

REF
671.32
1995
AMI

STUDY ON THE FORMATION OF OXIDES DURING HOT ROLLING OF M.S. BILLETS

BY

MD. AMIRUL MOMIN

A thesis submitted to the Department of Metallurgical Engineering, Bangladesh University of Engineering and Technology, Dhaka, in partial fulfilment of the requirements for the Degree of Master of Science in Engineering (Metallurgical).

October, 1995



BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY, DHAKA-1000,
BANGLADESH.



CERTIFICATE

This is to certify that this research has been carried out by the author under the supervision of Dr. Ehsanul Haque, Professor, Department of Metallurgical Engineering, BUET, Dhaka and Co-supervisor Mr. Md. Abdul Mannan, Superintendent Chittagong Steel Mills Ltd. Chittagong and it has not been submitted elsewhere for the award of any other Degree or Diploma.

E. Haque
Supervisor

m. a. moinin
Signature of the Author

Mannan
Co-supervisor

The undersigned examiners appointed by the Committee of Advanced Studies and Research (CASR) recommend to the Department of Metallurgical Engineering of the Bangladesh University of Engineering and Technology, Dhaka, the acceptance of the Thesis entitled "STUDY ON THE FORMATION OF OXIDES DURING HOT ROLLING OF M.S. BILLETS" submitted by Md. Amirul Momin, B.Sc. Engr. (Metallurgical) in partial fulfilment of the requirements for the Degree of Master of Science in Engineering (Metallurgical).

1. E. Haque
Prof. Dr. Ehsanul Haque
Dept. of Met. Engg.
BUET, Dhaka.

Chairman
(Supervisor)

2. Md. Mannan
Mr. Md. Abdul Mannan
Superintendent
Chittagong Steel Mills Ltd.
Chittagong.

Member
(Co-supervisor)

3. 29/10/95
Head
Dept. of Met. Engg.
BUET, Dhaka.

Member
(Ex-officio)

4. A.S.W. Kurny
Prof. Dr. A.S.W. Kurny
Dept. of Met. Engg.
BUET, Dhaka.

Member

5. Serajul Islam
Prof. Dr. Md. Serajul Islam
256/2A, Sultangonj (Nimtali)
Rayer Bazar, Dhaka.

Member
(External)

ACKNOWLEDGEMENT

The author expresses his profound indebtedness and sincerest gratitude to the thesis supervisor Dr. Ehsanul Haque, Professor, Department of Metallurgical Engineering, BUET, for his valuable suggestions, guidance and constant encouragement in all stages of the project as well as in preparing this thesis. The author is very much grateful to the thesis co-supervisor Mr. Md. Abdul Mannan, General Manager (steel), Chittagong steel Mills Ltd., for his kind help at various stages of this research work and the helpful discussions he had with him.

The author also wishes to thank Dr. A.S.W. Kurny, Professor, Department of metallurgical Engineering, for his inspiration. Thanks are also due to Mr. Mollah Ahmed Ali, Superintendent and Chief Instructor, Machine shop for his kind help in preparing sample specimens.

Appreciation also goes to Mr. Fazlul Haque Bhuiyan, Senior Foundry instructor, Mr. Md. Lutfur Rahman, Senior Crafts instructor, Mr. Binoy Bhushan shaha, Mr. Md. Abu Taher, Accounts Assistant-cum-Typist and other staff members in the Department for their kind help in different stages of this project.

Profound gratitude and appreciations are also due to the authority of Chittagong steel Mills Ltd., for granting study leave for Undertaking this work and also for the permission to use facilities at CSM, Chittagong.

The author also extends his full hearted thanks to Engineer Mirza Md. Rafiqul Islam, Managing Director, Chittagong Steel Mills Ltd., for his kind interest and full co-operation in the research work.

And above all, the author gives thanks to Almighty Allah for allowing him to complete his work.

Department of Met. Engg.
BUET, Dhaka.

MD. AMIRUL MOMIN

ABSTRACT

Chittagong steel Mills Ltd. (CSM) is the biggest steel Mill in Bangladesh. The heavy plate mill of the CSM has been running well below its rated capacity because of producing defective plates. Recently the rejection of about eighty percent plates produced in this section has created an alarming situation. It was found that out of the various types of defects, a high percentage of rejection was mainly due to scale pitting.

The present work describes a study on the formation and growth behaviour of oxide scales and their adherence characteristics during heating prior to rolling. The work is based on the data collected from the C.S.M. as well as experiments carried out in the BUET.

The formation of scales takes place constantly at high temperature and it reforms on descaling. If the iron oxides are adherent during the heating and rolling the possibility of rolled in scale is very high. So the probability of having the problem of scale pitting consistently or its recurring depends on the nature of scale adherence, which in turn depends on the atmospheric condition inside the furnace, surface condition of the ingot, descaling procedure, etc. It has been found that the scale adherence is much influenced by the improper fuel/air ratio in the heating furnace, a rough ingot surface, improper descaling method and some other contributing factors.

To overcome the scale pitting problems, optimum conditions have been established. Ingot surface should have a good finish; the fuel/ air ratio should be oxidizing (1:10) and a proper descaling method should be applied.

TABLE OF CONTENTS

CHAPTER-1	Page No.
1.1. Introduction	1
1.2. Rolling process	2
1.2.1. Preperation of rolling process	3
1.2.2. Rolling passes and drafts	4
1.2.3. Heating before rolling operation	5
1.3. Brief introduction of heavy plate mill problems	5
1.4. Objectives of the research	8
 CHAPTER-2 LITERATURE REVIEW:	
 2.1. Nature of scale formation during heating and oxidation loss in steel industry	 9
2.2. Oxidation mechanism	14
2.3. Mechanism of growth of oxide film	22
2.4. Oxidation behaviour of iron	23
2.5. The mechanism of scale formation on iron at high temperature	 25
2.6 Mechanism for the formation of voids at metal scale interface	 28
2.7. Condition for the initiation of oxide scale cracking and spallation	 29
 CHAPTER-3 EXPERIMENTAL DETAILS:	
 3.1. Introduction	 32
3.2. First phase of work	33
3.2.1. Sample preperation	33
3.2.2. Preperation of the furnace	33
3.2.3. Heating of the sample	34
3.2.4. Experimental procedures	35
3.3. Second phase of work	37
3.3.1. Experimental procedures	38

CHAPTER-4 EXPERIMENTAL RESULTS

4.1. First phase experimental results	39
4.2. Second phase experimental results	54

CHAPTER-5 DISCUSSION ON EXPERIMENTAL WORKS

5.1. Discussion on the 1st phase experimental work	63
5.2. Discussion on the 2nd phase experimental work	71

CHAPTER-6 CONCLUSIONS

6.1. Conclusions drawn from the present work	76
6.2. Remedial measures	77
6.3. Suggestions for further work	77

APPENDIX	79
----------	----

REFERENCES:	82
-------------	----

CHAPTER-1



1.1. INTRODUCTION:

Iron and steel industry is one of the basic industry of a country. The development and infrastructure of a country is very much dependent on iron and steel industry. The importance of iron and steel can be felt in any type of development work. It is said that per capita consumption of steel indicates the level of development of a country. But unfortunately per capita consumption of steel in Bangladesh is very insignificant which shows a very dismal picture of our development position, although consumption of steel is increasing.

Bangladesh has started with a poor base of manufacturing activities specially in primary iron and steel production. Indeed she made her first entry into this field as late as 1955-56, when the first re-rolling mill was setup in the country. At the initial stage Bangladesh had no facilities to produce steel ingot. At that time semifinished products (billet/bar etc) were imported and subsequently rolled out in rolling mills. But these rolling facilities were also limited in scale. With the increasing demand of steel products, people both in the private and in the public sectors were thought about the production of steel ingot. To meet up the increasing demand of finished products e.g., rod, bar, angle etc an integrated steel plant namely Chittagong Steel Mills Ltd.(CSM Ltd.) was established in the public sector.

CSM was setup during Pakistan second five years plan (1960-65). The mill was constructed by Kobe Steel Ltd. of Japan with an investment of Taka fifty crores and went into trial production on the 1st.Feb. 1967. Initially Chittagong Steel Mills Ltd. was set up with a steel ingot making capacity of 150,000 MT. which was raised to 2,50,000 MT in 1969-70. CSM Ltd. consists of following production units and its rated capacity along with planned product is shown in appendix

1.1. and appendix 1.2 show the actual production of CSM over the years. Mean while a good number of mini steel plants were also established in the private sector.

Many of these mills are facing problems due to formation of scale during rerolling. This often results in enormous production loss. The problem is particularly noticed in the heavy plate mill of the CSM.

This study is undertaken to identify the causes of undue scale formation leading to defective products and to find out the remedial measures. This will help to increase the production and reduce its cost. Thus the plant will be better suited to compete in the free market.

1.2. ROLLING PROCESS:

Direct casting of steel into smaller cross-section is not feasible in most cases. So ingots are cast and then they are rolled into a suitable form e.g. slab bloom or billet, for further processing. These are "semifinished sections. A slab is a flat piece intended for further rolling usually into strip, sheet or plate. Slabe has a rectangular cross section at area minimum width of 10" and an minimum thickness $3/2$ " however the width is always 3 or more times of the thickness which may be as much as 15". A bloom has a square cross section with a minimum size of 6" x 6" being intended for bar products. A billet is similar to bloom with a smaller cross-section from $1/2$ " upto the size of a bloom.

Rolling gives the following advantages:

a) Improvement of structural quality by breaking up the weak cast structure and converting it into a more uniform wrought one as the cross section is gradually reduced by working.

b) Improvement of surface quality by scaling off and rolling out of minor surface defects.

c) Improvement of material quality by welding of blow holes during rolling.

d) A good product is obtained by cropping off of piped, heavily segregated ingot tops and lowermost bottom parts, after the semifinished section is rolled.

In order to achieve these objectives of a rolling mill a number of steps are followed.

1.2.1. PREPARATION OF ROLLING PROCESS:

The initial materials are prepared to be further rolled by removing various surface flaws from previous manufacturing process. This is called surface conditioning and is an important operation, especially in the production of quality steels intended for the manufacture of vital components in various fields of industry. The ingots are conditioned after cooling or when hot depending upon the type of defect and grade of steel. Surface defects include scales, hairline cracks, rolling laps, nonmetallic inclusions, scratches etc. All of these flaws are revealed by inspection.

Chipping operations to remove surface flaws are performed by pneumatic hammers. Swing frame grinders are also used for surface conditioning operations. Grinder have a low production capacity in surface conditioning operations and the cost is higher than for chipping. At present flame scarfing surface conditioning is gaining an ever increasing popularity. Flame scarfing is performed either by hand or in special machines. At present, almost all types of steel are torch scarfed. The production capacity of hand torch scarfing is several times higher than chipping with pneumatic hammers. Highest out put is achieved, however, in flame scarfing

machines where surface defects are removed by an allover burning of the surface layer with an oxyacetylene flame.

The preparatory operations also include stacking of the conditioning products and their controlled cooling. Stacking of the products should be organized so as to ensure uninterrupted delivery of metal to the rolling mills in accordance with the rolling schedule.

1.2.2. ROLLING PASSES AND DRAFTS

Primary rolling is always carried out in individual "passes" back and forth through the reversing mill with space between the pair of rolls decreasing for each pass¹. This reduction in each pass is called "draft". Hence rolling results in a progressive reduction of a cross-sectional area of the section rolled, and a corresponding increase of its length.

The ingot is a casting and is therefore not strong. Hence the first few passes must be carried out with light drafts (reduction) and all four sides of the ingot should be rolled. Thus rolling starts almost invariably by one or two passes on one side, then the ingot is turned on its other side by the manipulators and this side is rolled.

Blooms are rolled from ingots of a nearly square cross section, hence the amount of rollout required from all ingot sides is uniform. This requires frequent turning over and pass practices 2-2-4-4-2-2-1 are common. Each dash represents a "turning". Heavy drafting such as 4-4-2-1 results in cracks.

Slabs are rolled differently, since the degree of thickness reduction on the flat side is high, a high amount of rolling on the slab face is required. However, unless slab edges are supported or occasionally rolled, they would tear or be squeezed together

producing an edge defect called "underfill". Modern, universal slabbing mills also have a pair of vertical rolls, which can roll the ingot edge while the face is being rolled. To ensure the best plastic properties of metal and high quality of final rolled products steels to be rolled are always heated prior to rolling.

1.2.3. HEATING BEFORE ROLLING OPERATION:

The heating of metal (ingots, billets) prior to hot rolling is one of the main processing operations. Properly chosen conditions and temperature ranges of heating make it possible to obtain required plasticity and proper metal structure. On heating, the metal structure becomes single phased and diffusion causes redistribution of impurities and levelling out of the metal composition (homogenization).

Overheating, burning, grain growth, fusing, decarburization, carburization, increased iron loss or some such defects are inevitable if the heating conditions are wrong. Improper heating may also result in the formation of cracks, fissures and scabs (due to the opening of subsurface blowholes). Heating is always of course accompanied by oxidation and scaling and its extent is sometimes unacceptable.

The heating process is dependent on the following principal parameters, the temperature range, duration and the rate of heating.

In many cases, the heating time and temperature for a certain steel is established on the basis of previous experience.

1.3 BRIEF INTRODUCTION OF HEAVY PLATE MILL PROBLEMS:

The heavy plate mill of CSM is running below the rated capacity level, lower than world average, resulting in high production cost. This is primarily caused by the facilities not being properly used.

Loss in operation time, leads to large maintenance expenses out of the reduced productivity. Decreased yields are caused by the unwanted defects of scale pitting. When there is excessive oxidation thick and adherent scales are formed during soaking prior to rolling. The failure to remove them through the descaling process, results in scale pitting on the plates during plate formation (rolling) from slabs. Thus a considerable quantity of finished steel products are rejected due to the scale pitting. This results in a higher production cost. Appendix 1.3. Shows a typical heavy plate mill performance. The highest as of today was in 1980-81 when 15,266 MT of steel plates were produced through it constituted only 25% of the rated capacity.

After a thorough investigation it was found that the plates were rejected due to the following types of defects².

1. Scale pitting
2. Non metallic inclusions (brick mark)
3. Pipe lamination
4. Cracks (Transverse and longitudinal)
5. Fissures
6. Under/over thickness
7. Blistering
8. Short breadth

Normally 80-90% of the plates produced from the CSM slabs are rejected, and 60-70% of the plates manufactured from the imported slabs are also rejected due to the above reasons.

A statistical data produced by a team from the Bangladesh University of Engineering and Technology on checking the records for more than 400 plates produced from local ingots and about 150 from foreign ingots are shown in Table 1.1. It reveals the percentages of defective plates due to individual types of fault.

Table - 1.1 : Heavy plate mill performance

Sl No	Types of defects observed	% of defective plates produced from	
		C.S.M. slab	Imported slab
1	Scale pitting	55	52
2	Non metallic inclusions (Brick mark)	40	20
3	crack (longitudinal)	6	Nil
4	crack (Transverse)	18	Nil
5	Pipe lamination	Not known	Nil
6	Fissures	8	Nil
7	under/over thickness	4	4
8	Blistering	1.5	Nil
9	Short breadth	2	2

It was observed that some plates were being rejected due to the simultaneous occurrence of a number of defects in them. On the other hand some defective plates were not being totally rejected and a considerable portion of it was being found to be acceptable. From the statistical analysis of the defects it is apparent that the high percentage of rejection was mainly due to two factors, viz scale pitting and non metallic inclusions. Rejection due to cracks was relatively low and that due to the pipe lamination could not be ascertained. No rejection due to improper chemical composition was reported. The origin of defects may be identified as having been caused mainly in two shops as follows:

1. Casting (Melting shop)

- a) Non metallic inclusions.
- b) Pipe lamination
- c) Cracks
- d) Fissures

2. Heavy plate mill:

- a) Scale pitting
- b) Cracks
- c) under/over thickness.

This research work is intended to look into the factors related to excessive scale formation and scale pitting.

1.4. OBJECTIVES OF THE RESEARCH:

The objectives of this research work is to study the following.

(a) The effects of three variables time, temperature and surrounding atmosphere on the nature of the scale formed.

(b) To suggest remedial measures against improper scale formation and subsequent defects (scale pitting) during hot working.

CHAPTER-2 LITERATURE REVIEW

2.1. NATURE OF SCALE FORMATION DURING HEATING AND OXIDATION LOSS IN AN STEEL INDUSTRY

Various types of scale are created by the chemical action of the steel and oxidizing compounds during the heating and rolling operation. The scale is always formed in three distinct layers, as shown in Figure 1. The growth of these multi-layered oxides develops chemically³. The thickness and distribution of the amount in each layer vary considerably. Sketch (1) shows a typical scale developed in a reheating furnace¹.

Oxidation¹ is explained by the two way diffusion of oxidizing gases and metal ions. The most typical furnace scale consists of three layers, Viz. inner (FeO), intermediate (Fe_3O_4) and outer (Fe_2O_3). The outer layer is the thinnest (10%). The scale layer on alloy steel ingots, apart from base metal oxides, also includes oxides of some alloying additions.

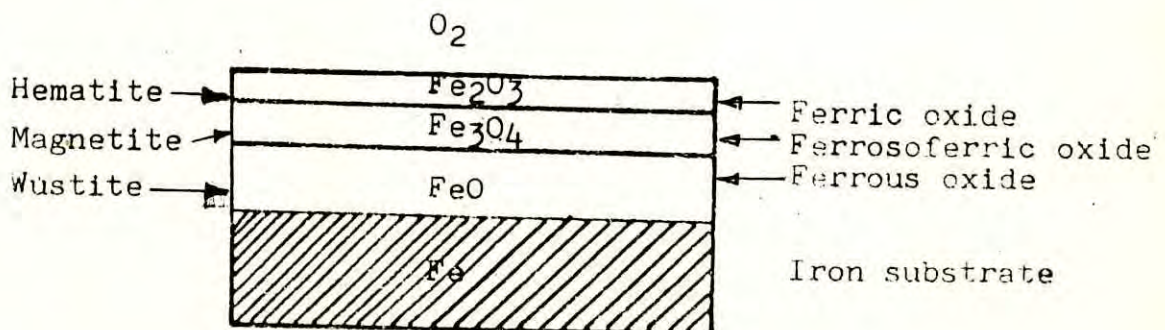


Fig. 1 : The complex oxide scale formed on iron at high temperatures.

The scale toughness depends on the chemical composition of metal and on the heating conditions. The scale layer on chromium-nickel and copper steel ingots firmly adheres to the metal.

In the heating and rolling of many different steel grades the scale will consist of many different oxides.

Table - 2.1. Showing element in the oxides scale.

<u>Element</u> %	<u>Slab</u>	<u>Scale</u>
C	.093	.000
S	.005	.000
Mn	.930	1.040
P	.011	.023
Si	.180	.430
Al	.023	.000
Cu	.220	.130
Ni	.180	.093
Cr	.040	.000
V	.044	.000
Nb	.010	.000

Table - 2.1 shows the chemical composition of a slab and of the scale that was formed on that slab in the reheating furnace. The chemical compositions of the scale vary considerably⁴ from place to place in the scale. Some elements (C, S, and Al) seem to be absent completely. Some elements (Mn, P, and Si) migrate to the scale. Other elements (Cu and Ni) seem to stay in the steel surface and

finally, Cr, V and Nb remain totally in the steel. The variations at the steel scale interface must depend considerably on the chemistry. These variations have a direct effect on the bonding strength of the interfaces and therefore will affect descaling requirements quite significantly.

The chemical action, time, temperature, and atmosphere produce scale particles of significantly different physical characteristics. Figure 2 shows schematically the surface topography of the typical scale particles. On the left is the crystalline formation produced by the chemical action. In the center the crystals are partially melted, and on the right the crystalline structure is completely melted back into a smooth surface. Enclosure (2) in the upper right represents the surface appearance between the steel and the FeO scale. This interface (c) between the FeO and the slab is like "sandpaper" or "orange peel" in appearance. The interface surfaces between the other layers (a) and (b) are very jagged interface of crystals. In the removal of scale by the descaler it is these interface that must be fractured free of the steel slab.

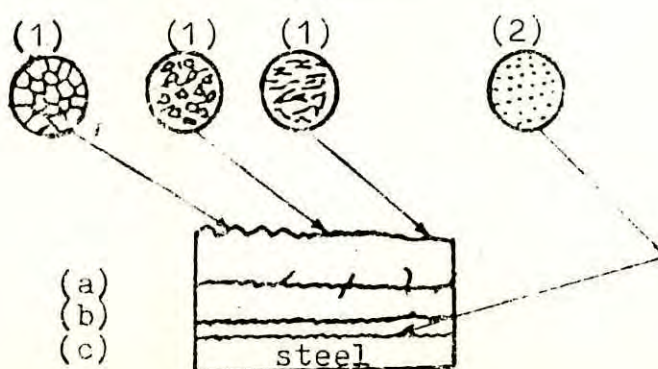


Fig. 2 : Different physical characteristics of oxide scale in iron at high temp.

Under normal circumstances the scale should break free between the FeO layer and the steel surface, and thus all the three layers are removed in one piece.

A change in surface area exposed to O_2 in the furnace will result in modifying the scale layers⁴. If the FeO layer is deprived of a steady flow of O_2 , the interface will begin to revert to Fe and FeO combination. This will totally change the interface bonding strength between the scale and steel.

The major factor in scale growth is temperature. The amount of scale growth⁴ is shown in Figure 3 where:

$$\frac{W}{A} = kt^2$$

where

W = Weight of Scale

A = Area of Sample

t = Temperature

K = Constant

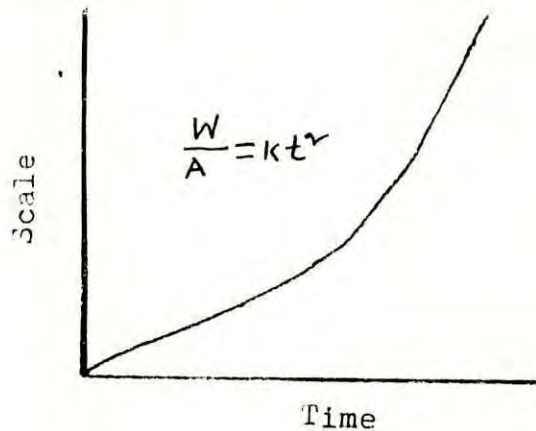


Fig. 3 : Scale growth in relation to time and temperature.

The "K" factor is also a function of temperature. At low temperatures the value is very small, and at high temperatures it is very high. In the furnaces and soaking pits the slab temperature varies considerably.

The "K" factors for each temperature and scale loss in relation to time are shown in Figure 4.

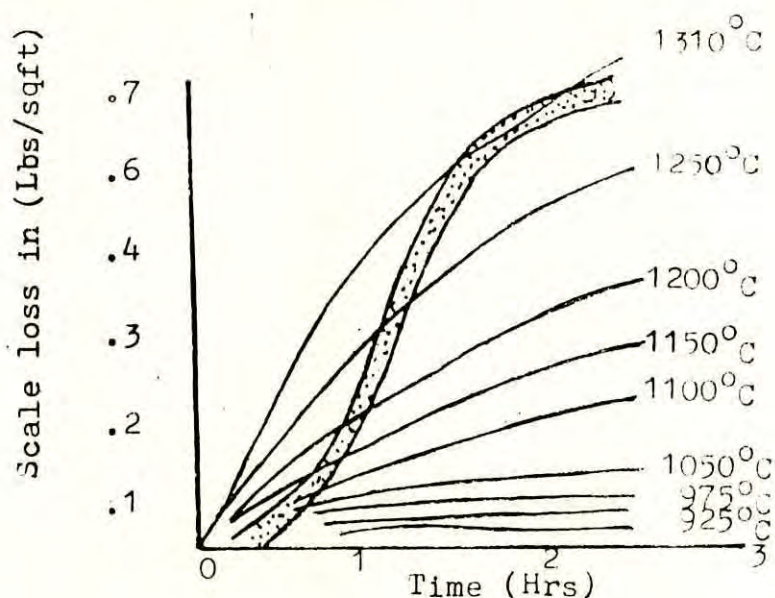


Fig. 4 : Scale loss in relation to time.

The shaded⁴ area is the approximate slab surface temperature as it passes through the furnace in about 2.5 hours. The early periods of heating will result in very little scale formation. Later in the heating and soaking zones the growth rate will increase rapidly. Most active scaling¹ takes place at 800-900°C and reaches its maximum at 1250-1350°C. So scale formation in a reheating furnace is a function of time and temperature, which is directly related to the push rate for each individual slab.

The main constituent gases of the furnace atmosphere are: oxidizing- O_2 , CO_2 , H_2O and SO_2 ; reducing- CO , H_2 and CH_4 and the neutral N_2 . Scale is also formed in a reducing atmosphere, but in this case it is more tough.

The amount of scale formed depends on the temperature, holding time at high temperature, heating rate and the furnace atmosphere. Scale formation is more intensive at temperatures³ above 900°-1000°C. The

amount of scale increases with the holding time at high temperature. The higher the heating rate the less the scale loss will be. Scale loss is also reduced if the furnace gases burn with a minimum of surplus air and to the fullest extent; the pressure should also be positive in the furnace.

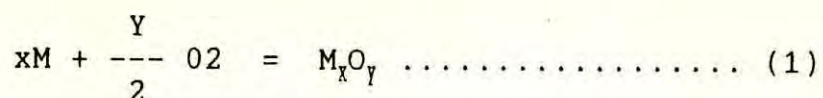
The ratio of the surface area of the metal to its volume also influences scale formation. The higher this ratio, the more scale is formed. This factor is of particular importance in heating sheet bar and sheet steel which have a large surface. Such items are heated to lower temperatures (800-900°C) to reduce scale loss.

Scale formation in heating is one large source of metal loss. A scale loss of 1 to 2 percent of the metal by weight may be expected upon normal operation of the heating facilities. The loss may reach 4 to 9 per cent if the facilities are not operating properly. Since the metal is reheated several times in rolling from the ingot to the finished product an average scale loss of 3 to 4 per cent is quite probable³. Scale formation is also a source of rolling defects; a rough surface is obtained if the scale is rolled into the metal.

2.2. OXIDATION MECHANISM⁵

To understand the causes and mechanism of intensive scale formation and scale pitting a theoretical knowledge about oxidation mechanism is necessary. This chapter deals with the relevant information about oxidation mechanism.

The reaction of metal with air or pure oxygen to form an oxide on the metal surface constitutes an important branch of corrosion. The oxidation of metals is a chemical reaction which can be represented by the equation.



where M = metals x & y = suitable numerical values

The oxygen atoms gain electrons to become oxygen ions and a stable oxide results, but such oxidation can only take place spontaneously if a decrease in free energy of the system occurs. The oxides of most metals are thermodynamically stable over wide ranges of temperature. However, silver forms only an oxide film below 180°C and gold is not oxidized at all, but at room temperature nearly all metals exposed to the air carry an invisible film of oxide.

Although the oxides are more stable at low temperatures, the rates of oxidation of metals in these temperature ranges are generally low. At higher temperatures, where the free energy decrease is smaller, the reaction is kinetically more favoured and rates of oxidation are considerably greater. The films are invisible when very thin, but over certain ranges of thickness of the oxide, interference colors can be produced. As the film becomes increasing thick, the colors disappear and a thick layer of oxide (which may flake off the metal on cooling) is obtained. If the metal strip is heated for long time a flaky oxide film is formed which may detach itself from the metal base during the oxidation or more probably, when the specimen cools down.

As an oxide is formed between a metal and its gaseous environment, a barrier develops through which reactants must pass if the oxide

film is to continue thickening. The rate of reaction may be governed by :

- a) The rate of transport of reactants through the oxide or,
- b) The rate of oxygen supply to the outer oxide surface, or,
- c) The rate at which the oxygen and metal react to give oxide.

If the rate of diffusion of the reactants through the oxide film is the slow step in the oxidation and so controls the speed of reaction, the oxidation rate will decrease as the thickness of the film increases.

Let y be the thickness of an oxide film at time t , and let it be increased to $y+dy$ in time $t+dt$. Then the rate of growth varies

inversely as the thickness i.e.

$$\frac{dy}{dt} = \frac{1}{y}$$

$$\text{or, } ydy = k_1 dt$$

$$y^2 = 2k_1 t + K_2$$

where K_1 and K_2 are temperature dependent constants. This is known as the parabolic law of oxidation.

The other simple oxidation laws are the logarithmic and rectilinear laws, which can be represented by the following equations:

$$Y = K_3 \log (K_4 t + K_5)$$

$$Y = K_6 t + K_7$$

The constants are all temperature dependent. The logarithmic law applies to metals which oxidize rapidly at first and then slowly, the film thickness becoming virtually constant. The rectilinear law shows that oxidation continues at an undiminished rate with time even though the oxide film is thickening.

The three laws are illustrated schematically in Fig. 5.

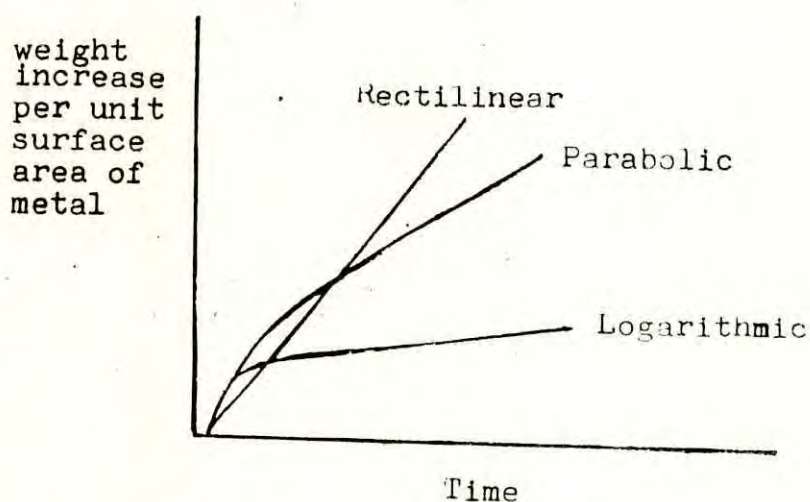


Fig. 5 : An illustration of the three main oxidation laws.

The parabolic law is frequently encountered in oxidation process of iron at temperatures above 200°C and copper at 800°C. Copper at 500°C shows a broken parabola Fig. 6. The logarithmic law applies to iron at temperature below 200°C, zinc below 400°C and aluminum at room temperature. The rectilinear law has been demonstrated for

oxidation of impure calcium (500°C), titanium ($650\text{--}950^{\circ}\text{C}$) and iron ($>900^{\circ}\text{C}$).

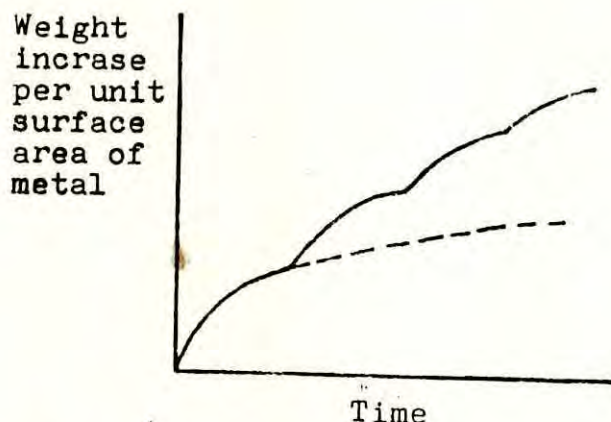


Fig. 6 : The broken parabola obtained on oxidizing copper at 500°C the dotted line indicates the probable oxidation curve of the film did not take place.

Originally it was suggested that metals could be expected to obey the parabolic oxidation law if the volume of oxide formed exceeded the volume of metal destroyed in producing it. In these circumstances, a compact oxide film offering relatively impermeable barrier between the gas and metal would result. If the contrary holds, that is the oxide volume is less than the original volume of metal, then the oxide layer can only cover the metal surface. If it is in tension, such tension will tend to crack the film and expose virgin metal at the base of the cracks and oxygen can pass directly through the non protective film. On this basis, it was predicted that Na, K, Ca and Mg should oxidize according to a rectilinear law and Al, Fe, Ni, Cu and Zn for instance would produce protective

oxide films. However, now known that a metal (e.g. Fe Iron) can behave in accordance with different laws over different ranges of temperature. In fact several of these metals can follow the logarithmic law of oxidation although no allowance was made for this.

In the rectilinear type of oxidation probably a very thin protective layer of oxide is first formed, but the stresses within the oxide become so great that when the oxide reaches a certain thickness it breaks down and the metal surface is exposed to the atmosphere. More oxide is then produced in contact with the metal, and after a similar time interval, the film is again disrupted so the process goes on a series of short periods of oxidation. Each period being followed by the break down of the film. The net result of such behavior is to produce an oxidation curve which is virtually a straight line Fig.7.

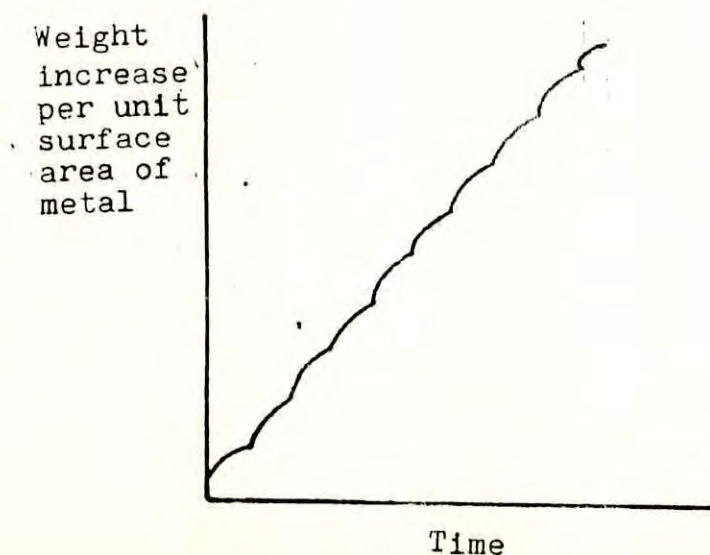


Fig. 7 : The rectilinear law of oxidation.

The broken parabola shown for copper oxidized at 500°C can be explained similarly. The oxide film reaches certain thickness at which it breaks down and so the corrosion rate speeds up again. At 800°C, where a smooth parabola is obtained the oxide is more plastic and is able to deform and reduce any stresses which have been generated within itself.

The rectilinear law⁵ probably holds good in the opening stages of oxidation of a metal which subsequently follows a different law. Thus the equation

$$\frac{dy}{dt} = K_1/y$$

indicates an infinite rate of thickening for $Y = 0$, but at the start of oxidation of a clean metal surface, the rate of reaction of metal and oxygen and not the rate of diffusion of reactants is the controlling factor. The rectilinear law appears to be followed at high temperatures of oxidation for many metals and also at lower temperatures provided the oxygen pressure is low, i.e. the gas is used up as fast as it arrives at the surface and so the gas pressure controls the rate of reaction.

Some metals obey the rectilinear law because the oxide formed is volatile and consequently has no protective capacity. If the pressure of the oxygen in the gas phase is less than the dissociation pressure of the oxide at the preboiling temperature then destruction of the metal can be prevented. This is of course, a perfectly general observation, namely that all metals can be prevented from oxidation if the oxygen pressure is made sufficiently low.

Probably the logarithmic law applies to metals having oxide films which carry cavities within themselves or which have broken away from the metal substrate⁵ Fig. 8. The blisters or cavities being formed by the stresses generated within the oxide during growth. As cavities form, the areas available for direct passage of the reactants through the film decreases and consequently the oxidation rate will fall off. The logarithmic law is obeyed by many metals at low temperatures where it may be expected that the oxide is rather brittle, cavities of this type have recently been observed by means of an electron microscope in an oxide film on tin.

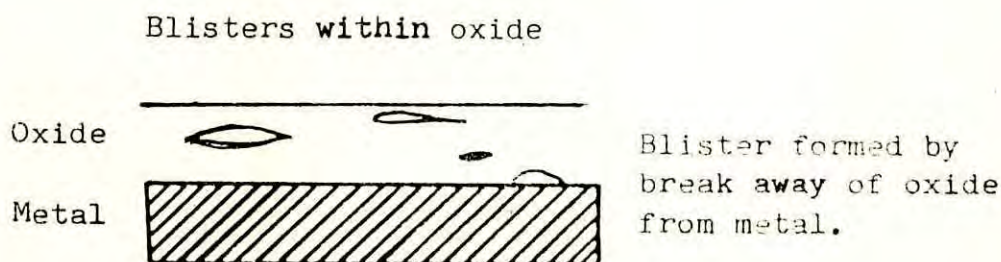


Fig. 8 : Possible types of blisters which may be present in an oxide film thickening in accordance with the logarithmic law.

There are several other laws of oxidation including the cubic law and the inverse logarithmic law. These laws may be represented as.

$$Y^3 = K_8 t + K_9$$

$$\frac{1}{Y} = K_{10} - K_{11} \log (K_{12} + t)$$

The cubic law applies to zirconium and the inverse logarithmic law to the later stages of oxidation of aluminum in humid oxygen.

2.3. MECHANISM OF GROWTH OF OXIDE FILM

There is a general agreement that the formation of an oxide layer is a diffusion process. The theoretical treatment of Wagner permits evaluation of the rate constant of oxidation from of certain crystal properties⁶. usually these values are not known so that it is still necessary to actually run oxidation tests and measures the rate. However, several systems have been investigated sufficiently to demonstrate the Wagners treatment to be correct. The oxides are composed of ionic crystals in which the components (anions cations and electrons) diffuse as charged particles.

Diffusion across the oxide layer forming on a pure metal requires a concentration gradient. This means that the oxide must vary from the stoichiometric composition even though no variation is detectable by existing analytical means.

The mechanism of diffusion of the reactants through an oxide film must now be considered, for if this does not take place oxides would presumably stop growing after the first layer of molecular thickness had been formed. Formerly it was assumed that oxidation involved the movement of oxygen towards the metal, but experiments⁶ on iron did not support this. A piece of clean abraded iron was taken and on it was placed a small patch of chromic oxide to act as a marker. Having heated the iron at a high temperature for a time it was found that the chromic oxide was situated at the boundary

between the iron oxide and the metal and not on top of the oxide. This indicates that growth of the film has taken place by migration of metal outward rather than oxygen inward.

2.4. OXIDATION BEHAVIOR OF IRON

The oxidation behavior of Fe and Fe alloys is normally discussed in terms of the migration of cations and electrons through defective oxides⁷. On this basis an explanation has been offered for the parabolic oxidation of Fe. It is considered however, that this simple mechanistic approach (i.e. the Wagner oxidation mechanism) whilst in itself correct, has a limited scope.

The oxidation and scaling of iron and its alloys (and other alloy systems) is known to be affected, sometimes markedly, by a number of experimental variables. These includes (a) the oxidizing atmosphere (b) the gas flow rate (c) surface preparation and pre-treatment and (d) the temperature. There are numerous examples in the literature of oxidation studies performed on nominally identical samples of steel under supposedly similar condition which have given widely differing results.

The investigation⁷ of the effect of the flow rate of the oxidizing atmosphere has produced surprisingly wide difference in results. Some authors have found that the oxidation rate increases with increase in gas flow rate upto a critical velocity and there after

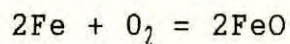
no further increase in the oxidation rate occurs. Other workers have observed a peak in the oxidation rate gas velocity curve. The peak shifting to higher gas velocities with increasing temperature. Still other workers have observed no change in oxidation rate with increase in gas flow rate.

The effect of changes in the oxidizing atmosphere upon oxidation behavior is clearer. It has been found, for example, that both the rate and the mechanism of oxidation of Fe and its low alloys differs markedly in O_2 , air, CO_2 and steam. The presence of very small amounts of gaseous impurities in any particular atmosphere are also known to have a Pronounced effect upon the oxidation of Fe and its alloys.

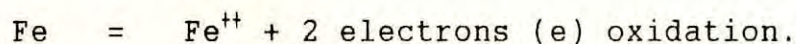
Several workers have been concerned with the effects of surface finish such work has been primarily concerned with specimens that have received the same preparation treatment upon all faces and edges, certain workers however have unfortunately polished only one face of the specimen whilst the other face remained in an as ground condition. It is known that radically different oxidation rates are possible with specimens finished in different ways, where as specimens prepared in an identical manner produce comparable results.

2.5. THE MECHANISM OF SCALE FORMATION ON IRON AT HIGH TEMPERATURE:

When iron is heated in an atmosphere containing available oxygen it becomes coated with a layer of black oxide scale FeO.



The above equation is only a simplified way of expressing the reaction. In fact atoms of iron have been oxidized whilst atoms of oxygen have been reduced. These processes are related to a transfer of electrons from atoms of iron to atoms of oxygen.



Iron at high temperature will continue to oxidize even though coated with an oxide skin. Iron atoms at the metal interface ionize (Fig.9),

releasing electrons which travel quickly to the surface of the scale. There the electrons react with oxygen from the air forming ions, O^{--} .

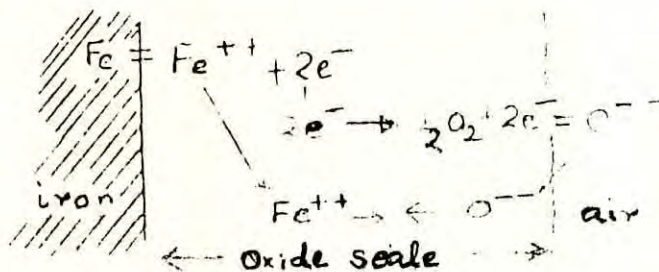


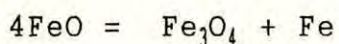
Fig. 9: The mechanism of oxidation or 'dry' corrosion

These oxide ions are then attracted towards the oppositely charged Fe^{++} ions. Although the equilibrium composition of the oxide skin is $Fe^{++} O^{--}$, that near the metal surface will contain an excess of Fe^{++} ions and that adjacent to the atmosphere will contain more oxygen ions. The rate at which new ions form depends upon their mobilities within the oxide film. That is, new Fe^{++} ions will form as existing ones diffuse away from the metal surface towards O^{--} ions. The more rapidly Fe^{++} ions move away, the more rapidly will iron atoms ionize to produce new Fe^{++} ions. Prolonged heating of large steel ingots for hot rolling produces a very thick layer of scale. The concentration gradients of Fe^{++} and O^{--} ions within this layer are often so great that three separate crystalline zones are found in layers.

From investigation⁵ it is now well established that the high

temperature oxidation of Fe leads to the formation of a two layered scale of Fe_3O_4 and Fe_2O_3 upto about 570°C and a three layered scale of FeO (wustite) Fe_3O_4 magnetite and Fe_2O_3 , hematite at higher temperatures.

Metals possessing more than one valency state (e.g. Cu and Fe) can form complex layers of oxide, containing oxides of several composition. Thus iron oxide at temperatures $> 600^\circ\text{C}$ is covered with an oxide scale consisting of three layers (Fig. 1) of which the FeO layer is the thickest. As the materials cool, the lower layer of FeO decomposes to give particles of iron embeded in a matrix of magnetite.



If the iron had been heated to some temperature lower than 570°C the FeO layer would have been absent, a duplex layer of Fe_3O_4 , Fe_2O_3 being formed. At still lower temperatures, Fe_2O_3 only is found⁵.

It is observed⁸ at lower temperatures 400 to 500°C , where FeO does not exist, and in a transition range, 580 to 600°C where FeO appears only after an incubation period. 45 minutes may elapse before the Fe predictable from the iron oxygen phase diagram becomes established as a continuous layer. At temperature of 650°C , and above FeO forms presumably from zero time.

2.6. MECHANISM FOR THE FORMATION OF VOIDS AT METAL SCALE INTERFACE⁸

According to wagners concept of the model for semiconducting compounds they are non stoichiometric being either metal-deficit or metal excess. Oxides like Cu_2O , CoO , Cr_2O_3 , NiO , FeO etc are said to be metal deficit. For instance, FeO , CoO and NiO are said to be stable with compositions, represented by $\text{FeO}_{1.055-1.190}$, $\text{CoO}_{1.0017}$ $\text{NiO}_{1.005}$ respectively and the metal-oxygen ratio in these oxides varies to a certain extent with temperature.

Oxidation of iron proceeds almost entirely by the diffusion of cations through vacant cation sites in the oxide lattice to the oxide gas interface. Iron oxide will contain some vacant cation sites in the lattice and there will be an equivalent number electron defects associated with these vacancies to maintain electrical neutrality. In oxidation, each oxygen atom will effect the migration of one Fe^{2+} ion and two electrons with the formation of one vacant Fe^{2+} site, or cation defects, and two vacant electron sites, electron defects. The defects will diffuse inwards through the scale and will be deposited at the metal-scale interface. As oxidation proceeds in an outwardly growing scale, metal ions are continuously transferred from the base metal to the gas-oxide interface through the growing oxide layer. In the absence of any mechanism to annihilate such vacancies, these vacancies will coalesce to form voids at the metal-scale interface thus resulting in a loss of contact between the scale and the bulk metal.

2.7. CONDITION FOR THE INITIATION OF ADHERENT OXIDE-SCALE CRACKING AND SPALLATION

The absence of adherent conditions either by spallation or cracking of the scale will lead to less scale pitting⁹.

For spalling the oxide film must fracture internally to produce through thickness cracks and adhesion must be lost at oxide metal interface. The processes leading to oxide spallation are discussed in terms of two routes as shown in Fig. 10. These diagrams represent an oxide stressed barely in compression as a result of a downward temperature change and whose linear co-efficient of thermal expansion is less than that of the metal.

Route 1 (Fig.10) is pertinent to the case where the oxide metal interface is more resistant to rupture than is the oxide layer itself during the temperature change, therefore, the increasing compressive stress box with the oxide and associated strain energy will be relaxed to some extent by the formation of inclined shear cracks. At a larger temperature change ΔT ($T_2 - T_0$), the subsequent buildup of stress and strain energy is relaxed by decohesion at the oxide metal interface with the release of a spall particle.

ROUTE II (Fig.10) considers the situation where the interface has a lower strength than the oxide layer. Here decohesion first occurs

Initiation of oxide-scale cracking and spallation

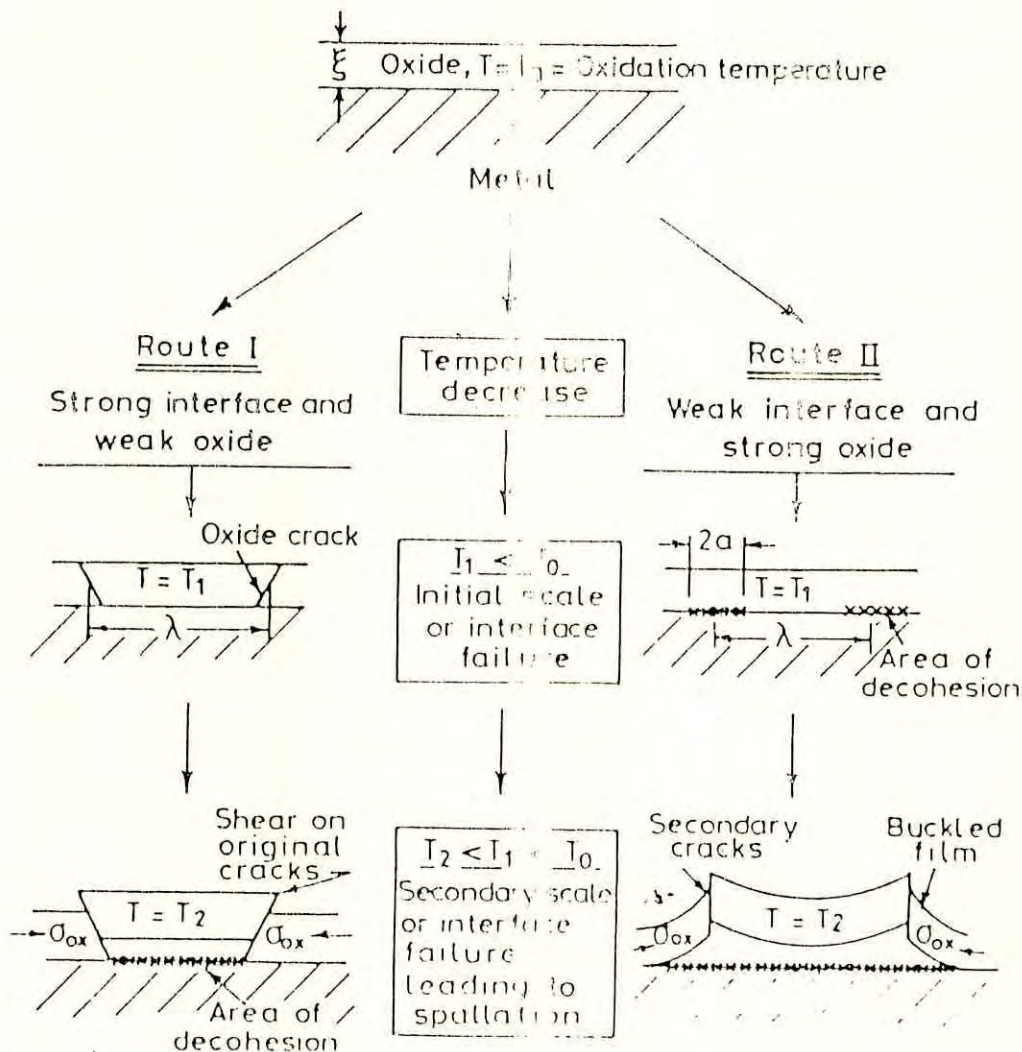


FIG. 10 Schematic diagram showing two routes leading to oxide spallation during a downward temperature change. *Route 1*: Compressive shear cracks first develop with subsequent decohesion at the oxide-metal interface. *Route 2*: Interfacial decohesion first occurs which leads to unstable buckling and decohesion.

in the absence of through thickness cracking of the oxide. This process produce progressive buckling and rumpling of the surface film as the temperature continues to decrease and the differential

strain between oxide and metal increases. At some stages relaxation of the stress and strain energy within the film occurs by the propagation of tensile cracks at the positions of maximum curvature of the surface oxide layer (Fig.10). If the oxide metal interface strength is sufficiently low, this process will again result in release of spalled oxide.

In order to initiate and develop cracks either within the oxide layer or at the oxide metal interface, it is necessary both to provide sufficient energy to offset the increase in surface energy on decohesion and also to generate sufficient stress to exceed the cohesive stress at the initiation site and subsequently at the tip of the propagation of a crack. The fact is that film cracking or loss of adhesion will result when the strain energy per unit volume of oxide contained in the layer of thickness equates to the work required either for internal cracking or for decohesion at the oxide metal interface.

CHAPTER-3 EXPERIMENTAL DETAILS

3.1. Introduction:

The research work aims to investigate the formation of scale pitting at the heavy plate mills of the SCM. It is apparent from the operation of the heavy plate mill that the scale on the ingot surface which is formed during heating prior to rolling is mainly responsible for the scale pitting. The formation of scales and its nature mainly depend on the following parameters.

- 1) Nature of the atmosphere prevailing in the furnace.
- 2) Surface condition of the ingot
- 3) Soaking temperature of the reheating furnace.
- 4) Soaking time of the ingot in the reheating furnace.

The possibility of scale pitting increases when the scale on the ingot surface is adherent and cannot be properly removed through descaling process prior to rolling for the plate formation. During rolling the scale is rolled with the ingot and as the oxide scale is harder than ingot surface it is pressed on the plate and as a result scale pitting occurs. For a clear understanding of the reasons for the formation of adherent scale and subsequent scale pit formation the research works were performed in two phases.

- 1) First phase of the work was carried out in a setup made at the BUET.
- 2) Second phase of the work was carried out in the CSM heavy plate mill.

3.2. FIRST PHASE OF WORK

The first phase of the work was to identify the parameter which is responsible for the formation of adherent scale on the steel ingot. It was performed in a small gas fired furnace by changing the various parameters, which influences the nature of the scale. This part of the experimental work was done only to observe the nature of the scale formed, i.e. whether the scales were adherent or not.

3.2.1. SAMPLE PREPARATION:

Samples for the first phase were collected from the CSM slab ingot. The chemical composition of steel used was as follows:

C%	= 0.20%
Mn	= 0.65%
Si	= 0.21%
S	= 0.040%
p	= 0.035%

Each sample was cut approximate to a size of 2" x 3" x 2", In order to study the effect of surface finish one surface of each sample was left as cast while another surface was given a plane finish. For 1st phase of the work 36 such samples were prepared. Each sample had one plane surface and another rough (as cast surface) and was marked accordingly.

3.2.2. PREPERATION OF THE FURNACE

The muffle furnace used for the experimental work, was made of a steel shell measuring 36" x 36" x 36". The furnace wall was lined with fire clay bricks. It was fitted with a moveable door and a gas burner was introduced through the back-wall. It also consisted of other accessories such as an air blower, a gas flow meter and 3 air flow meters, temperature controller etc. A combustion chamber was

provided in the lower part of the furnace. It had a chimney through which exhaust gas could be removed.

3.2.3. HEATING OF THE SAMPLES:

Experimental samples were heated by changing the following three parameters

- a) Furnace atmosphere
- b) Soaking temperature
- c) Soaking time.

a) Furnace inside Atmosphere: This is an established fact for natural gas that for full combustion, gas: air ratio should be 1: 10. For the experimental purpose fuel: air ratio considered are as follows:

- 1. Reducing atmosphere (gas: air = 1:7 & 1:9)
- 2. Neutral atmosphere (gas: air = 1:10)
- 3. Oxidizing atmosphere (gas: air = 1:12)

b) Soaking Temperature :

The ingots or billets should be heated before rolling so as to provide high plasticity and minimum resistance to deformation. It is best to use high heating temperature since in addition to the foregoing, this reduces the energy consumption in rolling, enables higher reduction to be applied and decreases the danger of breakage in the rolls or other components of the mill. On the otherhand high Soaking temperature may lead to intensive scale formation. Improper Soaking temperature may lead to the formation of adherent scales which is a probable source of rolling defects.

During experimental observations following soaking temperatures were considered.

1. 1050 - 1100°C.
- 2 1150 - 1200°C.
3. 1250 - 1300°C.

C. Soaking Time :

In the experimental observation soaking time was selected as 1 hr, 2 hrs and 3 hrs which was not selected on the basis of actual conditons at the CSM heavy plate mill soaking time, but only to study the effect of time. In CSM this soaking time varies from 10 to 130 hrs. Which was rather difficult to maintain in the labratory

3.2.4. EXPERIMENTAL PROCEDURE

The furnace was fired and the temperature was held within the required range and at the same time the desired flow of gas: air ratio was established through the burner.

The experiments were carried out in batches under reducing, neutral and oxidizing furnace atmospheres. Under each atmospheric condition three temperature ranges 1050-1100°C, 1150-1200°C and 1250-1300°C were used to study the nature of scale formation on plane and rough surfaces.

In the 1st set of experiment the oxidation of three samples No. 1, 2 and 3 was carried out at 1050 - 1100°C under reducing atmosphere (gas: air = 1:7) for 1 hr, 2 hrs and 3 hrs respectively. The oxidized specimens were removed and cooled in normal air and examined.

In the 2nd set of experiment the samples No. 4, 5 and 6 were put in the furnace at a temperature range of 1150 - 1200°C under reducing atmosphere (gas: air =1:7) oxidations of three samples were carried out for 1 hr, 2 hrs and 3 hrs respectively. The oxidized specimens were removed and cooled in normal air.

For the 3rd set of experiment samples No. 7, 8 and 9 were placed in the furnace with the same gas, air ratio of = 1:7 i.e. reducing atmosphere but the temperature was raised to 1250 - 1300°C. Oxidations of samples were carried out for 1 hr, 2 hrs and 3 hrs respectively. The oxidized specimens were removed and cooled, outside the furnace in normal air.

In the fourth set of experiment the samples No. 10, 11 and 12 were put in the furnace at a temperature range 1050-1100°C by maintaining the gas: air ratio 1:9. The oxidation of three samples were carried out by soaking for 1 hr, 2 hrs and 3 hrs respectively.

In the fifth set of experiment the oxidation of three samples No. 13, 14 and 15 was carried out at 1150-1200°C under reducing atmosphere (gas: air = 1:9). The three samples were oxidized under the above mentioned condition for 1 hr, 2 hrs and 3 hrs respectively.

In the sixth set of experiment samples No. 16, 17 and 18 were oxidized under reducing atmosphere (gas: air = 1: 9), and at temperature 1250 - 1300°C. The three samples were oxidized under this condition for 1 hr, 2 hrs and 3 hrs respectively and cooled, as usual in the normal air.

In the seventh set of experiment samples No. 19, 20 and 21, were oxidized under 1050 - 1100°C, under a neutral atmosphere of gas: air = 1: 10. Soaking time were 1 hr, 2 hrs and 3 hrs respectively. After heating for specified soaking time the samples were removed from the furnace and normalized as above.

In the 8th set of experiment samples No. 22, 23 and 24 were oxidized at temperature 1150 - 1200°C under the gas: fuel air = 1:10, soaking the samples for 1 hr, 2 hrs and 3 hrs respectively and removed from the furnace.

For the 9th set of experiment samples No. 25, 26 and 27 were oxidized at temperature $1250-1300^{\circ}\text{C}$ under the above mentioned neutral atmospheric condition of fuel: air = 1:10). Soaking the sample for 1 hr 2 hrs and 3 hrs respectively and removed from the furnace and cooled in normal air.

In the 10th set of experimental samples, No. 28, 29 and 30 were oxidized at temperature $1050 - 1100^{\circ}\text{C}$ under over oxidized fuel air ratio 1:12 soaking the sample for 1 hr 2 hrs 3 hrs respectively and removed from the furnace.

For the 11th set of experiment samples No. 31, 32 and 33 were oxidized at temperature $1150 - 1200^{\circ}\text{C}$ under over oxidized (fuel air = 1:12) flame, soaking the samples for 1 hr 2 hrs and 3 hrs respectively and cooled in normal air.

For 12th set of experiment samples No. 34, 35 and 36 were oxidized under over oxidized atmospheric condition (fuel air 1:12) at a temperature $1250 - 1300^{\circ}\text{C}$. Soaking the samples for 1 hr, 2 hrs and 3 hrs respectively and cooled in normal air.

3.3 SECOND PHASE OF WORK:

The works which were conducted in first phase provided some useful informations about the formation of adherent and non adherent scales. Second phase of works were carried out to investigate the plate condition after rolling.

It was observed that with the increase of soaking time scale adherence decreased. In case of CSM this soaking time is usually much longer in comparison to the maximum period of only 3 hrs. This leads to heavy oxidation and a thick layer of scale formation. At CSM the long soaking time is however a favourable point for the formation of non adherent scale according to the first phase experiments. It was also observed that with the increase of soaking

temperature the scale formed were comparatively less adherent.

In CSM these two parameters are maintained in a favourable way for the formation of non adherent scale. So for the second phase work these two parameters were excluded from further study. But these were recorded for proper understanding. From the first phase experimental observations it was revealed that the two important variables in the CSM responsible for the adherent scale formation are:

- 1) Reducing atmosphere
- 2) Rough ingot surface

And the probability of non adherent scale formation increased with the increase of oxidizing potential of the atmosphere used and with smoother surface finish.

On the basis of the above conclusion 2nd phase experimental works were performed to identify the effect of surface finish and atmospheric conditions on ingots using 139 samples at the CSM.

3.3.1. EXPERIMENTAL PROCEDURES

The ingots were preheated to the rolling temperature in reheating furnace before rolling. By changing furnace atmospheric condition and ingot surface condition adherent and non adherent scales were produced and then rolled as per normal procedure. After rolling the plate condition was observed to see whether it possessed scale pit or not. The following data were recorded for detailed analysis.

1. Fuel: air ratio
2. Ingot surface condition
3. Soaking temperature
4. Scale thickness
5. Degree of oxide adherence
6. Defects.

CHAPTER-4 EXPERIMENTAL RESULTS

4.1 1ST PHASE EXPERIMENTAL RESULTS:

The 1st phase experimental results are shown in table 4.1 to 4.12. In the table the adherence strength decreases with the increases of grading no. The macro photography of the oxidized samples are shown also in Fig. 4.1, to Fig. 4.36.

Table 4.1, 1st set experimental results.

Sam ple No	Gas : air	Temp °C	Soak ing hrs	Arbitrary scale adherence grading															
				As cast surface (Rough)								Plane surface							
				1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
1	1:7	1050 1100	1	✓								✓							
2	1:7	"	2	✓								✓							
3	1:7	"	3	✓								✓							

Table 4.2, 2nd set experimental results.

Sam ple No	Gas : air	Temp °C	Soak ing hrs	Arbitrary scale adherence grading															
				As cast surface (Rough)								Plane surface							
				1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
4	1:7	1150 1200	1	✓								✓							
5	1:7	"	2	✓								✓							
6	1:7	"	3	✓								✓							

Table 4.3, 3rd set experimental results.

Sam ple No	Gas : air	Temp °C	Soak ing hrs	Arbitrary scale adherence grading															
				As cast surface (Rough)								Plane surface							
				1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
7	1:7	1250 1300	1	✓								✓							
8	1:7	"	2	✓									✓						
9	1:7	"	3		✓								✓						

Table 4.4, 4th set experimental results.

Sam ple No	Gas : air	Temp °C	Soak ing hrs	Arbitrary scale adherence grading															
				As cast surface (Rough)								Plane surface							
				1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
10	1:9	1050 1100	1	✓									✓						
11	1:9	"	2		✓									✓					
12	1:9	"	3			✓								✓					

Table 4.5, 5th set experimental results.

Sam ple No	Gas : air	Temp °C	Soak ing hrs	Arbitrary scale adherence grading															
				As cast surface (Rough)								Plane surface							
				1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
13	1:9	1150 1200	1	✓									✓						
14	1:9	"	2			✓								✓					
15	1:9	"	3			✓									✓				

Table 4.6, 6th set experimental results.

[illegible]

Table 4.7, 7th set experimental results.

Sa mp le No	Gas: air	Temp °C	Soak ing hrs	Arbitrary scale adherence grading															
				As cast surface (Rough)								Plane surface							
				1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
19	1:10	1050 1100	1		✓								✓						
20	1:10	"	2			✓							✓						
21	1:10	"	3				✓								✓				

Table 4.8, 8th set experimental results.

[illegible]

Table 4.9, 9th set experimental results.

Sample No	Gas: air	Temp °C	Soaking hrs	Arbitrary scale adherence grading													
				As cast surface (Rough)								Plane surface					
				1	2	3	4	5	6	7	8	1	2	3	4	5	6
25	1:10	1250 1300	1					✓								✓	
26	1:10	"	2						✓								✓
27	1:10	"	3						✓								✓

Table 4.10, 10th set experimental results.

Sample No	Gas: air	Temp °C	Soaking hrs	Arbitrary scale adherence grading													
				As cast surface (Rough)								Plane surface					
				1	2	3	4	5	6	7	8	1	2	3	4	5	6
28	1:12	1050 1100	1			✓								✓			
29	1:12	"	2				✓								✓		
30	1:12	"	3					✓									✓

Table 4.11, 11th set experimental results.

Sample No	Gas: air	Temp °C	Soaking hrs	Arbitrary scale adherence grading													
				As cast surface (Rough)								Plane surface					
				1	2	3	4	5	6	7	8	1	2	3	4	5	6
31	1:12	1150 1200	1					✓								✓	
32	1:12	"	2						✓								✓
33	1:12	"	3							✓							✓

MACRO PHOTOGRAPHY OF
OXIDISED SAMPLE



Fig.4.1: Oxidized at 1050-1100°C
Fuel:air ratio =1:7
soaking time =1 hr.

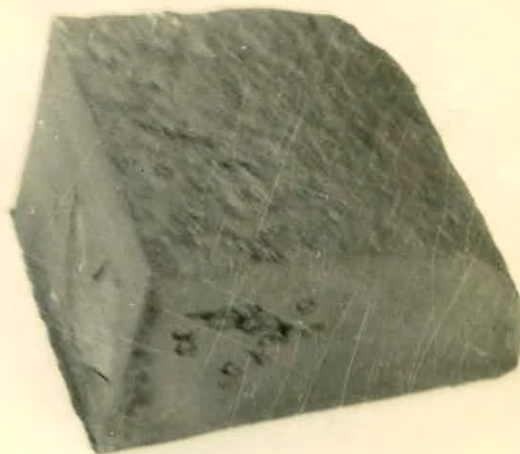


Fig.4.2: Oxidized at 1050- 1100°C
Fuel : air ratio =1:7
soaking time = 2 hrs.



Fig.4.3: Oxidized at 1050-1100°C
Fuel:air ratio =1:7
soaking time = 3 hrs.



Fig.4.4: Oxidized at 1150- 1200°C
Fuel : air ratio =1:7
soaking time = 1 hr.



Fig.4.5: Oxidized at 1150-1200°C
Fuel:air ratio =1:7
soaking time = 2 hrs.



Fig.4.6: Oxidized at 1150- 1200°C
Fuel : air ratio =1:7
soaking time = 3 hrs.



Fig.4.7: Oxidized at 1250-1300°C
Fuel:air ratio =1:7
soaking time = 1 hr.



Fig.4.8: Oxidized at 1250- 1300°C
Fuel : air ratio =1:7
soaking time = 2 hrs.



Fig.4.9: Oxidized at 1250-1300°C
Fuel:air ratio =1:7
soaking time = 3 hrs.



Fig.4.10: Oxidized at 1050- 1100°C
Fuel : air ratio =1:9
soaking time = 1 hr.



Fig.4.11: Oxidized at 1050-1100°C
Fuel:air ratio =1:9
soaking time = 2 hrs.



Fig.4.12: Oxidized at 1050- 1100°C
Fuel : air ratio =1:9
soaking time = 3 hrs.



Fig.4.13: Oxidized at 1150-1200°C
Fuel:air ratio =1:9
soaking time = 1 hr.



Fig.4.14: Oxidized at 1150- 1200°C
Fuel : air ratio =1:9
soaking time = 2 hrs.



Fig.4.15: Oxidized at 1150-1200°C
Fuel : air ratio =1:9
soaking time = 3 hrs



Fig.4.16: Oxidized at 1250- 1300
Fuel : air ratio= 1:9
soaking time = 1 hr.



Fig.4.17: Oxidized at 1250-1300°C
Fuel:air ratio =1:9
soaking time = 2 hrs.



Fig.4.18: Oxidized at 1250- 1300°C
Fuel : air ratio =1:9
soaking time = 3 hrs.



Fig.4.19: Oxidized at 1050-1100°C
Fuel:air ratio =1:10
soaking time = 1 hr.



Fig.4.20: Oxidized at 1050- 1100°C
Fuel : air ratio =1:10
soaking time = 2 hrs.



Fig.4.21: Oxidized at 1050-1100°C
Fuel:air ratio =1:10
soaking time = 3 hr.



Fig.4.22: Oxidized at 1150- 1200°C
Fuel : air ratio =1:10
soaking time = 1 hr.

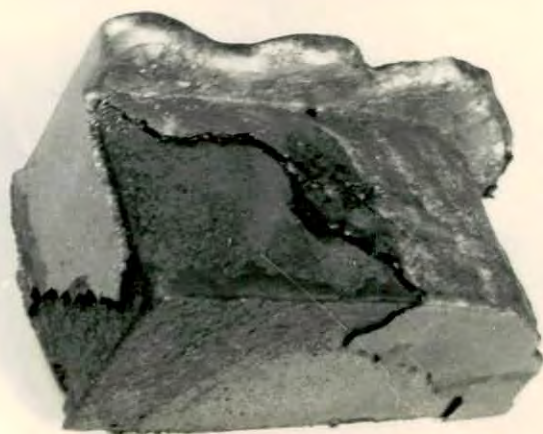


Fig.4.23: Oxidized at 1150-1200°C
Fuel:air ratio =1:10
soaking time = 2 hrs.



Fig.4.24: Oxidized at 1150- 1200°C
Fuel : air ratio =1:10
soaking time = 3 hrs.



Fig.4.25: Oxidized at 1250-1300°C
Fuel:air ratio =1:10
soaking time = 1 hr.



Fig.4.26: Oxidized at 1250- 1300°C
Fuel : air ratio =1:10
soaking time = 2 hrs.



Fig.4.27: Oxidized at 1250-1300°C
Fuel:air ratio =1:10
soaking time = 3 hrs.



Fig.4.28: Oxidized at 1050- 1100°C
Fuel : air ratio =1:12
soaking time = 1 hr.



Fig.4.29: Oxidized at 1050-1100°C
Fuel:air ratio =1:12
soaking time = 2 hrs.

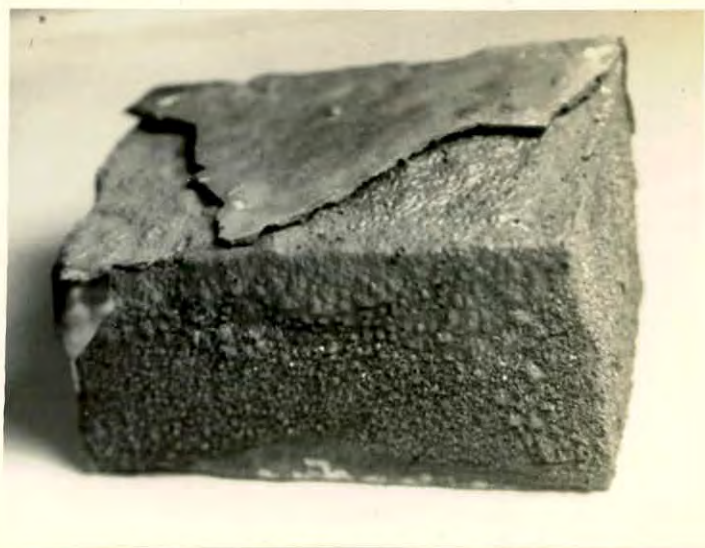


Fig.4.30: Oxidized at 1050- 1100°C
Fuel : air ratio =1:12
soaking time = 3 hrs.



Fig.4.31: Oxidized at 1150-1200°C
Fuel:air ratio =1:12
soaking time = 1 hr.

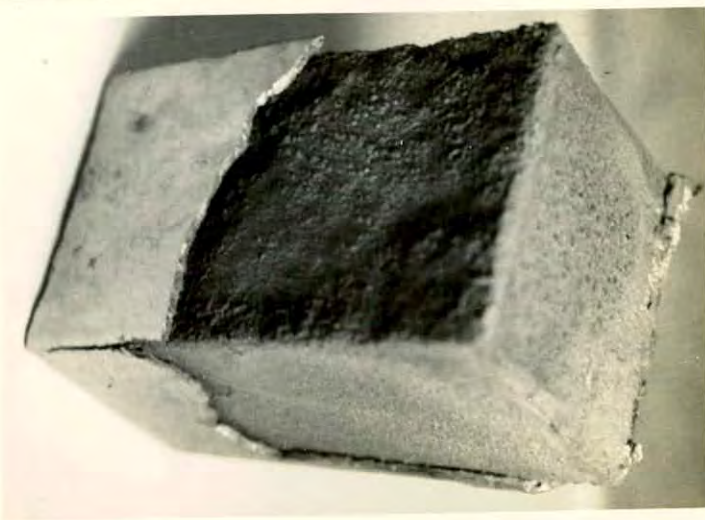


Fig.4.32: Oxidized at 1150- 1200°C
Fuel : air ratio =1:12
soaking time = 2 hrs.



Fig.4.33: Oxidized at 1150-1200°C
Fuel:air ratio =1:12
soaking time = 3 hrs.



Fig.4.34: Oxidized at 1250- 1300°C
Fuel : air ratio =1:12
soaking time = 1 hr.



Fig.4.35: Oxidized at 1250-1300°C
Fuel:air ratio =1:12
soaking time = 2 hrs.



Fig.4.36: Oxidized at 1250- 1300°C
Fuel : air ratio =1:12
soaking time = 3 hrs.

4.2. 2nd phase experimental results as obtained at the CSM are shown in table 4.14 to 4.17.

Table 4.14 : Experimental Observations under reducing atmosphere.

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
1	1:9	1300	1:12	1300	1:7	1280	Plane	6.94	F	-	
2	1:9	1310	1:10	1310	1:7	1300	"	6.74	F	Scp	
3	1:9	1310	1:10	1310	1:7	1300	"	6.79	F	-	
4	1:9	1310	1:10	1310	1:7	1300	"	-	F	-	
5	1:9	1310	1:10	1310	1:7	1300	"	-	A	Scp	
6	1:9	1300	1:10	1320	1:7	1280	"	6.94	A	-	
7	1:9	1300	1:10	1300	1:7	1280	"	-	A	-	
8	1:9	1310	1:10	1310	1:7	1270	"	-	A	Scp	
9	1:9	1310	1:10	1310	1:7	1270	"	-	F	-	
10	1:9	1310	1:10	1320	1:8	1270	"	-	F	-	
11	1:10	1310	1:9	1320	1:8	1270	Rough	6.84	A	Scp	
12	1:10	1300	1:9	1320	1:7	1270	"	-	A	Scp	
13	1:10	1300	1:10	1320	1:7	1270	"	-	A	Scp/BM	
14	1:10	1300	1:10	1310	1:7	1270	"	-	A	Scp	
15	1:8	1300	1:10	1300	1:7	1270	"	6.10	A	Scp	

The notations indicates, as follows:

F = Non adherent scale (Free)

A = Adherent scale

Scp = Scale pit

BM = Brick mark

TC = Transverse crack

LC = Longitudinal crack

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
16	1:8	1310	1:9	1310	1:7	1250	Rough	-	A	Scp	
17	1:8	1300	1:10	1300	1:7	1250	"	-	A	Scp	
18	1:8	1300	1:10	1300	1:7	1250	"	-	A	Scp	
19	1:8	1300	1:9	1300	1:7	1250	"	-	A	Scp	
20	1:8	1300	1:9	1300	1:7	1250	"	5.10	A	Scp	
21	1:8	1300	1:9	1300	1:7	1300	"	-	A	-	
22	1:8	1300	1:9	1300	1:7	1300	"	-	A	Scp	
23	1:8	1300	1:9	1300	1:7	1300	"	-	A	Scp	
24	1:8	1310	1:9	1300	1:7	1300	Plane	5.39	A	Scp	
25	1:8	1310	1:9	1300	1:7	1300	"	-	A	Scp	
26	1:9	1300	1:9	1300	1:7	1300	"	-	A	Scp	
27	1:8	1300	1:9	1300	1:7	1300	"	6.45	A	-	
28	1:8	1300	1:9	1300	1:7	1300	"	-	A	Scp	
29	1:8	1300	1:10	1300	1:7	1300	"	-	A	-	
30	1:8	1300	1:10	1300	1:7	1300	Rough	5.90	A	Scp	

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
31	1:8	1300	1:10	1300	1:7	1300	Rough	-	A	Scp	
32	1:8	1300	1:9	1300	1:7	1300	"	-	A	Scp	
33	1:8	1300	1:9	1300	1:7	1300	"	-	A	Scp	
34	1:8	1300	1:9	1300	1:7	1300	Plane	-	A	Scp	
35	1:9	1300	1:9	1300	1:7	1300	"	6.60	A	Scp	
36	1:9	1300	1:9	1300	1:7	1300	"	-	A	-	
37	1:9	1300	1:10	1300	1:7	1300	"	-	A	Scp	
38	1:9	1300	1:10	1300	1:7	1300	Rough	6.30	A	Scp	
39	1:8	1300	1:10	1300	1:7	1300	"	-	A	Scp	
40	1:7	1300	1:10	1300	1:9	1300	Plane	-	A	Scp	
41	1:7	1270	1:10	1300	1:6	1280	"	-	A	Scp	

Table 4.15 : Experimental Observations under oxidizing atmosphere.

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
1	1:14	1290	1:9	1300	1:6	1300	Plane	8.29	A	-	
2	1:14	1290	1:9	1300	1:5	1300	"	-	A	Scp	
3	1:14	1290	1:9	1300	1:5	1300	"	-	F	Scp	
4	1:14	1290	1:9	1300	1:5	1300	"	8.40	F	-	
5	1:14	1290	1:10	1300	1:6	1300	"	-	F	-	
6	1:14	1300	1:10	1300	1:10	1290	"	-	F	-	
7	1:13	1300	1:9	1300	1:7	1290	Rough	-	A	Scp	
8	1:13	1300	1:9	1300	1:7	1300	"	8.90	A	Scp	
9	1:13	1300	1:10	1300	1:7	1300	"	-	A	Scp	
10	1:14	1290	1:10	1320	1:6	1300	"	-	A	-	
11	1:14	1290	1:10	1320	1:6	1300	"	-	A	Scp	
12	1:14	1290	1:9	1310	1:6	1300	Plane	8.80	F	Scp	
13	1:14	1300	1:9	1310	1:6	1300	"	-	F	-	
14	1:14	1300	1:11	1310	1:6	1300	"	8.10	F	-	
15	1:13	1300	1:11	1305	1:6	1300	"	-	F	-	

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
16	1:13	1300	1:15	1300	1:6	1300	Plane	-	F	Scp/BM	
17	1:13	1300	1:15	1300	1:6	1300	"	7.70	F	-	
18	1:12	1300	1:15	1300	1:6	1300	"	-	F	Scp	
19	1:12	1300	1:15	1300	1:6	1300	"	-	F	-	
20	1:12	1300	1:15	1300	1:6	1300	"	7.5	F	-	
21	1:12	1300	1:15	1300	1:6	1300	Rough	-	A	Scp/Tc	
22	1:12	1300	1:15	1300	1:5	1300	"	-	A	Scp/Tc/ BM	
23	1:12	1300	1:15	1300	1:5	1300	Plane	-	F	Tc	
24	1:12	1300	1:15	1300	1:6	1300	"	7.6	F	BM	
25	1:15	1300	1:15	1300	1:6	1300	"	-	F	-	
26	1:15	1300	1:10	1300	1:6	1300	Rough	-	F	-	
27	1:15	1300	1:12	1300	1:6	1300	"	7.6	F	Scp/Tc	
28	1:14	1300	1:12	1300	1:6	1300	Plane	-	F	Tc	
29	1:10	1305	1:12	1320	1:6	1300	"	-	F	-	
30	1:10	1300	1:13	1320	1:6	1300	"	-	F	-	
31	1:13	1310	1:10	1300	1:5	1300	"	-	A	Scp	
32	1:13	1370	1:10	1325	1:5	1300	Rough	8.10	A	Scp/Tc	
33	1:13	1290	1:10	1330	1:5	1300	"	-	F	-	

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
34	1:16	1300	1:9	1320	1:7	1300	Plane	12.30	F	-	
35	1:16	1300	1:9	1320	1:7	1300	"	-	F	-	
36	1:15	1290	1:9	1320	1:8	1280	"	-	A	Scp	
37	1:14	1290	1:9	1320	1:8	1280	"	13.40	F	-	
38	1:14	1295	1:9	1315	1:7	1280	"	-	F	-	
39	1:14	1300	1:9	1315	1:7	1290	"	13.30	F	-	
40	1:14	1300	1:9	1315	1:7	1290	"	-	A	-	
41	1:14	1300	1:9	1300	1:9	1280	"	-	A	Lc/Tc	
42	1:16	1280	1:10	1320	1:6	1290	"	13.60	A	Scp	
43	1:16	1280	1:10	1320	1:6	1290	"	-	F	wavy	
44	1:16	1260	1:9	1310	1:8	1290	"	12.90	F	Tc/Lc	
45	1:14	1270	1:8	1320	1:7	1300	"	-	F	-	
46	1:10	1300	1:9	1310	1:7	1320	"	-	F	Tc	
47	1:12	1300	1:9	1310	1:7	1320	Rough	11.90	A	-	
48	1:12	1300	1:9	1310	1:7	1320	"	12.30	A	Scp	
49	1:12	1300	1:9	1320	1:7	1320	"	-	A	-	
50	1:12	1300	1:9	1320	1:7	1320	"	13.40	A	Scp/Tc	
51	1:11	1305	1:10	1320	1:8	1320	Plane	-	A	Tc/Lc	

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
52	1:11	1310	1:10	1320	1:8	1320	Plane	-	F	Tc	
53	1:11	1310	1:10	1320	1:8	1320	"	11.80	F	-	
54	1:11	1308	1:10	1325	1:8	1315	"	-	A	Tc/Scp	
55	1:11	1308	1:10	1325	1:8	1310	"	-	F	-	
56	1:10	1290	1:10	1330	1:5	1285	"	12.90	F	-	
57	1:10	1290	1:10	1330	1:5	1280	"	13.28	F	Tc	
58	1:10	1295	1:9	1330	1:5	1290	"	-	F	-	
59	1:11	1295	1:9	1330	1:5	1290	"	-	F	BM.TC	
60	1:11	1295	1:9	1330	1:5	1290	"	12.78	F	BM.TC	
61	1:11	1295	1:9	1330	1:7	1295	"	-	F	TC.LC	
62	1:10	1295	1:9	1330	1:7	1280	"	12.60	F	TC	
63	1:10	1295	1:10	1330	1:5	1280	Rough	-	F	-	
64	1:11	1295	1:10	1330	1:5	1290	Plane	13.63	F	LC	
65	1:10	1295	1:10	1330	1:5	1295	"	-	F	LC	
66	1:10	1300	1:10	1325	1:5	1300	"	-	F	-	
67	1:10	1300	1:10	1330	1:7	1290	Rough	-	F	TC	
68	1:10	1300	1:10	1325	1:7	1290	"	11.90	F	TC	
69	1:12	1300	1:10	1325	1:7	1290	"	-	F	TC	
70	1:12	1300	1:10	1330	1:7	1290	"	-	F	-	

Table 4.16 : Experimental observations under oxidizing atmosphere in case of rough ingot surface.

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
1	1:10	1300	1:10	1330	1:7	1295	Rough	-	A	Scp,Tc	
2	1:11	1300	1:10	1330	1:7	1295	(Just like small pox)	13.20	A	Ccp, Tc	
3	1:11	1305	1:10	1329	1:7	1295		-	A	Scp,Tc	
4	1:11	1302	1:10	1325	1:7	1295		-	A	Scp	
5	1:11	1302	1:10	1330	1:7	1290		13.60	A	Scp	
6	1:12	1302	1:10	1320	1:7	1290	"	-	A	Scp	
7	1:12	1300	1:10	1325	1:6	1285	"	-	A	Scp	
8	1:10	1300	1:10	1325	1:6	1285	"	-	A	Scp	
9	1:10	1297	1:10	1330	1:6	1290	"	13.80	A	Scp	
10	1:10	1295	1:10	1330	1:6	1290	"	-	A	Scp	
11	1:10	1295	1:14	1315	1:7	1300	"	-	A	Scp	
12	1:11	1295	1:14	1310	1:7	1300	"	-	A	Scp/Tc	
13	1:11	1295	1:11	1310	1:7	1290	"	12.80	A	Scp	

Table 4.17 : Experimental observations under oxidizing atmosphere in case of smooth ingot surface.

Ingot Sl No	Gas air ratio & Temp						Ingot Surface Condition	Scale Thickness mm	Degree of oxide Adherence	Defects	Remark
	Bottom zone		Top zone		Soaking zone						
	Gas: Air	°C	Gas: Air	°C	Gas: Air	°C					
1	1:11	1300	1:10	1310	1:7	1290	Plane	11.40	F	-	
2	1:10	1300	1:10	1320	1:9	1250	"	10.90	F	-	
3	1:10	1297	1:10	1320	1:6	1280	"	-	F	-	
4	1:10	1295	1:10	1325	1:6	1280	"	9.80	F	-	
5	1:11	1295	1:10	1330	1:6	1280	"	-	F	-	
6	1:11	1295	1:10	1330	1:6	1290	"	-	F	-	
7	1:9	1298	1:10	1330	1:6	1290	"	5.40	F	-	
8	1:9	1300	1:10	1330	1:6	1290	"	-	F	-	
9	1:9	1300	1:10	1325	1:6	1290	"	-	F	-	
10	1:10	1295	1:10	1325	1:6	1285	"	6.70	F	-	
11	1:10	1298	1:10	1325	1:6	1280	"	-	F	-	
12	1:12	1298	1:10	1325	1:6	1280	"	6.70	F	-	
13	1:12	1298	1:10	1325	1:6	1280	"	-	F	-	
14	1:12	1298	1:10	1325	1:6	1280	"	-	F	-	
15	1:12	1298	1:10	1325	1:6	1280	"	6.55	F	-	

CHAPTER-5 : DISCUSSION OF THE RESULTS

5.1. Discussion on 1st phase of experimental results:

(i) Sample surface finish: As a general practice the ingot surface must be scarffed either by hand or by machine scarffing before rolling to avoid subsequent scale pitting problem in rolling. But in the CSM there are no such facilities for scarffing, so the rolling operation is performed in the CSM with as cast ingot, whose surface is normally rough. This may be one of the causes of scale pitting. To identify the extent of scale adherence on heating due to rough ingot surface the samples were studied with a rough (as cast) surface and grinded smooth surface.

(ii) Soaking temperature : The ingots or billets are heated so as to provide high plasticity for deforming without cracking. It is best to use high heating temperature since in addition to the foregoing this reduces the energy consumption in rolling and decrease the danger of breakage in the rolls or other components of the mill. The high temperature however lead to intensive scale formation. Soaking temperature may lead to the formation of adherent scales which is a probable source of rolling defects.

In the CSM actually the rolling temperature is $1280-1300^{\circ}\text{C}$. Sometimes this temperature varies from $1050^{\circ}\text{C}-1300^{\circ}\text{C}$ due to unavoidable circumstances. So experimental soaking temperature

89305

range were selected between 1050°C - 1300°C to identify which temperature is suitable for the formation of non adherent scale.

(iii) SOAKING TIME:

Ingots or billets should be heated at specified temperature for a specified time to homogenize the temperature of the ingot interior and outer surface and 3 hrs is recommended for CSM. But in the CSM at present this soaking time varies from 10 to 130 hrs. Because the rolling is normally shut down from 2 PM to 6 PM, while the furnace is kept loaded with certain number of ingots and a temperature of 800°C is maintained to avoid danger the furnace. The number ingots that remain in the furnace varies from 48 to 60. Also on every rest or holiday or under some unavoidable circumstances rolling operation is shut down, but the furnace temperature of about 800°C is maintained with ingots inside it. So naturally some ingots are continued to be heated for a prolonged time which is not desired. This is also contrary to the specified design. It was however not possible to perform experimental observations at BUET under such unusually a long soaking time. In the experimental observation soaking time was taken as 1, 2 and 3 hrs which was selected only to have an idea of the effect of time.

(iv) First phase experimental results:

From 1st set of experimental observation (Table 4.1, Fig. 4.1 to

Fig. 4.3) it was found that for each sample both plane and as cast surfaces developed very thin and very adherent scales. As the scales were very thin and very adherent the scales could not be separated even by hammering and the scale thickness could not be measured.

The difference for the second set of experiment is only a change in the soaking temperature. From the experimental observation (Table 4.2, Fig. 4.4 to Fig. 4.6) it was observed that there was no remarkable change of the nature of the scale.

From the 3rd set of experimental observations (Table 4.3, Fig. 4.7 to Fig. 4.9) it appeared that there were no change in the adherence of scale even at the high temperature. In each case the scales were very thin and very adherent. In the reducing atmosphere (gas air ratio 1:7) the scales were very thin and adherent as such the effect of temperature or time on its thickness could not be detected. It was however observed that for smooth surfaces for in some of the samples (sample Nos. 3, 6, 8 & 9), the adherence decreased relatively.

From the 4th set of experimental works (Table 4.4, Fig. 4.10 to 4.12) it appeared that the scale adherence decreased with the increases of soaking time even though the scales were not peeted off. Scale thickness also increased with the increases of soaking time. In this experimental observation, however no remarkable

difference could be detected due to the variation of surface finish on the adherence characteristic. Any way this was an exceptional case.

From the 5th set of experimental observation (Table 4.5, Fig. 4.13 to Fig. 4.15) it was revealed that with the increase of soaking time the scale adherence decreased. It also showed that the scale formed on the smooth surface was comparatively less adherent than cast surface.

In case of 6th set of experimental observation (Table 4.6, Fig. 4.16 to 4.18) it appeared that there was remarkable change regarding of scale adherence with the increase of soaking time. The scale adherence decreased. From the experimental observation it was also revealed that for smooth surface scale adherence were comparatively less than on the rough (as cast) surface for same condition. Scale thickness increased with soaking time and were not much affected by surface finish.

From the 7th set of experimental observations (Table 4.7, Fig. 4.19 to 4.21) it was revealed that under this atmospheric condition with the increase of soaking time the scale adherence decreased for both smooth and as cast surfaces. But in case of smooth surface adherent strength was comparatively lesser.

From the 8th set of experimental observations (Table 4.8, Fig. 4.22

to 4.24) it was observed that the scale adherence decreased with the increases of soaking time and soaking temperature. It was also observed that for the same condition the scale adherence nature for smooth surface were comparatively less than as cast surface.

From the 9th set of experimental observation (Table 4.9, Fig. 4.25 to 4.27) it was observed that, with the increase of soaking temperature and soaking time scale adherence decreased. In case of smooth surface adherent strength is comparatively less than as cast surface.

From the 10th set of experimental observation (Table 4.10, Fig. 4.28 to 4.30) it was revealed that the scale adherence decreased with the increase of soaking time. It was also observed that for the same condition the scale for plane surface was comparatively less adherent than for as cast surface.

From the 11th set of experimental observation (Table 4.11, Fig. 4.31 to 4.33) it was observed that the scale adherence decreased with the increase of soaking time. For the same condition the scale adherence decreased in case of plane surface.

From the 12th set of experimental observation (Table 4.12, Fig. 4.34 to 4.36) it was clear that the scale adherence decreased with the increase of soaking time under the same atmosphere condition. The scale adherence was found to decrease in case of plane surface.

All the observations and experimental results are rearranged in a single table (table 5.1). The table contained all the experimental results of 36 samples. From the experimental observation it was revealed that reducing atmosphere (gas air = 1:7 to 1:9) was not suitable to non adherent scales, samples having either rough or plane surface. So it is clear that reducing atmosphere had negative effect on the formation of non adherent scale.

From the experimental observations samples treated under oxidizing atmosphere (sample S1 No. 19-36), it was clear that the nature of the scale formed was not dependent on any single parameter but had mixed influence of the parameters.

The most significant effect was of the oxidizing atmosphere. Thus with the increase of fuel air ratio the scale adherence strength decreased. From table 5.1 it was revealed that the scale adherence strength also decreased with higher soaking temperature and with the increase of soaking time. Smoother surface finish also provides less adherence.

So from the above discussion and experimental results it is clear that reducing atmosphere have negative effect on non adherent scale formation. While oxidizing atmosphere is necessary to form non adherent scale. Under oxidizing atmosphere other parameter such as surface finish, soaking time, soaking temperature also have marked influence on the formation of non adherent scale.

Table 5.1

Table	Sample Sl No	Macro photo graphy Fig. No.	Gas air ratio	Temp. °C	Soak ing time hrs	Arbitrary scale adherence grading															
						As cast surface (Rough)								Plane surface							
						1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
4.1	1	4.1	1:7	1050-1100	1	✓								✓							
	2	4.2	1:7	"	2	✓								✓							
	3	4.3	1:7	"	3	✓								✓							
4.2	4	4.4	1:7	1150-1200	1	✓								✓							
	5	4.5	1:7	"	2	✓								✓							
	6	4.6	1:7	"	3	✓								✓							
4.3	7	4.7	1:7	1250-1300	1	✓								✓							
	8	4.8	1:7	"	2	✓									✓						
	9	4.9	1:7	"	3		✓								✓						
4.4	10	4.10	1:9	1050-1100	1	✓									✓						
	11	4.11	1:9	"	2		✓									✓					
	12	4.12	1:9	"	3			✓								✓					
4.5	13	4.13	1:9	1150-1200	1	✓									✓						
	14	4.14	1:9	"	2			✓								✓					
	15	4.15	1:9	"	3			✓									✓				
4.6	16	4.16	1:9	1250-1300	1			✓								✓					
	17	4.17	1:9	"	2			✓									✓				
	18	4.18	1:9	"	3				✓									✓			

* Scale adherent grading : Adherent strength decreases with the increases grading no.

Table 5.1

Table	Sam ple Sl No	Macr o phot ogra phy	Gas air rati o	Temp. °C	Soak ing time hrs	Arbitrary scale adherence grading															
						As cast surface (rough)								Pne. surfce							
						1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8
4.7	19	4.19	1:10	1050-1100	1		✓								✓						
	20	4.20	1:10	"	2			✓							✓						
	21	4.21	1:10	"	3				✓							✓					
4.8	22	4.22	1:10	1150-1200	1		✓								✓						
	23	4.23	1:10	"	2				✓						✓						
	24	4.24	1:10	"	3					✓								✓			
4.9	25	4.25	1:10	1250-1300	1					✓						✓					
	26	4.26	1:10	"	2						✓						✓				
	27	4.27	1:10	"	3						✓							✓			
4.10	28	4.28	1:12	1050-1100	1			✓							✓						
	29	4.29	1:12	"	2				✓							✓					
	30	4.30	1:12	"	3					✓								✓			
4.11	31	4.31	1:12	1150-1200	1					✓						✓					
	32	4.32	1:12	"	2						✓							✓			
	33	4.33	1:12	"	3							✓							✓		
4.12	34	4.34	1:12	1250-1300	1				✓							✓					
	35	4.35	1:12	"	2							✓						✓			
	36	4.36	1:12	"	3								✓						✓		

5.2 DISCUSSION ON 2ND PHASE EXPERIMENTAL WORK:

It is apparent from the CSM heavy plate mill operation that the adherent scale on the ingot surface which is formed during heating before rolling is mainly responsible for scale pitting.

(i) Influence of adherent scale on scale pitting:

For the analysis of the effect of the nature of scale on scale pitting, all the data which were recorded for 139 of ingot samples were divided into adherent and non adherent group. It is shown in table -A, that out of 139 ingots examined, 70 ingots had adherent scale, and 69 ingots had non adherent scale. Ingot which possessed adherent scale gave 80% scale pit problems and 20% ingot had no scale pit problem. On the other hand for non adherent case only 7% showed scale pit problem and the rest 93% ingot gave scale pit free products.

So from the above results it is clear that adherent nature of the scale is mainly responsible for scale pit problems.

Table-A

Scale Nature	No. of ingot	Scale pit		Scale pit Free	
		No	%	No	%
Adherent	70	56	80	14	20
Non Adherent	69	5	7	64	93

(ii) Influence of Ingot surface Finish on Scale pitting:

The effect of surface finish on the formation of adherent scale and subsequent scale pitting problem in the 139 No. ingots under experimental observation with 52 ingots having rough surface and 87 ingots having plane surface are shown in Table B. It was observed that for rough surface 85% ingot possessed adherent scale and 15% ingot possessed non adherent scale. On the other hand ingots having good surface finish gave 30% adherent scale and 90% nonadherent scale.

So from the experimental observation it was revealed that adherent scale increased with the increase of surface roughness.

But it is also clear from the Table-B that for both rough and smooth surface adherent nature of scale gave higher percent scale pit in the product and nonadherent scale gave higher percentage of scale pit free product.

It was also revealed from this analysis that adherent scale for rough surface gave higher percentage of scale pit problem (91%) than adherent scale which formed on smooth surface (62%). So smoother surface have greater advantage to produce scale pit free product than rough surface whether adherent or nonadherent scales are formed.

So the surface condition of ingots are an important factor in decreasing the scale pitting.

Table-B

Ingot Surface	No. of Ingot	Adherent Scale						Non Adherent scale						Total %	
		No. of ingot	%	Scale pit		Scale pit free		No of ingot	%	Scale pit		Scale pit free		scp	Scp free
				No	%	No	%			No	%	No	%		
Rough	52	44	85	40	91	4	9	8	15	1	13	7	87	79	21
plane	87	26	30	16	62	10	10	61	70	4	7	57	93	23	77

For more clear understanding, of the influence of surface finish on the nature of scale formed and subsequent scale pitting problem individual experimental observation were carried out with. 13 very rough surface ingots (c.s.m product) and 15 fine surface ingots (concast product). They were heated before rolling under oxidizing atmosphere. It was observed that 13 rough ingot gave 100% adherent scale and subsequently produced 100% scale pit product. While 15 concast ingots with smooth surface gave 100% non adherent scale and subsequently produced 100% scale pit free product after rolling.

Table-C

Ingot Surface	No of Ingot	Adherent Scale						Non Adherent scale						Total %	
		No. of ingot	%	Scale pit		Scale pit free		No. of ingot	%	Scale pit		Scale pit free		scp	Scp free
				No	%	No	%			No	%	No	%		
Rough	13	13	100	13	100	-	-	-	-	-	-	-	-	100	-
plane	15	-	-	-	-	-	-	15	100	-	-	15	100	-	100

From this experimental observation it was concluded that for rougher surfaces, scales were adherent and gave the product scale pit problem even when the heating was carried out under oxidizing condition.

(iii) Effect of Furnace Atmosphere for the Formation of adherent scale and subsequent scale pit Problem:

The influence of furnace atmospheric condition (fuel: air ratio) on the formation of adherent scale and then subsequent scale pitting problem ingots were heated at different atmospheric conditions. The experimental observations were carried out in the following manner.

Assuming identical surface condition, 41 ingots were heated under reducing atmosphere and rolled out and 98 ingots were heated under oxidizing condition and rolled out. The experimental observation and results are furnished in table-D.

The 41 ingots which were heated under reducing atmosphere gave 85% adherent scales. These ingots gave 83% product with scale pit and 17% free of scale pit. Out of the 41 ingots 15% possessed non adherent scale which gave 17% product with scale pit and 83% product without scale pit.

The 98 ingots which were heated under oxidizing atmosphere showed adherent scale and in 36% nonadherent scale in 64%. In case of

adherent scale 77% ingot possessed scale pit and 23% product had no scale pit. In case of ingots having nonadherent scale, 6% gave scale pit problem and rest gave scale pit (94%) free product.

From the above experimental observations it is apparent that adherent nature of scales was influenced by furnace atmospheric condition (fuel: air ratio). It is clear that reducing atmosphere inside the furnace increased the probability for the formation of adherent scale and subsequently increased scale pitting problem. On the other hand oxidizing atmosphere inside the furnace increased the probability for the formation of non adherent scale ingots and subsequently decreased the percentage of scale pit problem.

Table-D

Atmos pheric condition	No.of Ingot	Adherent Scale						Non Adherent scale						Total	
		No.of ingot	%	Scale pit		Scale pit free		No.of ingot	%	Scale pit		Scale pit free		Scale pit %	Scp free %
				No	%	No	%			No	%	No	%		
Reducing	41	35	85	29	83	6	17	6	15	1	17	5	83	73	27
Oxidizing	98	35	36	27	77	8	23	63	64	4	6	59	94	32	68

CHAPTER-6 : CONCLUSIONS:

6.1 The following conclusions may be drawn from the present work;

- (1) Scale pitting on the plates occurs by the formation of adherent scales on the slabs and the failure to remove them through the descaling process prior to rolling for the plate formation.
- (2) The adherent character of the scale on the surface of the slabs depends on the nature of the atmosphere prevailing inside the furnace. Adherent nature increase under reducing atmosphere and decreases in oxidizing atmosphere.
- (3) Surface condition of the slab ingot is responsible to some extent for the degree of scale adhesion. Rough surface produces adherent scale and smooth surface finish produces non adherent scale.
- (4) Soaking temperature have marked influence on the formation of non adherent scale. Temperature of the reheating furnace should be maintained within the range of 1290°C to 1310°C .

6.2 REMEDIAL MEASURES

1. For the formation of non adherent scale the burner flames are to be adjusted to more oxidizing condition.
2. Rough surface of slab ingot must be scarffed before charging in the furnace. Unless the slab surface is smooth there will be a greater probability for the formation of adherent scale. So facilities must be developed in the CSM for scarffing the slab surface.
3. In the CSM descaling is carried out by water jet spraying at the rolling stand. The water pressure at the nozzles, their position, height and angle with the slab, nozzle configuration and alignment all play vital roles in the effectiveness of descaling. Nozzle blockage and improper gap and direction of the spray hamper scale removal. In order to make the best use of the present set up, the nozzles must be cleaned and aligned properly.

6.3 SUGGESTIONS FOR FURTHER WORK

- (1) Scale formed on different atmospheric condition should be studied by x-ray. This will elucidate the nature of scale formed at different atmospheric condition and their influence on scale adhesion

- (2) A study on the determination of chemical composition of the scale, which is formed at different atmospheric condition should be carried out, so that the seperate effects of the adherence strength of each type of scale may be assessed.
- (3) To conduct a techno-economic study on the introduction of continuous casting process for the improvement of better plate yield.

APPENDIX: 1.1. CAPACITY AND PRODUCT OF CSM LTD.

<u>NAME OF SHOP/UNITS</u>	<u>PRODUCTS</u>	<u>RATED CAPACITY (CSM. PLANNING CELL-1973)</u>
1. Melting shop	Steel ingots	2,50,000 MT
2. Blooming Mill	Billets, sheet bars	1,50,000 MT
3. Bar Mill	Billets, M.S. bar	55,000 MT
4. Sheet Mill	B.P. sheet and Thin plates	65,000 MT
5. Galvanizing shop	G.P. Sheets, CGI sheets	60,000 MT
6. Heavy plate	Heavy plates	60,000 MT

APPENDIX: 1.2. ACTUAL PRODUCTION.

Year	Melting shop	Bloomin g mill	Bar Mill	Sheet Mill	Thin plate	Galvan izingp plate shop	Heavy plate
66-67	18131	7199	2345	1276	-	592	-
67-68	68,217	54,091	25,032	12,890	1047	11,744	0
68-69	60,988	64,332	26,010	10667	5770	10092	0
69-70	54,139	50,486	23802	8435	4077	7343	0
70-71	40,105	34,666	18779	5957	23	6646	413
71-72	41,234	48,236	18266	6612	0	5778	194
72-73	67,966	46,297	27934	7073	4550	6603	6168
73-74	73,659	57,210	31460	5957	2242	4731	8503
74-75	76,412	7142	39708	4262	4312	4139	6883
75-76	90,400	70985	55518	6001	1004	7569	2142
76-77	102458	73500	57218	1582	8993	10663	11630
77-78	111000	106306	60954	3068	4395	6900	12112
78-79	121464	106561	100190	1977	8467	13691	14891
79-80	133315	92628	87842	851	6077	10414	12292
80-81	135733	90802	84942	1107	8284	8955	15266
81-82	107525	28655	90673	414	4498	7942	12310
82-83	47401	61983	25834	2263	855	6629	4318
83-84	78387	76784	56011	1233	2571	31351	7980
84-85	101419	64674	64155	819	3505	42097	8159
85-86	95512	76306	48053	699	1546	45202	7678
86-87	82072	61970	56047	1079	3284	33713	9434
87-88	70036	61526	42972	291	4584	32181	7637
88-89	86245	51983	50151	698	4331	26167	8173
89-90	75027	37875	38554	446	6148	13589	12630
90-91	57290	20104	24031	185	3364	8386	6557
91-92	36306	8967	64850	373	1311	7867	3438
92-93	7040	9784	11850	249	1553	6953	1963
93-94			4325	224	1790	3135	2467

APPENDIX 1.3 HEAVY PLATE MILL PERFORMANCE:

SL No	YEAR	PLATE PRODUCTION MT	% OF RATED CAPACITY*
1	1970-71	413	0.69
2	1971-72	194	0.32
3	1972-73	6168	10.28
4	1973-74	8503	14.17
5	1974-75	6880	11.47
6	1975-76	2142	3.57
7	1976-77	11630	19.38
8	1977-78	12112	20.19
9	1978-79	14891	24.82
10	1979-80	12292	20.49
11	1980-81	15266	25.44
12	1981-82	12310	20.52
13	1982-83	4318	7.20
14	1983-84	7980	13.30
15	1984-85	8159	13.60
16	1985-86	7678	12.80
17	1986-87	9434	15.72
18	1987-88	7637	12.73
19	1988-89	8173	13.62
20	1989-90	12630	21.05
21	1990-91	6557	10.93
22	1991-92	3438	5.73
23	1992-93	1963	3.27

* Fraction 0.05 and above are rounded off and below are dropped.

REFERENCES

1. Ferrous Production metallurgy
A.T. Peters,
Staff metallurgist, Inland steel company East Chicago, Indiana.
P. 267 to 268 (1982).
2. A Report on the problems of Heavy plate Mill at Chittagong steel Mills Ltd. submitted by Department of Metallurgical Engineering, BUET, Dhaka, p. 1-15 (1988)
3. Rolling practice, N. Perlov, Mirpublishers, Moscow p. 66-67.
4. "Rolled in scale the consistent problem" by David T. Blazevic.
President Hot rolling consultants Ltd. Home wood, Illinois, USA p. 1-3 (1987)
5. Corrosion of metals in gaseous environment U.R. Evans, the oxidation and corrosion of metals, London Edward Arnold Ltd. p. 1 to 10 (1960).
6. The mechanism of scale formation on iron at high temperature. By B.W. Dunnington, F.H. Beck and M.G. Fontana, Submitted for publication July 12, 1951. National association of corrosion Engineers. p. 2 to 8 (1951).
7. Some observations on the reproducibility of high temperature scaling behaviour of iron and low alloy steels.
By J.A. Von Fraunhofer and G.A. Pick up. The gas council, Watson house, London corrosion science, 966 vol. 10, p. 253 to 260 (1960)
8. Cyclic oxidation of cobalt based alloys.
Ph.D. Thesis, M.S. Islam. University of Liverpool, U.K. (Dept. of Met. Engg. BUET, Dhaka), p. 18, 19 (1978).
9. Conditions for the initiation of oxide scale cracking and spallation.
By H.E. Evans and R.C. Lobb
Corrosion science vol. 24, No.3.
p. 209 to 222 (1984).
10. Rolling mill practice
P. Polukhin, N. Fedosov, A. Korolyov and Y. Matveyev p. 34

11. Effect of cold work on the oxidation of iron from 400 - 650°C.
By D. Caplan and M. Cohen,
Division of Applied chemistry, national research council,
ottawa, canada corrosion science, 966 vol. 6, p. 321 to 335
(1966).
12. Plastic flow of iron oxide and the oxidation of iron.
By J.D. Mackenzie and C.E. Birchenall, Submitted for
publication November 13, 1957. National association of
corrosion Engineers p. 17 to 19 (1957).
13. Cavity formation in iron oxide,
By D.W. Juenker R.A. Meussner and C.E. Birchenall, Submitted .
for publication, November 9, 1956, National association of
corrosion Engineers p. 57 to 58 (1956).
14. Mechanism of Atmosphere Rusting,
By U.R. Evans and C.A.J. Taylor
Department of Metallurgy, Cambridge University cambridge
England, corrosion science, 1972, vol. 12, p. 227 to 246
(1972).
15. The influence of scale/metal interface characteristics on the
oxidation behaviour of iron at elevated temperature,
By S. Taniguchi and D.L. Carpenter, Department of Metallurgy
and Materials science University college cardiff, wales,
corrosion science, 1977, p. 15 to 26 (1977).
16. A new kinetic model for nucleation and growth of duplex oxide
scales on iron between 350 and 500°C
C.I. Howe, B. MCENANEJ and V.D. Scott. corrosion science, vol.
25, No. 3 p. 195 to 207 (1985).
17. The oxidation of Fe in the temperature range 450-550°C. The
pressure range 10^{-6} - 10^{-4} Torr. R.J. Hussey and M. Cohen,
corrosion science, 1971, vol. 11, p. 699 to 711 (1971).

