforcing bars during multi-pass hot rolling has been simulated by 3D finite element method and updating geometric method. The shape of inclusions is obtained under a variety of the inclusion sizes and positions. The inclusions elongate along rolling direction and compress along transverse direction. The inclusion sizes increase, the inclusion deformation gradually increases. The inclusion deformation also increases with the inclusion approaching the bars surface. As the rolling pass increases, the differences of inclusion shapes increase under various inclusion sizes and positions. [21]

Figure 2.14 shows the effect of rolling temperature on the relative plasticity index during hot rolling of steels. The relative plasticity index of inclusion increases while the rolling temperature rises. There exists a transition region, where the relative plasticity index changes rapidly. This trend agrees with the existing experimental results. [28]

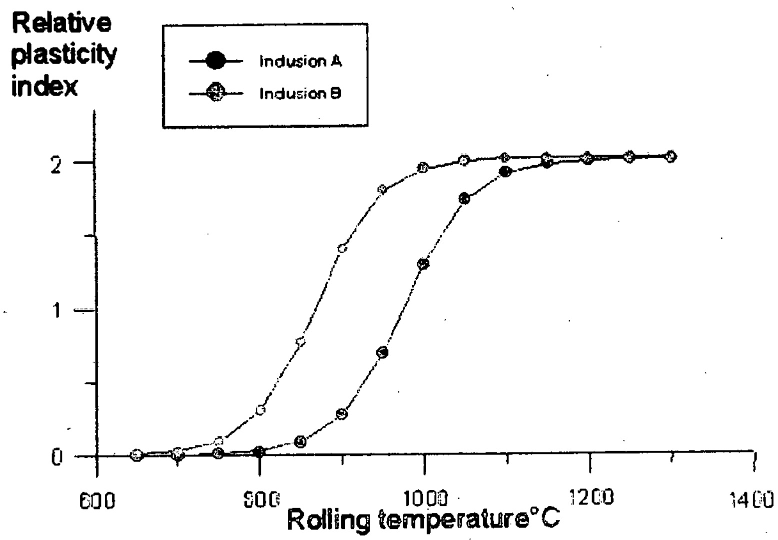


Figure2.14: Effects of rolling temperature on the relative plasticity index. [28]

Before rolling or forging inclusions are mainly four shape and size. As

(i) Sulphide type mainly plastic, (ii) Alumina type mainly hard refractory, (iii) Silicate type mainly strung and (iv) Globular oxide type.

After rolling or forging sulphide type inclusions are elongated and rolled out to some extent with the working direction. Alumina type inclusions retain their shape and particle size, refractory and brittle one breaks up. Silicate type inclusions are strung out. The size of the deformed samples (inclusions) indicates the degree of reduction of the material from ingot or billet to deformed bars. With increasing reduction, the plastic inclusions change shape and therefore their size, whilst the harder types of inclusions are not affected by reduction.

Inclusions in the form of thin films located on grain boundaries are especially dangerous for steel quality. These are usually low-melting oxysulphide inclusions precipitated in liquid state during steel solidification. They weaken the intergranular bonds, especially at elevated temperatures (red shortness).

Inclusion particles with sharp edges may also be quite dangerous; these are usually high-melting inclusions, i.e. with the melting point above the temperature of molten metal. They often serve as stress concentrators in the metal and as sites for the beginning of fracture. If such an inclusion passes onto the surface of an article (a ball bearing, rail, etc.) it will crumble out soon and cause premature failure of the article. Rounded off inclusion particles are considered less harmful. They are formed by substances which have a low melting point and are poorly wettable by the metal.

The size and distribution of inclusions change substantially in the subsequent process of rolling or forging. Some inclusions may form on grain boundaries during solidification and others, within grains. Some types of inclusions are disintegrated on plastic working and form chains oriented along the rolling axis; others are stretched out; still others remain unchanged, and so on. The result is that the apparent (as seen in the microscope) purity of steel from inclusions depends not only on their concentration in the metal, but also on the reduction in rolling, etc.

Chapter 3

Experimental procedure

3.1. Introduction

This study was based on analysis of samples collected from the process stream of a steel plant in Bangladesh. Care was always exercised to collect a representative sample. The plant concerned produces different grades of deformed bars from ingots produced in metal moulds and also from billets produced by a continuous casting machine respectively. The plant also has a ladle refining furnace and samples of continuously cast billets, both before and after ladle refining, were collected for study.

In this research work, the term ingot refers to the ingots produced by bottom pouring of vertical metal moulds. Normal standard billets refer to the billets produced in the continuous casting machines from molten steel that has not been refined in the ladle furnace, where as quality standard billets refer to the billets produced in the continuous casting machines from molten steel that has been subjected to ladle refining.

3.1.1. Ingot sample preparation

The process of manufacturing ingot consists of melting mild steel scrap (HMS-1) and sponge iron in a 5 MT induction melting furnace. Required amounts of ferroalloys are added to the molten metal to adjust the chemical compositions to required specifications. For the determination of the required amount of ferroalloy additions, samples of molten steel are collected from the furnace and analysed in an optical emission spectrometer (Model No:F_{20} ANALYZER) where 18 elements can be analysed simultaneously. The required amounts of ferroalloy addition are determined from this analysis.

Then the molten steel is tapped directly in to a tundish. The tundish is inserted between the furnace and the ingot mould to ensure a uniform metal stream while teeming from the top. The tundish has its own nozzle to regulate the flow. The tundish is filled to a certain depth which is maintained at the same level throughout the teeming period to eliminate the flow variation due to the varying ferro-static head in the tundish.

The tapping temperature is high enough to allow the additions and holding period in the tundish and still be left with adequate superheat to obtain good casting after teeming. Steel is teemed into a vertical runner which is connected at the bottom to a horizontal through runner that opens up in the bottom of the mould. A very large bulk of liquid steel is cast into permanent moulds to produce ingot.

3.1.2. Normal standard billet processing technology

The process of manufacturing normal standard billets consists of melting mild steel scrap (HMS-1) and sponge iron in 10 MT induction melting furnaces. Melt samples are analysed to determine the composition of the melt and also the required quantity of ferro-alloys to be added to the molten metal. The liquid metal thus produced is poured in a 20 MT capacity of ladle and is taken up to the continuous casting plant for casting 100 mm x 100 mm x 6000 mm steel billets. The billets thus produced in CCM are further analyzed in spectrometer to check the composition and if found satisfactory only then the billets are transferred to re-rolling unit for production of bars.

3.1.3. Quality standard billet processing technology

The process of manufacturing billets consists of melting mild steel scrap (HMS-1) and sponge iron in 10 MT capacity induction melting furnaces where required quantity of ferro-alloys are added. Samples of molten steel are collected from the furnace and these samples are analysed for the determination of ferro-alloy additions required. The liquid metal thus produced is poured in a 20 MT ladle and is taken to the ladle refining furnace through a ladle car running over rails.

In the ladle refining furnace nitrogen gas is blown through a porous plug in the bottom of the furnace. The potential benefits of bottom blowing are to accelerate slag metal reactions, improved iron yield and a higher manganese retention in steel. More efficient slag removal and lower nitrogen levels in steel are obtained with argon stirring, the plant in question however, uses nitrogen gas for blowing due to economic reasons. Non metallic inclusions of a certain size are removed and a uniform distribution of inclusions is obtained. The temperature of the liquid steel is raised to a desired temperature.

Three different purging pressures and three different time of purging were used as the variables Table 3.1). Variation of time of purging at a fixed purging pressure, or variation of purging pressure for a fixed time would have been more appropriate to reach any conclusion. This study, as has already been mentioned, is based on samples collected from the process stream of a steel plant. In actual plant operation, purging time and pressure depends on many factors including the tapping temperature.

Table 3.1: Variation of purging pressure and time:

Heat No	Purging Pressure(bar)	Purging Time (minute)
QB-1	3.30	33
QB-2	3.00	35
QB-3	4.00	40

Control of sulphur is exercised through the addition of lime in the ladle refining furnace. Samples are then collected for further analysis in the spectrometer. Additions are made if necessary, and when the composition is found ok the liquid steel with ladle is taken up to the continuous casting plant for casting 100mm x 100mm x 6000mm steel billets. The billets thus produced in CCM are further analyzed in spectrometer to check the composition and if found satisfactory only then the billets are transferred to re-rolling unit for production of bars.

3.2. Sample for chemical test

Samples were collected from the different positions of ingots or billets. The analyses were performed using spectrochemical methods. Reinforcing bars are graded on the basis of carbon equivalent value.

The following formula is used to calculate the carbon equivalent value, where the chemical symbols represent the percentages of each element: [22]

Carbon equivalent value =C + Mn/6 + (Cr + Mo + V)/5 + (Ni + Cu)/15

3.3. Sampling

Steel ingots or billets produced commercially are heterogeneous in nature. The exact distribution of the non-metallic inclusions in an ingot or billet is not known, but experience suggests that it could vary from top to the bottom or first to the end and also centre to the outside of an ingot or billet. Differences between ingots or billets in the same cast have also been observed. The magnitude of these differences is largely dependent on steel making and casting conditions.

The sampling procedure of any heterogeneous material is of great importance. Two methods of sampling are advocated:

a. Random sampling:

In which selection of the material is done in such a way that every portion of the bulk has an equal chance of appearing in the sample

b. Representative sampling:

In which the sample is deliberately taken in a systematic way so that each portion of the sample represents a corresponding portion of the original bulk.

Representative sampling is best achieved by taking samples equally spaced throughout the bulk. This method of sampling is perfectly satisfactory.

3.4. Tension tests

The different diameter bars were produced into standard tensile bars specimens with a nominal diameter. The tensile specimens were then tested with a Universal Tensile Testing Machine (Model No: TUK-100 and TUT-40) to obtain data on yield strength (Y. S.), ultimate tensile strength (U. T. S.), percentage of elongation (%EL), and percentage of reduction in area (% RA). [23]

3.5. Bending tests

To determine the bendability of the bars, bars of different diameters are bent around corresponding pin dia as shown in below:

Table.3.2.Requirement of bend test:

Diameter of bar	Required pin dia for bend test	
	Grade 40	Grade 60
10, 12, 16 mm	4 times dia	4 times dia
20 mm	5 times dia	5 times dia
22, 25 mm	5 times dia	6 times dia
28, 32 mm	5 times dia	8 times dia
Method of bending 90^0 around the pin.		

The results are recorded as satisfactory or unsatisfactory. [22]

3.6. Inclusion assessment

Inclusions present at three levels of each of ingots or billets were microanalysed:

Level 1- (near the top of the ingots or first casting of billets)

Level 2- (mid-height of the ingots or middle casting of billets)

Level 3- (near the bottom of the ingots or end casting of billets)

All samples in each level were used for microanalysis work.

Inclusion content may be determined either by a macroscopic or a microscopic method. The advantages of microscopic method are that, the character or type of inclusions may be determined and extremely small inclusions are revealed.

Specimens or metallographic assessment of inclusions were prepared by using standard techniques of polishing. Extreme care was used to ensure that the inclusions are retained. It was not always possible to retain all the inclusions. However, this did not affect the result because the cavities in the samples were counted as inclusions. [24]

Inclusions count method:

Inclusion count methods are dividing into four distinct groups

Microscopic methods based on visual comparison of fields with standard charts

Microscopic methods based on direct counting and possibly measurement of inclusions

Micro radiographic methods

Magnetic methods

Microscopic methods based on direct counting and possibly measurement of inclusions:

Many investigators have preferred to estimate the inclusion content in steels without recourses to arbitrary standards, primarily because the personal factor in these methods is reduced to as rational a basis as possible. Several methods have been suggested for assessing the inclusion content in steel. The method in which inclusions were counted given here:

Method Proposed by Kjerrmann: [25]

This method is simple and direct, but it has the following disadvantages

It is tedious and time consuming;

It does not take inclusions less than 0.002 mm into account; and

It gives the final count as "number of inclusions per square millimeter" which renders the results subject to variations due to errors in counting the very small inclusions.

In this method a sample is taken from the centre of the middle ingot or billet of the cast. Specimens were taken from its longitudinal and transverse section. The cut surface is then prepared for micro examination. The specimen is examined at a magnification of X200, and micrographs were taken and the number of inclusions per square milimetre was recorded.

3.6.1. Optical assessment of inclusions

A total of 54 (3 routes x 2 surface x 3 positions x 3 variables) samples were selected for microscopical assessment.

1. Each specimen was placed on the stage of the microscope so that the specimen surface is perpendicular to the optical axis.
2. Inclusions were examined by using magnifications of 200X and 400X.
3. 50 optical micrographs in each sample was recorded and
4. Finally total numbers of inclusions per square centimeter were counted on the micrographs.

Table.3.3. Operating conditions:

	Low magnification	High magnification
Objective lens	X200	X400
Number of fields	50	50
Area per field	0.16 mm^2	0.04 mm^2
Total area assessed	8 mm^2	2 mm^2
Step size between x fields	0.475 mm	0.2375 mm
and y fields	0.335 mm	0.1675 mm

The low magnification measurements on all ten ingots or billets were repeated to obtain a measure of the reproducibility of assessment. Examination of the duplicate series of ingots or billets was restricted to the lower magnification examination. For ease of presentation the results from all these assessments have been simplified and the numbers of inclusions above and below 20 μm are recorded. This division is considered suitable not only because different magnifications were used but also because this seems a practical boundary between large and small inclusions.

The largest oxide inclusions observed in the surface examined from each ingot were about 100 μm diameter.

The larger inclusions were generally segregated to the midradial position in the top and mid height regions of the ingot, and to the centre line position at the bottom. A few large oxides did occur, however, at positions near at the ingot surface. The sulphide inclusions were rarely larger than 50 μm and were generally less than 10 μm in diameter and their distribution was much more uniform than the oxides.

3.6.2. Scanning Electron Image Analysis

The SEM can automatically perform image analysis and with the advanced of chemical characterization using Energy Dispersive X-ray (EDX) detector (Model No: XL-30).

Operating condition:

1. Specimen was placed on the stage of the scanning electron microscope so that the specimen surface is perpendicular to the optical axis.
2. Images were examined by using magnifications of 1000X.
3. Images were chemically micro-analysed by use of energy dispersive x-ray detector

Chapter 4

Experimental results and discussion

4.1. Experimental results

Many pencil ingots of different heats, normal standard billets and quality standard billets were chemically analyzed. The chemical compositions of the samples were observed. Heats chosen for further work was based on carbon equivalent factor as determined from the above.

The chemical compositions of three pencil ingots, three normal standard billets and three quality standard billets from different heats are listed bellow.

4.2. Chemical composition

Table. 4.1. Required chemical ingredients:

Heat No	Casting Type	Chemical Composition						Carbon Equivalent =%C+%Mn/6
		%C	%Mn	%Si	%P	%S	%N	
PI-1	Pencil Ingot	0.34	1.10	0.27	0.03	0.04	0.02	0.52
PI-2		0.35	1.14	0.24	0.05	0.04	0.02	0.54
PI-3		0.35	1.09	0.30	0.03	0.05	0.02	0.53
NB-1	Normal	0.32	1.15	0.25	0.04	0.04	0.02	0.51
NB-2	Standard	0.33	1.16	0.25	0.03	0.04	0.02	0.52
NB-3	Billet	0.32	1.07	0.28	0.03	0.05	0.02	0.50
QB-1	Quality	0.33	1.06	0.27	0.04	0.04	0.03	0.51
QB-2	Standard	0.31	1.16	0.31	0.03	0.04	0.03	0.50
QB-3	Billet	0.30	1.30	0.26	0.03	0.05	0.03	0.49

4.1.1. Pencil Ingot Analysis

Three different heat numbers of pencil ingots, inclusions present at three levels (top, midheight and bottom) of each ingot were micro-analyzed. Two samples in each level were used for microanalysis work. Inclusions per samples were analyzed.

Level 1: Near the top of the ingot

Level 2: Near the midheight of the ingot

Level 3: Near the bottom of the ingot.

Inclusions size distribution

Table 4.2. Number of inclusions and size distribution of Pencil Ingot. ***Heat No: PI-1*:**

Ingot Position	Level	Inclusions Size, µm					Total Number of Inclusions /cm^2
		3-10	11-20	21-30	31-40	>40	
Top	1	30000	1600	625	235	00	32460
Midheight	2	45000	5445	718	353	50	51566
Bottom	3	48050	8333	815	423	55	57676

Table 4.3. Number of inclusions and size distribution of Pencil Ingot. ***Heat No: PI-2*:**

Ingot Position	Level	Inclusions Size, µm					Total Number of Inclusions /cm^2
		3-10	11-20	21-30	31-40	>40	
Top	1	5000	625	625	321	12	6583
Midheight	2	19285	1428	714	400	60	21887
Bottom	3	19700	2307	812	450	78	23347

Table 4.4. Number of inclusions and size distribution of Pencil Ingot. ***Heat No: PI-3*:**

Ingot Position	Level	Inclusions Size, µm					Total Number of Inclusions /cm^2
		3-10	11-20	21-30	31-40	>40	
Top	1	13200	1222	729	205	5	15361
Midheight	2	22312	1562	900	356	86	25216
Bottom	3	32200	1853	1500	425	145	36123

4.1.2. Billet casting analysis

Inclusions present at three levels of each cast were microanalysed. Under normal casting conditions, the size, shape and distribution of inclusions were constant in all the lengths, except

- For the first few meters which correspond to the filling of the tundish. At this time, the temperature of the steel is too low to allow a good separation of the inclusions. Also, given that the steel is exposed in air, there is re-oxidation.
- For the last few meters which correspond to the draining of the tundish. At this time the top layer containing the inclusions which have floated out is entrained into the mould.

A random sample (except at start and end of cast) will be representative of the entire cast on one strand as the conditions of cast while the following parameters are constant.

- Protection of steel stream from the ladle
- depth of bath in tundish
- Casting speed

The segregation of inclusions depend only the number of inclusions and the machine geometry. This is a continuous process and similar to the progress of solidification.

Sample collections:

Level 1: First Casting Billet

Level 2: Middle Casting Billet

Level 3: End Casting Billet

4.1.2. Normal Standard Billet Casting Analysis:

Table 4.5. Number of inclusions and size distribution of normal standard billet. ***Heat No: NB-2:***

Casting Position	Level	Inclusions Size, µm					Total Number of Inclusions /cm^2
		3-10	11-20	21-30	31-40	>40	
First casting	1	10200	2514	925	21	12	13672
Middle Casting	2	9506	1973	789	51	00	12319
End Casting	3	15421	3241	945	60	56	19723

Table 4.6. Number of inclusions and size distribution of different normal standard billet. ***Heat No: NB-1, NB-2, and NB-3: (middle casting position)***

Heat No	Inclusions Size, µm					Total Number of Inclusions /cm²
	3-10	11-20	21-30	31-40	>40	
NB-1	11250	625	253	00	00	12128
NB-2	9506	1973	789	51	00	12319
NB-3	8333	1510	365	00	00	10208

4.1.3 Quality Standard Billet Casting Analysis

Table 4.7. Number of inclusions and size distribution of quality standard billet. ***Heat No: QB-2:***

Casting Position	Level	Inclusions Size, µm					Total Number of Inclusions /cm²
		3-10	11-20	21-30	31-40	>40	
First casting	1	11225	253	00	00	00	11478
Middle Casting	2	10500	625	00	00	00	11125
End Casting	3	12365	856	00	00	00	13221

Table 4.8. Number of inclusions and size distribution of different quality standard billet. ***Heat No: QB-1, QB-2, and QB-3: (middle casting position)***

Heat No	Inclusions Size, µm					Total Number of Inclusions /cm²
	3-10	11-20	21-30	31-40	>40	
QB-1	12812	546	78	00	00	13436
QB-2	10500	625	00	00	00	11125
QB-3	40250	3000	00	00	00	43250

4.3. Mechanical properties

Tests were performed by Universal Tensile Testing machine and the results are presented in table 4.9

Table 4.9. Results of physical properties.

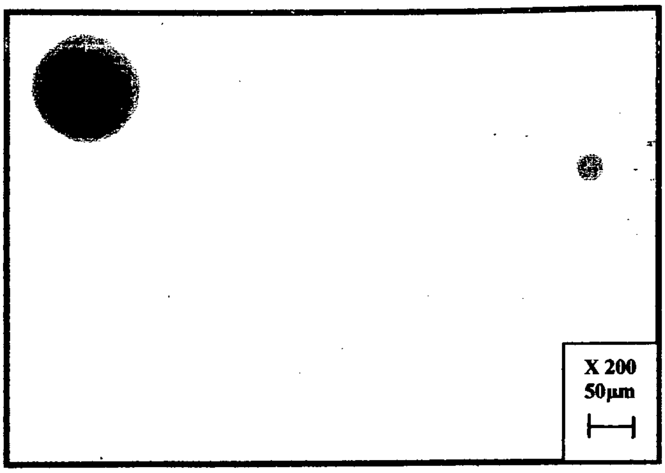
Heat No	Selected Diameter mm	Test Piece	Yield Strength MPa	Ultimate Tensile Strength MPa	Percentage of Elongation in 203mm	Percentage of Reduction in Area	Bend Test
PI-1	10	3	418	658	10	28	Ok
PI-2	10	3	422	646	11	26	Ok
PI-3	10	3	414	622	13	30	Ok
NB-1	10	3	415	672	15	35	Ok
NB-2	12	3	443	678	15	30	Ok
NB-3	10	3	419	670	14	34	Ok
QB-1	20	3	475	741	18	40	Ok
QB-2	16	3	472	713	19	40	Ok
QB-3	20	3	484	720	18	42	Ok

4.4. Discussion

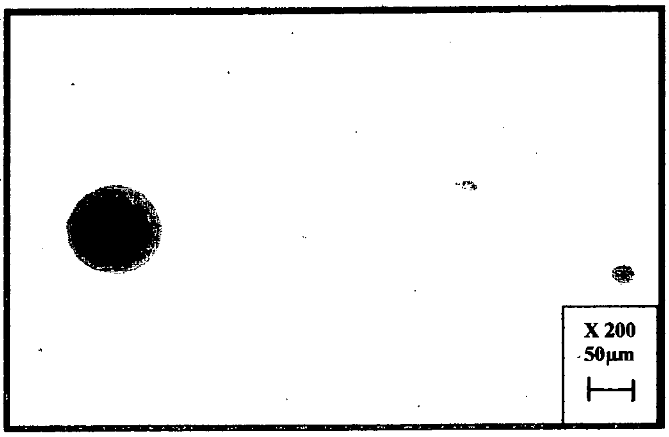
Number and size distribution

Inclusions present at three levels of each pencil ingot or billets were micro analyzed. Top, midheight, and bottom of a pencil ingot or first, middle and end casting of billets were used for microanalysis work. The locations of inclusion analysis were randomly selected. The inclusions varied in size and shape. The inclusion size distribution and total number of inclusions/cm^2 were observed and recorded.

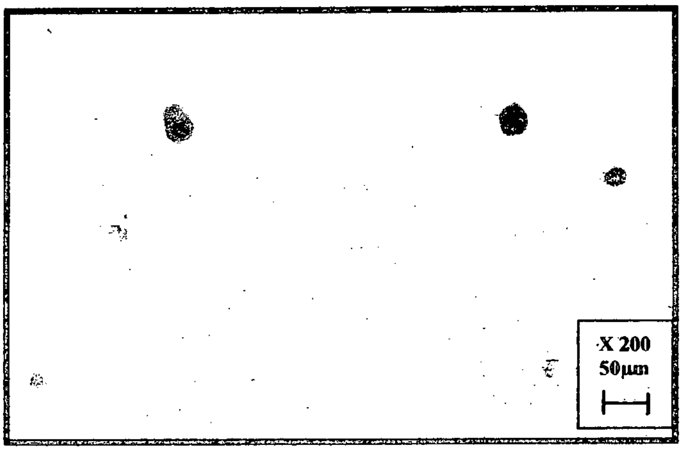
In pencil ingot samples top, midheight and bottom portions showed inclusions .At the top the total number of inclusions were smaller than at midheight or at the bottom.(Fig..4.1)



(a)



(b)



(c)

Fig: 4.1. Optical micrograph showing inclusions (a) top, (b) midheight and (c) bottom position of a pencil ingot. Heat No: PI-2

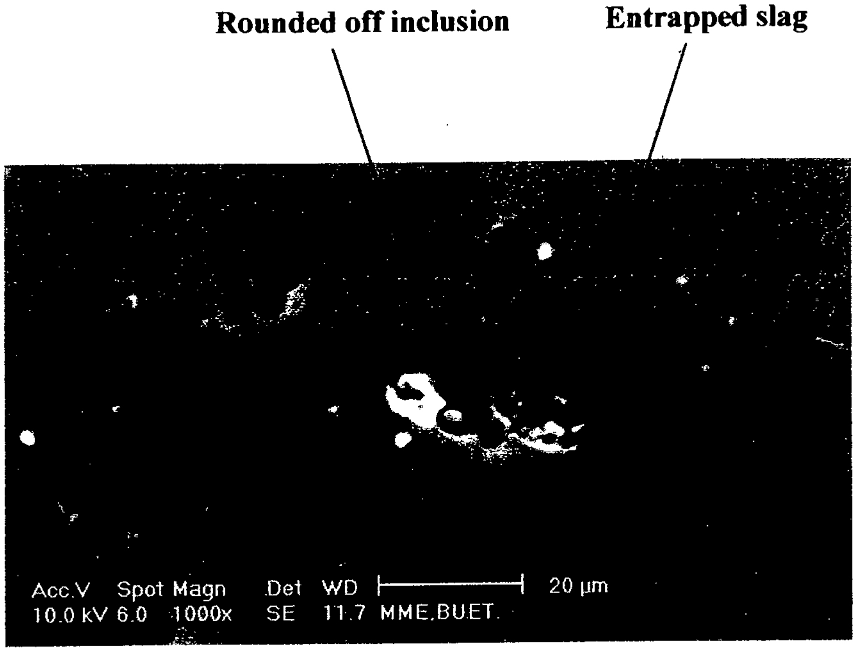
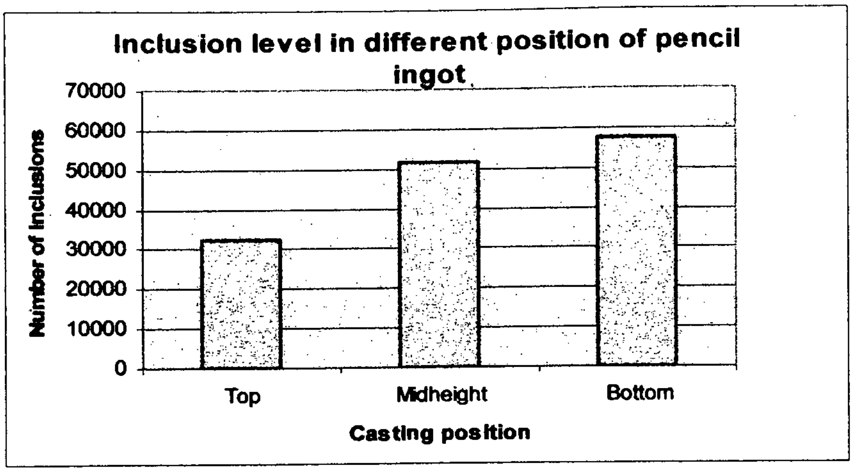
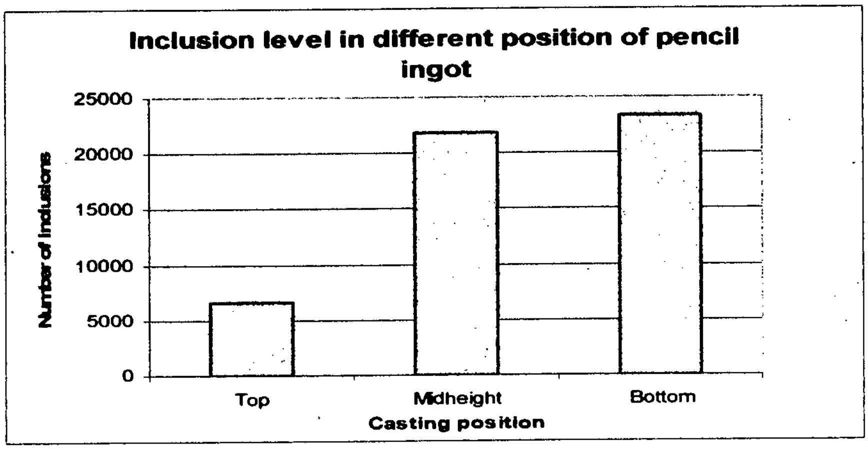


Fig: 4.2. SE image showing rounded off inclusions and entrapped slags in pencil ingot.

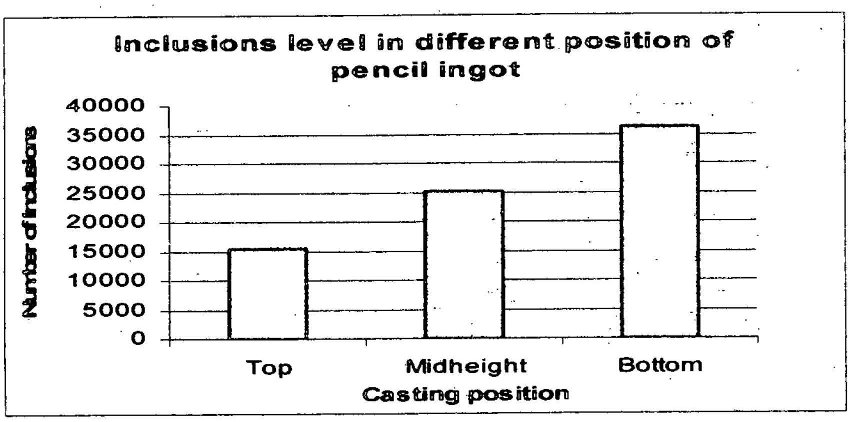
At the top and bottom positions the inclusions are not representative for the larger ingot. At mid-height casting positions, inclusions are representative for grading of structural steel. In general at top position of larger castings inclusions are higher than mid-height and bottom position. In this research work, the pencil ingots were small (bottom: 100 mm x100 mm, top: 75 mm x75 mm and height maximum: 1500 mm). While casting the tapping temperature was low to ensure better quality casting by minimizing pipe formation. So rapid solidification occurred and inclusions were not properly separated from bottom to top. At bottom and mid-height positions of all pencil ingots, inclusions were about the same. But at top position total numbers of inclusions were lower. The explanation for this, however, would require further experimentation.



(a) PI-1



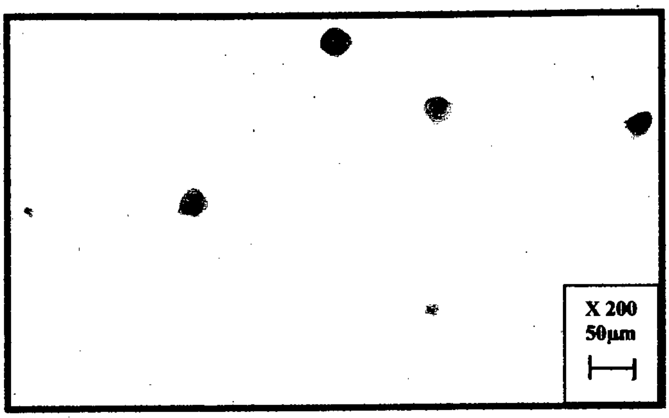
(b) PI-2



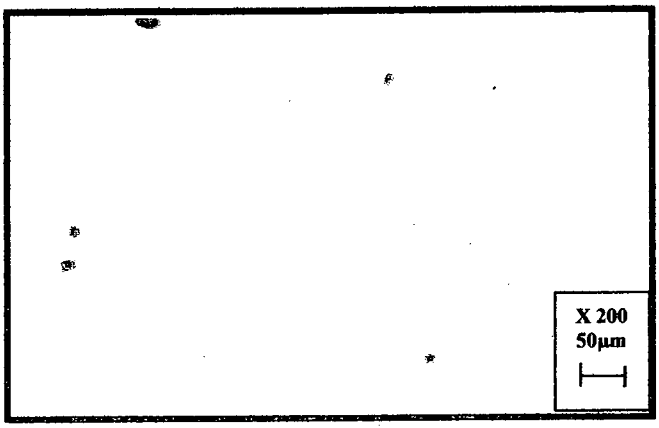
(c) PI-3

Fig: 4.3. Number of inclusions in different positions of pencil ingots.

In normal standard billet inclusions are count at three level first, middle and end casting of billets. At the first casting billets total number of inclusions are higher than middle casting billets but lower than end casting billets.



(a)



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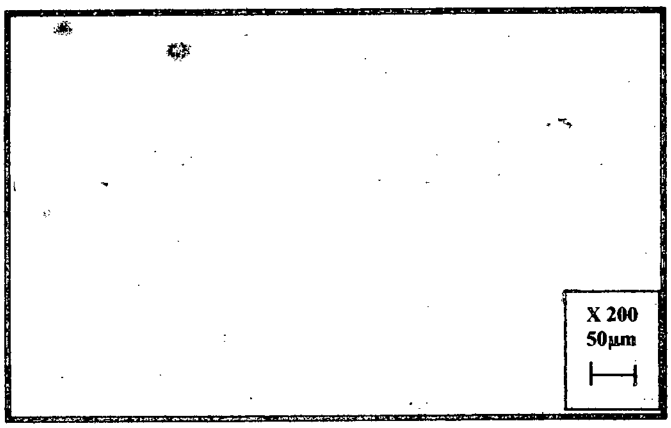
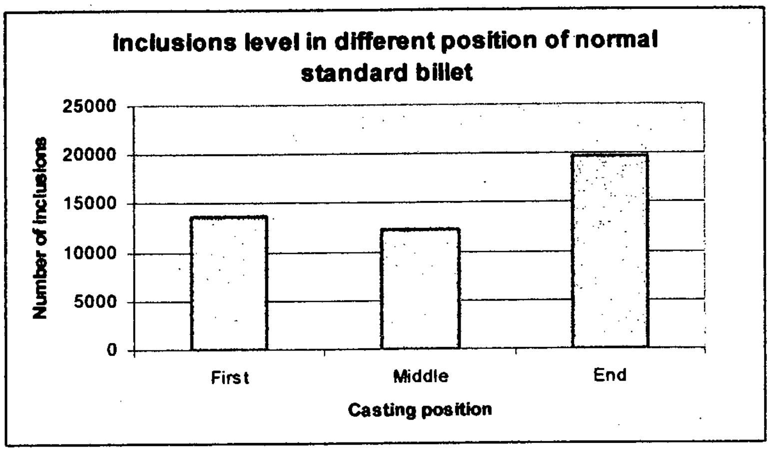


(c)

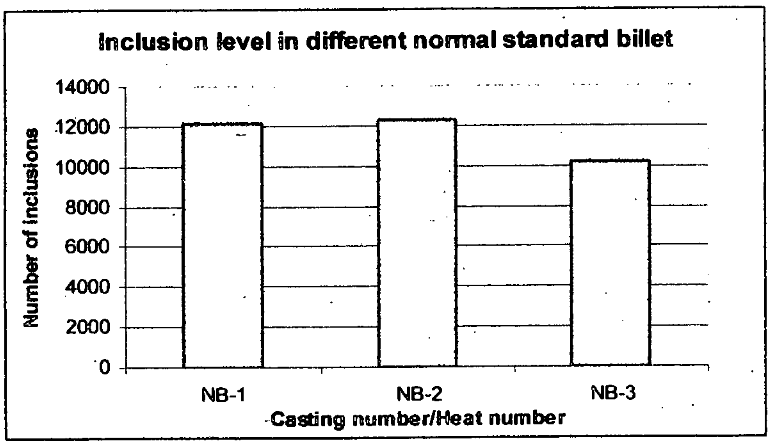
Fig: 4.4.Optical micrograph showing inclusions (a) first, (b) middle and (c) end casting of normal standard billet. Heat No: NB-2.

When the ladle nozzle is opened, the liquid metal from the bottom of the ladle gradually fills up the tundish. There is no change in inclusions floating up according to Stokes law. While time passed the inclusions are floating up. The speed of floating up to the surface of molten metal depends on size of the inclusions, viscosity of the liquid metal and specific gravity of the liquid metal and inclusions. As first casting billets there is no inclusions removed. While time passed the inclusions are rise up to the surface of molten metal.

The inclusions with larger size and lower specific gravity float up easily. So the middle casting contains inclusions that are smaller in size and lower in number. But at the end of casting all the floating up inclusions are present at the top level of molten metal.



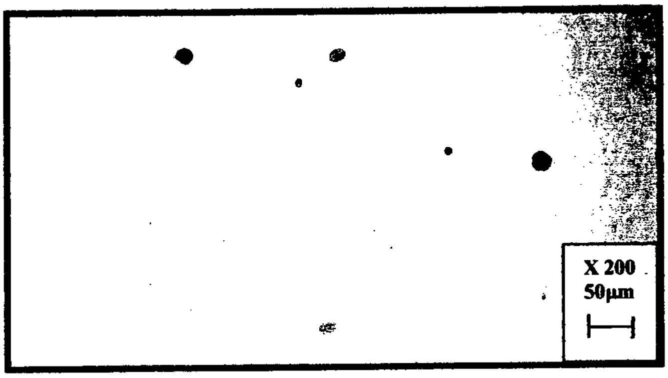
(a)



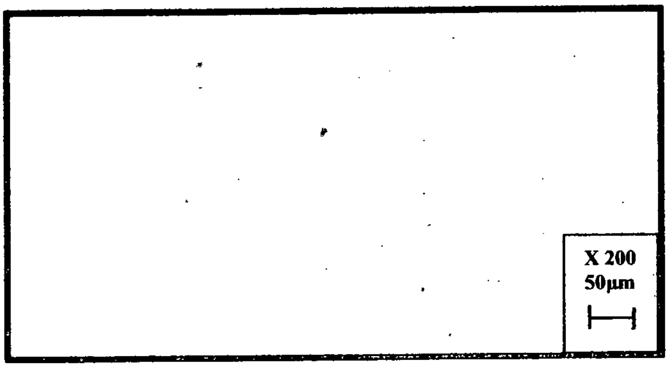
(b)

Fig: 4.5. Number of inclusions in (a) different position of normal standard billets (Heat No.NB-2) and (b) different normal standard billets.

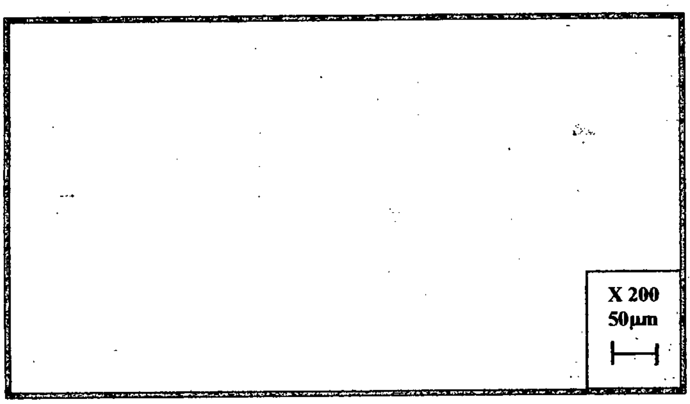
So end casting billets contains inclusions that are bigger in size and more in number, in normal standard billet castings the number of inclusions are lower in number than in pencil ingot. Effective ladle and tundish metallurgy ensure better removal of inclusions from the liquid metal. In quality standard billet first and middle casting contains near about same number of inclusions. But end casting billets contain more and larger inclusions than the first or the middle casting.



(a)



(b)



(c)

Fig: 4.6. Optical micrograph showing inclusions (a) first, (b) middle and (c) end casting of quality standard billet. Heat No: QB-2

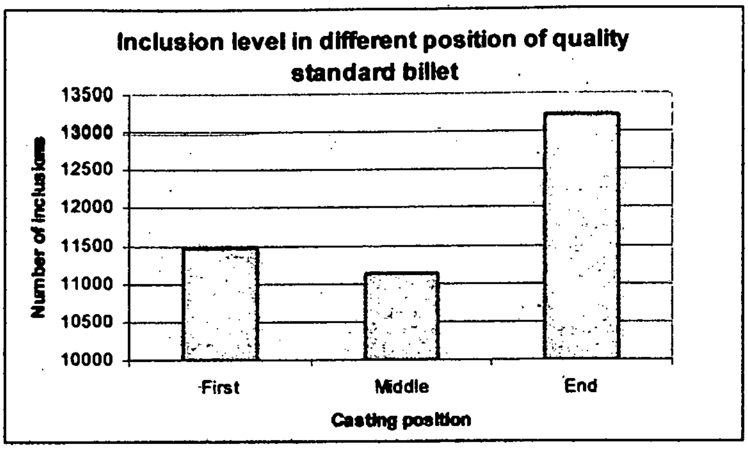


Fig: 4.7.Number of inclusions in different position of quality standard billet.QB-2

In the case of quality standard billet processing technology, the liquid metal is treated in ladle refining furnace. Inert gas is blown from bottom and stirring of the metal together with a specially prepared slag is carried out. The treatment of steel with the slag cleans the metal from unwanted impurities. In ladle refining furnaces the following actions are performed-

(a) Reducing the content of phosphorus;

(b) Diminishing the concentration of oxygen and non-metallic inclusions;

(c) Desulphurization and de-oxidation of the metal; and

(d) Removal of harmful impurities and improvement of the surface of billets.

During metal treatment the following processes take place:

1) De-sulphurization; upon treatment with synthetic slag, the content of sulphur in the metal usually decreases to 0.002-0.010%.

2) De-oxidation; since $a_{(FeO)}$ in synthetic slag is negligible, the oxidation degree of the metal decreases by 33-50%.

3) Removal of non-metallic inclusions; experience was shown that the total concentration of non-metallic inclusions can be reduced roughly by 50% upon synthetic slag treatment.

An essential advantage liquid metal treatment is that the process takes up some time. The whole procedure can be completed during arcing through the ladle refining furnace. The liquid metals are stirred by purging and the output quality of steelmaking plants is raised, some other operations, such as desulphurization and deoxidation, are also performed in the ladle.

In ladle refining furnace, the larger sizes inclusions are removed easily. Most of the inclusions are present at 3-10mm size ranges.

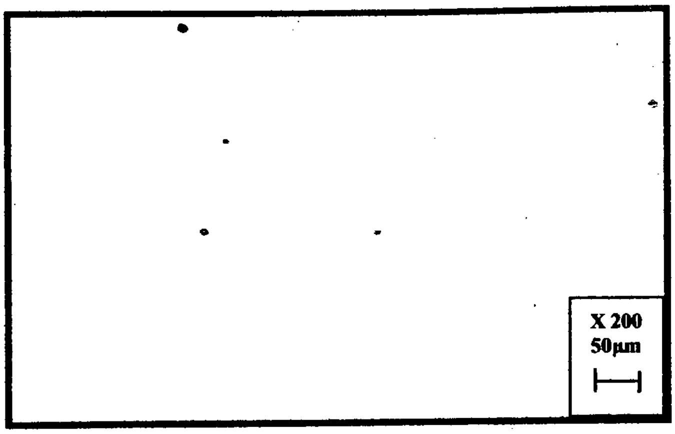


Fig: 4.8. Optical micrograph showing inclusions in quality standard middle casting billet.

The SEM analysis also confirms the presence of smaller inclusions in quality standard billets.

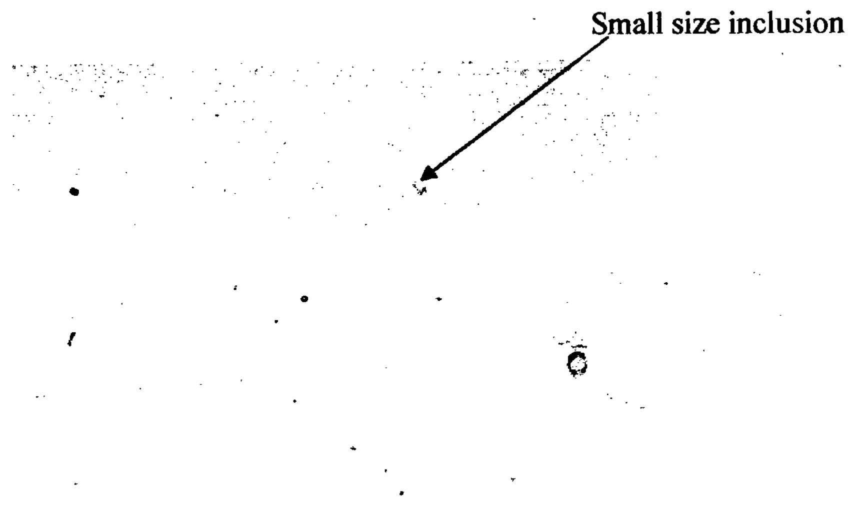
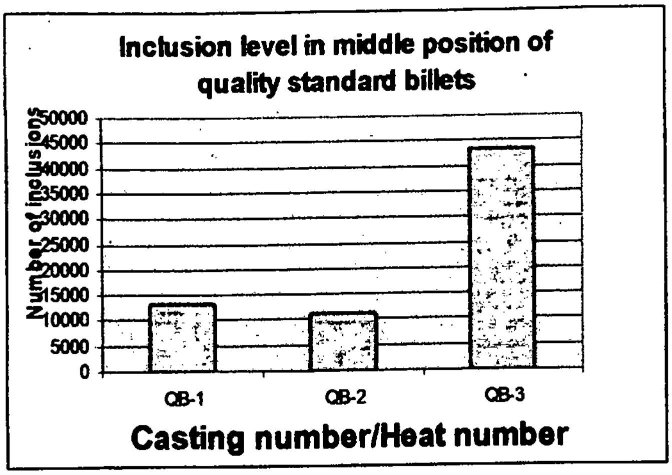


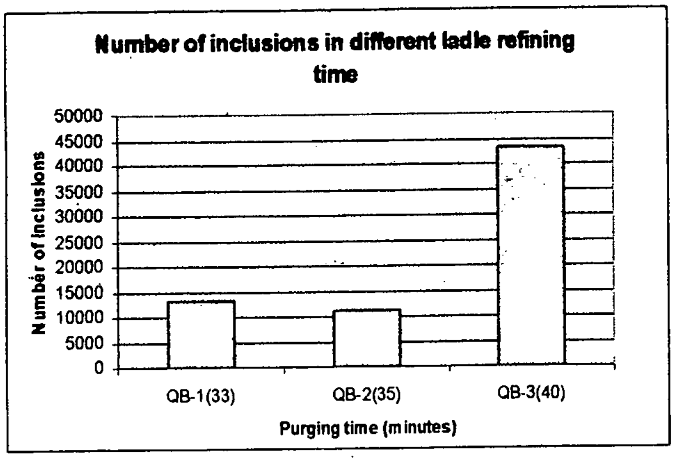
Fig: 4.9. SE image showing inclusions in quality standard middle casting billet.

Ladle metallurgy incorporates inert gas stirring for homogenization of temperature and composition of the steel in the ladle as well as for floatation of inclusions. For continuous casting, inert gas stirring of steel in the ladle has become a standard practice to improve the quality of steel.

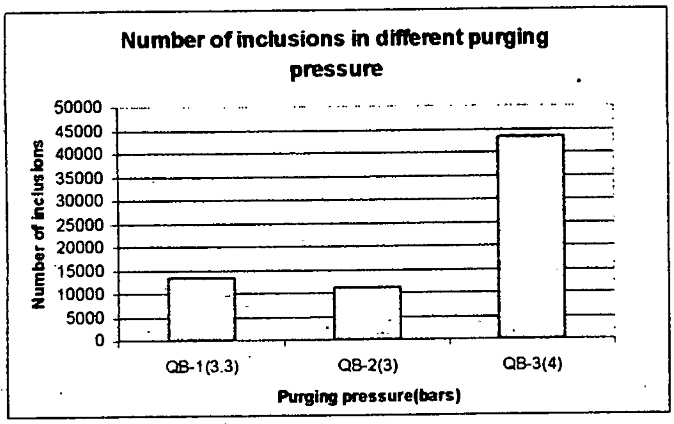
Better removal of nonmetallic inclusions, harmful elements and gases ensures the better quality of steel properties.



(a)



(b)



(c)

Fig: 4.10. Number of inclusions (a) different quality standard billets, (b) different ladle refining time and (c) different purging pressure in quality standard billets.

Size distribution of inclusions:

It is interesting at this stage to estimate the total number of inclusions in the different size ranges in the whole pencil ingot or billets.

The optical micrograph shows larger than 100 μm inclusions in size. During pencil ingot processing, there are produce huge number and larger sized inclusions.

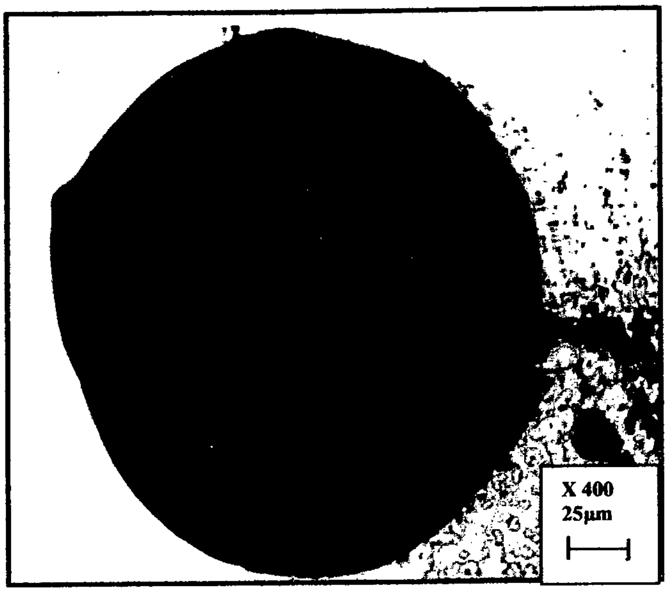
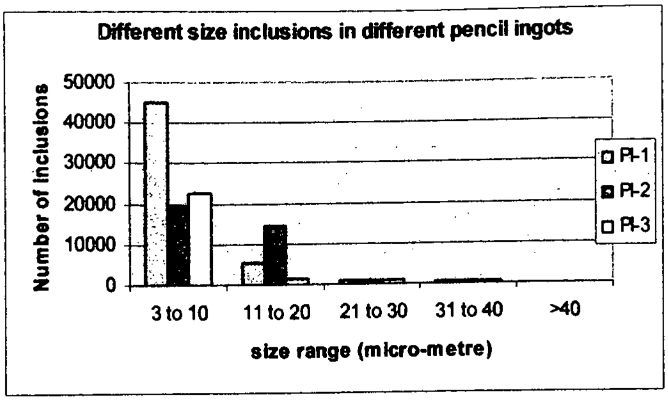


Fig: 4.11. Larger than 100 μm inclusion size in pencil ingot.

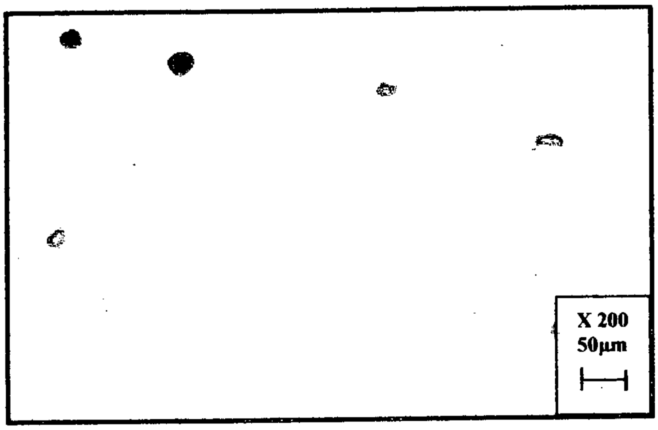
The Scanning Electron image also introduces the larger size inclusions types.

Fig: 4.12. SE image showing >40 μm inclusion size in pencil ingot.

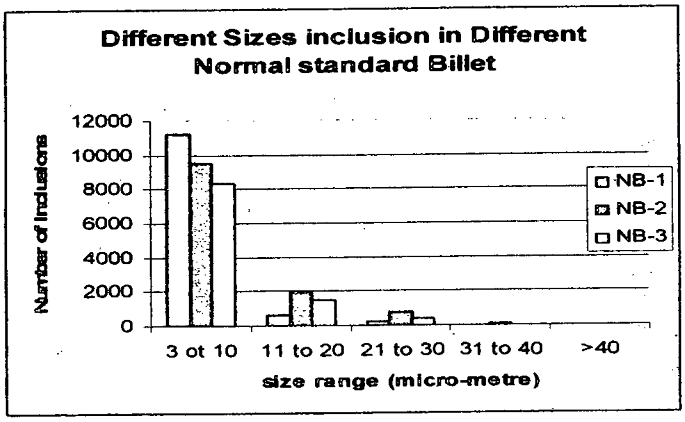


(a)

In normal standard billets, at the first and end casting billet contains larger sized inclusions while the middle casting billets contain smaller sized inclusions.



(b)



(c)

Fig: 4.13.Fig: (a) showing different sizes of inclusions present in different pencil ingots and (b) and (c) showing different sizes of inclusions present in different normal standard billets.

For quality standard billet processing technology through ladle refining furnace larger than 20 μm are not present in the castings.

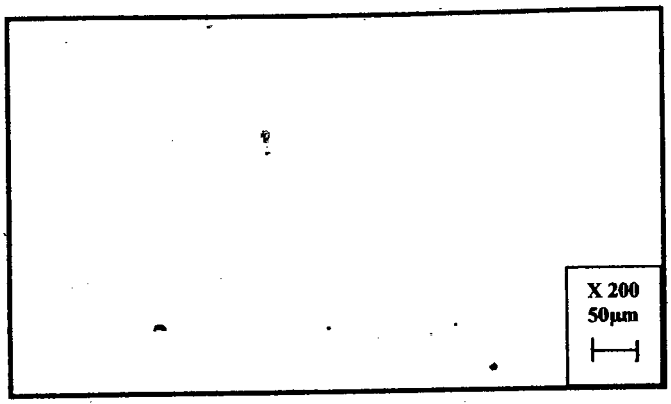


Fig: 4.14. Micrograph showing inclusion size in quality standard billet after ladle refining.

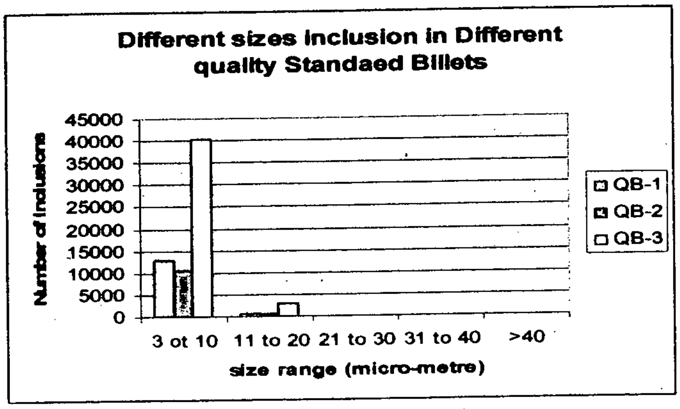


Fig: 4.15. Different sizes of inclusion present in different quality standard billets.

The SEM analysis ensures smaller size of inclusions present in quality standard billets.

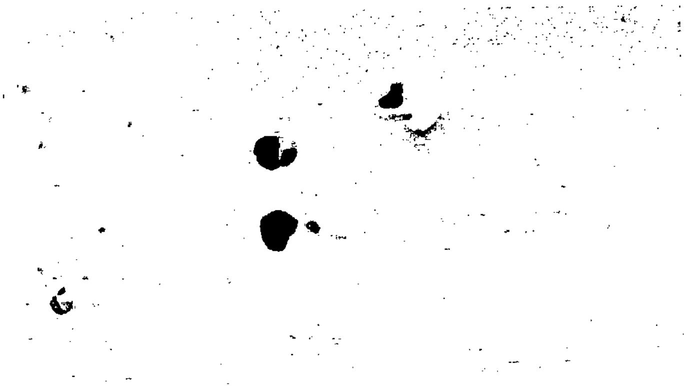


Fig: 4.16. SE image showing inclusion size in quality standard billet after ladle refining.

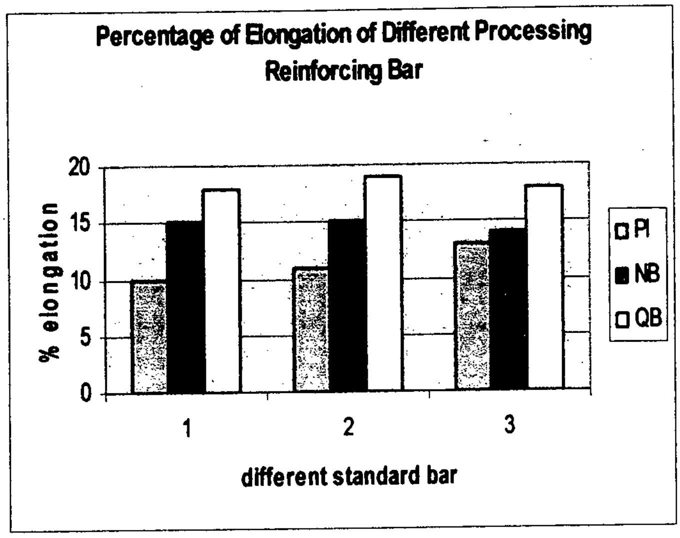
The reinforcing bar produced from quality standard billet (QB-3) content higher inclusions. During ladle refining the purging pressure was 4 bars and purging time was 40 minutes (higher than QB-1 and QB-2).This purging pressure was vigorous stirring for long time which disintegrate or destruction the previous formed inclusions. This condition impairs the process of removal inclusions from the molten metal.

The effect of nitrogen on steel properties can be either detrimental or beneficial, depending on the other alloying elements present, the form and quantity of nitrogen present, and the required behavior of the particular steel product. In general, however, most steel products require that nitrogen be kept to a minimum. High nitrogen content may result in inconsistent mechanical properties in hot-rolled products. The maximum solubility of nitrogen in liquid iron is approximately 450 ppm, and less than 10 ppm at ambient temperature. Nitrogen present in steel as nitride compounds and interstitial solid solution.

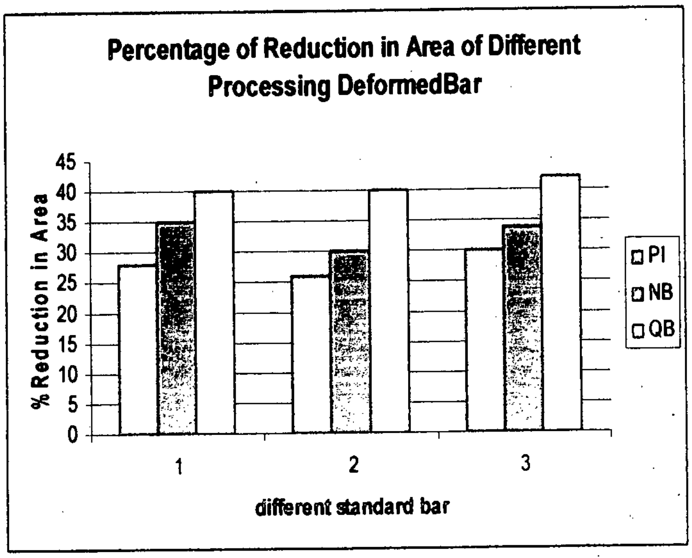
During ladle refining operation nitrogen gas was purged through the purging plug from bottom of the ladle and the molten metal picked up nitrogen. In the case of pencil ingot or normal standard billet processing, the maximum nitrogen content was 0.02 but quality standard billet content 0.03. So the reinforcing bars produced from quality standard billets give higher yield strength and ultimate tensile strength due to higher content of nitrogen.

All the reinforcing bars from pencil ingot show a low value of %elongation and %reduction in area Fig:4.18.These may be due to the presence of slags and larger non-metallic inclusions and also presence of larger sizes inclusions causes' lower % elongation and % reduction in area.

Reinforcing bar from the normal standard billets contain lower number and smaller sized inclusions then pencil ingot. The sizes of inclusions are smaller, so the %elongation and %reduction in area is higher than pencil ingot.



(a)



(b)

Fig: 4.18. Percentage of elongation (a) and percentage of reduction in area (b) of different processing reinforcing bar.

It can be seen from Table.4.10. that although the strength is higher than other two types of bars, the reinforcing bars obtained from quality standard billets show better %elongation and %reduction in area and also better toughness than the rods produced from pencil ingots or normal standard billets. Higher N_2 content generally decreases ductility with increase in strength. But the higher ductility may be due to the lesser amount of slags and inclusions in the deformed bars.

The following figure ensures which types of reinforcing bars are used in structural purposes.

Table 4.10:Overall experimental results and comments.

Heat No.	Chemical compositions		Carbon equivalent	Total number of inclusions/ cm^2	Mechanical properties				Selected final diameter (mm)	comments
	%C	%Mn	%C+%Mn/6		Y.S MPa	T.S MPa	%EL	%RA		Reinforcing bars from QBs are followed structural uses.
PI-1	0.34	1.10	0.52	51566	418	658	10	28	10	
NB-2	0.33	1.16	0.52	12128	443	678	15	30	12	
QB-1	0.33	1.06	0.51	13436	475	741	18	40	20	

N.B: Reinforcing bars produced from quality standard billets are more reliable due to lower inclusions content and better mechanical properties.

When the pencil ingot or billet is hot worked by rolling or forging the inclusions are strung out in the direction of working. Before rolling or forging inclusions are mainly four shape and size. As

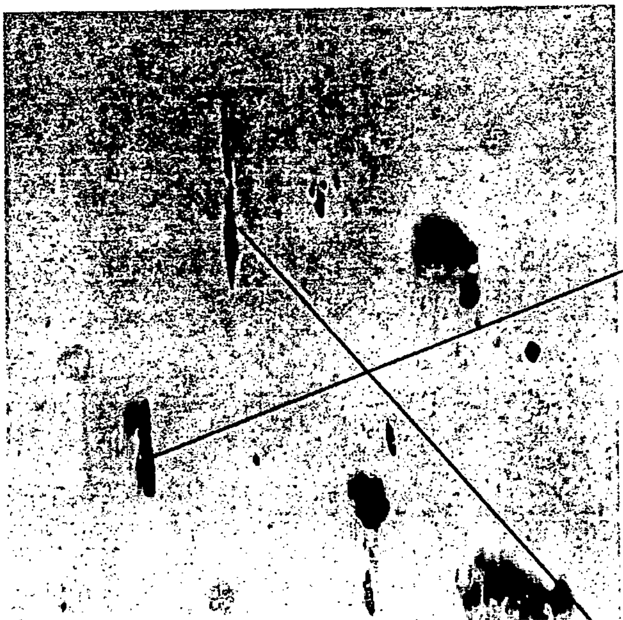
a) Sulphide type mainly plastic

b) Alumina type mainly hard refractory

c) Silicate type mainly strung

d) Globular oxide type

After rolling or forging Sulphide type inclusions are elongated and rolled out to some extent with the working direction.



EDX results:

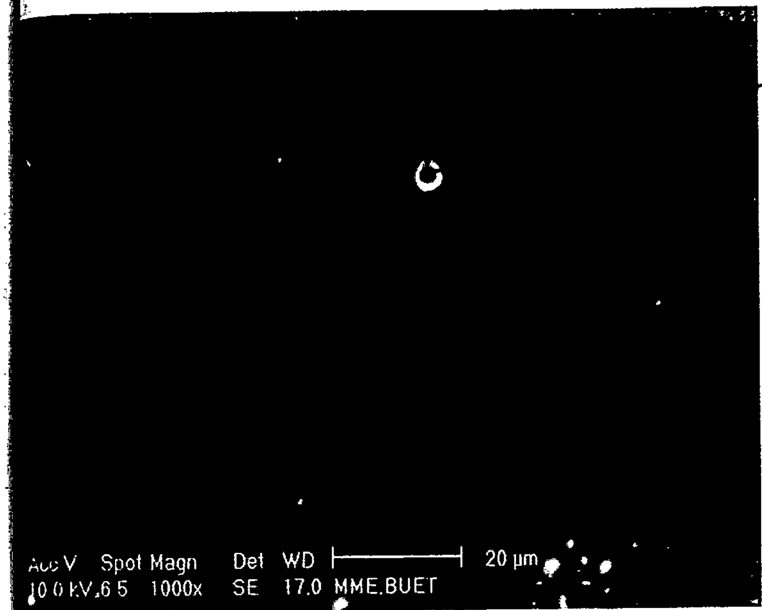
Element	% of wt	Comments
Fe	23.84	May be Sulphides (FeS, MnS)
Mn	35.46	
Si	0.39	
Al	0.32	
O	1.94	
S	26.17	
C	11.88	

Fig: 4.19: SE image sulphide and carbide types inclusions; inclusions distinguished by EDX and depending on their shapes after rolling.

EDX results:

Element	% of wt	Comments
Fe	65.89	May be carbides (Fe_3C,Mn_3C) and some oxides
Mn	23.74	
Si	0.52	
Al	0.45	
O	1.98	
S	0.38	
C	7.11	

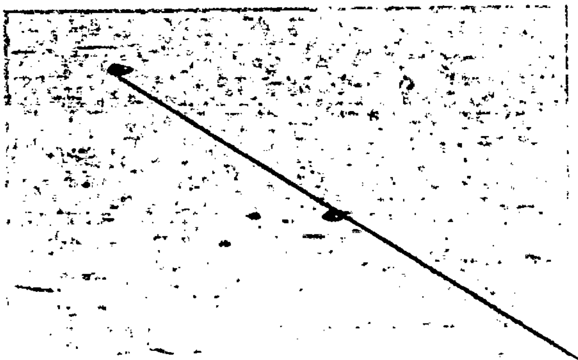
Alumina type inclusions are retaining their shape and particle size, refractory and brittle one breaks up.



EDX results:

Element	% of wt	Comments
Fe	62.72	Mainly carbides type
Mn	24.72	
Si	1.14	
Al	2.06	
O	0.00	
S	0.00	
C	9.36	

Fig: 4.20: SE image carbide types inclusion; inclusions distinguished by EDX and shape did not changed after rolling.

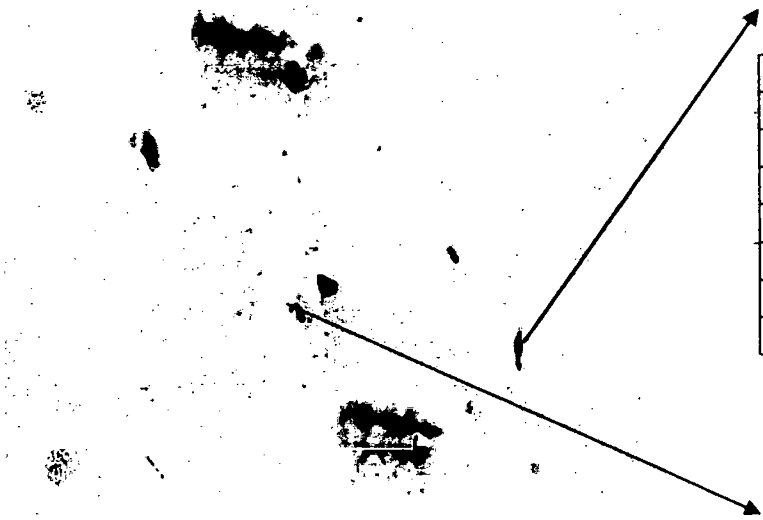


EDX results:

Element	% of wt	Comments
Fe	67.44	May be carbide and alumina types
Mn	22.23	
Si	0.28	
Al	2.38	
O	1.34	
S	0.00	
C	6.33	

Fig:4.21: SE image carbide and alumina types inclusion; inclusions distinguished by EDX and shape did not changed after rolling.

Silicate type inclusions are strung out.



EDX results:

Element	% of wt	Comments
Fe	52.18	carbide (Fe_3C, Mn_3C), silicate and alumina types
Mn	19.04	
Si	7.12	
Al	3.69	
O	0.00	
S	0.00	
C	17.94	

EDX results:

Element	% of wt	Comments
Fe	53.3	carbide (Fe_3C, Mn_3C), silicate and oxide(FeO, MnO, SiO_2 , Al_2O_3) types
Mn	24.26	
Si	1.10	
Al	0.91	
O	7.51	
S	0.00	
C	12.92	

Fig: 4.22: SE image oxides and carbides types inclusions; inclusions distinguished by EDX and depending on their shapes after rolling.

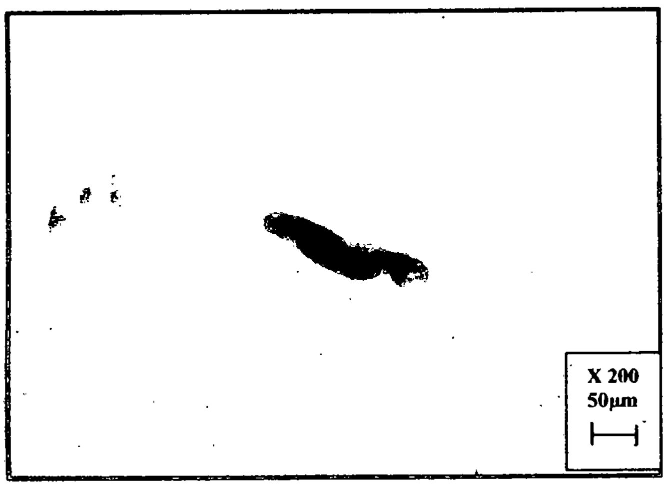


Fig: 4.23. Optical micrographs showing inclusion is elongated and rolled out to some extent with the rolling direction.

The size of the samples indicates the degree of reduction of the material from ingot or billet to deformed bars. With increasing reduction, the plastic inclusions change shape and therefore their size, whilst the harder types of inclusions are not affected by reduction.

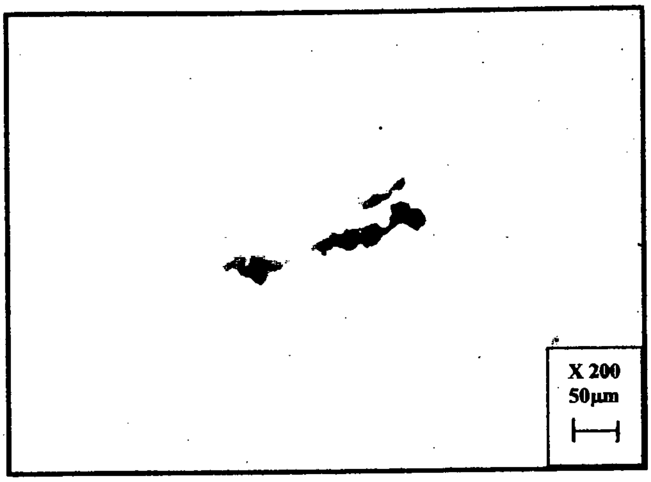


Fig:4.24. Optical micrographs showing slags in the reinforcing bars produced from pencil ingot.

A low concentration of inclusions in steel is not, by itself, the guarantee of high quality. With the total low concentration of inclusions, they may be concentrated in particular place of an ingot or casting.

Inclusions in the form of thin films located on grain boundaries are especially dangerous for steel quality. These are usually low-melting oxysulphide inclusions precipitated in liquid state during steel solidification .They weaken the intergranular bonds, especially at elevated temperatures (red shortness).

Inclusion particles with sharp edges may be also quite dangerous; these are usually high-melting inclusions, i.e. with the melting point above the temperature of molten metal.

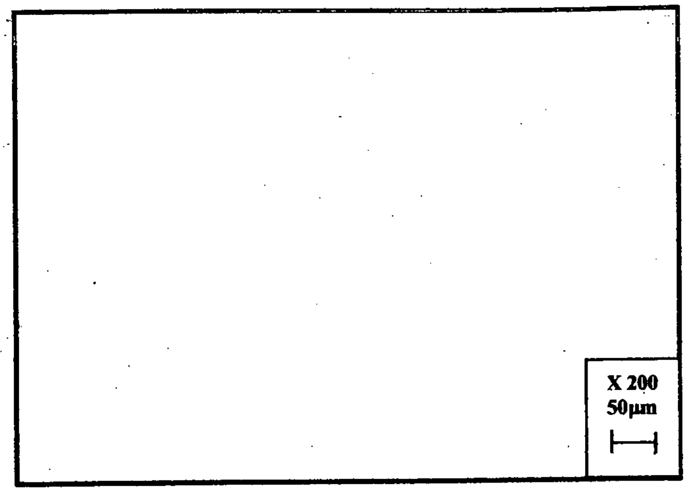


Fig: 4.25: Optical micrographs showing sharp edge inclusions in the reinforcing bar.

They often serve as stress concentrators in the metal and as sites for the beginning of fracture. If such an inclusion passes onto the surface of an article (a ball bearing, rail, etc.) it will crumble out soon and cause premature failure of the article.

Rounded off inclusion particles are considered less harmful. They are formed by substances which have a low melting point and are poorly wettable by the metal.

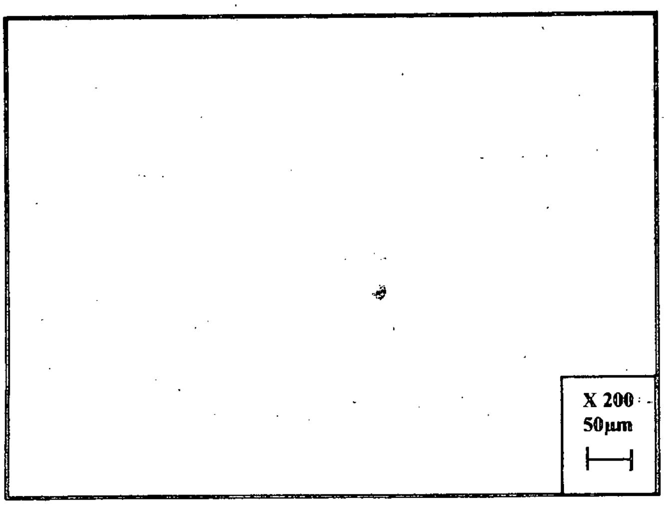


Fig: 4.26: Optical micrographs showing rounded off inclusions in the reinforcing bar.

The size and distribution of inclusions changed substantially in the subsequent process of rolling or forging. Some inclusions may form on grain boundaries during solidification and others, within grains. Some types of inclusions are disintegrated on plastic working and form chains oriented along the rolling axis; others are stretched out; still others remain unchanged, and so on. The result is that the apparent (as seen in the microscope) purity of steel from inclusions depends not only on their concentration in the metal, but also on the reduction in rolling, etc.

CHAPTER 5

SUMMARY AND CONCLUSION

During pencil ingots processing there was a little or no metallurgical control and these ingots contain different (higher in number and larger in sized) amount of inclusions and give uncertain physical and mechanical properties. Billets (Direct casting/Normal standard) were unrefined and contain slags, inclusions and inhomogeneity in structure. Final products from these billets give inferior physical properties. Billets (Quality standard) produced through proper refining (molten metal treated with synthetic slag and purging pressure and time adjusted properly) give clean and refined liquid steels or a little slags and inclusions. Finished products from these billets have better mechanical properties.

a) The reinforcing bar produced from quality standard billets (laddle refining steel) is recommended to use for structural works provided it fulfil the requirements of structural steels. The higher strength may be due to the combined effect of N_2 and less amount of inclusions but the higher ductility is mainly due to less amount and also smaller sizes of inclusions

b) The reinforcing bars produced from normal standard billets give inconsistent results and are not suitable for use as reinforcement steel for structural works although random samples may satisfy yield strength requirement.

c) Pencil ingots produced under no metallurgical control give sub-standard reinforcing bars and are not acceptable.

SUGGESTIONS FOR FURTHER STUDIES

In secondary steelmaking process, laddle refining practice with lime in the ladle, coupled with inert gas purging helps control of slag inclusions in addition to ensuring chemical and thermal homogeneity and better properties of steel. Attempts were made to establish a relationship between inclusion content and the physical properties. From this research work the followings can be suggested for further studies:

- At bottom and mid-height positions of all pencil ingots, inclusions were about the same. But at top position total numbers of inclusions were lower. The explanation for this requires further experimentation.
- Study of behaviour of inclusions during hot or cold rolling.
- Relate fracture mechanics with inclusions and establish a standard fracture behaviour of inclusions.
- Effects of inclusions on the corrosion behaviour of structural steels in different corrosive environment.
- Specify the permissible size, shape and distributions of inclusions for satisfactory different structural uses.
- Study the effects of inclusions on welding performance.

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