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# 1. INTRODUCTION

## 1.1. Background

During the steel-making process, scale is formed in the reheating furnace prior to hot-rolling and is used to oxidize away some surface imperfections, but if the resultant scale cannot be removed prior to rolling (by the usual method of hydraulic descaling), serious surface quality problems can arise. These quality problems appear to be important for both stainless and carbon steels.

Specific industry observations (by members of the Center for Iron and Steel-making Research at Carnegie Mellon University) which were used to determine the direction of this work are as follows: For stainless steels, difficult-to-remove scale forms on an 11.5% chromium stainless steel (similar to Type 409, but without titanium stabilization) at the reheating temperature of 1210°C, if the total reheating time is substantially longer than the usual 4 hour. This effect is reportedly not found for the Type 304 stainless steel. However, the scale on type 304 is also sometimes difficult to remove.

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## 1.2. Problem

It is expected that scale removal is not only influenced by the thickness of the scale formed, but also by the micro-structural characteristics of the scale; i.e. the nature and distribution of the different phases present in the scale as well as the interfacial morphology of the scale. Roughening of the scale-steel interface during scale growth in the reheating furnace is expected to cause strong scale adhesion, leading to poor scale removal (by hydraulic descaling) prior to hot rolling. Thus the micro-structural characteristics have to be understood for efficient removal of the scale.

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### **1.3. Objectives of investigation**

The goals for this literature survey are to:

- Identify possible mechanisms for the formation of sticky scale
- Identify mechanisms of scale removal during hydraulic descaling
- Obtain information regarding the mechanisms of mechanical descaling

The objectives are then to allow scale growth under simulated reheating furnace conditions, where the temperature, gas atmosphere and mass transfer to the sample surface are controlled and then to assess scale adhesion at temperature.

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## **1.4. Research methodology**

### **1.4.1. Introduction**

From the general observations above, the main parameters to consider in this study are the reheating conditions (time, temperature and gas atmosphere) and the steel grade (both major alloying elements and impurities). The underlying idea is that the nature of the scale-metal interface must affect the removability of the scale. Hence, possible mechanisms, which can lead to interfacial roughening, will be surveyed first, followed by observations on the mechanism of hydraulic as well as mechanical descaling.

### **1.4.2. Need for new methods**

The importance of the descaling mechanism in this study concerns ways in which descalability may be evaluated in the laboratory. The usual method of using actual high-pressure sprays is not simple to implement in a laboratory. Thus simpler methods - for example, bending the sample around a set radius immediately upon removal from the furnace, and subsequently measuring the amount of scale remaining— may suffice. This of course, all depends on the nature of the descaling mechanism (mechanical or thermal).

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## 2. LITERATURE SURVEY

### 2.1. Interfacial roughening mechanisms

#### 2.1.1. Pure metals

##### 2.1.1.1 VOID FORMATION AT THE SCALE-METAL INTERFACE

Both carbon and stainless steels generally oxidize by a cationic diffusion mechanism (Kofstad, 1988). As an example, the classical scale growth mechanism of iron is as follows: Iron is oxidized to  $\text{Fe}^{2+}$  at the scale-metal interface, releasing two electrons. Both the  $\text{Fe}^{2+}$  cation and the electrons travel through the scale to the outside atmosphere. At the scale-gas interface, the electrons react with oxygen (or other oxidants such as  $\text{CO}_2$  or  $\text{H}_2\text{O}$ ) to produce  $\text{O}^{2-}$  anions, which then react with the  $\text{Fe}^{2+}$  to form new  $\text{FeO}$ . In this way, the new scale forms at the scale-gas interface, while the iron atoms are removed from the scale-metal interface.

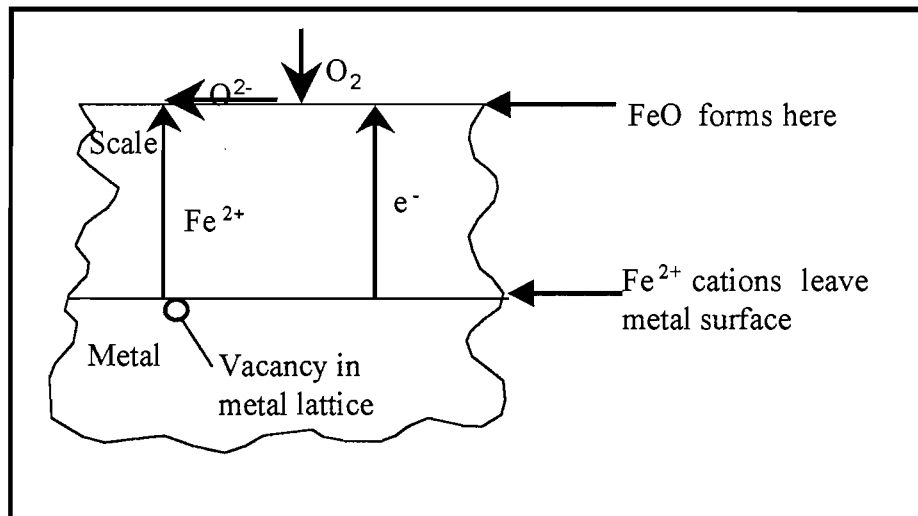


Figure 1: Schematic depiction of processes occurring during scale growth by cationic diffusion

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At high temperatures, three oxides of iron are stable in the scale and form in parallel layers. The innermost layer with the lowest oxygen content is wustite (FeO) followed by magnetite (Fe<sub>3</sub>O<sub>4</sub>) and then hematite (Fe<sub>2</sub>O<sub>3</sub>). For oxidation in the temperature range 700-1250°C, the average FeO/ Fe<sub>3</sub>O<sub>4</sub> / Fe<sub>2</sub>O<sub>3</sub> thickness ratios were 95:4:1 (Sheasby, *et al*, 1984) (Ajersch, 1992). At lower temperatures (below 570°C), wustite is not stable and magnetite grows at the expense of the wustite (Sheasby ,*et al*, 1984).

### 2.1.1.2 RATE OF SCALE GROWTH

Initially after exposure of a base metal, the oxygen mass transfer to the outer scale surface controls the rate of scale growth since cation (Fe<sup>2+</sup>) transfer is relatively efficient at small-scale thicknesses. The rate of scale growth is thus linear — the thickness of the scale formed is directly proportional to time and independent of the scale thickness. During this period, only FeO is present (appendix 1) up to a thickness of between 0.4 and 0.5 mm, beyond this thickness, the rate-controlling step becomes the arrival of Fe<sup>2+</sup> at the interface. Then the scale growth rate changes from linear to parabolic, i.e. the square of the scale thickness is proportional to time, at constant temperature (Sachs and Tuck, 1968).

The initial scaling rates are reduced by reducing the oxygen input/fuel ratio to the furnace, thereby lowering concentrations of free oxygen in the combustion products from about 3 to 1.5 %. The predicted weight gains during reheating in the industrial reheat furnace indicate that oxidation rates during reheating are intermediate between linear and parabolic, especially during reheating with high oxygen/fuel ratio. (Abuluwefa, *et al*, 1997).

The classic Wagner theory (Rapp, 1984) predicts parabolic kinetics, if the oxidation rate is controlled by diffusion (of cations, in this case) through the scale — and good

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agreement between actual scale growth rates and those predicted from diffusivities is indeed found, especially at temperatures higher than about 0.75 times the absolute melting point of the scale. This means the scale growth rate is often controlled by the rate of diffusion through the scale, which should serve to smooth any irregularities in the scale — since the scale thickens more rapidly at points where it is locally thinner. In this way, parabolic scale growth should imply a smooth scale layer, and hence a smooth scale-metal interface.

However, even in the case of pure metals undergoing parabolic scale growth, the basic scale growth mechanism can lead to roughening of the interface. As indicated above, cationic oxidation proceeds by continuous removal of iron atoms from the scale-metal interface, which means that vacancies are continually created in the metal lattice at this position. These vacancies are generally eliminated by climb of the misfit dislocations at the scale-metal interface (Rapp, 1993).

These misfit dislocations are present at the interface because of the epitaxial relationship between the scale and the underlying metal (i.e., with specific crystal planes and crystallographic directions of the scale parallel to those in the underlying metal) (Boggs, 1973; Rapp, 1984; Taniguchi, 1985; Pieraggi & Rapp, 1988). The dislocations are required to accommodate the misfit between the metal and scale lattices. If the process of vacancy elimination is somehow prevented, the vacancies can condense to form voids at the scale-metal interface — roughening that interface. Vacancy elimination can, for example, be prevented by the loss of epitaxy between the scale and the metal, or through immobilization of the dislocations by cations of "reactive elements" (such as yttrium) (Pieraggi, et al, 1995)

Void formation at the interface appears inevitable for the conditions during reheating. The extent of void formation is expected to increase as the extent of scaling increases, but it is not clear what the effect on descalability will be. On the one hand, voids may act as stress raisers, promoting loss of scale adhesion (Taniguchi, 1985). This may be an explanation for the observation that thinner scales (formed at lower

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temperatures) on aluminum-killed steels are more difficult to remove by hydraulic descaling (Morris, *et al*, 1996). Conversely, because voids interrupt the movement of cations, scale growth slows down above voids causing the scale-metal interface to become uneven, which might in fact aid adhesion by a mechanical keying action (Kofstad, 1988).

Epitaxy may be lost if the metal undergoes a phase transformation (which disrupts the crystallographic relationship between the metal and the scale). This may play a role during the scaling of iron or carbon steel during heating, where a temporary reduction in the scale growth rate is observed when the temperature passes through approximately 900°C to 975°C, perhaps because the ferrite-austenite phase transformation causes separation between the scale and the metal (Sachs, and Tuck, 1970).

## **2.1.2. Alloys**

### **2.1.2.1 INTRODUCTION**

Interfacial roughening may occur when the alloying element (or impurity) is less likely to be oxidized (is more noble) than the iron matrix, or equally when it is more likely to be oxidized (is more reactive).

The general condition for interfacial stability, when one element in an alloy is considerably more reactive, and forms most of the scale (e.g. Cr<sub>2</sub>O<sub>3</sub> formation on stainless steel) was stated by Wagner (1959). The qualitative result is that, if diffusion of the reactive element in the oxide is rate determining (for scale growth), the scale-metal interface is stable and should remain planar, whereas if diffusion in the alloy is comparatively slow, the interface is destabilized and roughens (due to internal oxidation leading to the formation of Cr<sub>2</sub>O<sub>3</sub> in the metal substrate) (Gibbs, 1981). For iron-chromium alloys, a stable interface is only expected if the chromium content



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exceeds a critical value, which is in the range of 13 - 25% (Hindham and Whittle, 1982) — thus commercial stainless steels (which have lower chromium contents) are generally expected to have unstable (i.e. roughened) scale-metal interfaces. This is experimentally observed for the oxidation of Fe-Cr alloys in oxygen (Whittle and Wood, 1967) and in water vapour (Fujii and Meussner, 1963, 1964). Thus some degree of interfacial instability is always expected for the stainless steels and certainly for the carbon steels, where the low concentrations of reactive elements (such as silicon, aluminum and manganese) imply that internal oxidation (Douglass, 1995) will be prevalent. Since some interfacial instability is expected, the question becomes what the effect of alloying elements and impurities will be on the extent (and physical size) of that instability. This is discussed in the following sections, classified according to the main mechanisms (fayalite formation, nickel enrichment, decarburization and internal oxidation), which are observed for commercial steels.

#### **2.1.2.2 FAYALITE FORMATION**

With high (> 0.25 %) silicon concentrations in the steel, a film of silica might form at the metal-oxide interface and thus slow the rate of scaling (Sachs and Tuck, 1970). Because an oxygen activity gradient exists between the scale and the metal, atomic oxygen diffuses from the scale into the steel and reacts with the dissolved silicon to form an internal precipitate of  $\text{SiO}_2$  (Sachs and Tuck, 1970). FeO and  $\text{SiO}_2$  form a eutectic system (with melting point around  $1180^\circ\text{C}$ ); the eutectic composition is close to fayalite ( $\text{Fe}_2\text{SiO}_4$ ) as shown in the figure below. This means that, for reheating temperatures greater than  $1180^\circ\text{C}$ , a liquid oxide is present next to the metal; this causes the interface to break down, apparently because the fayalite melt wets and penetrates the grain boundaries of the steel. The resulting inter-penetration of scale and metal renders the scale difficult to remove (Palin, 1965).

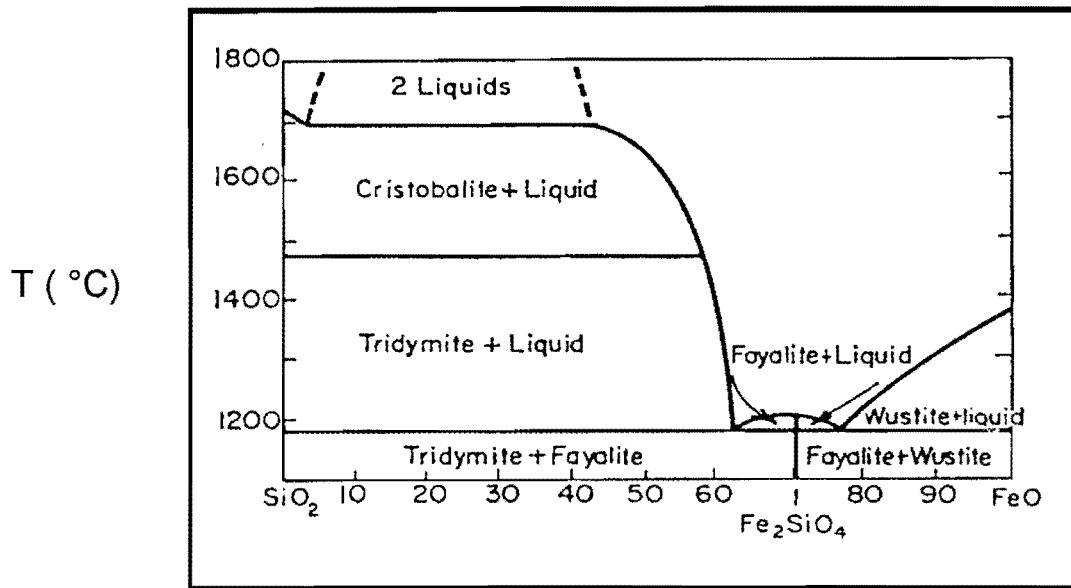


Figure 2 : SiO<sub>2</sub> - FeO phase diagram (Levin, et al, 1964)

### 2.1.2.3 NICKEL ENRICHMENT

Nickel is less easily oxidized than iron and is thus rejected by the scaling reaction and is concentrated at the metal-oxide interface. Because nickel does not diffuse rapidly back into the steel, a discontinuous nickel-rich layer, which extends into the scale, is formed. The presence of Ni entanglement in the scale makes the scale adherent during rolling, so that it is sometimes forced into the metal, giving rise to surface defects (Zittermann, *et al*, 1982). Ni in steel having a bulk Ni concentration of 0.14% segregates in the sub-scale to a concentration as high as 5-6% after oxidation. Increasing the Ni content may result in extensive internal oxidation and the appearance of a Fe-Ni spinel phase next to the oxide-metal interface (Boggs, 1973).

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#### 2.1.2.4 INTERNAL OXIDATION

Internal oxidation of elements, which are more reactive than iron may occur within the metal behind the metal-scale interface, by reaction with oxygen. This results in oxide particles distributed within the metal grains. For example, in the case of stainless steels with high Cr contents, a protective film of  $\text{Cr}_2\text{O}_3$  is formed which inhibits the outward diffusion of Fe. However, the film often breaks down and a spinel ( $\text{FeCr}_2\text{O}_4$ ) forms at the scale-metal interface, which allows some oxidation to occur, although the rate is much slower than with ordinary steels (Fujii and Meussner, 1964).

#### 2.1.2.5 DECARBURIZATION

In general, the oxidation rate of steels is much slower than that of pure iron and it is not easy to establish which component is responsible for this effect. Carbon is peculiar in that it forms a gaseous reaction product. The main effect of carbon on oxidation rates is to make the rates more erratic. Thus carbon reacts with the iron oxides to produce CO and  $\text{CO}_2$ . A concentration of CO or  $\text{CO}_2$  in pores in the scale might cause cracking leading to an increase in the scaling rate. On the other hand, the gases might fill the gaps and voids, thus preventing healing of the defects and slowing the rate of oxidation (Sachs, and Tuck, 1970). Even very low carbon contents are sufficient to cause some pores to form at the interface; the pores are only absent if the carbon content is lower than 50 PPM for the oxidation of carbon steel at  $950^\circ\text{C}$  (Sheasby, *et al*, 1984).

#### 2.1.2.6 IMPLICATIONS FOR THIS WORK

It appears likely that all of the mechanisms briefly surveyed here — fayalite formation, entanglement due to interfacial enrichment with nobler elements, carbon monoxide bubble formation, and internal oxidation — will be present to some extent

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under the reheating conditions considered in this work. Two likely exceptions are CO formation in the case of stainless steels and fayalite formation in the case of aluminium-killed steels— but even with these excluded, several mechanisms leading to interfacial roughening are expected to operate simultaneously. As a result, the descalability of stainless steels is expected to depend strongly on their composition, and especially the level of impurities (with both noble and reactive impurities being important). It also means that it will probably be impossible to assign causes of interfacial roughening or poor descalability to a single factor. The implication is that the steel grades, which are to be used in the experimental work, need to be chosen with care.

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## 2.2. Descaling

### 2.2.1. Introduction

The two legs of this project are the development of the interfacial morphology and the effect of this morphology on the behaviour during descaling. This section deals with the mechanism of hydraulic descaling. A typical plate mill plant-descaling rig includes a pump, which is driven by an electric motor. The typical pump pressure used is about 0.7 MPa (Iscor Vanderbijlpark). High pressure water is delivered by the pump to a header bar over the steel, from which water is sprayed through a series of nozzles. Both pump and motor are fitted to a base frame mounted on anti-vibration mountings (Morris, *et al*, 1996).

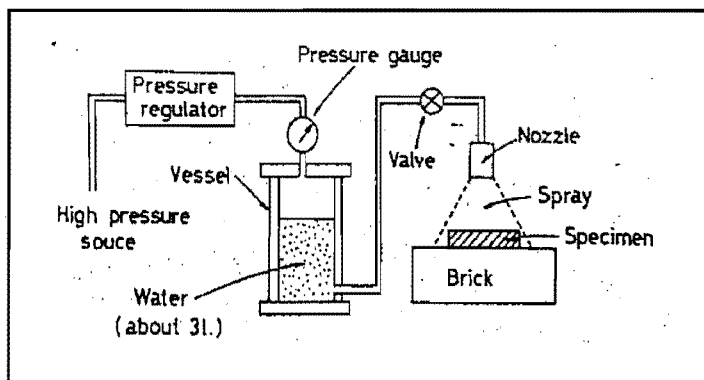


Figure 3 : Diagram of set-up during hydraulic descaling (Matsuno, 1980)

### 2.2.2. Header diameter

Larger diameter (150 mm) header pipes with the same water pressure and flow rate have lower water velocities and thus, less turbulent flow than smaller diameter headers. Less turbulence produces better descaling action since more kinetic energy remains in the spray. Modern descaling systems attempt to achieve an optimum velocity of 1.2 meters a second in the header (Grigg, *et al*, 1985).

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### 2.2.3. Nozzles and attachments

The nozzle system consists of a welded steel body, with a long tapered bore to promote smooth water flow, a replaceable nozzle tip and a threaded locking nut to retain the tip. The orifice tips should be self-aligning to ensure a consistent offset-angle and the orifices should be made of tungsten carbide to increase the working life (Grigg, *et al*, 1985).

The impact pressure is a maximum when the nozzle impact angle is vertical because a scouring action is produced on chipped scale, which removes the scale. The main benefit from inclining the nozzle towards the approaching steel is that detached scale is directed away from the rolling stand. An elliptical orifice results in better descaling because a relatively constant impact pressure across the jet width can be maintained. The water jet width and thickness increase linearly with nozzle height. The impact pressure of the jet increased linearly with system pressure (Sheridan and Simon, 1995).

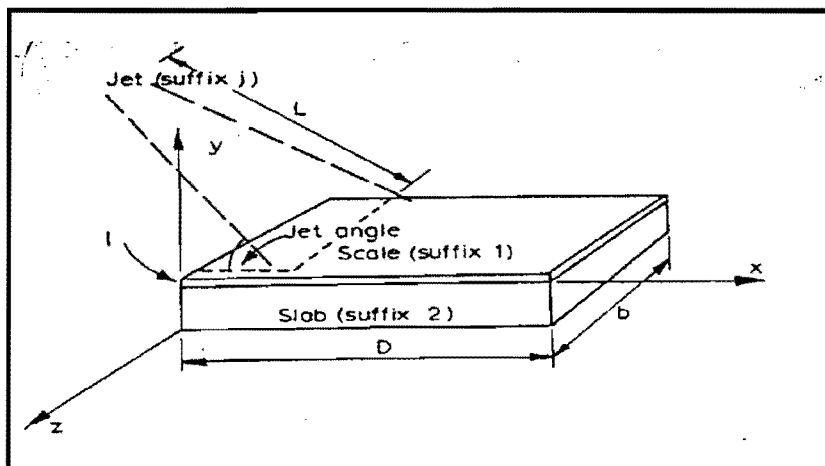


Figure 4 : Diagram indicating offset angle of cooling jet (Sheppard, and Steen, 1970)

Figure 5 below gives as a function of the nozzle diameter:

- $Q_c \cdot f_c$  or the actual descaling capacity of the nozzle which is the flow rate through coherent part (orifice) of nozzle  $Q_c$  (litre/m<sup>2</sup>) multiplied by the actual momentum flux density  $f_c$  (MPa) ; ( $f_c$  is the jet force divided by the jet cross sectional area)
- $Q_c \cdot f_c / (Q_t \cdot f)$  or the efficiency of the nozzle, i.e. the ratio between the actual descaling capacity at the impact and the theoretical descaling capacity, which is equal to the total flow rate of the nozzle  $Q_t$  multiplied by the theoretical jet momentum flux density.

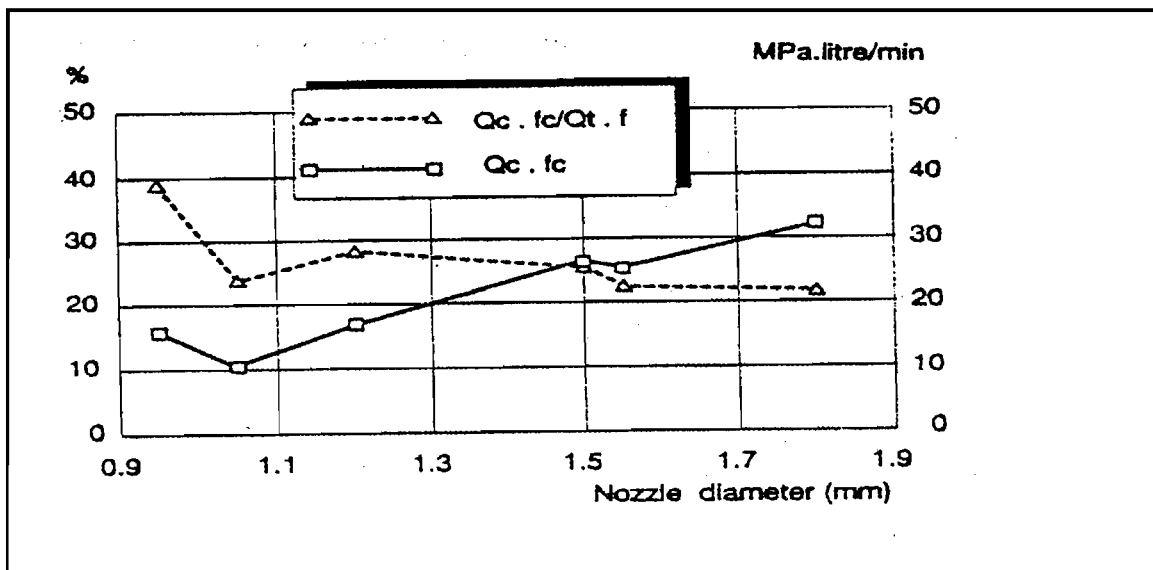


Figure 5 : Influence of nozzle diameter (Sheridan and Simon,1995)

The descaling capacity increases with the nozzle diameter but the efficiency is better for smaller nozzles. Thus it is better to use a larger number of nozzles of smaller diameter with a reduced pitch between the nozzles.

#### 2.2.4. Distance and water pressure

The distance between the nozzle and the steel surface should be as small as practically possible and the delivered water pressure should be maximized to ensure that sufficient descaling is obtained in the shortest time possible. Raising the jet velocity considerably increases the descaling efficiency that is until the change in

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pressure across the spray nozzle attains a value of 400 kN/m<sup>2</sup> (0.4 MPa), when no significant increase in efficiency occurs (Sheppard and Steen, 1970).

### **2.2.5. Effect of scale temperature**

The effect of scale surface temperature on the efficiency of scale removal originates in the crack initiation and propagation behavior during air-cooling. When scale formed at high temperature is subjected to air-cooling, tensile stress is developed at the scale surface resulting from thermal contraction. When this tensile stress exceeds the fracture stress of the scale, the cracks initiated at the scale surface continuously propagate towards the scale-metal interface (Fukutsuka, *et al*, 1981). The scale surface temperature influences the removability of especially the scale on the silicon-killed steel slabs.

### **2.2.6. Mechanism of high pressure water descaling**

#### **2.2.6.1 INTRODUCTION**

Two principal effects may predominate, namely thermal effects as a result of water-cooling, and secondly, the mechanical effect of the water jet in striking the steel, thereby breaking the scale and removing it by direct force. The hydraulic removal of the scale is dependent on both the impact pressure and quantity of applied water per unit area. The total stress produced in the scale by descaling is the sum of the thermal stress produced by the cooling action of water jets, the stress due to the impact force of the jet and the depositional compression stress (the oxidation stress). The amount of stress in the scale is closely related with the hydraulic scale removal (Matsuno, 1980). The importance of the descaling mechanism in the present work concerns ways in which descalability may be evaluated in the laboratory. If there is no clarity on the descaling mechanism, there is no alternative to using actual high-pressure water sprays (which is not simple to implement in a laboratory). However, if the mechanism is primarily mechanical, simpler methods — for example, bending



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the sample around a set radius immediately upon removal from the furnace, and subsequently measuring the amount of scale remaining (as used by Tuck and Barlow [1972]) — may suffice.

#### **2.2.6.2 THERMAL EFFECTS**

The difference in temperature between the descaling water and the scaled steel or between the atmosphere and the scale when the steel is removed from the furnace is such that a thermal gradient can exist within the scale leading to thermal contraction of the surface of the scale. This causes the scale to curl and break away from the steel, i.e. the thermal shock created by the high energy impact breaks the scale and the short contact time limits the temperature drop of the product (Sheridan and Simon, 1995). The depth of scale, which would experience cooling as a result of the effects of the water jets, is only about 0.2 mm (as calculated in appendix 2).

Reducing the sample speed under the descaling spray increases the effective volume of the water applied to a given area of steel, reducing the temperature further and increasing the descaling action. The formation of steam, leading to disruption of the scale, may be capable of supplying sufficient energy to the scale to fracture the metal / scale interface. This mechanism depends however on the ability of the water to penetrate pores and fissures within the scale and thus produce an undercutting effect.

#### **2.2.6.3 MECHANICAL PRESSURE EFFECTS**

When considering the mechanical pressure effects on the other hand, it is noted that the force exerted by the water jets on the surface of the scale can produce displacement of the scale if porosity or detachment exists as a result of either scaling conditions or thermal contraction effects. This mechanism would be expected in friable scales, and would be of increased effectiveness at higher water pressures. In addition, the force of the water jet on the scale may crush and fragment the scale,

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following which the pieces of scale are removed by the water. In practice, these mechanisms may all contribute to scale removal:

- The stagnation pressure of the water jet forces water beneath the scale
- The generation of steam undermines and ruptures the scale

The figure below illustrates that, for low-carbon aluminium-killed steel, successful descaling relies on the impact pressure being high enough, supporting the mechanical removal mechanism. However, in the case of steels which are more difficult to descale - such as those containing significant amounts of nickel or silicon - a trade-off between impact pressure and the total amount of water is found: as the figure shows, successful descaling of the high silicon steel requires an impact pressure of 1.5 MPa at a lower water consumption (i.e. shorter dwell time beneath the jet), but successful descaling is obtained at an impact pressure of 0.5 MPa if more water (longer time) is used at 25 //m<sup>2</sup> specific water flow rate.

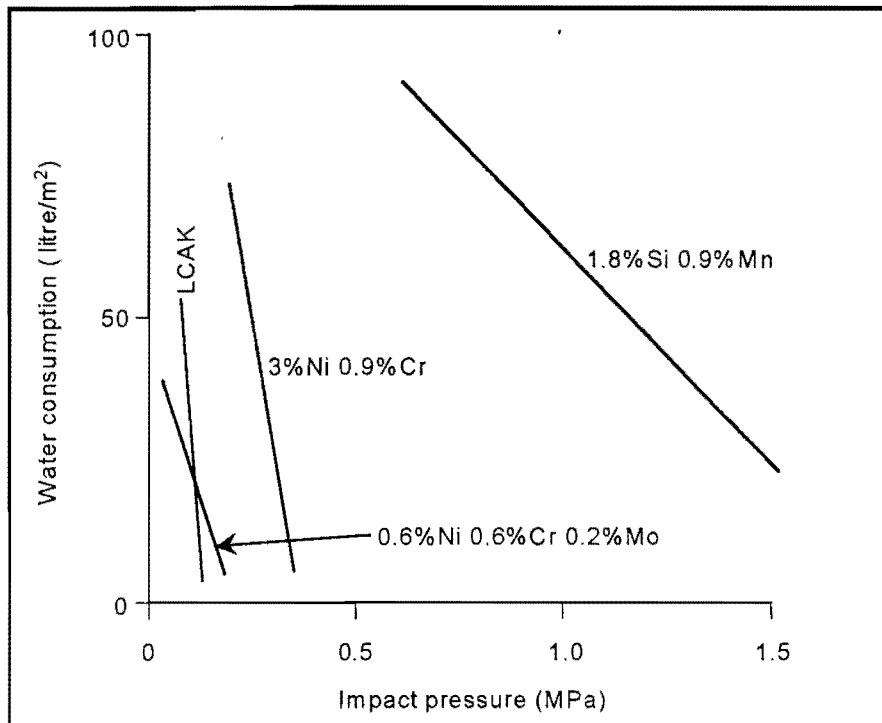


Figure 6 : Conditions required for successful descaling of four steel grades (low-carbon aluminium-killed steel, and 3 alloy steels). Descaling is successful if the actual jet impact and water consumption lie to the right of the relevant line. (Sheridan and Simon, 1995)

However, the effect of time suggests that a comparatively slow process - such as heat transfer through the scale, may contribute to descaling of these steels. The envisaged role of heat transfer is to quench the scale, causing it (or its outer layers) to contract, scale failure being the result of thermal shock (Sheridan and Simon, 1995). The pressure and time required to obtain successful descaling for given reheating conditions could be related by the parameter  $\Delta p t / l^2$  — where  $\Delta p$  is the header pressure,  $t$  the time allowed for descaling, and  $l$  the scale thickness. The dependence on the square of the scale thickness is in accordance with the control by heat conduction through the scale (Sheppard and Steen, 1970).

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In further indirect support of a possible role of thermal shock, Fukutsuka, *et al* (1981), ascribed the poorer descaling of silicon-killed steel to the great prevalence of through-thickness cracks in the scale on this steel — these cracks can give stress relief during cooling (Tominaga, *et al.*, 1982), so improving the resistance to thermal shock.

However, some quantitative indication of whether thermal shock is a feasible mechanism can be obtained by estimating the time required to cool the scale to a given depth. Assuming that conduction through the scale is rate determining, the time to cool the scale is of the order of  $l^2/\alpha$ , where  $l$  is the scale thickness and  $\alpha$ , the thermal diffusivity. Using literature values for the thermal conductivity and density of wustite (Akiyama *et al.*, 1992) and its heat capacity (FACT, 1998), the thermal diffusivity is estimated to be some  $8.3 \cdot 10^{-7} \text{ m}^2/\text{s}$  in the vicinity of  $1100^\circ\text{C}$ . Based on the typical thickness of the jet of around 8 mm, and slab speeds of 0.1 to 1 m/s (Morris, *et al*, 1996), the time that the scale is exposed to the water jet is between 8 and 80 ms — which, based on the thermal diffusivity, is only sufficient time to quench the outer 80 - 250  $\mu\text{m}$  of the scale (compared with the total scale thickness which may be 2 mm or more after reheating). This very limited cooling of the scale does bring the validity of the thermal shock mechanism into doubt (appendix 2).

This is further illustrated in cases where larger pieces of scale are observed upon their removal from the slab at the descaler. It is observed that the surface, which had been in contact with the steel, still glows red-hot, indicating that the full thickness of the scale had not been cooled down - suggesting that thermal shock was not effective. In addition, the observed removal of scale at positions up to several millimeters ahead of the jet also suggests a mechanical action (Blazevic, 1987).

Thus it does seem that the mechanism of hydraulic descalers is largely mechanical and that a mechanical means of descaling at temperature should yield an adequate simulation of in-plant hydraulic descaling.

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## 2.2.7. The effect of interfacial roughening on descaling

According to Asai, *et al.* (1997), the effect of impurities on the removability of primary scale on 0.02 and 0.1 mass percentage Si mild steels was investigated using hydraulic descaling tests and the results were as follows:

- The scale / metal interface becomes uneven for steels containing a small amount of Ni ranging to 0.05 mass %, and the unevenness is independent of oxidation temperature as well as Si content of steel. Thus the removability of primary scale reduces by an addition of a small amount of Ni. However, comparing the residual thickness of 0.05 and 0.1 mass % Ni steels, the thickness of both was almost equal (Asai, *et al.*, 1997).
- The cause of poor descaling in Si-added steel has been explained so far in terms of the fact that the impact by high-pressure water spray is reduced by the existence of molten eutectic compound (fayalite) between steel and scale. The fayalite increases the unevenness of the interface thus leading to poor descaling.

The qualitative indications are that steels with roughened interfaces, whether as a result of nickel enrichment or of fayalite formation, are more difficult to descale. However, it is not clear whether interfacial roughness correlates in a quantitative way with descalability or even what an appropriate way of quantifying the interfacial roughness is.

In one study, the interfacial roughness was quantified by determining the length of the scale-metal interface relative to a smooth interface (expressed as a ratio) (Asai, *et al.*, 1997). This roughness index was determined by examining cross-sections under an optical microscope. Some of the results are shown in figure 6 below. This figure (redrawn from the data of Asai, *et al.*, 1997) indicates the amount of residual scale after hydraulic descaling, together with the measured roughness index, for

steels with low and high nickel and silicon contents, which had been reheated at 1100°C and 1200°C in air. The figure shows the expected effect of silicon and nickel to increase the interfacial roughness and that descaling is less efficient if the interfacial roughness is larger.

This work does indicate that this simple roughness index might be useful but also hints at its limitations. For example, the figure shows that the higher-nickel higher-silicon steels which had been reheated at 1200°C had (as expected) the worst descaling behavior, but their roughness index was similar to that of steels with less silicon, and of the same composition reheated at the lower temperature. This suggests that something more than just the roughness index may be required. A contender could be a measure of the absolute size of the roughness (Asai, *et al*, 1997).

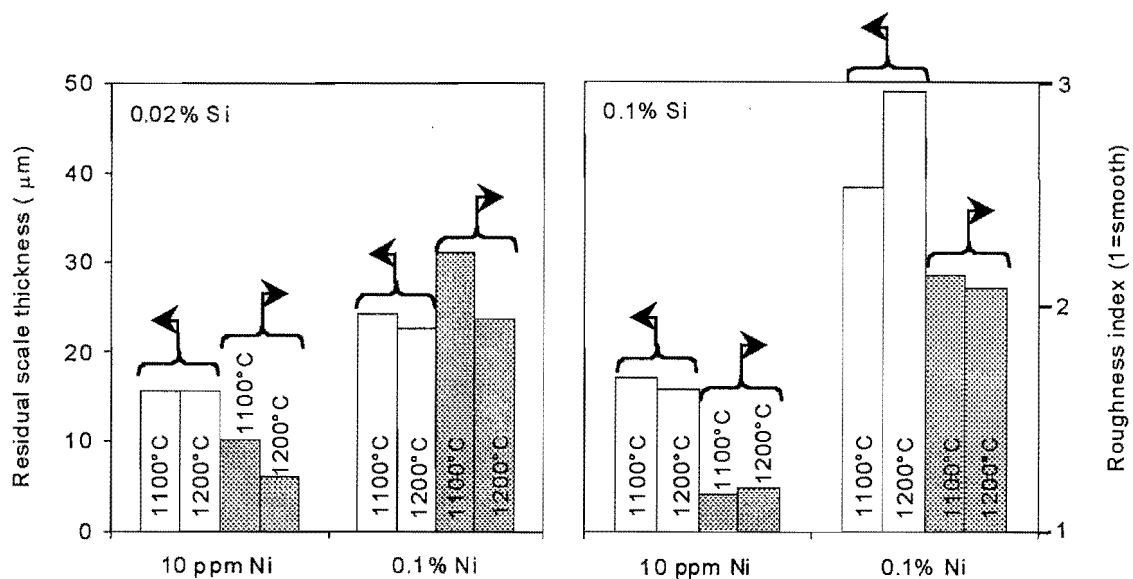


Figure 7 : Effect of steel composition and reheating temperature on the interfacial roughness and descaling behavior for steels containing 0.02% or 0.1% Si, together with 10ppm or 0.1 Ni %. The clear columns give the average thickness of scale remaining after descaling (plotted against the left-hand axis), and the shaded columns give the relative roughness of the scale-metal interface (right-hand axis) (Asai, *et al*, 1997).

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## 2.2.8. Mechanical Descaling

The above arguments indicate that the action of hydraulic descalers is largely mechanical. And as stated, this means that a mechanical means of descaling at temperature should yield an adequate simulation of in-plant hydraulic descaling. A number of papers relating to mechanical removal of the scale on stainless steel coils have been published (Liekmeier, 1982; Tuck, *et al*, 1972; Garceau, 1997; Ito, *et al*, 1997 and Fresnedo, *et al*, 1996). However apparently no systematic research has been performed on mechanical descaling methods therefore, an optimal descaling method has not yet been established.

The principle basically is to remove the relatively thick shield of coarse oxide by deforming the sample whilst still hot (i.e. approximately at the furnace temperature [1200°C]). This latter requirement is in contrast with available commercial mechanical descaling methods, where the samples are allowed to cool to room temperature before descaling (Engell and Peters, 1957). Tuck and Barlow (1972), used a hot bend test to assess the quantity of adherent scale remaining on a particular specimen after being deformed at the furnace temperature. A simple test rig was designed and built to bend hot specimens by a constant deformation of approximately 32 mm at their center. This degree of deformation was sufficient to remove some of the scale but not all. Thus after oxidation, each specimen was removed from the furnace, immediately bent in the jig and allowed to cool naturally. Loose scale was removed from the convex face, the specimen weighed and the convex face shot blasted to remove the fine shield of oxide that is very firmly adhered to the steel surface.

The force required to deform the specimens (100mm x 50mm) in this project was calculated to be 2.5kN (section 3.3). Thus the impact pressure of the mechanical descaler was 0.5 MPa (force / Area). Using equation A3.2 in appendix 2, the compressive strain exerted was calculated to be  $2.6 \times 10^{-6}$ . This value is of the same order as the calculated compressive strain ( $5.3 \times 10^{-6}$ ) for a typical commercial

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hydraulic descaler (calculations shown in detail in appendix 2). Based on these calculations, it can be assumed that mechanical descaling is likely to yield the same results as hydraulic descaling.

However, depending on the amount of scale and the surface roughness of the steel, the crust can be easy or difficult to remove by the mechanical descaling process. But regardless of the amount or nature of the scale, it should be completely removed if the quality of the steel is to be maintained.



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## 2.3. Reheating temperature

While high reheating temperatures (giving thicker scales) were found to increase scale adhesion, especially for silicon-killed steels, this was not observed for all grades (Palin, 1965). For aluminium-killed steels, lower reheating temperatures gave thinner scales, which were more difficult to remove (Morris, *et al.*, 1996) whereas steels susceptible to entanglement (silicon and nickel-containing steels) developed more entanglement at higher temperatures, with a negative effect on descalability. An indication of the complex effect of reheating temperature is given by the results of Sheppard and Steen (1970), which are redrawn in figure 8 below. This figure shows a combination of header pressure ( $\Delta p$ ) and time ( $t$ ) required to remove a scale of thickness  $l$ , for different reheating temperatures —clearly scales formed at 1000°C are more difficult to remove than those formed at both higher and lower reheating temperatures.

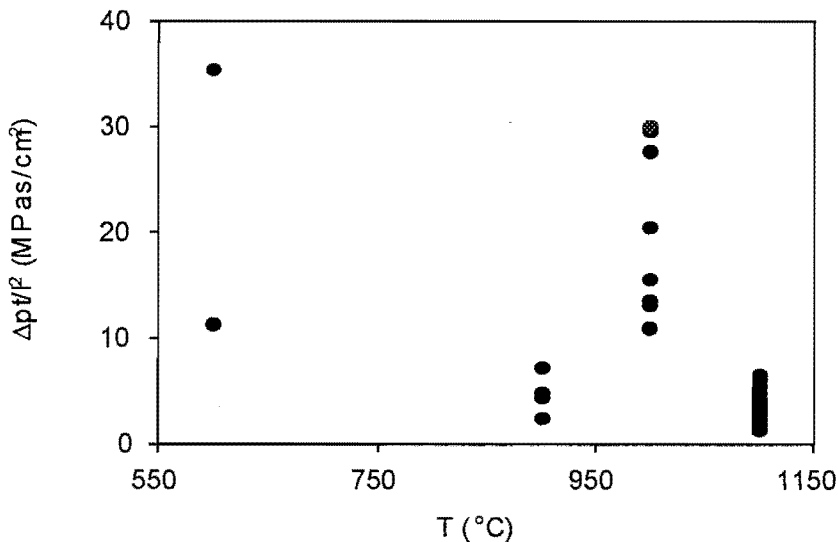


Figure 8 : Combinations of header pressure ( $\Delta P$ ) and time ( $t$ ) required to remove scale with thickness  $l$  from carbon steel which had been reheated at different temperatures.

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In addition to the nominal reheating temperature, the temperature cycle to reach this is expected to be of importance. For example, Fukutsuka *et al.* (1981), found that the amount of residual scale on slabs following descaling did not correlate well with either reheating time or temperature alone, but best with an integrated “degree of overheating” (DOH). DOH was defined as follows:

$$\text{DOH} = \int_{t_o}^{t_e} (T - T_o) dt$$

Where  $T_o$  is the critical temperature for overheating (1100°C yielded the best results),  $t_o$  is the time when the slab reaches  $T_o$ , and  $t_e$  is the time when the slab is removed from the furnace.

In addition to the thermal history of the slab as it reaches the reheating temperature, possible fluctuations in the surface temperature are expected to affect the scale — Abuluwefa *et al.* (1997b) found more cracks in the scale on samples which had passed through an actual reheating furnace, compared with samples exposed to nominally the same thermal cycle and atmosphere under laboratory conditions. The cracks were ascribed to temperature fluctuations in the furnace (temperature cycling is indeed a well-established method to induce cracking of oxide scales [Schutze, 1995]).

## 2.4. Reheating time

The plant observation that the descalability of the 11.5% chromium stainless steel is severely reduced by extended reheating emphasizes the importance of this variable. While this observation suggests a monotonic decrease in descalability with increased reheating time, this is not always the case. For carbon steel slabs, the amount of residual scale after descaling increased with reheating time, but peaked at just over two hours, dropped to a minimum at 2.5 hours, and then increased again. Hence time was varied in the first number of experiments, whilst keeping to the typical reheating temperature profiles used in stainless steel plants (Palin, 1965).

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## 2.5. Composition of gas atmosphere

### 2.5.1. Effect of water vapour

In oxidizing gas mixtures containing water vapour, the scale remained attached to the metal surface for considerably longer reaction times than in the absence of vapour, and the scale growth progressed in accordance with the parabolic rate for pure Fe (Sheasby, *et al*, 1984). This might imply that the presence of water vapour in the oxidizing gas enhanced the rate of creep in the scale and thus increased the rate of scale growth. Pores which developed (probably due to mechanical cracking) at the metal / scale interface in the presence of water vapour migrated away from the interface as the oxidation progressed. In the scales formed on the Fe-C alloys the pores migrated into the wustite layer and became larger. When H<sub>2</sub>O was absent, the scale was detached from the metal, preventing further Fe transport across the scale. However, oxidation continued in the detached FeO and Fe<sub>3</sub>O<sub>4</sub> resulting in a thinner layer of FeO and thicker layers of Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub>. Thus the presence of water vapour in the laboratory furnace atmosphere is essential to achieve scale growth that is comparable to that in the reheating furnace (Sheasby, *et al*, 1984). To obtain a product gas containing 3% excess oxygen for example, the other components of the gas mixture can be calculated by a mass balance and based on these calculations; about 16% water vapour is required in the gas mixture (appendix 3).

### 2.5.2. Effect of oxygen

The amount of free oxygen has a strong effect on the scaling of carbon steels apparently because the initial period of scale growth (lasting an hour or more) often exhibits linear kinetics, where the scaling rate is limited by gas-phase oxygen diffusion to the scale surface (Abuluwefa, *et al*, 1996, 1997a). Despite this, the

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extensive study of Morris *et al.* (1996) did not find a large effect of excess oxygen (in the range 1-6%) on the descalability of carbon and low-alloy steels. Whilst it appeared important to test possible effects of the amount of excess oxygen, in the initial experiments gas atmospheres with 3% excess oxygen were used – this appeared to be typical of many reheating furnaces. Because of probable mass transfer control (for the initial scaling of carbon and stainless steels), the gas flow rate across the sample should be controlled to yield similar mass transfer constants to those encountered in a reheating furnace. Control of the gas flow rate is important in the case of stainless steels also due to the formation of the volatile species  $\text{CrO}_3$ , which is expected to form at temperatures above 1000°C (Stearns *et al.*, 1974).

## 2.6. Surface finish

During oxidation studies, ground or polished surfaces are often used, to provide a reproducible starting condition. However, the nature of the surface finish can affect the scaling process. For scales which grow by cationic diffusion, the new scale grows on top of the old, which implies that the original surface finish is trapped at the scale-metal interface. Only in the case of extensive internal oxidation is the surface finish expected to be less important (Boggs, 1973). In this project, hot-rolled plate samples were used and thus the samples had the same surface finish and starting condition.

## 2.7. Effect of cooling conditions after descaling

The microstructure of the scale cross-section is greatly affected by the rate of cooling of the samples in the case of carbon steel. If wustite is the only phase formed in the early stages of oxidation, the wustite layer decomposes and oxidizes to form relatively thick hematite and magnetite layers during slow cooling of the sample (Sheasby, *et al.*, 1984). Thus, the slower the cooling rate the greater the volume fraction of higher oxides in the scale.