Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

Doctoral thesis

by

Jerker Sterneland

Stockholm
May 2002
Division of Metallurgy
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Abstract

The reduction behaviour of the olivine iron ore pellet MPBO was studied in laboratory scale at KTH as well as in the LKAB experimental blast furnace. Initially, a new reduction-under-load, or so-called reduction/softening/melting, test equipment was developed. Experiments using different reducing conditions, corresponding to different radial positions of the blast furnace, were conducted. The experiments included different temperature profiles, reducing atmospheres and mechanical loads applied on the sample bed to simulate the varying conditions in the blast furnace process. The progress of reduction was investigated, as well as the processes of sintering and contraction during reduction. A model of the carburisation (pick-up of carbon by the reduced iron) and melt-down process during rapid contraction was presented.

Laboratory testing of MPBO pellets was compared with results from the LKAB experimental blast furnace. The reduction of iron ore pellets in the experimental blast furnace was surveyed by a dissection of the furnace after quenching. The high temperature phenomena occurring when reducing the MPBO pellet, with limited softening and a short temperature range of the melting process, resulting in a thin cohesive zone, were found to be the same in laboratory tests and in the experimental blast furnace. The reduction down through the burden of the experimental blast furnace was similar, but not identical to the results of the RUL experiments. The differences were found to be due to different reducing conditions. Therefore, it was concluded that a simulation of the reduction occurring in the blast furnace can be performed in laboratory scale, provided the experimental conditions are correctly chosen.

Finally, a modification to further improve the properties of the MPBO pellets was examined. With the aim to improve the blast furnace process, coating of blast furnace pellets was investigated in laboratory scale, as well as in the LKAB experimental blast furnace. Olivine, dolomite and quartzite were used as coating agents. In laboratory scale the sticking prevention action of the different coating materials was verified, in established test methods as well as in new test methods, modified for blast furnace conditions. Testing of the coated pellets in the experimental blast furnace revealed several advantages; significantly reduced blast furnace flue dust generation, improved gas utilisation and a smoother blast furnace operation with a potential for a lowered fuel rate.

Keywords: Olivine, pellets, pellet testing, reduction/softening/melting, MPBO, blast furnace, reduction, quenching, dissection, coating, sticking, coated pellets.
Supplements

This thesis is based on the following supplements:

**Jerker Sterneland** and Ashok Lahiri:
*Contraction and melt down behaviour of olivine iron ore pellets under simulated blast furnace conditions*

**Jerker Sterneland** and Ashok Lahiri:
*New equipment for simulation of blast furnace reduction*
Presented at the 58th Ironmaking Conference, Chicago, IL, USA, March 1999.

**Jerker Sterneland** and Mats Hallin:
*The use of an experimental blast furnace for raw material evaluation and process simulation*
Presented at the 6th Japan-Nordic Countries Joint Symposium, Nagoya, Japan, November 2000.

**Jerker Sterneland**, Margareta Andersson and Pär Jönsson:
*Comparison of iron ore reduction in an experimental blast furnace and a laboratory scale simulation of the blast furnace process*
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**Jerker Sterneland** and Pär Jönsson:
*The use of coated pellets in optimising the blast furnace operation*
ISRN KTH/MSE--02/15--SE+METU/ART
Contributions to the supplements by the author:

**Supplements I and II:**

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<tr>
<td>Evaluation of experiments</td>
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**Supplement III:**

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**Supplement IV:**

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**Supplement V:**

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<td>80 %</td>
</tr>
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<td>Writing</td>
<td>75 %</td>
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The author also contributed to the following works:

Lawrence Hooey, Jerker Sterneland and Mats Hallin:

*Evaluation of operational data from the LKAB experimental blast furnace*
Presented at the 60th Ironmaking Conference, Baltimore, MD, USA, March 2001.

Lawrence Hooey, Jerker Sterneland and Mats Hallin:

*Evaluation of high temperature properties of blast furnace burden*
Proceedings of 1st International Meeting on Ironmaking, pp. 205-220.
Presented at the 1st International Meeting on Ironmaking, Belo Horizonte, Minas Gerais, Brazil, September 2001.

Mats Hallin, Lawrence Hooey, Jerker Sterneland and Dag Thulin:

*LKAB:s experimental blast furnace and pellet development*
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Jerker Sterneland and Magnus Andersson:

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SUPPLEMENT IV - Comparison of iron ore reduction in an experimental blast furnace and
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Jerker Sterneland, Margareta Andersson and Pär Jönsson

SUPPLEMENT V - The use of coated pellets in optimising the blast furnace operation.
Jerker Sterneland and Pär Jönsson
Chapter One

Introduction

Ever since the beginning of blast furnace ironmaking, more than half a millennium ago, the aim of the blast furnace process has, of course, been to produce iron. Increased commercialism during the time of industrialisation, especially in the past century, refined the ironmaking and steelmaking processes into more and more sophisticated techniques. Thereby, also the demands on ironmaking and steelmaking have sharpened. Today, the aim of the blast furnace is not only to produce iron but also to furnish the steel mill with correct amounts of hot metal, with a specified, consistent composition, at the right time and at a low cost. A smooth operation of the blast furnace, leading to high quality consistent hot metal, low production costs, and the possibility of reaching a high productivity, is to a large extent depending on the shape and position of the cohesive zone. A narrow cohesive zone maximises permeability and thereby also productivity, while a position of the zone deep in the furnace promotes a larger extent of indirect reduction, minimises heat consumption, lowering the production costs and provides the possibility of stabilising hot metal composition over time. The shape and position of the cohesive zone, and its melting down behaviour, are in turn governed by the properties of the iron ore in combination with the operation practice. Therefore, testing of iron ore is important to achieve the goals of blast furnace ironmaking. Laboratory testing of iron bearing materials for use in the blast furnace has four main areas of interest:

- optimisation of iron ore mix (mainly in sinter production),
- optimisation of mixing of charge materials (sinter, pellets, briquettes and coke),
- improvement of properties, especially high temperature properties, of iron bearing materials; and
- increasing knowledge of the complex phenomena occurring in the blast furnace.

Many of the reduction, softening and melting tests described in literature have been developed with the aim to find out the optimum iron ore mix and sinter chemistry, or to find out the best way of mixing different types of iron bearing materials in the blast furnace. Some tests are used to improve the high temperature properties of iron ore agglomerates. The laboratory equipment used in this investigation is of the fourth category; it was developed mainly to study the complex high temperature phenomena occurring in the burden of the blast furnace. Fundamental studies to increase the knowledge of the complex process of reduction in the blast furnace was therefore the aim of the first part of this work, the laboratory reduction studies covered by Supplements I and II. The laboratory
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studies included investigations of the influence on reduction by different reducing conditions, simulating different radius positions in the blast furnace, by the load applied etc. To describe the processes of reduction and melting of the olivine pellets, several experiments were interrupted at different stages of the reduction, and the materials obtained were studied under microscope.

Information of the reduction behaviour of iron oxides can also be obtained by using an experimental pilot-plant blast furnace. Supplement III is included in this thesis to describe the features and performance of such an equipment, the LKAB experimental blast furnace, and how it can be used as a research tool. The LKAB experimental blast furnace, used in parts of this work, is large enough to simulate a commercial size blast furnace, of which an example is given in Supplement III, and at the same time small enough to be economically operated for research purposes. The comparatively small size of the furnace provides opportunities for quenching and dissecting the furnace, so that up-dated information on the reduction degree etc. of iron oxides at different positions in the furnace can be obtained. However, since experiments in an experimental blast furnace still can be quite costly, it would also be very useful to carry out trials in a small laboratory furnace to determine what trials that are worthwhile to perform in the experimental blast furnace. Therefore, the second part of this work, covered by Supplement IV, aims to compare the results of laboratory reduction experiments with results obtained from the experimental blast furnace. Except for the progress of reduction during the different experiments, also the softening and melting behaviours are compared.

Finally, the third part of this thesis, presented in Supplement V, gives an example of how the experimental blast furnace can be used in developing new blast furnace products. The experimental blast furnace is regularly used in campaigns, often followed by an excavation of the furnace after nitrogen quenching. During earlier dissections of the furnace a sticking, or clustering, phenomenon was observed. The pellets of the burden were found to start sticking together in the middle and lower shaft, before the material entered the cohesive zone. If clustering of the particles in a pellet bed is pronounced, the descending movement of the ferrous burden layers in the blast furnace can be disturbed. In the worst case the burden descent can be held up, resulting in hanging, followed by a slip when the burden suddenly again moves downwards. Another possible consequence of clustering in the pellet layers is the forming of gas channels. As the blast furnace process continuously improves and the fuel rates are minimised, the need for stability in the blast furnace process has increased. One way of improving the process stability is to improve the flows of solids and gases through the blast furnace shaft, resulting in an improved and more uniform gas/solid contact. In the third part of this work the effects of coating of the MPBO pellet is analysed, by the use of the experimental blast furnace as well as laboratory tests. The aim is to optimise the blast furnace process, by smoothing the operation.
The usage of laboratory experiments and the experimental blast furnace in developing new blast furnace products is schematically exemplified below.

![Diagram](image)

This sketch shows, in principal, how the experimental blast furnace and laboratory experiments were used in *Supplement V* of this thesis. For other purposes, however, the steps of development can be quite different, and involve also other activities.
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace
Chapter Two

Literature survey, part I: Laboratory testing

Laboratory testing is extensively used to investigate different properties of iron bearing materials. Standardised test procedures aiming to describe the quality of iron bearing materials are very useful, as long as it is kept in mind that quality in this sense often is a measure of consistency or optimal performance under certain, often idealised, conditions but not a guarantee for satisfactory behaviour in the blast furnace.

Laboratory tests aiming to predict the blast furnace performance have often been questioned. The blast furnace burden is from the stockline and down through the furnace exposed to a changing environment, in terms of temperature, gas composition etc. The temperature profile and gas composition contours in a blast furnace are the results of very complicated interactions among properties of coke, ore and fluxes, burden distribution as well as gas distribution, and other operational parameters. Therefore, the conditions iron bearing materials are exposed to during the descent in the blast furnace are very much determined of the material itself, i.e. gas compositions and temperature profiles within the blast furnace changes with the properties of the burden materials used. It is reasonable to suggest that without a complete knowledge of the state of the furnace from the stockline to the melting, one cannot predict the behaviour of pellets and the performance of the blast furnace. Therefore laboratory testing can be misleading, and the results should be evaluated with care.

Nevertheless, laboratory testing aiming to predict blast furnace performance can still be used to qualitatively determine if a burden material has a potential as blast furnace feed material. In this respect laboratory testing can be considered as an effective and inexpensive way of testing materials compared to possibly costly failures in larger scale. Also, many fundamental studies can, and sometimes have to, be performed in laboratory scale, such as studies of mechanisms of interactions between different materials etc. As the focus of this investigation was the behaviour of olivine pellets, the literature survey was concentrated on pellets as the blast furnace burden material.

2.1 STANDARDISED METHODS OF TESTING IRON ORE PELLETS

Improved testing techniques are continually being developed, especially regarding high temperature properties of iron bearing materials. However, in the present state of the art, there is no single laboratory test, which can approximate the very complex successive processes to which the burden is exposed in its descent from the distributor to the melting zone. A true simulation in laboratory scale appears to be impossible; final optimisation of blast furnace smelting is still achieved only in the blast
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furnace itself. Consequently, the investigator is obliged to consider a number of chemical, physical and metallurgical tests in conjunction with each other. Such combination of test results might then be correlated, usually satisfactorily, with actual performance data for a given furnace with a given composition of the burden.

Worldwide co-operation within the International Organization for Standardization, ISO, has led to the standardisation of several test methods to describe the quality of iron oxides, where the following are considered the most important for blast furnace pellets:¹

**Physical tests**
- ISO 3271 – Determination of tumble strength. Measurement of strength and abrasion resistance (fractions of >6.3 mm and <0.5 mm respectively) after tumbling.
- ISO 4700 – Determination of crushing strength (also known as cold compression strength, CCS). Measurement of the average force required to crush a pellet particle.

**Metallurgical tests**
- ISO 4695 – Determination of reducibility. Measurement of reducibility, isothermally at 950 °C.
- ISO 4698 – Determination of relative free-swelling index. Measurement of swelling index after isothermal reduction at 900 °C.
- ISO 7992 – Determination of reduction properties under load. Measurement of reducibility, resistance to gas flow and bed contraction during isothermal reduction under load at 1050 °C.
- ISO 13930 – Dynamic test for low-temperature reduction-disintegration. Measurement of strength and abrasion (fractions of >6.3 mm (LTD) and <0.5 mm respectively) after low temperature reduction (isothermally at 500 °C) and simultaneous tumbling.

2.2 REDUCTION, SOFTENING AND MELTING TEST METHODS

2.2.1 Survey of existing reduction, softening and melting test methods
The standardised test methods for measuring reducibility, pellet strength and swelling properties etc., are important tools for quality control of pellet production, but not for predicting the behaviour of a burden material in the blast furnace.² To simulate the reduction in the transition zone between the solid and liquid phases of the blast furnace several test methods have been developed, which all could be designated the name of reduction/softening/melting tests, or softening/melting tests only. Although several equipment for this purpose have been developed, with similar apparatuses and procedures - all with the aim of simulating the high temperature region of the blast furnace - none have yet been standardised. One reason is that in these kinds of simulations it is sometimes difficult to choose the experimental conditions because of lack of knowledge of the conditions in the high temperature zones of the blast furnace. Furthermore, when the experimental conditions have been chosen according to
the operation of one blast furnace, those conditions would probably not apply to another furnace, and, to make things even worse, they are also expected to change from time to time. Because of the complexity and diversity of the conditions in the blast furnace, standardisation of reduction/softening/melting tests is not likely to be considered in a near future.\cite{3}

During softening and melting tests, differential pressure across the sample bed, height of sample bed, composition and flow rates of inlet and exit gas, and weight of the sample are continuously measured. Subsequently, the degree of iron oxide reduction (or oxygen loss) and bed contraction are calculated. The data are usually presented in the form of graphs of differential pressure, degree of reduction, and degree of bed contraction versus sample or furnace temperature, or testing time. From these graphs various temperatures that correspond to the start of softening, maximum pressure differential, start of melting and end of melting are obtained. The degrees of reduction at specific temperatures are also estimated.

To better express the softening and melting properties of iron bearing materials Mu et al suggested several melting down test indices.\cite{4} Several proposals for a definition of a single index to comprehensively characterise the softening and melting behaviours have been suggested:

- Tayama et al defined an "S-value" based on the area under the differential pressure versus temperature curve from the start of softening to end of melting.\cite{5}
- Clixby defined a critical zone of temperature and reduction in which a rapid rise in differential pressure is experienced by the sample.\cite{6}
- Yamaoka et al defined an average permeability resistance coefficient over the softening and melting temperature range.\cite{7}
- Gudenau et al calculated a dimensionless pressure parameter to represent the differential pressure versus temperature curve during the entire test.\cite{6}

So far, no standardisation has been made, and none of the above definitions has managed to be adopted worldwide.

The results of reduction, softening and melting tests can be used for:

- evaluation of the effects of using different types of materials on the cohesive zone configuration in the blast furnace under constant operating conditions,
- evaluation of the effects of using different blast furnace operating practices and coke properties on the cohesive zone configuration when using a constant iron bearing burden; and
- modification of the production conditions for the iron bearing material in order to obtain the desired softening and melting characteristics.

In Table 2.1 several test procedures found in the literature\cite{2,5,6,8-15} are listed.
Table 2.1. Specifications of test procedures found in literature.

**Group 1: Constant heating rate and gas composition**

<table>
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<tr>
<th>Laboratory</th>
<th>Sample weight (g)</th>
<th>Bed diam. (mm)</th>
<th>Bed height (mm)</th>
<th>Coke layers</th>
<th>Heating rate (°C/min)</th>
<th>max T (°C)</th>
<th>Gas composition</th>
<th>Flow rate (Nm/min)</th>
<th>Load (kg/cm²)</th>
</tr>
</thead>
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<tr>
<td>BHP, Australia [6]</td>
<td>500</td>
<td>55</td>
<td>130</td>
<td>13 (g)</td>
<td>Constant</td>
<td>6</td>
<td>1200</td>
<td>Constant</td>
<td>15</td>
</tr>
<tr>
<td>BHP International Group, Australia [8]</td>
<td>87</td>
<td>42</td>
<td>42</td>
<td>13 (g)</td>
<td>Constant</td>
<td>10</td>
<td>1000</td>
<td>Constant</td>
<td>7.2</td>
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<tr>
<td>British Steel, Teesside, Gr. Britain [6,10,15]</td>
<td>800</td>
<td>90</td>
<td>66</td>
<td>1</td>
<td>Constant</td>
<td>3,35,6</td>
<td>1350</td>
<td>Constant</td>
<td>60</td>
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<td>Japanese laboratory [8]</td>
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<td>42</td>
<td>35</td>
<td>Yes</td>
<td>Constant</td>
<td>10</td>
<td>1100</td>
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<td>Kobe, Japan [6]</td>
<td>500</td>
<td>75</td>
<td>50</td>
<td>Constant</td>
<td>10</td>
<td>1100</td>
<td>Constant</td>
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<td>MEFOS, Luleå, Sweden [2,6]</td>
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<td>70</td>
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<td>5</td>
<td>1400</td>
<td>Constant</td>
<td>30</td>
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<tr>
<td>Nippon Steel, Hirohata, Japan [5,6]</td>
<td>105</td>
<td>70</td>
<td>70</td>
<td>Constant</td>
<td>5, 3.6 or 2.8</td>
<td>1500</td>
<td>Constant</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>CSM, Italy [6]</td>
<td>500</td>
<td>70</td>
<td></td>
<td></td>
<td>Only N₂, pre-reduced sample</td>
<td>3</td>
<td>1400</td>
<td>Only Ar, pre-reduced sample</td>
<td>50</td>
</tr>
<tr>
<td>USSR [13]</td>
<td>30</td>
<td>60</td>
<td>Constant</td>
<td>10</td>
<td>1300</td>
<td></td>
<td></td>
<td>low</td>
<td>Constant 2</td>
</tr>
<tr>
<td>Burghardt, Germany [6]</td>
<td>1200</td>
<td>125</td>
<td>Isothermal</td>
<td>-</td>
<td>1050</td>
<td></td>
<td>Constant</td>
<td>40</td>
<td>83</td>
</tr>
<tr>
<td>Hoogovens [6]</td>
<td>500</td>
<td>71</td>
<td>88</td>
<td>Isothermal</td>
<td>-</td>
<td>1050</td>
<td>Constant</td>
<td>40</td>
<td>30</td>
</tr>
</tbody>
</table>

**Group 2: Constant heating rate and varying gas composition**

<table>
<thead>
<tr>
<th>Laboratory</th>
<th>Sample weight (g)</th>
<th>Bed diam. (mm)</th>
<th>Bed height (mm)</th>
<th>Coke layers</th>
<th>Heating rate (°C/min)</th>
<th>max T (°C)</th>
<th>Gas composition</th>
<th>Flow rate (Nm/min)</th>
<th>Load (kg/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centre de Res. Minérales, Canada [8]</td>
<td>150</td>
<td>42</td>
<td>60</td>
<td>4</td>
<td>Constant</td>
<td>6,7</td>
<td>1600</td>
<td>Var. 4 steps</td>
<td>9</td>
</tr>
<tr>
<td>Dofasco Hamilton, Ontario, Canada [8]</td>
<td>500</td>
<td>75</td>
<td>32/32</td>
<td>4</td>
<td>Constant</td>
<td>5</td>
<td>1600</td>
<td>Var. 4 steps</td>
<td>20</td>
</tr>
</tbody>
</table>
### Table 2.1. Continued.

#### Group 3: Varying heating rate and constant gas composition

<table>
<thead>
<tr>
<th>Laboratory</th>
<th>Sample weight (g)</th>
<th>Bed diam. (mm)</th>
<th>Bed height (mm)</th>
<th>Coke layers</th>
<th>Heating rate (°C/min) max T (°C)</th>
<th>Gas composition</th>
<th>Flow rate (Nl/min)</th>
<th>Load (kg/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BHP Central Research Lab., Australia [8]</td>
<td>500-950</td>
<td>75</td>
<td>75</td>
<td>15/25</td>
<td>Varying</td>
<td>10/4, 3/2</td>
<td>1200</td>
<td>Constant 1</td>
</tr>
<tr>
<td>Centro Giuluppo Materiali, Italy [8]</td>
<td>500</td>
<td>70</td>
<td>60-70</td>
<td>?</td>
<td>Varying</td>
<td>6/0/7</td>
<td>1600</td>
<td>Constant 0,5</td>
</tr>
<tr>
<td>CNRM [6]</td>
<td>30</td>
<td>30</td>
<td>30</td>
<td>Varying</td>
<td>Lin., 5 to 0.5</td>
<td>1300</td>
<td>Only N₂, pre-reduced sample</td>
<td>Constant 2</td>
</tr>
<tr>
<td>Kobe, Japan [6]</td>
<td>3 particles</td>
<td>42</td>
<td>monolayer</td>
<td>Varying</td>
<td>8,6/0,6,6</td>
<td>1500</td>
<td>Constant 2</td>
<td>Constant 0,25</td>
</tr>
<tr>
<td>Nippon Koken, Kehin, Japan [6]</td>
<td>500</td>
<td>75</td>
<td>65</td>
<td>Varying</td>
<td>5/3/5</td>
<td>1600</td>
<td>Constant 30</td>
<td>Constant 1</td>
</tr>
<tr>
<td>Nippon Steel, Japan [6]</td>
<td>500</td>
<td>70</td>
<td>70</td>
<td>Varying</td>
<td>10/5</td>
<td>1400</td>
<td>Constant 30</td>
<td>Constant 1</td>
</tr>
<tr>
<td>RWTH Aachen, Germany [6]</td>
<td>50</td>
<td>30</td>
<td>basket</td>
<td>Varying</td>
<td>10/4</td>
<td>1600</td>
<td>Constant 30</td>
<td>No</td>
</tr>
<tr>
<td>RWTH Aachen, Germany [6]</td>
<td>400</td>
<td>60</td>
<td>75</td>
<td>Varying</td>
<td>10/4</td>
<td>1600</td>
<td>Constant 30</td>
<td>Class V, 1,1</td>
</tr>
<tr>
<td>Tammann, Brazil [8]</td>
<td>500</td>
<td>60</td>
<td>120</td>
<td>20/20</td>
<td>Varying</td>
<td>10/4, 3/2/5</td>
<td>1600</td>
<td>Constant 1</td>
</tr>
<tr>
<td>Usininas Research Centre, Brazil [8]</td>
<td>500</td>
<td>60</td>
<td>120</td>
<td>20/20</td>
<td>Varying</td>
<td>5/3/5/2</td>
<td>1400</td>
<td>Constant 2</td>
</tr>
</tbody>
</table>

#### Group 4: Varying heating rate and gas composition

<table>
<thead>
<tr>
<th>Laboratory</th>
<th>Sample weight (g)</th>
<th>Bed diam. (mm)</th>
<th>Bed height (mm)</th>
<th>Coke layers</th>
<th>Heating rate (°C/min) max T (°C)</th>
<th>Gas composition</th>
<th>Flow rate (Nl/min)</th>
<th>Load (kg/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Armco, Ohio [6,13]</td>
<td>10-12 part.</td>
<td>48</td>
<td>monolayer</td>
<td>Varying</td>
<td>6/4/3/2</td>
<td>1320</td>
<td>Var. 8 steps</td>
<td>26-36</td>
</tr>
<tr>
<td>Bethlehem [6]</td>
<td>10 partic.</td>
<td>60</td>
<td>monolayer</td>
<td>Varying</td>
<td>3,3/6,6</td>
<td>1550</td>
<td>Var. 4 steps</td>
<td>18-40</td>
</tr>
<tr>
<td>British Steel, Teeside, Gr. Britain [8,11]</td>
<td>450-750</td>
<td>80</td>
<td>60</td>
<td>25/25</td>
<td>Varying</td>
<td>9,2/7,8,9,2/5,4</td>
<td>800</td>
<td>Var. 2 steps</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>800</td>
<td>100</td>
<td>70</td>
<td>Yes</td>
<td>Varying</td>
<td>10/4, 3/2/5</td>
<td>Var. 4 steps</td>
<td>25-50</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>1000</td>
<td>100</td>
<td>90</td>
<td>Yes</td>
<td>Varying</td>
<td>5/2/5</td>
<td>Var. 4 steps</td>
<td>22-41</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>600-700</td>
<td>75</td>
<td>70</td>
<td>Yes</td>
<td>Varying</td>
<td>5,5/6/6,7</td>
<td>Var. 4 steps</td>
<td>22-45</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>500</td>
<td>70</td>
<td>70</td>
<td>Yes</td>
<td>Varying</td>
<td>5,7/1,3,5/7</td>
<td>Var. 4 steps</td>
<td>7-30</td>
</tr>
<tr>
<td>LKAB Melmerget, Sweden [2,8,14]</td>
<td>300</td>
<td>50</td>
<td>50</td>
<td>No</td>
<td>Varying</td>
<td>9,5/8</td>
<td>Var. 5 steps</td>
<td>23-45</td>
</tr>
<tr>
<td>Nippon Koken, Fukuyama, Japan [6,8]</td>
<td>450-650</td>
<td>80</td>
<td>60</td>
<td>25/25</td>
<td>Varying</td>
<td>9,8/6</td>
<td>Var. 4 steps</td>
<td>25-45</td>
</tr>
<tr>
<td>Sumitomo, Japan [6,8]</td>
<td>700</td>
<td>70</td>
<td>100</td>
<td>Varying</td>
<td>10/0/5</td>
<td>1550</td>
<td>Var. 4 steps</td>
<td>25-45</td>
</tr>
</tbody>
</table>
2.2.2 Limitations of conventional reduction, softening and melting tests

The majority of the reduction, softening and melting test methods that have been developed aim to simulate the blast furnace conditions completely or to some extent. Although the softening and melting tests attempt to meticulously reproduce the time-temperature-gas composition-stress cycles experienced by the iron bearing materials in the blast furnace, they do have some limitations. For instance, heat transfer and oxygen transfer reactions between burden materials and gases in the blast furnace are interrelated,\cite{16} but both gases and solids are heated externally in the reduction, softening and melting tests. Consequently, heat transfer in the furnace and its possible effect on the kinetics of iron oxide reduction cannot be accurately simulated. Further, the operating conditions found in the blast furnace are so complex and diverse that they can only be obtained during operation of a blast furnace. In the attempts to simulate those conditions in the laboratory tests some limitations, or assumptions, therefore have to be accepted. A further drawback of the reduction, softening and melting test methods is that, as mentioned earlier, there is yet no standardised method to predict the blast furnace performance based on the test results, therefore the data obtained by different test methods are often not comparable.

When the iron burden layers soften during their descent through the furnace, the permeability decreases, and therefore the gas flow rate through the fused layers decreases too. The effects of loss in permeability on gas flow, and perhaps, on the reduction kinetics are not accurately simulated in the existing test methods, since they often employ constant gas flows. Further, laboratory tests are often conducted at approximately atmospheric pressure, while the gas pressure inside the blast furnace can be two to four times higher. Some investigators have reported on the effects of alkali and sulphur on the softening and melting behaviour under laboratory conditions, but the true effects in the blast furnace are not clearly known.\cite{6,10,17}

Some limitations are caused by experimental difficulties. Among the reduction, softening and melting test methods found in literature, as shown by Table 2.1, the following limitations have been found:

- Constraints in sample or crucible size.
- Constant instead of varying rate of temperature increase.
- Restrictions in maximum temperature attainable.
- Constant instead of varying reducing gas composition.
- Only nitrogen and carbon monoxide, and no carbon dioxide or hydrogen, considered in the reducing gas.
- Too low gas flow rates.
- No, too small, or constant mechanical load.
One or more of these constraints makes a precise prediction of the behaviour of the burden in the blast furnace impossible. In *Table 2.1* the test procedures found in the literature were divided into four groups, classified by applied heating rate and gas composition. The test methods of the first two groups use a constant heating rate, which is not considered to be the case in the blast furnace. Group 3 applies varying heating rates but uses a constant reducing gas (often with 30% CO/70% N\(_2\)), which is indeed not suitable for blast furnace simulations either. In group 4, the test methods are the closest to be able to predict the behaviour of the burden in the blast furnace. Although they apply varying heating rates and gas profiles, there are still some limitations: often the applied load is at a constant level, some do not use hydrogen in the reducing gas, some use too small samples (monolayers) or too small crucibles to get the realistic aerodynamic properties of a packed bed. Most of the test methods also use a stepwise manner in changing heating rates and gas profiles.

### 2.2.3 Selection of test conditions for reduction, softening and melting tests

The selection of test conditions will be a function of the intended application of the test results, and depends on whether a qualitative simulation of the softening and melting phenomena is sufficient, or a more quantitative simulation for obtaining accurate data regarding softening and melting temperatures, degree of reduction, and permeability is required. Test precision, equipment and operating costs, and ease of operation also need to be taken into consideration. Test methods which use constant conditions, i.e. temperature, heating rate, load, gas composition and flow rate etc., often require simpler equipment, incur fewer operation costs and are easier to use in comparison to those which use time dependent, variable conditions. Therefore, these tests can be carried out more frequently and may be sufficient for routine quality monitoring. On the other hand, evaluation of new types of iron bearing materials and/or blast furnace operating practices probably requires more quantitative simulations of the softening and melting phenomena, involving complex time dependent conditions in the testing.

### 2.3 SUMMARY

In this chapter testing of pellets, in general terms, and test methods of reduction, softening and melting behaviour, in particular, were discussed on the basis of what could be found in literature. A lot of standardised test methods for pellets and other types of iron bearing materials have been developed, and are frequently used as a control measure of quality consistency of iron bearing materials. When it comes to simulation of the whole blast furnace process, and especially the high temperature region of the furnace, there is no standardised test method available. The reason is that the blast furnace process is too complex to be imitated in a simplified test. A simulation of the blast furnace process requires advanced equipment that can handle continuously changing heating rates, gas compositions and load programs up to temperatures higher than all standardised testing methods of iron bearing blast furnace burden materials. Therefore, a lot of different reduction, softening and melting test methods are proposed in the literature, all including more or less simplifications and...
limitations. The limitations of conventional reduction, softening and melting test methods were outlined in this literature survey, in order to facilitate the choice of design for a new laboratory reduction equipment and to make the best selection of test conditions to simulate the blast furnace process in laboratory scale.
Chapter Three

Literature survey, part II: Experimental blast furnaces

3.1 THE DEFINITION OF AN EXPERIMENTAL BLAST FURNACE
There is no universal convention of what an experimental blast furnace really is. However, generally the expression describes a blast furnace, which is built and used for research purposes. Thus, an experimental blast furnace differs from a commercial blast furnace in the sense that the main output is not hot metal, but research results. On the other hand, an experimental blast furnace is distinguished from laboratory research equipment by including the actual blast furnace process, and not a simulation thereof.

3.2 PURPOSES OF USING EXPERIMENTAL BLAST FURNACES
The are numerous purposes of using an experimental blast furnace. Generally, the purposes can be categorised into four main groups:
A. Developments of the blast furnace process as well as new techniques to be used in blast furnace ironmaking.[18-26]
B. Comparison of different types of burden materials.[18-21,23,27-28]
C. Development of burden materials.[18-21,23]
D. Fundamental studies of the blast furnace process.[18-23,27,29]

In group A) furnaces have been used for studies of injection techniques, mostly studying coal, natural gas or oil injection, and different process concepts, such as oxygen enrichment and the concept of the oxygen blast furnace. Comparison of burden materials, group B), has been used to characterise different types of burden materials, and to analyse the difference between materials. Examples are comparisons of acid and basic pellets, pellets and sinter, pellets/sinter versus lump ore, and comparing different qualities of coke etc. Group C) purposes have mostly been the development of new types of pellets and modified sinters, but also testing of cold-bonded pellets, the use of pre-reduced burden, finding the optimal size of burden materials etc. Fundamental studies, group D), include for instance studies of the influence of alkali, swelling of pellets, mechanisms for dissolution of silicon, manganese and phosphorous in the hot metal etc.

3.3 EXPERIMENTAL BLAST FURNACES HISTORICALLY
During the years, there have been several experimental blast furnaces in use for research purposes. Most of them came about during the nineteen fifties, sixties and seventies, when there was an extensive development of the blast furnace process. The research performed was somewhat different
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace
depending on the company or institute that was operating the furnace, but most research activities
were among the four research areas described above. In Table 3.1 all experimental blast furnaces
referred to in literature are presented.\textsuperscript{[18-31]} Most of the experimental blast furnaces listed in the table
do not exist today. They were shut down due to lack of financing or that the objectives of the
individual projects had been reached.

Table 3.1. Experimental blast furnaces found in literature.\textsuperscript{[18-31]}

<table>
<thead>
<tr>
<th>Years of operation</th>
<th>Working volume (m$^3$)</th>
<th>Hearth diameter (m)</th>
<th>Tuyeres</th>
<th>Blast temp. (°C)</th>
<th>Top pressure (bar, gauge)</th>
<th>Productivity (tHM/m$^3$ × 24h)</th>
<th>Fuel rate (kg/tHM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>British Steel, Great Britain</td>
<td>1988-1998</td>
<td>1</td>
<td>0.5</td>
<td>800</td>
<td>-</td>
<td>1.7</td>
<td>800</td>
</tr>
<tr>
<td>CRM, Belgium</td>
<td>1953-1972</td>
<td>8.6</td>
<td>1.4</td>
<td>1000</td>
<td>1.5</td>
<td>4.3</td>
<td>560</td>
</tr>
<tr>
<td>US Bureau of Mines, USA</td>
<td>1955-1967</td>
<td>8.6</td>
<td>1.4</td>
<td>1360</td>
<td>2.5</td>
<td>5</td>
<td>580</td>
</tr>
<tr>
<td>US Steel, USA</td>
<td>1961-1968</td>
<td>9.6</td>
<td>1.2</td>
<td>980</td>
<td>-</td>
<td>4</td>
<td>600</td>
</tr>
<tr>
<td>ICM, China</td>
<td>1958-1964</td>
<td>17.5</td>
<td>1.5</td>
<td>1250</td>
<td>-</td>
<td>4</td>
<td>600</td>
</tr>
<tr>
<td>ICM, China</td>
<td>1964-1955-1981</td>
<td>0.8</td>
<td>0.5</td>
<td>800</td>
<td>-</td>
<td>6.4</td>
<td>700</td>
</tr>
<tr>
<td>Tokyo University, Japan</td>
<td>1967-</td>
<td>3.9</td>
<td>0.6</td>
<td>850</td>
<td>-</td>
<td>4.0</td>
<td>680</td>
</tr>
<tr>
<td>NKK, Japan</td>
<td>1970-1989-</td>
<td>2.3</td>
<td>1.0</td>
<td>890</td>
<td>-</td>
<td>2.5</td>
<td>670</td>
</tr>
<tr>
<td>Sumitomo, Japan</td>
<td>1989-</td>
<td>0.9</td>
<td>0.9</td>
<td>1050</td>
<td>-</td>
<td>7.4</td>
<td>670</td>
</tr>
<tr>
<td>Nippon Steel, Japan</td>
<td>1967-</td>
<td>0.3</td>
<td>0.4</td>
<td>1300</td>
<td>-</td>
<td>0.6</td>
<td>620</td>
</tr>
<tr>
<td>Kawasaki, Japan</td>
<td>1997-</td>
<td>8.2</td>
<td>1.2</td>
<td>800</td>
<td>-</td>
<td>1.2</td>
<td>530</td>
</tr>
<tr>
<td>LKAB, Sweden</td>
<td>1989-</td>
<td>-</td>
<td>1.2</td>
<td>1300</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

1) Oval cross-section.
2) Sector of a circle, 90 degrees.

Generally, preparing of data for evaluation were manually performed at the experimental blast
furnaces during the nineteen fifties and sixties. This was a time consuming duty, which limited the
amount of process data and analyses. In 1968 US Steel installed the first process computer, which
was said to be the first blast furnace process computer ever.\textsuperscript{[20]} Below some additional information of the experimental blast furnaces listed in the Table 3.1 are given.

British Steel (presently Corus), Teesside, Great Britain
The furnace was built for studies of the coal injection technique. Injection of iron ore fines and fluxes
has also been studied. The last experimental campaign was in 1998, and the furnace is currently under
dismantling.\textsuperscript{[30]} It had a comparatively small volume and, therefore, a high fuel rate. Devices for
measuring during operation were few, and most of the information of the burden behaviour was
obtained from dissections of the furnace.\textsuperscript{[18]}

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CRM, Ougrée, Belgium
An oval shaped furnace built in 1953 was in 1965 rebuilt to an 8.6 m$^3$ experimental blast furnace. In this furnace many successful experiments were conducted (comparing different iron bearing materials and coke qualities) before it was shut down in 1972. The furnace was at that time considered the best pilot scale furnace in Europe ever.\[18,19\]

United States Bureau of Mines, Bruceton, USA
Before the LKAB furnace was built, this equipment was considered the best pilot-scale blast furnace ever. It was intensively used in 1959-1965, for testing of new sinter and pellet types, and also for studying techniques of fuel injection.\[21,27\]

US Steel, Monroeville, USA
The furnace was erected in 1961 as a copy of the successful USBM furnace. It was used internally in US Steel until 1968. There were many research activities, but no results were published externally.\[20\]

ICM, Beijing, China
A 17.5 m$^3$ furnace, built in 1958, has been experimentally used for development of the so-called “3-high” concept, i.e. high top pressure, high blast temperature and high humidity of the blast, and for testing of titanium and vanadium containing ores. This is a relatively large unit for experimental purposes. However, it is presently used as a commercial furnace.\[19,31\]

Tokyo University, Tokyo, Japan
The working volume of the furnace was originally 0.5 m$^3$, but after rebuilding in 1970 it was enlarged to 0.8 m$^3$. From 1955 to 1981 the furnace was used once or twice a year. Many research projects have been conducted and over 800 students have been involved in the operation of the furnace, as a part of their education. A special feature was the use of a fibre scope for direct optical observations inside the furnace.\[28,29\]

NKK, Kawasaki, Japan
A small furnace of 0.6 m$^3$ working volume was first installed in 1967. In 1970 the furnace was rebuilt and enlarged to 0.8 m hearth diameter and 3.2 m$^3$ working volume, and has after that been further enlarged to 1.0 m and 3.9 m$^3$ respectively.\[23,24\] This equipment has had a large influence on the blast furnace process development at NKK, and in developments of new techniques, such as the oxygen blast furnace.\[24\]

Sumitomo, Kokura, Japan
Originally, this was an experimental smelting furnace. It was rebuilt to an experimental blast furnace
and used primarily for the development of simultaneous injection of high rates of PCI with fine ore and fluxes, and for testing of the concept of oxygen blast furnace.\cite{25,26}

**Nippon Steel, Kimitsu, Japan**

The experimental blast furnace of Nippon Steel is a $90^\circ$ sector of a furnace. It has a plasma torch for heating of the blast, so the blast temperature reached can be very high, and another for heating of the stagnant coke region. Because of the use of plasma torches, the operation time is limited to between 40 and 100 hours (of which it takes 24 hours to stabilise the operation). The furnace was built for studies of injection of coal and ore in the tuyere, and is still in use.\cite{22}

**Kawasaki, Chiba, Japan**

A small, single tuyere, experimental blast furnace of $0.3 \text{ m}^3$ working volume has been used for studies of injection of ore and coal.

### 3.4 THE SIZE OF AN EXPERIMENTAL BLAST FURNACE UNIT

In *Figures 3.1 to 3.4* some of the experimental blast furnaces described in the literature are shown. Regarding size there is no general definition of an experimental blast furnace, which could be seen in *Table 3.1* and in the examples of experimental blast furnaces shown in *Figures 3.1 to 3.4*. The size of an experimental blast furnace has to be decided based on the purposes of the equipment. The unit has to be large enough to provide a realistic blast furnace process, including the formation of raceways of a suitable size, a cohesive zone, a stagnant coke zone (dead man) etc. The expression realistic in this sense means that the blast furnace process has to be established with a limited fuel rate. If the fuel rate is too high, too much reducing gas will be produced, resulting in incomparable results of the reduction behaviour of different types of burden materials, and the influence of large amounts of ash produced will also be significant. The smaller the size, the higher will be the heat losses, per unit production, and thereby the fuel rate. On the other hand, the size of the furnace must not be too large, as the costs of investments and operation of the furnace increase with the increasing size. All typical equipment and monitoring instruments that are necessary for the operation of a commercial blast furnace is also needed for an experimental blast furnace, as the process of the experimental blast furnace is indeed the blast furnace process. Since the product of an experimental blast furnace, as mentioned earlier, is the research results, in the form of data, samples and phenomena etc., even more equipment, monitoring and measuring instruments, often demanding for a high precision, need to be installed, either for collection of data, or samplings and observations. The auxiliaries of an experimental blast furnace is described in detail for the case of the LKAB experimental blast furnace in Chapter 7 of this thesis, and in *Supplement III*. 

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3.5 SUMMARY

The blast furnace process is continuously under development, and therefore the experimental blast furnace is still a valuable research tool. In the past, there has been an extensive use of experimental blast furnaces worldwide. A lot of important results and developments have been gained, although historically the data acquisition possibilities were limited. In that sense, the conditions are more advantageous today. On the other hand, due to increasing competition in the ironmaking business, leading to rationalisations and downsizing of research activities, there seems not to be a propitious financial situation for an experimental unit of the size of an experimental blast furnace. Therefore, there are not many activities in experimental blast furnaces presently, with the exception of the LKAB experimental blast furnace.
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

Figure 3.3. The NKK experimental blast furnace, before and after enlargement in 1970.[23]

Figure 3.4. The CRM experimental blast furnace, 1965-72.[18]
Chapter Four

Calculation of reduction degree

This chapter includes the definition of the reduction degree and derivations of the calculations of oxygen removal, carbon deposition and carbon gasification during reduction. These calculations were necessary to obtain the reduction degrees in the laboratory studies presented in Chapter 6 and in Supplements I and II. The definition of the reduction degree is essential in examining the process of reduction in laboratory scale, as well as in samples from the experimental blast furnace, as conducted in Supplement IV, and summarised in Chapter 8.

4.1 DEFINITION OF REDUCTION DEGREE

Reduction degree, \( R \) (\%), was calculated according to the following definition:

\[
R (\%) = \frac{m_{\text{Acc Rem}}}{m_{\text{Ori} \text{ Ori}}} \times 100
\]

(4.1)

where \( m_{\text{Acc Rem}} \) = accumulated mass of oxygen removed from sample (g); and
\( m_{\text{Ori} \text{ Ori}} \) = original content of oxygen present as iron oxides (g).

Original content of oxygen in the examined pellet type, MPBO, is 28.6 per cent of the sample weight (94.7 % Fe\(_2\)O\(_3\) and 0.7 % FeO).\(^{[32]}\) In the laboratory tests two different approaches were used to continuously register the oxygen removed during reduction: 1) gas analysis of inlet and outlet gases, followed by oxygen balance calculation (as described below); and 2) the measured weight loss of the sample (corrected for the influence of carbon deposition at low temperatures and carbon gasification at high temperatures). In the case of interrupted experiments, the measured and calculated weight losses were verified by weighing the samples after reduction. In some cases, the attained degree of reduction was also verified by comparisons of chemical analyses.

The reduction degrees of the samples from the experimental blast furnace were also determined by two different methods: 1) phase identification under microscope; and 2) chemical analyses of the samples. From metallogographical investigations the extent of different iron oxide/iron phases can be determined by image analysis. The information of the different fractions can then be used to calculate the reduction degree by equation (4.2), which gives the reduction degree of sample \( i \), \( R_i \):
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\[ R_i (\%) = v_{\text{Magnetite}} \times R_{\text{Magnetite}} + v_{\text{Wüstite}} \times R_{\text{Wüstite}} + v_{\text{Iron}} \times R_{\text{Iron}} \]  

(4.2)

where \( v_j \) is the volume fraction of iron oxide/iron phase \( j \), and \( R_x \) is the reduction degree of the respective oxide and iron phases, i.e. 11.1 % for magnetite, 29.6 % for wüstite and 100 % for metallised iron (hematite is excluded in equation (4.2), since its degree of reduction is zero, per definition). This method to determine the reduction degree is further described in Supplement IV.

### 4.2 Calculation of Oxygen Removal

For the samples from the experimental blast furnace the oxygen removal could be determined by either chemical analysis or phase identification, as described above. Monitoring oxygen removal by measuring the weight loss during laboratory reduction is a straightforward exercise. Calculating oxygen removal from inlet and outlet gas analyses are somewhat more complicated. In the theoretical treatment of oxygen removal from calculation of change in gas analysis, a gas mixture of \( \text{CO}, \text{CO}_2 \) and \( \text{N}_2 \) is, for simplicity, first dealt with. After that, the presence of \( \text{H}_2 \) in the gas phase is included. If the reducing gases are treated as ideal gases the oxygen removal can be calculated from gas analyses of inlet and outlet gases. The mass flow of oxygen, \( \dot{m}_O \), in a \( \text{CO}/\text{CO}_2/\text{N}_2 \) gas mixture is at any time given by:

\[
\dot{m}_O = M_O \times v_{\text{CO}} + 2 \times M_O \times v_{\text{CO}_2} = (v_{\text{CO}} + 2 \times v_{\text{CO}_2}) \times M_O
\]

(4.3)

where \( \dot{m}_O \) = mass flow of oxygen in the dimension of (mass/time)

\( M_O \) = molar mass of oxygen (g/mole)

\( v_i \) = molar flow of species \( i \) in the dimension of (moles/time)

The relation between molar flow, \( v_i \), and volume flow, \( \theta_i \) (dimension volume/time) is

\[
v_i = \frac{\theta_i}{V_0}
\]

(4.4)

where \( V_0 \) represents the standard volume of an ideal gas at room temperature (dimension volume/mole). Thus the mass flow of oxygen can be expressed by:

\[
\dot{m}_O = \left( \frac{\theta_{\text{CO}}}{V_0} + 2 \times \frac{\theta_{\text{CO}_2}}{V_0} \right) \times M_O = \left( \theta_{\text{CO}} + 2 \times \theta_{\text{CO}_2} \right) \times \frac{M_O}{V_0}
\]

(4.5)

For species \( i \) the volume flow, \( \theta_i \), is given by:

\[
\theta_i = \frac{I\%_i}{100} \times \theta
\]

(4.6)
where $[\%i]$ is the volume percentage of species $i$ and $\theta$ is the total volume flow of gas. This gives:

$$m_O = (\frac{[\%CO]}{100} \times \theta + 2 \times \frac{[\%CO_2]}{100} \times \theta) \times \frac{M_O}{V_0} = (\frac{[\%CO]}{100} \times \theta + 2 \times \frac{[\%CO_2]}{100} \times \theta) \times \frac{\theta \times M_O}{100 \times V_0} \tag{4.7}$$

Due to carbon deposition at low temperatures, $2CO \Rightarrow CO_2 + C$, and the reverse reaction (Boudouard or carbon gasification reaction) at higher temperatures, the total volume flow of the reducing gas, $\theta$, is not constant. At any time the total amount of gas (volume flow) is given by the relationship between nitrogen content of the gas phase and the volume flow of nitrogen (which is expected to be constant):

$$\theta = \frac{\theta_{N_2}}{[\%N_2]} \times 100 \tag{4.8}$$

This relation applied to equation (4.7) gives:

$$m_O = (\frac{[\%CO]}{100} + 2 \times \frac{[\%CO_2]}{100}) \times \frac{\theta_{N_2} \times M_O}{[\%N_2] \times V_0} \tag{4.9}$$

The oxygen removal due to reduction can then be calculated as:

$$m_{O, Rem} = m_{O, Outlet} - m_{O, Inlet}$$

$$= (\frac{[\%CO]^{Outlet}}{[\%N_2]^{Outlet}} + 2 \times \frac{[\%CO_2]^{Outlet}}{[\%N_2]^{Outlet}}) \times \frac{\theta_{N_2} \times M_O}{[\%N_2]^{Outlet} \times V_0}$$

$$- (\frac{[\%CO]^{Inlet}}{[\%N_2]^{Inlet}} + 2 \times \frac{[\%CO_2]^{Inlet}}{[\%N_2]^{Inlet}}) \times \frac{\theta_{N_2} \times M_O}{[\%N_2]^{Inlet} \times V_0}$$

$$= \left(\frac{[\%CO]^{Outlet} + 2 \times [\%CO_2]^{Outlet}}{[\%N_2]^{Outlet}} - \frac{[\%CO]^{Inlet} + 2 \times [\%CO_2]^{Inlet}}{[\%N_2]^{Inlet}}\right) \times \frac{\theta_{N_2} \times M_O}{V_0} \tag{4.10}$$

For a reducing gas mixture containing hydrogen the oxygen removal in the form of $H_2O$ must also be taken into account in equation (4.10):
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

\[ \dot{m}_{O,Rem} = \left( \frac{[\% CO]_{Outlet} + 2 \times [\% CO_2]_{Outlet} + [\% H_2O]_{Outlet}}{[\% N_2]_{Outlet}} \right) \times \frac{\theta_{N_2} \times M_O}{V_0} \] (4.11)

During reduction experiments the inlet and outlet gases were analysed for contents of CO, CO\(_2\) and H\(_2\). After heating the sample, before the reduction program starts at 400 °C, the moisture content of the sample is expected to be zero. Therefore, a hydrogen balance from inlet and outlet gases can be performed in order to substitute the content of H\(_2\)O by H\(_2\) in equation (4.11):

\[ \text{Molar flow of inlet} (H_2 + H_2O) = \text{Molar flow of outlet} (H_2 + H_2O) \] (4.12)

The inlet flow of H\(_2\)O, \(\theta_{H_2O}^{Inlet}\), is assumed to be zero. Using equations (4.6) and (4.8) together with (4.12) a suitable expression for the H\(_2\)O content of the outlet gas is obtained:

\[ \frac{[\% H_2O]_{Outlet}}{[\% N_2]_{Outlet}} = \frac{[\% H_2]_{Inlet}}{[\% N_2]_{Inlet}} \times \frac{[\% H_2]_{Outlet}}{[\% N_2]_{Outlet}} \] (4.13)

This expression is inserted into equation (4.11) to give:

\[ \dot{m}_{O,Rem} = \left( \frac{[\% CO]_{Outlet} + 2 \times [\% CO_2]_{Outlet} - [\% H_2]_{Outlet}}{[\% N_2]_{Outlet}} \right) \times \frac{\theta_{N_2} \times M_O}{V_0} \] (4.14)

The mass of oxygen removal can now be calculated by integrating the removal rate over time:

\[ m_{O,Rem} = \int_0^t (\dot{m}_{O,Rem}) \times dt \] (4.15)

During a time increment of constant oxygen removal rate, the solution to the integral in equation (4.15) is simply the product of the oxygen removal rate, \(\dot{m}_{O,Rem}\), and the time, \(t\), of the increment.
under consideration:

\[
m_{O,\text{Rem}} = \dot{m}_{O,\text{Rem}} \times t
\]  

(4.16)

Accumulated oxygen removal, finally, is calculated by summing up the mass of oxygen removal for each time increment \((t_i)\):

\[
m_{O,\text{Rem}}^{\text{Acc}}(t) = \sum_{i=1}^{T} (\dot{m}_{O,\text{Rem}} \times t_i)
\]  

(4.17)

where \(T\) is the total number of time increments.

### 4.3 MASS BALANCE OF CARBON

Similarly as for the oxygen removal calculation the extent of the solution loss reaction, i.e. carbon deposition at low and carbon consumption at high temperatures respectively, can be calculated by a mass balance of carbon. Thus, the rate of carbon deposition can be expressed as:

\[
\dot{m}_{C,\text{Dep}} = \dot{m}_{C,\text{Inlet}} - \dot{m}_{C,\text{Outlet}}
\]

\[
= \left( \frac{[\% CO]^{\text{Inlet}} + [\% CO_2]^{\text{Inlet}}}{[\% N_2]^{\text{Inlet}}} \times \frac{[\% CO]^{\text{Outlet}} + [\% CO_2]^{\text{Outlet}}}{[\% N_2]^{\text{Outlet}}} \right) \times \frac{\theta_{N_2} \times M_C}{V_0}
\]  

(4.18)

Carbon deposition is then at all times given by integrating the removal rate over time:

\[
m_{C,\text{Dep}} = \int_{0}^{t} (\dot{m}_{C,\text{Dep}}) \times dt
\]  

(4.19)

For time increments of constant carbon deposition rate, the accumulated carbon deposition is given by summing up of all time increments, similar to equation (4.17) for oxygen removal:

\[
m_{C,\text{Dep}}^{\text{Acc}}(t) = \sum_{i=1}^{T} (\dot{m}_{C,\text{Dep}} \times t_i)
\]  

(4.20)

where a positive value represents carbon deposition and, consequently, negative values represent carbon consumption.
4.4 COMPARISON OF DIFFERENT MEASURING METHODS

As was mentioned earlier, both the weight loss and the changes in the composition of the reducing gas were measured to monitor the oxygen removal during reduction in the laboratory tests. In Figure 4.1, two examples of the results of the different methods of monitoring oxygen removal are shown. The agreement between the two methods was found to be good. The accuracy of the measurements was also verified by weighing of the samples from the interrupted experiments, to check the attained reduction degree after interrupted reduction. The agreement between measured weight loss during reduction and control measuring of samples after reduction was generally within 2 g, i.e. the deviation was less than 2 percentage units in reduction degree, which was generally also the range of deviation when comparing with chemical analyses.

![Figure 4.1](image.png)

Figure 4.1. Two examples of monitoring weight loss during reduction, measured and calculated from the change in gas composition (from Supplement I, wall profile experiments).

Also for samples from the experimental blast furnace two different methods to determine the reduction degree were used, i.e. phase identification under microscope and chemical analyses of the samples. The accuracy of the phase identification method to determine the reduction degree was verified by comparing results from the standard procedure of identifying reduction degree, i.e. from chemical analysis, for samples collected at the same position during the blast furnace dissection. The comparison can be seen in Figure 4.2, which shows the degree of reduction in different layers of the furnace and at different radial positions. The deviation between the different methods of calculating the degree of reduction was generally less than 8 percentage units.
4 CALCULATION OF REDUCTION DEGREE

Figure 4.2. Comparison of reduction degree calculated from chemical analysis and calculated from phase identification respectively, for three different sample positions (from Supplement IV).

4.5 SUMMARY

The most important feature of the ironmaking process, i.e. the removal of oxygen from the iron oxides, was theoretically treated in this chapter. An understanding of the theoretical part of reduction is essential to be able to numerically describe the degree of reduction and carbon deposition etc. in the laboratory reduction tests as well as in the blast furnace process.
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Chapter Five

Experiments, Part I: Laboratory tests

In this chapter, a description of the laboratory reduction-under-load tests used in Supplements I and II is given. Results of the experiments are given in Chapter 6 (as well as in Supplements I and II).

5.1 MATERIALS

The iron bearing material examined in this study was the olivine iron ore pellet MPBO produced by LKAB in Malmberget. MPBO is, together with KPBO (same type of pellet, produced by LKAB in Kiruna and Svappavaara), the dominating blast furnace feed at the Swedish iron plants and is also exported. The chemical composition of MPBO has been slightly adjusted during the years. Table 5.1\(^{[32]}\) shows the chemical composition of MPBO at the time when the experiments were carried out.

Table 5.1. Chemical analysis of the MPBO pellet (weight per cent)\(^{[32]}\)

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>SiO(_2)</th>
<th>CaO</th>
<th>MgO</th>
<th>Al(_2)O(_3)</th>
<th>TiO(_2)</th>
<th>MnO</th>
<th>Na(_2)O</th>
<th>K(_2)O</th>
<th>V(_2)O(_5)</th>
<th>P(_2)O(_5)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>66.8</td>
<td>2.00</td>
<td>0.37</td>
<td>1.40</td>
<td>0.42</td>
<td>0.30</td>
<td>0.06</td>
<td>0.04</td>
<td>0.02</td>
<td>0.21</td>
<td>0.02</td>
</tr>
</tbody>
</table>

The coke, placed in layers above and under the pellet bed, was produced by SSAB Oxelösund AB. Originally the coke was of "fist" size, but the coke used in experiments was crushed to sizes of 10 to 15 mm to make up a suitable bed layer in the sample crucible.

5.2 EQUIPMENT

Reduction-under-load experiments were conducted in a newly developed reduction equipment. The features of the equipment were the following. To be able to closely simulate the reducing conditions as experienced by iron bearing materials in the blast furnace, it was possible to continuously govern the furnace temperature, gas composition and flow rates, and the applied load over the sample during reduction. A variety of parameters were registered during the test, including bed shrinkage and weight loss. Each parameter was registered ten times per second, and averaged minute values were produced. The size of the sample crucible (graphite, with perforated bottom and lid) was 80 mm in diameter and 125 mm in height. The sample bed size was 500 g, corresponding to a bed height of roughly 55 mm. Above and below the pellet bed 30 mm layers of coke were placed. In addition to external preheating of the reducing gas, a gas preheating chamber was placed below the sample crucible, where also a melt collector was placed. A ceramic tube surrounded the crucible and the preheating chamber, which all were placed on a balance, for continuous measurement of the weight loss during reduction. A 15 kW tube furnace, consisting of six Kanthal Super elements and an isolation of ceramic fibre
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materials, was used for heating. The furnace was placed around the ceramic tube and a loading ram above the sample crucible applied the intended load onto the sample bed. The physical outlook of the equipment is schematically shown in Figure 5.1.

![Figure 5.1. A schematic view of the reduction-under-load equipment.](image)

5.3 EXPERIMENTAL CONDITIONS

Simulation of the blast furnace process was made for a typical blast furnace of intermediate size, with strong central gas flow and slightly higher wall flow than the major part of the burden, as in most blast furnaces. Consequently, this simulated a blast furnace process with a W-shaped cohesive zone.

To be able to simulate actual reducing conditions in the blast furnace, it is of course necessary to know what conditions to simulate. The parameters that were considered the most important, with respect to the behaviour of iron bearing materials during reduction, were the following:

- rate of temperature increase
- rate of reducing gas flow
- change in reducing gas composition
- change in mechanical stress
These parameters were interpreted for three different radial positions of the blast furnace; the extremes of centre and wall, and the intermediate mid-radius position (radius ratios of 0.1, 0.9 and 0.5 respectively). Values of these parameters are, when they are found in the literature, often given as a function of height in the blast furnace. Measured descent velocities in combination with estimated average residence time for iron bearing materials were therefore used to interpret the time dependence of the various parameters. For a commercial blast furnace with a throat diameter of 8.8 m the descent velocity has been measured to 6-7 m/h,

\[33\]

while the average residence time for iron bearing materials from stockline to the dripping zone in a blast furnace of a somewhat smaller size has been found to be between six to eight hours.\[34,35\] In the following sections an explanation of the choice of values for each reduction parameter will be given.

5.3.1 Rate of temperature increase

As the solids descending through the blast furnace are heated by the ascending gas, according to the second law of thermodynamics there has to be a difference between the temperature of solids and the temperature of the gas phase, since heat can only flow naturally from a higher to a lower temperature potential. However, in literature it is reported that the difference in temperature between solids and gas in a large part of the furnace, often referred to as the thermal reserve zone, is negligible.\[16,31,36\] This zone is situated in the middle of the furnace shaft, and often it occupies up to half of the furnace height. The temperature in the thermal reserve zone is approximately 900 to 1000 °C. The cause of the temperature constancy of the zone is that the heat of exothermic and endothermic reactions are balanced. Therefore, the temperature difference between solids and gas in this zone is small. Above and below the thermal reserve zone, where the solid materials are being heated by the gas phase, the temperature difference between solids and gas of course is higher, due to the nature of heat transfer. The temperature difference here is often 100 to 200 °C, whereas in the vicinity of the raceways (combustion zone) it is considerably higher, since the flame temperature of the raceway often exceeds 2000 °C. A schematic picture of the solids and gas temperature profiles is shown in Figure 5.2.

As previously mentioned, the heating pattern at the time of descent of the burden in the blast furnace greatly differs according to the radial position in the furnace, but is also influenced by the furnace operating conditions. The temperature has a strong effect on the morphology of the iron oxide reduction reaction. Turkdogan reported that the mode of iron oxide reduction changes from uniform internal reduction to topochemical with increasing temperature.\[37\] The extent of internal reduction and topochemical reduction is, in turn, expected to significantly affect the softening and melting characteristics. The selection of a temperature program for laboratory tests is, therefore, crucial in order to correctly simulate the reduction, softening and melting phenomena in the blast furnace.
Many blast furnaces were quenched and dissected throughout the world in the nineteen seventies. The majority of the dissections were carried out in Japan, and much effort was put into the evaluation of the dig-outs in order to get a clearer view of the internal state of the blast furnace during operation. As a result of this work conclusions were drawn concerning the temperature profiles within the blast furnace. The temperature profiles used for the experiments in the present work are shown in Figure 5.3. The profiles were chosen from an interpretation of Figure 5.4, which shows results from a numerical analysis of the blast furnace operation using a two-dimensional mathematical-kinetic model.

Reduction below 400 °C is assumed to be suppressed by the slow kinetics at low temperatures. The reduction programs were therefore started at 400 °C. Since the heating rates chosen were different for the three experimental conditions the length of each reduction experiment varied with the intended radial position. Figure 5.5 shows the three temperature profiles as a function of time from start of reduction, and in Table 5.2 the rates of temperature increase and time for completion of experiments are shown.
Figure 5.3. Temperature profiles chosen for the blast furnace simulation.

Figure 5.4. Longitudinal distributions of temperature of solids in a blast furnace ($\zeta$ denotes the radius ratio $r/R$). $^{[33]}$

Figure 5.5. Temperature profiles used in reduction-under-load experiments.
Table 5.2. Rate of temperature increase and time for completion of the reduction-under-load tests for the different temperature profiles used.

<table>
<thead>
<tr>
<th>Temperature interval (°C)</th>
<th>Rate of temperature increase (°C/min)</th>
<th>Wall profile</th>
<th>Mid-radius profile</th>
<th>Centre profile</th>
</tr>
</thead>
<tbody>
<tr>
<td>400 - 800</td>
<td></td>
<td>4</td>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>800 - 1000</td>
<td></td>
<td>5</td>
<td>1.2</td>
<td>5</td>
</tr>
<tr>
<td>1000 - 1600</td>
<td></td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Time for complete test (min)</td>
<td></td>
<td>260</td>
<td>320</td>
<td>193</td>
</tr>
</tbody>
</table>

5.3.2 Rate of reducing gas flow
The flow distribution of reducing gases through the blast furnace is affected by the radial particle size distribution, void fraction distribution and by the shape of the cohesive zone as well as the deadman coke column. These parameters, in turn, are partly or totally governed by the radial distribution of ore and coke layers, and the thickness of them. Under the conditions prevalent in the blast furnace stack, though, the reduction kinetics are expected to be independent of the prevailing gas velocity. For laboratory purposes Edström found that a relatively low critical gas velocity exists, above which a further rise has very little influence on the rate of reduction, given as a linear gas velocity of 0.12 m/s. Yamaoka et al and Yoshinaga et al have independently obtained similar values of the critical gas velocity in laboratory scale by using a gas velocity in excess of 0.1 to 0.13 m/s. Gas velocity is defined as the gas flow rate per unit cross sectional area of the sample bed, and hence, the larger the bed diameter, the greater the gas flow needed to achieve the specified gas velocity. Since high gas flow rates can cause longitudinal temperature gradients and increased operating costs, it is desirable to use the minimum gas flow rate necessary to achieve the specified gas velocity. The total gas flow rate used in this work, therefore, was minimised to the critical gas velocity of 0.1 m/s.

5.3.3 Reducing gas composition cycle
The gas composition can vary widely depending of the radial position in the blast furnace. There are several reasons for this variation. Most important is, of course, the difference in temperature, which makes the equilibrium gas composition change drastically in the temperature range of 800 to 900 °C. Above this temperature CO is the stable gas phase in the system Fe-C-O and all carbon dioxide formed by reduction of iron oxides will be reduced back to CO by carbon according to the Boudouard reaction (CO$_2$ + C $\rightarrow$ 2 CO). Below 800 °C the stability of carbon dioxide increases as temperature decreases and the extent of the Boudouard reaction will therefore be limited.

Another reason for the radial difference in gas composition is the non-uniform distribution of the charge materials, e.g. in the centre of a centre working furnace there is often excessive amounts of coke, with respect to iron bearing materials, resulting in a gas phase high in carbon monoxide. For each of the three temperature profiles used two reduction potentials were chosen to study the effect of reducing gas composition, as shown by the CO content versus temperature in Figure 5.6.
The hydrogen content of the reducing gas was coupled to the carbon monoxide content by using a hydrogen content of one tenth of the carbon monoxide content. The two reducing gases were balanced by carbon dioxide to 40 volume per cent, the rest being nitrogen. These proportions of the reducing gas were arrived at after studying operating data from several blast furnaces.\cite{34,39}

5.3.4 Mechanical stress cycle

The applied load during reduction-under-load experiments should of course also be decided from estimations of the stress experienced by iron bearing materials in the blast furnace at different temperatures, and from the cross sectional area of the sample bed. The load distribution in the blast furnace, however, is difficult to measure. Estimations of the load distribution can be derived from the Janssen equation.\cite{5,40} Figure 5.7.a shows an example of the gas pressure in a blast furnace determined by the measured static pressure distribution, and Figure 5.7.b shows the vertical stress in the blast furnace, as calculated by Kanayama et al using the Janssen theory.\cite{40}

From Figure 5.7.b the vertical stress distribution was chosen giving the loading pattern for the laboratory experiments. In the blast furnace, the radial load distribution in the lumpy zone is not expected to vary considerably. Loading patterns with only minor differences between the three reducing environments corresponding to the centre, wall, and the mid-radius position, were therefore chosen, Figure 5.8. Nevertheless, since the heating patterns were different for the three cases, the loading patterns resulted in different mechanical stress at a given burden temperature, just as expected in the blast furnace. Figure 5.9 shows the applied load as a function of temperature.
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

Figures 5.7.a and b. Distribution of gas pressure and vertical stress in the blast furnace.

Figure 5.8. Vertical stress profiles used for blast furnace simulation.

Figure 5.9. Profiles of the mechanical load applied, as a function of temperature, for the different temperature profiles.

5.4 SUMMARY

This chapter covered the description of the laboratory blast furnace simulating equipment developed at KTH, based on data from a literature study. A summary of the experimental parameters used in the reduction tests performed was also given.
Chapter Six

Laboratory tests: Reduction behaviour of MPBO

This chapter comprises the findings of the laboratory reduction experiments conducted at KTH, and is a summary of Supplements I and II.

6.1 REDUCTION EXPERIMENTS
Reduction experiments were conducted with three different temperature profiles, and two different gas compositions for each temperature profile, i.e. six complete reduction experiments. The continuous progress in degree of reduction was calculated from gas analyses of inlet and outlet gases, and also measured by the weight loss of the sample due to oxygen removal during reduction. Figure 6.1 displays the degree of reduction during the experiments. For each of the temperature profiles the increase in the reduction degree was faster for the sample treated with the stronger reducing gas (i.e. a higher CO/CO\(_2\) ratio) than for the weaker. The reduction was fastest for the centre samples and slowest for the mid-radius samples, a combined effect of the different heating rates and gas composition profiles.

![Figure 6.1. Reduction degree as a function time for the different temperature profiles.](image-url)
Figure 6.2 shows the contraction of the sample beds as a function of time. Due to the higher heating rate, the contraction started first for the centre samples and, consequently, last for the mid-radius samples. The contraction was for all temperature profiles first started for the samples treated with the stronger reducing gas. The final contraction, however, was more rapid for the samples reduced under the weaker gas.

![Figure 6.2: Sample bed contraction, as a function of time for the different temperature profiles.](image)

6.2 REDUCTION WITHOUT LOAD
Reduction experiments without load were conducted in order to study the effect of mechanical load. Figure 6.3 shows an example of the volume change obtained from measurements of the samples reduced without load, together with the corresponding contraction curve for the reduction-under-load test under the same reducing conditions.

The behaviour of samples reduced without load was found to initially resemble that for samples reduced with load applied. Thus both the swelling and the initial shrinking of the particles of the sample bed were found to be independent of the applied load. The driving force for the shrinking behaviour of the particles is the energy minimisation that is obtained by minimising the total surface energy of the material during elimination of the pore structure. At higher temperatures the contraction rate declined for the samples treated without load, whereas the bed contraction for the samples reduced under load continued.

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6.3 REDUCTION EXPERIMENTS INTERRUPTED AT DIFFERENT TEMPERATURES

Reduction experiments were interrupted at various temperatures and contraction degrees. Figure 6.4 shows at what stages, i.e. temperatures and bed contraction degrees, the experiments were interrupted, for the case of the wall profile. The pellets from interruptions up to 1000 °C showed little or no tendency of sticking. With increasing temperature the sticking, or sintering, among the pellets of the sample bed increased. Pellets from experiments interrupted at 1360 °C or higher were impossible to separate without tools. In Figure 6.5 a photo of pellet samples from the first four interruption is shown, together with an unreduced pellet. The size of the individual particles increased in the beginning (swelling) but started to decrease above 1000-1200 °C. Fine cracks in the particles were found to sinter together during reduction, while larger cracks grew. The shape of each individual particle was not deformed, but the original spherical shape was principally retained.

At high temperatures, the pellets around the periphery of the sample bed started to melt partially, as a result of carburisation (pick-up of carbon by the iron) taking place in contact with the crucible wall. At even higher temperatures carburisation and partial melting also occurred at the coke interfaces. The carbon contents of the iron shells were analysed. Table 6.1 shows the carbon contents of shells and of metal, which had already melted before interruption at high temperature.
Figure 6.4. Sample bed contraction for interrupted experiments I11-I17, together with contraction results of complete experiments.

Figure 6.5. Pellet samples from interrupted experiments (unreduced pellet and the first four samples of interruptions under strong gas, shown in Figure 6.4). The size (diameter) ranges from about 13 (unreduced) to 11 mm.

Table 6.1. Specification of carbon contents in samples from some of the experiments (weight per cent).

<table>
<thead>
<tr>
<th>Exp. no.</th>
<th>Temperature at interruption (°C)</th>
<th>Range of C content of metallic shell (%)</th>
<th>Carbon content of melted iron (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I11</td>
<td>1000</td>
<td>0.01 – 0.02</td>
<td>-</td>
</tr>
<tr>
<td>I12</td>
<td>1145</td>
<td>0.02 – 0.03</td>
<td>-</td>
</tr>
<tr>
<td>I13</td>
<td>1255</td>
<td>0.02 – 0.05</td>
<td>-</td>
</tr>
<tr>
<td>I14</td>
<td>1360</td>
<td>0.02 – 0.05</td>
<td>4.6</td>
</tr>
<tr>
<td>I15</td>
<td>1430</td>
<td>0.02 – 0.06</td>
<td>4.4</td>
</tr>
<tr>
<td>I22</td>
<td>1420</td>
<td>0.02 – 0.04</td>
<td>4.3</td>
</tr>
<tr>
<td>I32</td>
<td>1090</td>
<td>0.02 – 0.03</td>
<td>-</td>
</tr>
</tbody>
</table>

As can be seen from Table 6.1 small amounts of melt were obtained even at 1360 °C. This was found in contact with the graphite crucible wall, but not at the coke interfaces. This was probably a result of a somewhat higher temperature at the crucible wall compared to the top and bottom of the crucible, where the coke was placed. The limited carbon contents found in the metallic shells led to the conclusion that diffusion of carbon in the solid metal was slow, i.e. slower than the melting and separation of a portion of already carburised iron, whereas the high carbon contents in the melted part of the samples indicate that carburisation was fast in the liquid state. Also the slag phases entrapped in the pellet cores were analysed after interruption of the reduction. The slag compositions, in combination with studies of phase diagrams, indicated that the slag should have been in the liquid state at temperatures above 1400 °C.
6.4 MODE OF REDUCTION

A series of photos from the metallographical studies is included in Supplement II. It was confirmed that the reduction process proceeded topochemically, from the pellet surface to the centre, not only for the pellet but for the higher oxides also in each grain. In Figures 6.6 and 6.7 the topochemical reduction is shown for one hematite grain and for a pellet particle respectively.

![Figure 6.6. Pellet reduced to 9 per cent degree of reduction, interrupted at 590 °C, showing a hematite grain with the border reduced to magnetite. Magnification 50 times.](image1)

![Figure 6.7. Pellet reduced to 59 per cent degree of reduction, interrupted at 1145 °C, showing a border of iron around the core of wüstite. Magnification 50 times.](image2)

Reduction of hematite via magnetite and wüstite to iron was found to proceed in a stepwise manner in spite of a gas phase strong enough to reduce the oxides even further. The reason for the stepwise behaviour of the reduction was believed to be a locally increased partial pressure of carbon dioxide in the outer layers of the particle, caused by reduction reactions in the core. Thus, further reduction of the outer layers was prevented.

In the initial stage, some reduction of wüstite was observed to proceed in an internal mode inside the wüstite grains, instead of starting at the surface and producing a border of iron around the wüstite grains. This phenomenon was believed to be a result of nucleation taking place at sites in the grains where, for example, a crystal defect makes nucleation more favourable than at the outer surface of the grain.

From the olivine grains magnesia was found to diffuse to the surrounding wüstite phases, while the magnesia in the olivine was replaced by FeO. This results in a homogeneous solid solution of the wüstite phase, with an increased melting point (or melting range). However, the wüstite was molten in samples interrupted at 1430 °C and above.
6.5 CHARACTERISATION OF THE CONTRACTION BEHAVIOUR

All contraction curves started with an initial expansion, followed by shrinking at an almost constant rate, starting at 1000 °C to 1200 °C, and finally rapid contraction from 1420 °C to 1450 °C. The contraction behaviour of the MPBO pellet was therefore divided into three separate stages, as shown by Figure 6.8, with the first stage including the swelling, i.e. a negative contraction. The contraction was due to the thermally increased mobility of the iron produced, resulting in sintering of the particles, in the later parts of the second stage in combination with the continuously increasing mechanical load applied on the sample bed, causing a continued shrinking of the particles. The mechanical load also assisted in sintering between the particles. Metallographical investigations verified that the start of contraction origins from the shrinking of the individual particles caused by sintering of the metallic iron shell, and that sintering also took place between the individual particles.

The last stage of contraction was caused by a carburisation/melting process proceeding over a temperature range. The start of rapid contraction took place at roughly the same temperature, 1420-1450 °C, and contraction degree, 55-60 per cent, for all reducing conditions. Neither the difference in load nor the degree of reduction affected the start of rapid contraction. Therefore the rapid contraction was found not to be caused by collapsing of pellets but caused by melting. The analyses of metallic shells and examination of samples from interrupted experiments indicated that the rapid contraction at high temperatures was initiated by carburisation and partial melting of the metal. However, the melting of the slag phase occurred at temperatures not much lower than the start of rapid contraction, why melting of the slag phase might also have contributed to the higher rate of contraction during the rapid contraction stage. In that case, a combined effect of carburising of reduced iron and melting of the slag phase could be initiating the rapid contraction. In Figures 6.9.a and b, the microstructure of one pellets reduced to 81 per cent degree of reduction, interrupted at 1430 °C, is shown. The metallic iron shell is densely sintered, and partially melted. The oxide slag phases inside the pellet have formed a rim inside the shell, resulting in large cavities in the centre of the pellet.
Figures 6.9.a and b. Microstructure of the sample interrupted at 1430 °C, degree of reduction 81 per cent. Left photo, 9.a, shows the surface of the pellet and in the right photo, 9.b, the inside of the metallic shell is shown.

The temperature for the start of rapid contraction was, as previously mentioned, found to be the same for both strong and weak reducing gases. When the rapid contraction had started, though, the rate of contraction was higher, at all reducing profiles, for the samples treated with the weaker reducing gas.

### 6.6 PROCESS OF MELT-DOWN

At temperatures above 1400 °C carburisation of the metallic iron is fast, and due to the increasing temperature the metal in contact with carbon will eventually melt. The process of carburisation and melt-down during rapid contraction is schematically shown in Figure 6.10.

When carburisation of the metal shells starts (a), the particles in contact with the graphite crucible, or coke, will eventually melt (b). At start of melting only very small parts of the particles are in contact with solid carbon and the melting rate is low. The melting rate will then accelerate due to increasing contact surfaces between the iron and the carbon (c), as the melted parts drips away from the interface. Possibly also the liquid carburised metal penetrating down through the sample bed helps in accelerating the process by carburising the metal not in direct contact with carbon. Finally, the outer metallic shell of the particle becomes very thin and breaks (d). The core of liquid slag retained inside the shell is released and penetrates down through the sample bed (e). In the coke layer below the sample the iron oxide of the slag phase will be quickly reduced due to the high temperature and the favourable kinetics of the liquid/solid phase reaction. This process of carburisation, melting and collapsing will repeat for the next layer of particles now exposed to the carbon surfaces of the coke layers (f) or the crucible wall.

During the rapid contraction of the carburisation/melt-down stage it was observed that no actual softening occurred, but the particles continued to sinter until melt-down started. During sintering of particles the voidance of the bed was decreased, but there were still channels for the gas to pass through the sample bed, and thus some permeability remained. Therefore, it was concluded that the
permeability of an iron bearing layer of olivine pellets in the cohesive zone of the blast furnace might not be so seriously impaired as is implied by the softening process theory.

![Diagram](image)

Figure 6.10. Schematic picture of the continuous process of carburisation and melt-down.

### 6.7 SUMMARY

Laboratory reduction experiments under load, as well as without load and interrupted experiments, were used to describe the behaviour of olivine pellets under simulated blast furnace conditions. The process of reduction was described by obtained reduction curves and by metallographical studies. Under the reducing conditions used, the reduction of the MPBO pellets was found to proceed topochemically, and to take place in stages, i.e. no more than two iron oxide/iron phases existed at the same time.

The contraction of a sample bed of MPBO pellets proceeded in three different stages, referred to as swelling, shrinking and rapid contraction respectively. The swelling period represents an expansion of the pellets. Increasing sintering of metallic iron shells, accompanied by pore diffusion, resulted in shrinking of the individual particles, and the start of contraction of the sample bed. At higher temperatures carburisation and partial melting of iron in contact with carbon, possibly in combination with melting of the slag phase, initiated rapid contraction. During rapid contraction the pellets picked up carbon and melted. The process of melting was found to be comparatively fast, and the phenomenon normally referred to as softening was not found to occur.
Chapter Seven

Experiments, Part II: Experimental blast furnace trials

The first part of this chapter, based on Supplement III, describes the LKAB experimental blast furnace and how it is operated. The second part gives a summary of the test conditions of the experimental blast furnace trials included in this thesis, in Supplements IV and V. The results are treated in Chapters 8 and 9.

7.1 THE LKAB EXPERIMENTAL BLAST FURNACE

The development of the blast furnace process has been accelerating in the last few decades. One widespread opinion in the second half of the twentieth century, that the blast furnace process will be left behind, when the new and better alternative ironmaking processes have been developed, has come to nought. With development of computer technology, more advanced control systems have led to a new dimension of blast furnace ironmaking. Along with new technologies of injections of fuels in the blast furnace tuyeres, combined with high blast temperature and oxygen enrichment, for replacing large amounts of coke, high top pressure furnaces and higher quality of raw materials etc., the efficiency of the blast furnace process has increased tremendously, and alternative ironmaking processes are still two steps behind. For large volumes of ironmaking there is actually no other process that can compare with the blast furnace.

As the blast furnace process has developed, the demands for consistent hot metal quality, high productivity, reliability and undisrupted operation have increased. Thereby the need for a step in between laboratory scale metallurgical testing and full-scale tests of new blast furnace burden materials and operating concepts has become more and more evident. An experimental blast furnace is a way to reduce the risks before full-scale tests are performed in a commercial blast furnace. The possibilities of operating an experimental blast furnace in a broad range of process concepts can also allow to get a faster progress in the development of burden materials and to test various concepts of new techniques, which are too risky to be tested directly in a full-scale blast furnace.

In 1994, a feasibility study was initiated by LKAB to evaluate the possibilities to design and build an experimental blast furnace. The study was done by Mefos (the metallurgical research foundation in Luleå, Sweden) and LKAB in co-operation, and discussions were made with experts from all over the world. In October of 1996 the LKAB board of directors decided to build the furnace and in 1997 the experimental blast furnace was blown-in for the first time. Since then the experimental blast furnace has been regularly operated in campaigns of four to ten weeks, twice a year. So far there have been
nine campaigns conducted in the furnace and in total over 430 days of operation. Over 40 different types of pellets (commercial as well as trial pellets), several types of sinter and lump ores have been tested – as constituting 100 % of the iron bearing burden or in burden mixtures (i.e. different pellet mixtures, pellet/sinter mixtures, pellet/sinter/lump ore mixtures etc). Some examples of different process concepts tested are:

- 100 % pellets as ferrous burden with injection of PCI in the range of 0 to 165 kg/tHM.
- Simultaneous injection of PCI or oil together with a slag former amount of up to 36 kg/tHM. The slag volumes have been varied between 85 to 190 kg/tHM.
- Sinter/pellets as ferrous burden with pellet ratios from 20 % to 60 %. Oil injection rates have been up to 85 kg/tHM and the slag volumes have been between 170 to 220 kg/tHM.
- High oxygen and oil injection process concept using a sinter/pellet burden, reaching 42.3 % of oxygen in the blast and an oil injection rate above 200 kg/tHM.

7.1.1 Plant layout

The layout of the experimental blast furnace is shown in Figure 7.1. All typical equipment required for a blast furnace have been installed, and the monitoring and measuring means are even much more extensive. Figure 7.2 shows a cross-section of the furnace body, and in Figure 7.3 a photo of the plant is shown.

![Figure 7.1. Layout of the experimental blast furnace.](image)

The general specifications and operating parameters of the experimental blast furnace are summarised in Table 7.1. The furnace has a working volume of 8.2 m³ and a hearth diameter of 1.2 m, the outside view shown in Figure 7.4. From the tuyere level to the stock line the height is 5.9 m, and there are three tuyeres placed with 120 degrees separation. The tuyeres have a diameter of 54 mm, resulting in a blast velocity of 150 m/s at normal blast volume. The furnace is equipped with systems for injecting pulverised coal, oil and other injection materials.
Table 7.1. Experimental blast furnace specifications.

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Working volume</td>
<td>8.2 m³</td>
</tr>
<tr>
<td>Hearth diameter</td>
<td>1.2 m</td>
</tr>
<tr>
<td>Working height</td>
<td>5.9 m</td>
</tr>
<tr>
<td>Tuyeres</td>
<td>54 mm diameter (×3)</td>
</tr>
<tr>
<td>Top pressure, gauge</td>
<td>up to 1.5 bar</td>
</tr>
<tr>
<td>Charging</td>
<td>Modified bell type</td>
</tr>
<tr>
<td>Burden distribution</td>
<td>Movable armour</td>
</tr>
<tr>
<td>Injection</td>
<td>Coal, oil, slag formers</td>
</tr>
<tr>
<td>Blast</td>
<td>up to 2000 Nm³/h</td>
</tr>
<tr>
<td>Max. blast temp.</td>
<td>1300 °C</td>
</tr>
<tr>
<td>Furnace crew*</td>
<td>5/shift</td>
</tr>
<tr>
<td>Tapping volume</td>
<td>1.3-1.8 tHM/tap</td>
</tr>
<tr>
<td>Tap time</td>
<td>5-10 min.</td>
</tr>
<tr>
<td>Tapping interval</td>
<td>60 min.</td>
</tr>
<tr>
<td>Fuel rate</td>
<td>510-540 kg/tHM</td>
</tr>
<tr>
<td>Quenching N₂ gas flow</td>
<td>300-400 Nm³/h</td>
</tr>
</tbody>
</table>

*excluding sampling/research staff

The blast is normally preheated to 1170-1250 °C in pebble bed heaters, shown in Figure 7.5. There are two pebble bed heaters, working in cycles. The raw materials system consists of four bins for pellets, sinter or lump ore, one bin for coke and two small bins for slag formers. Each material is weighed separately according to the actual recipe. The material is transported to a receiving hopper at the furnace top by a skip. Below the receiving hopper, there is a pressure equalising lock hopper. The furnace top pressure can be controlled up to 1.5 bar (150 kPa) overpressure.

The experimental blast furnace is equipped with a bell top. There is a moveable armour for burden distribution control. Two mechanical stock rods are used to monitor the burden descent and to control the charging into the furnace. The top gas is transported through the uptakes and down-comer to a dry dust catcher. The gas is further cleaned in a venturi scrubber and a wet electrostatic precipitator, before it is flared in a torch. Dry flue dust is collected in a storage container, sampled twice a day and analysed for the chemical composition. The wet flue dust (sludge) is collected in a thickener and sampled when the thickener is emptied by a suction truck.

The furnace has one tap hole, which is opened with a pneumatic drill. After each tap, the tap hole is closed with a hydraulic mud gun. The hot metal and the slag are tapped into a sand bed, Figure 7.6. Hot metal and slag are sampled, as shown in Figure 7.7, and analysed after every tap. After solidification, the pig iron and slag are transported to the SSAB steel plant, where the pig iron is charged as scrap to the BOF converter. To facilitate dig-outs and repairing, the hearth is detachable and can be separated from the furnace in one to two hours (see Figure 7.4).
7.1.2 Comparison with commercial blast furnaces

Compared to a commercial blast furnace, the experimental blast furnace has less compression of the burden, shorter gas and iron ore residence time, higher heat losses (per tonne of hot metal), lower hot metal temperature and higher hot metal silicon content.\footnote{[41]} Despite these differences, the experimental blast furnace proceeds a complete ironmaking process as done in the commercial blast furnace, all the functions proceeded including reduction, softening, melting, desulphurising, alkali re-circulation and so on, are the same. In addition, some abnormal operations such as scaffolding, hanging, channelling and other typical disturbances may also occur in the experimental blast furnace, depending on the operation conditions and the burden quality. The chemical and thermal treatment of the pellets in the experimental blast furnace can therefore be said to simulate a full-scale blast furnace. As to now, the experience is that the experimental blast furnace is a very sensitive tool for detecting differences in properties for different burden materials. The response time is much shorter for the experimental furnace compared to a commercial furnace.

The main difference is perhaps higher fuel rate for the experimental blast furnace due to its high heat losses, which results in a higher rate of reducing gas to iron ore. In order to get a fuel rate close to that reached in a commercial scale furnace, the experimental blast furnace was designed to use higher blast temperature and higher intensity of the operation than that used in a normal commercial scale furnace. To further decrease the heat losses, ceramic refractory insulation of the furnace has been used where possible, and only the tuyeres and the lower bosh region immediately above the tuyeres are water-cooled. Thereby the heat loss is minimised and, consequently, the fuel rate is kept relatively low, 510 to 540 kg/tHM, which is comparable to production blast furnaces. An example of a comparison of the blast furnace process in the experimental blast furnace with a commercial blast furnace is given in Supplement III.

7.1.3 Quenching and dissection of the furnace

Shut-down and quenching of the experimental blast furnace is performed by flushing the burden column with nitrogen gas. The subsequent dissection of the furnace is carried out by first removing the furnace top, and then carefully removing the burden material layer by layer, from stockline down to the hearth. Each burden layer uncovered is examined, and the appearance and nature of every separate layer are documented by photographs and video shots, in addition to written documentation.

Before quenching the blast furnace several basket samples are introduced into the burden layers, in a carefully pre-determined manor. The individual baskets contain about 600 grams of raw material, and provide the opportunity to study the behaviour of several different types of burden materials, at different levels in the furnace, after each dissection. Figure 7.8 shows an example of basket samples found again in the burden during dissection. The material from each basket sample is examined chemically, physically and, if necessary, also by microscope; and is compared to samples of the “bulk” material next to the individual baskets.
Figure 7.2. Cross-section of the experimental blast furnace.

Figure 7.3. Overview of the experimental blast furnace plant.

Figure 7.4. The hearth of the experimental blast furnace (detachable).
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

Figure 7.5. Pebble bed heater.

Figure 7.6. Tapping into a sand bed for solidification.

Figure 7.7. Slag sampling during tapping.
7 EXPERIMENTS PART II: EXPERIMENTAL BLAST FURNACE TRIALS

Figure 7.8. Two basket samples together with one briquette, found again in the burden during dissection (this example is from the shaft part of the furnace).

7.2 EXPERIMENTAL CAMPAIGNS

Brief information of the operating parameters in the experimental blast furnace trials studied in this work will be given below.

7.2.1 Operation conditions and temperature profiles when using MPBO pellets in the experimental blast furnace

The olivine pellet MPBO is used as a reference material in the experimental blast furnace. Therefore, operational data with this type of pellet in the furnace are available from all experimental blast furnace campaigns. Table 7.2 displays the operating parameters in different campaigns. In Chapter 8 (and Supplement IV) data from the dissection after Campaign A are used for comparing with laboratory reduction results from the first part of this work. Data from campaigns B to E are used to study the internal temperature and gas composition profiles, for comparison with laboratory simulation conditions as well as with commercial furnaces. The main differences of the operating conditions among the different campaigns were the coal injection and oxygen enrichment rates used. While Campaign A was run on all-coke operation, Campaigns B and C were run with a moderate coal injection rate and Campaigns D and E with comparatively high injection rates. Another operational
difference was that during Campaign A the blast furnace was wall-working, while in the later campaigns the operation was centre-working.

The reproducibility of the vertical probe temperature measurements can be observed in Figure 7.9, which shows the measurement results on different days during Campaign E. The ability to attain similar conditions in different campaigns is considered good. Figure 7.10 gives a comparison of the in-burden wall temperature profile between different campaigns. The burden composition used was almost the same, but different operating parameters were used in different campaigns, resulting in different in-burden temperature profiles. However, the differences in temperature between campaigns with similar operating conditions, for instance between Campaigns B and C, and between Campaigns D and E respectively, were small, in the range of 50 °C in most of the furnace shaft.

Table 7.2. Blast furnace operating parameters prior to shut-down of Campaign A, and during Campaigns B, C, D and E respectively – averages of 24 hours stable (representative) operation.

<table>
<thead>
<tr>
<th>Campaign no.</th>
<th>Campaign A</th>
<th>Campaign B</th>
<th>Campaign C</th>
<th>Campaign D</th>
<th>Campaign E</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Productivity (tHM/m³/24h)</strong></td>
<td>4.9</td>
<td>4.3</td>
<td>4.1</td>
<td>4.5</td>
<td>4.3</td>
</tr>
<tr>
<td><strong>Productivity (tHM/m³/24hr)</strong></td>
<td>35.5</td>
<td>30.9</td>
<td>29.8</td>
<td>32.6</td>
<td>31.2</td>
</tr>
<tr>
<td><strong>MPBO pellets (kg/tHM)</strong></td>
<td>1396</td>
<td>1381</td>
<td>1389</td>
<td>1384</td>
<td>1387</td>
</tr>
<tr>
<td><strong>Quartzite (kg/tHM)</strong></td>
<td>10</td>
<td>7</td>
<td>7</td>
<td>15</td>
<td>18</td>
</tr>
<tr>
<td><strong>Limestone (kg/tHM)</strong></td>
<td>50</td>
<td>33</td>
<td>42</td>
<td>53</td>
<td>48</td>
</tr>
<tr>
<td><strong>BOF slag (kg/tHM)</strong></td>
<td>51</td>
<td>52</td>
<td>54</td>
<td>36</td>
<td>46</td>
</tr>
<tr>
<td><strong>Coke (kg/tHM)</strong></td>
<td>515</td>
<td>442</td>
<td>439</td>
<td>407</td>
<td>403</td>
</tr>
<tr>
<td><strong>Temperature (°C)</strong></td>
<td>1208</td>
<td>1178</td>
<td>1171</td>
<td>1169</td>
<td>1198</td>
</tr>
<tr>
<td><strong>Coal injection, PCI (kg/tHM)</strong></td>
<td>-</td>
<td>74</td>
<td>87</td>
<td>121</td>
<td>132</td>
</tr>
<tr>
<td><strong>Oxygen enrichment (%)</strong></td>
<td>-</td>
<td>1.9</td>
<td>1.5</td>
<td>3.6</td>
<td>3.5</td>
</tr>
<tr>
<td><strong>Moisture (g/Nm³)</strong></td>
<td>40</td>
<td>26</td>
<td>27</td>
<td>23</td>
<td>23</td>
</tr>
<tr>
<td><strong>Temperature (°C)</strong></td>
<td>121</td>
<td>202</td>
<td>210</td>
<td>209</td>
<td>199</td>
</tr>
<tr>
<td><strong>Pressure (bar, gauge)</strong></td>
<td>0.7</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td><strong>ηCO (%)</strong></td>
<td>48.7</td>
<td>48.8</td>
<td>47.9</td>
<td>46.9</td>
<td>46.8</td>
</tr>
<tr>
<td><strong>CO (%)</strong></td>
<td>23.0</td>
<td>22.6</td>
<td>22.5</td>
<td>25.4</td>
<td>25.2</td>
</tr>
<tr>
<td><strong>CO₂ (%)</strong></td>
<td>21.8</td>
<td>21.6</td>
<td>20.7</td>
<td>22.5</td>
<td>22.2</td>
</tr>
<tr>
<td><strong>H₂ (%)</strong></td>
<td>2.4</td>
<td>2.6</td>
<td>2.8</td>
<td>3.6</td>
<td>3.8</td>
</tr>
<tr>
<td><strong>Temperature (°C)</strong></td>
<td>1416</td>
<td>1460</td>
<td>1435</td>
<td>1468</td>
<td>1408</td>
</tr>
<tr>
<td><strong>C (%)</strong></td>
<td>4.5</td>
<td>4.6</td>
<td>4.4</td>
<td>4.5</td>
<td>4.4</td>
</tr>
<tr>
<td><strong>Si (%)</strong></td>
<td>0.66</td>
<td>1.17</td>
<td>1.54</td>
<td>1.71</td>
<td>1.23</td>
</tr>
<tr>
<td><strong>S (%)</strong></td>
<td>0.065</td>
<td>0.032</td>
<td>0.030</td>
<td>0.032</td>
<td>0.067</td>
</tr>
<tr>
<td><strong>Volume (kg/tHM)</strong></td>
<td>146</td>
<td>136</td>
<td>137</td>
<td>140</td>
<td>148</td>
</tr>
<tr>
<td><strong>CaO (%)</strong></td>
<td>30.5</td>
<td>30.9</td>
<td>34.1</td>
<td>32.6</td>
<td>33.1</td>
</tr>
<tr>
<td><strong>SiO₂ (%)</strong></td>
<td>34.5</td>
<td>34.0</td>
<td>32.8</td>
<td>32.9</td>
<td>35.7</td>
</tr>
<tr>
<td><strong>MgO (%)</strong></td>
<td>17.0</td>
<td>18.7</td>
<td>17.3</td>
<td>18.2</td>
<td>17.3</td>
</tr>
<tr>
<td><strong>Al₂O₃ (%)</strong></td>
<td>12.8</td>
<td>14.6</td>
<td>14.5</td>
<td>15.2</td>
<td>13.1</td>
</tr>
</tbody>
</table>

1) Utilisation of furnace volume (working volume from stockline to tuyere level).
2) Output per unit hearth area.
7.2.2 Coated blast furnace pellets

When testing coated pellets in the experimental blast furnace, most of the operating parameters, shown in Table 7.3, were chosen to resemble the operation of the SSAB Tunnplåt no. 3 blast furnace.
in Luleå. The operating parameters were basically unchanged during the whole test period, the intention was to keep all of them on the same level during all periods of the trial.

The trial was divided into five different periods:
1. MPBO-2  Reference period using pellets without coating
2. MPBO-O  Olivine coated MPBO-3 pellets
3. MPBO-D  Dolomite coated MPBO-3 pellets
4. MPBO-Q  Quartzite coated MPBO-3 pellets
5. MPBO-3  Reference period using pellets without coating

MPBO-2 and MPBO-3 are basically the same type of pellets. Both are olivine pellets with addition of olivine and a small amount of limestone, and in the MPBO-3 pellet also a small amount of silica is added. Both pellets have been operated at SSAB Tunnplåt (Luleå) and SSAB Oxelösund in Sweden, and at Fundia Koverhar in Finland, without any significant difference in blast furnace operation.

Results of the trials are given in Chapter 9.

Table 7.3. Blast furnace operating parameters during the trials.

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duration (h)</td>
<td>85</td>
<td>83</td>
<td>48</td>
<td>68</td>
<td>27</td>
</tr>
<tr>
<td>Blast temperature (°C)</td>
<td>1198</td>
<td>1197</td>
<td>1198</td>
<td>1197</td>
<td>1197</td>
</tr>
<tr>
<td>Wind rate (Nm³/h)</td>
<td>1590</td>
<td>1589</td>
<td>1591</td>
<td>1590</td>
<td>1570</td>
</tr>
<tr>
<td>Coal injection, PCI (kg/tHM)</td>
<td>133</td>
<td>131</td>
<td>123</td>
<td>127</td>
<td>122</td>
</tr>
<tr>
<td>Oxygen enrichment (%)</td>
<td>3.3</td>
<td>3.4</td>
<td>3.5</td>
<td>3.4</td>
<td>3.4</td>
</tr>
<tr>
<td>Blast moisture (g/Nm³)</td>
<td>26</td>
<td>26</td>
<td>27</td>
<td>27</td>
<td>27</td>
</tr>
<tr>
<td>Flame temp. (calculated, °C)</td>
<td>2188</td>
<td>2195</td>
<td>2201</td>
<td>2201</td>
<td>2204</td>
</tr>
<tr>
<td>Top pressure (bar, gauge)</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
</tbody>
</table>

7.3 SUMMARY
The features and operation of the experimental blast furnace was comprehensively described in the first part of this chapter. Thereafter, the operation of the furnace during the different trials studied in this work was presented.
The main purpose of this part of the work was to study how well laboratory data compare with data from an experimental blast furnace. The laboratory results, given in Chapter 6, were compared with data obtained by the dissection of the experimental blast furnace after Campaign A, and some operating parameters from Campaigns B to E (sequential campaign numbers 1, 4, 5, 8 and 9 respectively). A detailed description of this work is given in Supplement IV.

8.1 EXPERIMENTAL PARAMETERS
The parameters for the laboratory reduction-under-load (RUL) experiments were chosen to simulate the blast furnace process in a commercial blast furnace, as was described in Chapter 5. Therefore, the reducing programs had longer reduction time compared to the experimental blast furnace, which has a shorter through-put time of iron bearing materials compared to most commercial blast furnaces. In Figure 8.1, where reduction temperature versus time is compared for the experimental blast furnace and the laboratory experiments, the differences in temperature/time cycles can be seen. The heating rate of the centre position of the reduction-under-load experiments resembled the vertical temperature profiles at the wall of the experimental blast furnace.

Figure 8.1. Reduction temperature versus time in the experimental blast furnace, compared to the set-up of the RUL experiments. Temperature measurements in the experimental blast furnace are for the wall position of the furnace radius.
8.2 COMPARISON OF REDUCTION DEGREE

As a consequence of the differences in reduction time, comparisons of reduction data between laboratory and experimental blast furnace results were not made on a time basis, but based on the expected position of the laboratory sample in the blast furnace that was simulated. In Figure 8.2 an example of the comparisons of reduction degrees between laboratory results and the experimental blast furnace is shown as a function of vertical position in the furnace.

Figure 8.2. Comparison of reduction degree in the experimental blast furnace and RUL experiments, as a function of distance from stockline, mid-radius position. The dimensionless distance for the RUL results were based on the total reduction time of the experiment.

As can be seen in Figure 8.2, initially the reduction is fast in the experimental blast furnace. This is followed by a very low reduction rate at a reduction degree from 25 to 30 per cent, and finally again a high reduction rate (disregarding the last, minor, reduction probably occurring in the liquid phase). The same observation is often given in literature describing the reduction profile in a blast furnace.\[16,33\] The region including very little reduction is generally referred to as the chemical reserve zone. The extent of the chemical reserve zone is depending on the geometry of the blast furnace, i.e. the height of the internal burden column for a specific blast furnace. The chemical reserve zone in the experimental blast furnace was from the dissection data estimated to stretch from 1.0 m to 2.0 m below the stockline. There is also a thermal reserve zone in the shaft of the blast furnace, where there is only little heat transfer from the ascending blast furnace gas to the descending burden. The thermal reserve zone often includes the chemical reserve zone, but also a part of the region of initial reduction above the chemical reserve zone and what is called the indirect reduction zone below the chemical reserve zone. Using data from the vertical temperature probings, the thermal reserve zone was found to include the distance from a depth of less than 1 m down to 3.5 m, or in some cases even beyond 4 m, below the stockline, when operating the experimental blast furnace with the MPBO pellet as the
For the wall and the mid-radius positions, the deviation in reduction between laboratory experiments and the experimental blast furnace was not considerably large. However, the shape of the reduction curves was different. In the experimental blast furnace, the reduction was restricted in the middle part of the furnace, corresponding to the chemical reserve zone as described above. Though a thermal reserve zone was applied in the mid-radius reduction-under-load experiments, a chemical reserve zone behaviour was not observed, as shown in Figure 8.2. This was due to the reducing gas potential used in the experiments, which was somewhat too high to restrict the reduction in the thermal reserve zone. The effect of the limited gas flow in the centre of the experimental blast furnace, restricting the reduction rate, in combination with the experimental set-up of RUL-experiments with extremely strong reducing gas and a high heating rate, was a strong deviation in reduction after the wüstite reduction stage for the centre sample.

The differences described above are due to differences in reducing conditions. The gas compositions were somewhat different, when comparing the levels of the different gas components between the laboratory tests and the results of the experimental blast furnace gas measurements (Campaigns D and E). In the laboratory tests the contents of CO in the gas used were lower than that found in the experimental blast furnace. However, the CO₂ contents were also lower, resulting in a higher CO/CO₂ ratio. This is demonstrated in Figure 8.3, where the gas ratios (CO/[CO+CO₂] × 100) in the laboratory tests and experimental blast furnace have been inserted into the Fe-C-O equilibrium diagram. It can be seen that the gas ratios below 900 °C were all higher for the laboratory tests, compared to the conditions of experimental blast furnace Campaign D and E.

The lower gas ratios below 900 °C in the experimental blast furnace is caused by the equilibrium between iron and wüstite, governing the gas ratio at low temperatures. At higher temperatures the CO gas is much more stable compared to CO₂, and almost all CO₂ produced is converted to CO, resulting in a gas ratio of close to 100. The effect of the lower gas ratios at low temperatures was found in the restricted reduction in the middle part of the experimental blast furnace. Similarly, the fast increase in gas ratio at 900 to 1000 °C, for the experimental blast furnace conditions compared to the laboratory tests, was reflected in a faster reduction in the lower part of the furnace.

The differences in reduction time for the laboratory tests compared to the experimental blast furnace is partly compensated for when comparing degree of reduction as a function of distance in the furnace, as shown in Figure 8.2. The similar gas ratios thereby results in similar reduction for the wall and mid-radius positions, when comparing by the distance in the furnace. For the centre position, on the other hand, the conditions were quite opposite between laboratory and the experimental blast furnace, resulting in large differences in observed reduction degree.
8.3 REDUCTION MODE

In Figure 8.4 the extent of the different iron oxides/iron phases down through the furnace is shown for the mid-radius positions of the experimental blast furnace. It can be seen that the hematite was quickly reduced in the upper part of the blast furnace. However, the reduction of magnetite to wüstite was found to start before complete reduction of hematite to magnetite. Similarly, iron started to form long before all magnetite was reduced to wüstite. Thus, in the major part of the blast furnace shaft three different iron oxide/iron phases co-existed.

For the laboratory reduction-under-load samples, the reduction was found to take place in stages, i.e. reduction to a lower oxygen containing iron oxide phase did not occur until the previous reduction step was completed, which was described in Chapter 6 and in Supplement II. Therefore, at no time more than two iron oxide (or iron) phases co-existed. As shown in Figure 8.4, in the experimental blast furnace there was a clear over-lapping in the reduction process resulting in three co-existing phases (i.e. hematite/magnetite/wüstite or magnetite/wüstite/iron) for longer periods, in this case from 0.3 to 3.5 m below stockline.
8 COMPARISON OF REDUCTION BETWEEN THE EXPERIMENTAL BLAST FURNACE AND LABORATORY EXPERIMENTS

8.4 SOFTENING AND MELTING

In the reduction-under-load experiments it was found that the pellet particles studied retained their original spherical shape until melting started. During heating the metallised iron in the particles of the pellet bed was found to pick up carbon in contact with coke, and thereby eventually melt because of the lowered melting temperature of the iron phase. There was an increasing, and especially at very high temperatures extensive, sintering between the particles, lowering the void fraction of the pellet bed. There was also sintering within pellets, causing a shrinking of the individual particles and thereby a contraction of the bed. What is normally referred to in literature as softening, though, was not found to occur.

In Figure 8.5 a sample from a reduction experiment interrupted at a temperature of 1430 °C, and a contraction degree of 69 per cent, is shown. Some particles of the sample bed have been mechanically deformed, but most of them show little, or no, tendency of deformation. The particles around the periphery are partially melted. The main reason for the start of melting at the periphery was probably a higher temperature at the crucible wall, since the heat was supplied from the outside of the sample crucible by the furnace. Carbon pick-up by the metallic iron decreases its melting point leading to partial melting.
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

In *Figure 8.5* the cross-section of some pellets can also be seen, where samples have been removed for chemical analyses as well as for metallographical investigations. The pellets consist of a metallic shell with a hollow core. Inside the metallic shell, there was slag adhering to the iron. In Chapter 6 the microstructure in one of the samples removed were presented.

Even though the reduction was found to be considerably faster in the experimental blast furnace, the mechanism of contraction of the pellet bed due to sintering and the melt-down process, without considerable deformation, was found to be the same. *Figure 8.6* shows an example of the cohesive zone formation in two pellet layers, while *Figure 8.7* shows an examples of the underside of the cohesive zone, with partly melted metal where it has been in contact with coke.
Figure 8.6. Two consecutive pellet layers constituting the cohesive zone.

Figure 8.7. The underside of the cohesive zone, with partly melted metal where it has been in contact with coke. Note, the next pellet layer to be melted in the top right corner, the originally spherical shape virtually unchanged.
The melting of the metallised pellets was found to be rapid, occurring only in two consecutive layers of the burden, i.e. only two layers contained semi-melted material, or what is normally referred to as cohesive zone formation. The absence of significant softening during reduction/melt-down is believed to be a special feature of this type of pellet, since in later experimental blast furnace excavations other types of burden materials have been found to behave differently during reduction and melt-down. In many cases, using other types of pellets, there has been extensive softening of pellets in the high temperature region of the furnace, resulting in several (in extreme cases up to 7-8) layers of what was defined as the cohesive zone.

8.5 SUMMARY

The very narrow range of the melting process was in agreement with what was found in the laboratory experiment, and is considered as an important advantage of the MPBO pellet, together with the start of melting occurring at a high temperature. The narrow melting range results in a narrow cohesive zone in the blast furnace, which in turn gives a low pressure drop. The high melting temperature obtained in the laboratory tests gives a cohesive zone low in the blast furnace, thereby providing more space for gaseous reduction. Thus, the laboratory experiments were considered to simulate the high temperature phenomena of the blast furnace in a realistic way.

Comparing reduction in laboratory scale with blast furnace reduction can be appropriately made as long as the reduction parameters for laboratory scale testing are correctly chosen. The deviation in reduction behaviour observed in this work was mostly due to the experimental parameters chosen for laboratory testing deviating from the conditions of the experimental blast furnace, which in turn was caused by the sequence of the experiments. Therefore, if laboratory reduction-under-load tests are considered for simulation of the blast furnace process, those could certainly be adequately performed by adjusting the laboratory reduction parameters, i.e. temperature/gas composition/time programs, for the blast furnace process in question.

In order to simulate the blast furnace operation in laboratory scale it is, of course, important to know the conditions in the blast furnace process. Although information of temperature and gas composition at various levels in commercial blast furnaces can be found in the literature, this information may only be valid for these furnaces measured under their conditions, such as raw materials used, operational parameters and strategies applied. Probings, excavations and dissections of the experimental blast furnace can provide more useful information of the state inside the blast furnace. Using such information to adjust the test parameters used in laboratory tests a much better simulation of the blast furnace process could be made in laboratory scale.
Chapter Nine

The use of coated pellets in optimising the blast furnace operation

During dissections of the experimental blast furnace a sticking, or clustering, phenomenon of pellets has been observed. The ferrous burden was found to start sticking together in the middle and lower part of the shaft, before the material entered the cohesive zone. If clustering of the particles in a pellet bed is pronounced, the smooth descending movement of the ferrous burden in the blast furnace can be disturbed.

The mechanism of sticking during reduction of iron oxides is mainly due to the freshly reduced iron on the pellet surfaces sintering together with the neighbouring pellets at the contact points. One way of preventing the sintering between pellets is therefore to keep the iron surfaces of individual pellets apart. This can be achieved by covering the outer layer of the pellets with a low-reactive (under blast furnace reducing conditions) coating material. By using coated pellets, it should be possible to decrease the clustering tendency, at least above the cohesive zone of the blast furnace.

This chapter is based on Supplement V, which describes the possible effects of using coated blast furnace pellets and the results obtained by laboratory testing of coated pellets, as well as testing in the LKAB experimental blast furnace.

9.1 COATING OF PELLETS
Coated pellets for this experiment were made by coating regular MPBO (LKAB olivine pellets) with different types of coating materials. The chemical analyses of the pellets, as well as the coating materials used, are shown in Table 9.1. MPBO-2 and MPBO-3 are basically the same type of pellets, both are olivine pellets with addition of olivine and a small amount of limestone, and in the MPBO-3 pellet also a small amount of silica is added. The MPBO-3 pellet was used as the base pellet for the coating experiments, while both uncoated MPBO-2 and MPBO-3 were used as reference materials in the experimental blast furnace.

Olivine, quartzite and dolomite, respectively, were mixed with 9% of bentonite as a binding phase, and applied to the pellets, in an amount of 4 kg of solid coating materials per ton of pellet product. Chemical analyses of the base pellets and the coated pellets are given in Table 9.2, where chemical analyses of the pellets sampled at the blast furnace site are also given. As can be seen from the data
the amount of coating materials could be accurately controlled, and the coating materials were found to well stay on the pellet surfaces even after storage, transport, handling and screening (undersize <6 mm screened off before charging to the blast furnace).

### Table 9.1. Chemical analysis of oxide pellets and coating materials (weight per cent).

<table>
<thead>
<tr>
<th>Material</th>
<th>MPBO-2</th>
<th>MPBO-3</th>
<th>Olivine</th>
<th>Quartzite</th>
<th>Dolomite</th>
<th>Bentonite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe (%)</td>
<td>66.6</td>
<td>66.6</td>
<td>5.0</td>
<td>0.3</td>
<td>1.0</td>
<td>3.8</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>1.78</td>
<td>2.00</td>
<td>42.20</td>
<td>98.00</td>
<td>2.00</td>
<td>56.30</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>0.32</td>
<td>0.22</td>
<td>0.80</td>
<td>0.02</td>
<td>29.50</td>
<td>2.83</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>1.48</td>
<td>1.42</td>
<td>49.50</td>
<td>0.09</td>
<td>21.00</td>
<td>3.73</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>0.29</td>
<td>0.29</td>
<td>0.44</td>
<td>1.00</td>
<td>0.37</td>
<td>18.60</td>
</tr>
<tr>
<td>TiO₂ (%)</td>
<td>0.39</td>
<td>0.37</td>
<td>0.03</td>
<td>0.03</td>
<td>0.00</td>
<td>0.83</td>
</tr>
<tr>
<td>MnO (%)</td>
<td>0.06</td>
<td>0.05</td>
<td>0.00</td>
<td>0.01</td>
<td>0.10</td>
<td>0.06</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.29</td>
<td>0.09</td>
<td>0.57</td>
</tr>
<tr>
<td>V₂O₅ (%)</td>
<td>0.26</td>
<td>0.25</td>
<td>0.02</td>
<td>0.01</td>
<td>0.00</td>
<td>0.05</td>
</tr>
<tr>
<td>P₂O₅ (%)</td>
<td>0.017</td>
<td>0.017</td>
<td>0.030</td>
<td>0.011</td>
<td>0.050</td>
<td>0.160</td>
</tr>
</tbody>
</table>

### Table 9.2. Compositions of pellets before and after coating (weight per cent). Results shown are a) chemical analysis before coating, b) expected analysis after coating (calculated), c) chemical analysis of pellets after coating, and d) chemical analysis of samples taken at the blast furnace site, i.e. after storing (outside 4 to 6 weeks), transport, handling and on-size screening (+6 mm).

<table>
<thead>
<tr>
<th>Material</th>
<th>Sample</th>
<th>Coating</th>
<th>SiO₂ (%)</th>
<th>MgO (%)</th>
<th>CaO (%)</th>
<th>Fe (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPBO-3</td>
<td>a) Base material</td>
<td>None</td>
<td>2.00</td>
<td>1.42</td>
<td>0.22</td>
<td>66.60</td>
</tr>
<tr>
<td></td>
<td>b) Theoretical</td>
<td>Olivine</td>
<td>2.16</td>
<td>1.60</td>
<td>0.22</td>
<td>66.33</td>
</tr>
<tr>
<td></td>
<td>c) At pellet plant</td>
<td>Olivine</td>
<td>2.16</td>
<td>1.65</td>
<td>0.26</td>
<td>66.39</td>
</tr>
<tr>
<td></td>
<td>d) At BF site</td>
<td>Olivine</td>
<td>2.15</td>
<td>1.64</td>
<td>0.20</td>
<td>66.44</td>
</tr>
<tr>
<td>MPBO-0</td>
<td>b) Theoretical</td>
<td>Dolomite</td>
<td>2.01</td>
<td>1.50</td>
<td>0.31</td>
<td>66.33</td>
</tr>
<tr>
<td></td>
<td>c) At pellet plant</td>
<td>Dolomite</td>
<td>2.01</td>
<td>1.50</td>
<td>0.38</td>
<td>66.49</td>
</tr>
<tr>
<td></td>
<td>d) At BF site</td>
<td>Dolomite</td>
<td>1.98</td>
<td>1.50</td>
<td>0.29</td>
<td>66.55</td>
</tr>
<tr>
<td>MPBO-D</td>
<td>b) Theoretical</td>
<td>Quartzite</td>
<td>2.37</td>
<td>1.42</td>
<td>0.22</td>
<td>66.33</td>
</tr>
<tr>
<td></td>
<td>c) At pellet plant</td>
<td>Quartzite</td>
<td>2.42</td>
<td>1.40</td>
<td>0.20</td>
<td>66.24</td>
</tr>
<tr>
<td></td>
<td>d) At BF site</td>
<td>Quartzite</td>
<td>2.50</td>
<td>1.44</td>
<td>0.19</td>
<td>66.24</td>
</tr>
</tbody>
</table>

### 9.2 INVESTIGATION OF THE COATING LAYER UNDER MICROSCOPE

The theoretical thickness of the coating layer, when using an amount of coating material of 4 kg/ton of pellets, has been calculated to be 0.010 mm (0.008 mm for olivine due to its higher density), assuming a uniform layer on an ideal sphere with a diameter of 10.5 mm. The actual coating layers were investigated in scanning electron microscope (SEM). In Figure 9.1 an example of the outer part of the olivine coated pellets is shown. The coating layer thickness observed is in the range from zero up to 0.100 mm. As expected, there were also some areas of the pellet surfaces examined not covered by coating at all, as shown by the right particle in Figure 9.1.
9 THE USE OF COATED PELLETS IN OPTIMISING THE BLAST FURNACE OPERATION

9.3 STICKING TESTS

To investigate the sticking behaviour of the coated pellets, different types of reduction/sticking tests were carried out, of which three are presented here:

1. **HYL Standard test**
   The first test method used was the HYL Standard test, normally performed on all DR pellet deliveries. At most DR plants, the upper limit of the sticking index is 30.

2. **BFS (Blast Furnace Sticking) test**
   Since the HYL Standard test is well-known and has been widely accepted as a testing method for DR pellets, it constituted the basis for the development of a modified test for the coated blast furnace. The hydrogen-based gas mixture in the HYL Standard test (55% H₂, 21% CO, 14% CO₂ and 10% N₂) was changed to a carbon monoxide/nitrogen based gas mixture (2% H₂, 40% CO and 58% N₂) in the BFS test. The results of the sticking tests are given in Table 9.3. A significant effect of the coating materials on the sticking behaviour can be seen, when comparing with the results for the uncoated pellet, which experiences a sticking index in the range of 45 to 50. Olivine and dolomite coatings seem to be the best, SI around 3, while quartzite is a somewhat less effective coating material, with an SI value of 8. These results clearly indicate that all of the three types of coated pellets have a very low sticking tendency.

Figure 9.1. SEM image of surface layer of olivine coated pellet. On left particle the coating layer (dark grey) is from zero up to 0.100 mm in thickness, while the surface of the right particle was not covered by coating. Scale mark is 600 μm.
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

<table>
<thead>
<tr>
<th>Test method</th>
<th>Measured properties</th>
<th>MPBO-3</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>HYL Standard</td>
<td>Sticking index, SI</td>
<td>45</td>
<td>3</td>
<td>3</td>
<td>8*</td>
</tr>
<tr>
<td></td>
<td>Reduction time (min)</td>
<td>60</td>
<td>61</td>
<td>70</td>
<td>66*</td>
</tr>
<tr>
<td>BFS</td>
<td>Sticking index, SI</td>
<td>50*</td>
<td>3*</td>
<td>4*</td>
<td>8*</td>
</tr>
<tr>
<td></td>
<td>Reduction time (min)</td>
<td>160*</td>
<td>163*</td>
<td>147*</td>
<td>169*</td>
</tr>
</tbody>
</table>

Despite two to three times longer reduction time in the blast furnace gas composition compared to DR gas composition, the resulting sticking index values were almost identical. These results indicate that the sticking index is probably independent of the reduction velocity.

3. Modified ISO 7992 (reduction under load) test

To examine if a standardised reduction test under load, ISO 7992[1], which is commonly used for testing reduction behaviour of blast furnace burden materials, could also be used for sticking tests, the ISO 7992 test was appended with a drop test for measuring sticking after reduction. The ISO 7992 test was conducted at three different levels of temperature, 1050 °C, 1000 °C and 950 °C. Results of these tests are shown in Figure 9.2. As can be seen in the figure, the influence of reduction temperature on the sticking behaviour is distinct in the temperature range from 950 °C to 1050 °C, both for uncoated and coated pellets. At 950 °C all the coated pellets have SI values lower than 8, while the uncoated has a value of 22. At 1000 °C the values are in the range of 17 to 40 for coated pellets, compared to 71 for uncoated pellets. Finally, at 1050 °C both the uncoated and the quartzite coated pellets became severely sintered (95 in sticking index), while a sticking preventing effect was still achieved by the olivine and dolomite coatings, resulting in SI values of 47 and 35 respectively.

![Figure 9.2. Sticking index after reduction-under-load test at different temperatures (modified ISO 7992[1] test).](image_url)
9.4 EXPERIMENTAL BLAST FURNACE TRIALS WITH COATED PELLETS

The experimental conditions of the trial were described in Chapter 7.

9.4.1 Flue dust generation

Table 9.4 shows the amounts of flue dust collected, its composition, and the estimated contributions of different sources to the flue dust. A significant decrease in blast furnace flue dust, collected in the dry dust catcher cyclone, was observed during the trials with coated pellets, from 5.4 kg/tHM for the MPBO-2 and 4.4 kg/tHM for the MPBO-3 pellets down to below 3 kg/tHM for the coated pellets. The mass balances show that less pellet fines accounted for 0.8 to 1.0 kg/tHM of the decrease in flue dust amounts when using coated pellets compared to uncoated, or inversely expressed: pellet material as the flue dust leaving the furnace decreased by about two thirds.

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flue dust, dry (kg/tHM)</td>
<td>5.4</td>
<td>2.9</td>
<td>2.7</td>
<td>3.0</td>
<td>4.4</td>
</tr>
<tr>
<td>Fe (%)</td>
<td>21.6</td>
<td>13.8</td>
<td>n.a.</td>
<td>13.3</td>
<td>21.8</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>11.1</td>
<td>15.9</td>
<td>n.a.</td>
<td>20.8</td>
<td>17.7</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>16.2</td>
<td>14.1</td>
<td>n.a.</td>
<td>12.1</td>
<td>14.2</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>4.3</td>
<td>9.2</td>
<td>n.a.</td>
<td>6.3</td>
<td>6.8</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>3.0</td>
<td>4.2</td>
<td>n.a.</td>
<td>4.0</td>
<td>4.0</td>
</tr>
<tr>
<td>TiO₂ (%)</td>
<td>0.2</td>
<td>0.2</td>
<td>n.a.</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>MnO (%)</td>
<td>0.3</td>
<td>0.4</td>
<td>n.a.</td>
<td>0.4</td>
<td>0.3</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.3</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.4</td>
<td>0.6</td>
</tr>
<tr>
<td>V₂O₅ (%)</td>
<td>0.3</td>
<td>0.3</td>
<td>n.a.</td>
<td>0.3</td>
<td>0.2</td>
</tr>
<tr>
<td>P₂O₅ (%)</td>
<td>0.1</td>
<td>0.1</td>
<td>n.a.</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td>C (%)</td>
<td>20.4</td>
<td>26.0</td>
<td>n.a.</td>
<td>31.2</td>
<td>16.5</td>
</tr>
<tr>
<td>From pellets (kg/tHM)</td>
<td>1.5</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.5</td>
<td>1.3</td>
</tr>
<tr>
<td>From coke (kg/tHM)</td>
<td>1.4</td>
<td>0.9</td>
<td>n.a.</td>
<td>1.1</td>
<td>0.9</td>
</tr>
<tr>
<td>From limestone (kg/tHM)</td>
<td>1.0</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.4</td>
<td>0.8</td>
</tr>
<tr>
<td>From BOF-slag (kg/tHM)</td>
<td>1.0</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.5</td>
<td>0.7</td>
</tr>
<tr>
<td>From quartzite (kg/tHM)</td>
<td>0.5</td>
<td>0.3</td>
<td>n.a.</td>
<td>0.3</td>
<td>0.7</td>
</tr>
<tr>
<td>From olivine coating (kg/tHM)</td>
<td>-</td>
<td>0.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>From quartzite coating (kg/tHM)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.2</td>
<td>-</td>
</tr>
</tbody>
</table>

It was also observed that the amounts of fine particles formed by coke fines as well as the lumpy slag formers charged were all lower for the periods with coated pellets and with the wet MPBO-3 pellet than for the period with dry MPBO-2 pellet. The cause is believed to be the combined effects of the two following factors:

1) The improved gas flow in the shaft of the furnace when using coated pellets. Less clustering, and de-clustering, as well as less channelling certainly gave a more uniform gas flow with a lower gas velocity locally, therefore less fine particles were picked up by the gas.
2) High moisture contents of the pellets used in all periods except for the MPBO-2 period. The higher moisture content of the coated pellets resulted in a lower top gas temperature, due to the energy demanded for vaporisation of the water. Assuming that the gas phase behaves ideally, the
lowering of the top gas temperature by 10 °C represents a decrease of 2 % of the top gas velocity (and 4 % for a 20 °C decrease). The lowered top gas velocity possibly resulted in less fine particles blown out of the furnace.

9.4.2 Sticking

Probe samples were taken out of the blast furnace during operation. The lower shaft probe, i.e. the third probe from the top, shown in the left part of Figure 7.2 (Chapter 7), was used to take samples at a level of 3.4 m below stockline. A SEM image of a sample of olivine coated pellets, reduced to 70 % reduction degree, is shown in Figure 9.3. A layer of the coating material can be observed on the surfaces of the pellet. Beneath the coating layer, the outer layer of the pellets is completely reduced to iron, whereas in the interior, not shown in the figure, there was still some amount of wüstite.

![Figure 9.3. SEM image of surface layer of reduced olivine coated pellet, collected by a shaft probe, 3.4 m below stockline. Inside the coating layer (dark grey) is reduced iron (reduction degree 70 %). Scale mark is 100 μm.](image)

An inclined probe, shown in the right part of Figure 7.2 (Chapter 7), was used to collect samples from the cohesive zone in the lower part of the blast furnace. The temperature in this region was, from vertical temperature probings, estimated to be in the range of 1200 to 1300 °C. Even at these high temperature levels, the sticking phenomenon had been successfully prevented by the use of coating of the pellets, as shown by the pellet samples in Figures 9.4 to 9.7. Inversely, the sticking observed is significantly worse for the uncoated pellets than for the coated ones. Also the sticking when using quartzite coated pellets is more pronounced than for olivine or dolomite coated pellets, in agreement with laboratory sticking test results at higher temperatures, Figure 9.2.
Figure 9.4. Probe samples taken from the cohesive region of the blast furnace, uncoated pellets.
Upper sample: 95 % reduction degree, 0.4 % C.
Lower sample: 98 % reduction degree, 1.9 % C.

Figure 9.5. Probe samples taken from the cohesive region of the blast furnace, olivine coated pellets.
Upper sample: 94 % reduction degree, 0.6 % C.
Lower sample: 97 % reduction degree, 1.9 % C.

Figure 9.6. Probe samples taken from the cohesive region of the blast furnace, dolomite coated pellets.
Upper sample: 91 % reduction degree, 0.3 % C.
Lower sample: 98 % reduction degree, 1.7 % C.

Figure 9.7. Probe samples taken from the cohesive region of the blast furnace, quartzite coated pellets.
Upper sample: 95 % reduction degree, 0.2 % C.
Lower sample: 96 % reduction degree, 0.8 % C.
9.4.3 Gas utilisation

The gas utilisation, $\eta_{CO}$, was at the highest levels, also with the lowest standard deviation, when using coated pellets. A high value of $\eta_{CO}$ means that more CO is oxidised to CO$_2$ in the furnace shaft. Thereby more heat released by oxidising CO is utilised in the process and the fuel demand is decreased by the increased indirect reduction.

The increase in gas utilisation when using coated pellets can be considered as a result of improved reduction conditions by the use of coated pellets, achieved by a smoother burden descent and less channelling in the blast furnace shaft, in turn caused by a decreased sticking in the pellet layers. The lowered variations in gas utilisation could also be taken as evidence of the improved gas-solid contact, with less channelling and smoother burden descent.

9.5 SUMMARY

Most of the issues of coated blast furnace pellets, as sketched in Figure 9.8, were exposed during the laboratory and experimental blast furnace trials. All coated blast furnace pellets generated less blast furnace flue dust, and resulted in a higher gas utilisation and a smoother blast furnace operation. The decreased sticking tendency, as shown by laboratory as well as experimental blast furnace test results, was attributed the improved furnace performance, possibly in combination with less dust generation inside the blast furnace.

![Figure 9.8. Schematic view of possible effects of using coated pellets in the blast furnace.](image-url)
Chapter Ten

Concluding discussion

This work has been focused on the reduction of LKAB olivine pellets in laboratory scale and in an experimental blast furnace.

Initially, a literature survey was conducted to determine the best design of a laboratory simulation of the blast furnace process. Based on this information, together with previous experience of reduction-under-load experiments, a laboratory equipment for simulation of the blast furnace process was constructed at KTH. This equipment was then used to investigate the behaviour of olivine pellets under simulated blast furnace conditions. In Supplement I the contraction behaviour of a pellet bed during reduction was examined by conducting reduction-under-load tests as well as reduction experiments without load. In this way the processes of swelling, shrinking and sintering, and finally carburisation and melt-down was explained. In Supplement II the mode of reduction of the olivine pellet was established by studying the microstructures of samples from interrupted reduction experiments. The metallographical examinations of the interrupted experiments also verified the findings of the previous investigation, and an explanation of the carburisation and melt-down process could be proposed.

Later, an attempt was made to compare the results of the laboratory reduction experiments with trials conducted in the LKAB experimental blast furnace. Supplement III was included in the thesis to illustrate the features of the experimental blast furnace, and how it is used for research purposes. In Supplement IV the comparison of reduction of the MPBO pellet in laboratory scale and in the experimental blast furnace is made. This study showed that the behaviour of iron oxides in the blast furnace, regarding progress of reduction and the softening/melt-down properties, can be simulated in laboratory scale, as long as proper reducing conditions are used. Therefore, correctly planned laboratory experiments could be useful to screen what experiments are worthwhile to conduct in the experimental blast furnace.

Finally, Supplement V gives an example of how the experimental blast can be used in optimising the blast furnace process. From observations made during dissection of the furnace, the need to prevent sticking, or clustering, of pellets in the blast furnace shaft was realised. The method of coating, i.e. covering the pellet surfaces with a thin layer of a less reactive material, was then tested in laboratory scale to establish the sticking preventing effect of the coating. In the experimental blast furnace the decreased sticking tendency of coated MPBO pellets was verified, and a more stable blast furnace
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

process, with higher gas utilisation, was obtained. In addition, there was also less generation of blast furnace flue dust during the trials with coated pellets in the experimental blast furnace, which makes the use of coating even more favourable.
Chapter Eleven

Conclusions

LABORATORY TESTS
The examination of the reduction behaviour of the olivine MPBO pellets have revealed the following:

- The reduction process of the MPBO pellets was found to proceed topochemically, from the pellet surface to the centre, not only for the pellet but for the higher oxides also in each grain.
- The reduction was found to take place in stages, i.e. the hematite pellet was first reduced completely to magnetite, then to wüstite and finally to iron.
- The contraction of a bed of MPBO pellets proceeded in three different stages, referred to as swelling, shrinking and rapid contraction respectively. The swelling period represents an expansion of the pellets. Shrinking took place at an almost constant rate up to 55 to 60 per cent of contraction before the rapid contraction started at 1420 to 1450 °C.
- Increasing sintering of metallic iron shells, accompanied by pore diffusion resulting in shrinking of the individual particles, was found to be the cause for start of contraction.
- Carburisation, resulting in partial melting of iron in contact with carbon, initiated rapid contraction, possibly in combination with melting of the slag phase. During the investigation the phenomenon normally referred to as softening was not found to occur.
- Except for the sintering between pellets, the pellets retained their original spherical shape until melting started, which is attributed the dissolution of magnesia into the wüstite phase, thereby increasing the melting temperature of the wüstite. For the blast furnace process this observation is very important, since it implies that the permeability of a layer of olivine pellets in the cohesive zone of the blast furnace might not be so impaired as is generally stated by the softening process theory.
- The very narrow range of the melting process is considered as an important advantage of the MPBO pellet, together with the start of melting occurring at a high temperature, 1420 to 1450 °C. The narrow melting range results in a narrow cohesive zone in the blast furnace, which in turn gives a low pressure drop. The high melting temperature gives a cohesive zone low in the blast furnace, thus providing more space for gaseous reduction.
COMPARING LABORATORY REDUCTION WITH THE EXPERIMENTAL BLAST FURNACE

The high temperature phenomena occurring when reducing the MPBO pellet, were found to be the same in laboratory tests and in the experimental blast furnace:

- Extensive softening of the MPBO pellets was not found to take place either in the experimental blast furnace or in the RUL furnace.
- Cohesive zone formation was found to be very restricted when using the MPBO pellet in the experimental blast furnace. Only two layers of semi-melted material indicates a fast melting of the metallised pellets, which was also observed in the RUL experiments.

There were also differences between the results of laboratory reduction and the experimental blast furnace:

- The reduction down through the burden of the experimental blast furnace was similar, but not identical to the results of the RUL experiments. The differences were found to be due to different reducing conditions.
- In the RUL experiments, at all time during reduction only two phases (iron oxides or iron) co-existed and the reduction was found to take place in stages. In the experimental blast furnace, most of the time during reduction three phases co-existed. This distinction was also believed to be due to the differences in reducing conditions.

The comparison of the experimental blast furnace with laboratory scale reduction-under-load experiments showed that a simulation of the reduction in the blast furnace can be performed in laboratory scale, provided the experimental conditions are correctly chosen. To perform such a simulation it is very important to know the internal state of the blast furnace. This information can be provided from the experimental blast furnace.

THE USE OF COATED PELLETS IN OPTIMISING THE BLAST FURNACE OPERATION

Based on the results of the investigation of the effects of coating of blast furnace pellets the following overall conclusions have been made:

- Dust generation was decreased by 32 up to 50 % when using coated pellets in the blast furnace. The amounts of pellet fines in the flue dust were only one third compared to operation with uncoated pellets.
- Sticking was prevented by the coating material. This was verified in different types of laboratory tests, as well as by studying probe samples taken from the experimental blast furnace.
- Gas utilisation was higher for all coated pellets, 47 to 48 % compared to 46.4 to 46.8 %. The variations in gas utilisation were lower, indicating a smoother blast furnace operation and a potential of getting a lower fuel rate.
Future work

Laboratory reduction experiments
Future work in laboratory simulation of the blast furnace process should include modification of the test conditions of reduction, softening and melting experiments to imitate the operation of the experimental blast furnace. The results can further verify the conclusion that it is possible to make a simulation of blast furnace reduction in laboratory scale. Laboratory reduction, softening and melting tests can then provide more accurate and valuable knowledge of pellet properties, especially high temperature properties, as a very useful simulation tool for the experimental blast furnace as well as for commercial blast furnaces.

Experimental blast furnace
By adding temperature indicators (so called tracers, or markers) to the burden before quenching of the experimental blast furnace it should be possible to establish the temperature profile of the complete cohesive zone region. Thereby, in a future work more comparisons between laboratory tests and the experimental blast furnace can be done, with more emphasis on temperatures of cohesive mass formation, start of melting and final melting, which are valuable results of reduction-under-load (or reduction, softening and melting) tests.

The gas composition profile along the height of the experimental blast furnace should be monitored by vertical gas probings, to provide an increased knowledge of the blast furnace interior, and to establish proper experimental conditions for laboratory simulations of the process.

Coating of blast furnace pellets
In the future work lies further evaluation of the trials performed in the experimental blast furnace. Especially samples taken out of the furnace will be studied in more detail under microscope to investigate the mechanism of sticking and the behaviour of coating materials in the blast furnace, and to survey the distribution of alkali components in pellets and in coating materials in different parts of the furnace.

Future work must also include verification of the results of this investigation in a commercial blast furnace. Many areas of research needs to be covered, particularly in optimisation of the coating:

- Refining of the coating technique.
- Evaluate best location of coating (at the pellet plant, in the transport chain, at the blast furnace site etc).
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

- Map out what materials that are advantageous as coating agents, and which is the best choice.
- Determine the proper choice of material as binder and the optimal amount in the coating mixture.
- Determine optimal size distribution of coating agent.
- Optimise amounts of coating material to be added and optimal water addition.

All studies mentioned above should be combined with necessary fundamental studies.

In investigating the sticking phenomenon also some work to establish the best laboratory testing method for blast furnace pellets remains, as well as determining the critical limit of sticking in the blast furnace process.

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SUPPLEMENT I

Jerker Sterneland and Ashok Lahiri

“Contraction and melt down behaviour of olivine iron ore pellets under simulated blast furnace conditions”

In the present study the contraction behaviour of the olivine iron ore pellet MPBO during reduction under load has been investigated. Three different programmes of reducing conditions have been used, corresponding to three different radial positions of the blast furnace. The reducing programmes have included different temperature profiles, reducing atmospheres, and mechanical loads applied to the specimen bed. The results of reduction under load experiments have shown that contraction has roughly three different stages: initial expansion, followed by contraction at a certain rate, and eventually a third stage with contraction at a higher rate. The process of contraction is comprehensively discussed. The influence of the different reducing conditions is outlined.

At the time the work was carried out, Mr Sterneland was in the Department of Metallurgy, Royal Institute of Technology, S-100 44 Stockholm, Sweden and Professor Lahiri was in the Department of Metallurgj, Indian Institute of Science, Bangalore, India. Mr Sterneland is now with LKAB Malmberget, S-983 81 Malmberget, Sweden. Manuscript received 26 June 1998; accepted 26 February 1999.

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INTRODUCTION

It is well known that iron bearing materials exposed to increasing temperature and mechanical load under reducing conditions, such as those in the blast furnace, contract and finally collapse during meltdown. The behaviour of iron bearing materials during contraction and meltdown is thought to depend both on the chemistry and structure of the material under consideration, and on the reducing conditions, i.e. temperature, mechanical load, reducing gas composition, etc. Other factors that might be important are the reducing history of the material and the prevailing degree of reduction.

Increasing temperature reduces the yield stress of most materials, finally resulting in breakdown. Increasing mechanical load will obviously also result in breakdown of the material subjected to it. The role of reduction with regard to contraction and breakdown is partly to change the structure of the material, i.e. grain sizes, pore structures, etc., but also to change the actual material, from iron oxides to lower oxides and finally to metallic iron, thereby continuously altering the total yield stress of the particles.

Smooth blast furnace operation and the possibility of reaching a high productivity are to a large extent dependent on the shape and position of the cohesive zone. A narrow cohesive zone maximises permeability and thereby also productivity, whereas a zone positioned deep in the furnace minimises heat consumption, lowering the production costs, and provides the possibility of stabilising hot metal composition over time. The size and position of the cohesive zone are in turn governed by the properties of the iron ore in combination with the operating practice. Therefore, testing of iron ore is important to achieve optimum blast furnace ironmaking.

In the present work it is intended to investigate the phenomena occurring during reduction under load, especially contraction and meltdown, which are of decisive importance for the size, shape, and position of the cohesive zone in the blast furnace. The aim of the study is to characterise the behaviour of olivine pellets during reduction under simulated blast furnace conditions.

EXPERIMENTAL SETUP

A number of experiments have been performed to establish the dependence of temperature, load, and reducing gas composition profiles on the contraction and meltdown behaviour of an olivine pellet during reduction. The experiments are actually of the reduction/softening/melting type but will, for simplicity, be referred to as reduction under load (RUL) tests. They are not to be confused with the standardised RUL test (ISO 7992) which ends at a temperature of 1050°C.

Experimental conditions

To investigate how the different reducing conditions at different radial positions in the blast furnace affect the reduction/softening/melting behaviour of pellets during reduction, six reducing programmes have been established.

From the literature concerning various measurements in blast furnaces during operation, dissection of blast furnaces, and modelling of the conditions in the blast furnace based on results of dissections,1–4 the parameters of rate of temperature increase, reducing gas composition, and mechanical stress at different radial positions in the blast furnace can be characterised as shown in Table 1.

The chosen programme will hereinafter be referred to as wall, midradius, and centre profiles, respectively. It is important, though, to bear in mind that the different temperature profiles, gas composition profiles, and load patterns often vary widely from furnace to furnace and also from time to time in the same furnace.

Table 1 Rough characterisation of some conditions at different radial positions in blast furnace

<table>
<thead>
<tr>
<th>Radial position</th>
<th>Wall</th>
<th>Midradius</th>
<th>Centre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rate of temperature increase</td>
<td>Low</td>
<td>Medium</td>
<td>High</td>
</tr>
<tr>
<td>Reducing gas potential</td>
<td>Medium</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Mechanical load</td>
<td>High</td>
<td>Medium</td>
<td>Low</td>
</tr>
</tbody>
</table>
Contraction and meltdown of olivine pellets

1 Temperature profiles used in reduction under load experiments

2 Six different composition profiles of carbon monoxide, corresponding to three temperature profiles

Reducing gas composition

Reduction experiments were conducted by applying two different reducing gas potentials for each temperature profile. The gas composition profiles for carbon monoxide are shown in Fig. 2. The hydrogen content of the reducing gas has been coupled to the carbon monoxide content by using a hydrogen content of one tenth of the carbon monoxide content. The two reducing gases have been balanced by carbon dioxide to 40 vol.-%, the rest being nitrogen. Also, the proportions of the reducing gas have been arrived at after studying operating data from the Swedish blast furnaces.

Table 2 Rate of temperature increase and time for completion of reduction under load tests for different temperature profiles used

<table>
<thead>
<tr>
<th>Rate of temperature increase, K min⁻¹</th>
<th>Time for complete test, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>400–800 °C</td>
<td>800–1000 °C</td>
</tr>
<tr>
<td>Wall</td>
<td>4</td>
</tr>
<tr>
<td>Midradius</td>
<td>12</td>
</tr>
<tr>
<td>Centre</td>
<td>12</td>
</tr>
</tbody>
</table>

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loss during reduction. A 15 kW tube furnace consisting of six Kanthal Super elements and an insulation of ceramic fibre materials is used for heating. The furnace is placed around the ceramic tube and a loading ram above the specimen crucible applies the intended load to the specimen bed.

During reduction, a computer program attached to the furnace equipment controls the furnace temperature, gas compositions and flowrates, and the applied load over the specimen bed. The computer also registers a variety of parameters during the test, including bed shrinkage and weight loss. Each parameter is registered 10 times per second and averaged per minute values are produced.

RESULTS

Gas analysis
During experiments the inlet and outlet gas analyses of hydrogen, carbon monoxide, and carbon dioxide were measured using an IR heat conductivity gas analyser. Measurements were conducted with a time interval of 5 min, except during the highest rate of temperature increase (12 K min\(^{-1}\)) where the interval was 3 min. The changes in reducing gas composition between inlet and outlet gases for carbon monoxide, carbon dioxide, and hydrogen contents are shown, as a function of temperature, for one experiment at each of the temperature profiles in Fig. 5.

As expected, an increase in the change in carbon dioxide content of the reducing gas corresponds well to an opposite effect in the analysis of carbon monoxide. The oscillating behaviour of the change in gas analyses of carbon monoxide and carbon dioxide has been registered in all experiments.

This behaviour cannot be explained and is believed to be a characteristic of the measuring technique used rather than of the actual process.

Calculated and measured weight loss
Based on the gas analyses of inlet and outlet gases, the weight loss of the specimen bed owing to removal of oxygen during reduction has been calculated by a simultaneous carbon and oxygen balance. The calculated weight losses are shown in Fig. 6, together with the actual weight losses measured by the load measuring device. As can be seen, in all six experiments the agreement between measured and calculated weight loss is good.

Degree of reduction
For each experiment the average of the calculated and measured weight losses were used to calculate the progress of reduction in terms of the degree of reduction \(R, \%\), defined as the proportion of oxygen removed in relation to the original oxygen content of the iron oxide

\[
R = \frac{m_{\text{accO,rem}}}{m_{\text{O,ori}}} \times 100
\]

where \(m_{\text{accO,rem}}\) is the accumulated mass of oxygen removed from the specimen, g, and \(m_{\text{O,ori}}\) is the original content of oxygen present as iron oxides, g.

Figure 7 gives the results of calculations of the degree of reduction. For each of the temperature profiles the increase in the degree of reduction is faster for the specimen treated at higher temperatures, respectively, will be obtained.

A carbon balance from the measured change in inlet and outlet gas compositions can be used to calculate the extent of carbon deposition/gasification. From the calculated rate

Carbon deposition/gasification
During reduction the change in gas analysis between inlet and outlet gases is mainly caused by reduction reactions converting carbon monoxide in the reducing gas to carbon dioxide. Part of the change in reducing gas composition, though, is caused by the Boudouard reaction

\[
2\text{CO} \leftrightarrow \text{CO}_2 + \text{C}
\]

The thermodynamic stability of the reactants of the Boudouard reaction changes with temperature. At low temperatures, carbon dioxide is the stable gas component, while at high temperatures (above 750–800°C) the stability is shifted to the left hand side of equation (2). In applications where carbon monoxide is present at a partial pressure above equilibrium, carbon can be precipitated from the gas phase if the nucleation possibilities and kinetics of the Boudouard reaction are favourable. At higher temperatures, carbon monoxide can be generated from carbon dioxide in an environment of surplus carbon.

In reduction under load experiments, therefore, carbon deposition and carbon gasification, at low and high temperatures, respectively, will be obtained.
of carbon deposition/gasification the accumulated rate of carbon deposition/gasification is arrived at by integrating the deposition/gasification rate over time. Figure 8 shows the rate of carbon deposition and gasification, together with the accumulated carbon deposition/gasification throughout the experiments conducted. In all cases the rate of carbon deposition, or precipitation, increases drastically at 600–700°C to reach a maximum value at around 750–800°C. This is a characteristic of the Boudouard reaction, which has been reported in several investigations.\(^3\) The maximum rate of carbon deposition ranges between 140 and 240 mg min\(^{-1}\), depending on the gas composition used. This represents 3–5% of the amount of carbon in the carbon monoxide phase of the reducing gas. As a comparison, the reduction reactions consume 5–10% of the carbon monoxide in the gas. In the vicinity of the
investigations. Carbon gasification then continues for the remainder of the reduction.

The accumulated carbon deposition/gasification curves, given by the time integral of the rate of carbon deposition/gasification, naturally show an increasing mass of carbon deposition up to where the instantaneous carbon deposition turns to gasification. The maximum amount of accumulated carbon deposition is a function not only of the rate of carbon deposition but also of time. Therefore, the maximum value of accumulated amount of carbon deposition is not the highest for the centre profile, which has the highest rate of deposition at all times, but is higher for both the wall and midradius profiles, which are treated for a longer time before carbon gasification starts. Maximum values for these experiments range from 6.5 to 7 g of accumulated carbon, whereas for the centre profile the values are slightly lower (from 5 to 5.5 g).

The total accumulated mass of carbon deposition then decreases and eventually turns to carbon gasification. Once again, the time dependence of the accumulated amount of carbon deposition/gasification results in higher total amounts of carbon gasification for the midradius profile, despite lower (or equal) rates of gasification, because of the longer time in the temperature range 900–1000 °C. Therefore, the total amounts of carbon gasification for the midradius profile are 7–9 g, whereas the other experiments stop between 4 and 5 g of total carbon gasification.

A discontinuity in the carbon deposition curves can be spotted at 800 °C for the centre profile and, more distinctly, at 800 and 1000 °C for the midradius profile. This is once again a consequence of change in rate of temperature increase at these points.

**Contraction**

For the six complete meltdown experiments, Fig. 9 shows the contraction of the specimen bed as a function of temperature. The contraction is given as the percentage change in specimen bed height compared with the original value according to

\[
C = \frac{\Delta H}{H_0} \times 100
\]

where \(\Delta H\) is the displacement, or change, in specimen bed height, mm, and \(H_0\) is the original specimen bed height, mm.

All contraction curves start out with an initial expansion, by 3–4% for the wall and midradius profiles and by 7–8% for the centre profile. The expansion starts first for the specimens treated with a strong reducing gas. The initial expansion is followed by a contraction at an almost constant rate, starting at somewhere between 1000 and 1200 °C. Also, the start of contraction is experienced first by specimens treated with a strong reducing gas. At higher temperatures there is a turning point to contraction at a higher rate, which is somewhat higher for specimens treated with weak reducing gas.

**DISCUSSION**

All graphs in the figures presented in the results section were given as a function of temperature. While studying the graphs it is important to bear in mind that specimens from the different radial positions simulated have been treated under different temperature cycles. The temperature dependent graphs shown do not therefore represent the same time scale. In Fig. 10 the specimen bed contraction is plotted as a function of time. For the different temperature profiles the specimen bed contraction starts at different times. This result implies that, in blast furnaces with temperature profiles corresponding to the wall, midradius, and centre profiles used in this investigation, the studied material would contract at different heights of the different furnaces, provided the descent of charge materials...
was approximately even. Thus, these results applied to blast furnace operation would lead to the formation of a W shaped cohesive zone.

In Fig. 11 the degree of reduction is plotted as a function of time. It can be seen that, at the same elapsed time, the degree of reduction is at first higher for the midradius profile than for the wall profile, despite higher reducing potential of the gas for the wall profile. The reason for this behaviour is of course the difference in rate of temperature increase. At the time when the midradius profile has the higher degree of reduction, the prevailing temperature is higher, thus increasing the rate of reduction. This shows that the degree of reduction is not only dependent on the reducing atmosphere but also on the temperature cycle under which the material is treated.

**Characterisation of contraction behaviour**

The appearance of the contraction curves shown in Fig. 9 can be divided roughly into three separate stages. First there is swelling and no contraction. Then there is up to 55–60% contraction at a certain rate (1420–1450 °C), before the third stage continues with contraction at a high rate.

In order to study the effect of mechanical load applied to a specimen during reduction, additional reduction experiments were carried out without load. The experiments were interrupted at different temperatures, and the change in volume of the specimens was registered. Figure 13 shows the volume change obtained from measurements of specimens treated without load, together with the corresponding contraction curves for reduction under load tests under the same reducing conditions. The progress in the degree of reduction was, for all cases, similar with and without load.
At the start of the reduction experiments the behaviour of specimens treated without load closely resembles that of specimens treated under load. The resemblance includes the initial expansion period and also the beginning of contraction. For the wall profile the contraction behaviour does not start to deviate until 25% contraction, or volume decrease, has been reached, while the deviation occurs earlier for the midradius and centre profiles. This was not due to the applied load, since this was the highest for the wall profile. It was observed that the deviation starts at 64–66% reduction in all cases.

**Swelling**

During stage 1 the specimen bed contraction is negative, i.e. the specimen bed expands. This is due to swelling, a well known, and unavoidable, phenomenon in the reduction of iron ore pellets. Swelling of iron ore pellets is the outcome of the reduction process and is affected by all the parameters that govern the reduction behaviour of iron ore. Among the reduction parameters, the reduction temperature, reducing gases, and their partial pressures have been reported to have a significant effect on the swelling behaviour of an iron ore pellet. The process of swelling normally includes thermal expansion, phase transformation, and growth of fibrous iron or iron whiskers. Thermal expansion and volume change during phase transformation are inherent properties of the ore, and are generally considered to be very small compared with the volume expansion due to growth of iron whiskers.

Nucleation and growth of iron whiskers take place at selected sites, such as edges, corners, etc., in the microstructure. Thus, the swelling is dependent on the number of nucleation sites for iron whisker growth. The freshly reduced iron migrates towards these nuclei, and hence growing whiskers push the adjacent grains, resulting in an increase in the pellet volume. An increased reduction rate results in a faster production of metallic iron in the form of whiskers. This explains the observation that, for all the temperature profiles in this investigation, swelling starts first for the specimens treated with the stronger reducing gas (Fig. 9), which are also observed to have the highest reduction rate (see Fig. 7).

The rate of sintering of iron is rapid above 1000°C (Ref. 7), where the mobility of iron ions is more than 100 times the mobility at 600°C (Ref. 3). Increasing movement of grain boundaries starts to reduce dislocations in the lattice structure and, thus, retards the growth of iron whiskers. Therefore, the observed expansion decreases and shrinking starts at temperatures around 1000°C (Fig. 9).

Some investigators have reported maximum swelling in a certain range of reduction, e.g. between 45 and 50% (Ref. 3) or between 60 and 70% reduction, or at a certain temperature, 1000°C (Ref. 7), or temperature range, 900–1000°C (Ref. 3). In Fig. 14 the bed contraction is plotted as a function of the degree of reduction for the present investigation. As can be seen, the maximum values of swelling are obtained in a wide range of reduction from 15 to 55%. In Fig. 9 it is shown that the temperature of maximum swelling also varies greatly from 800 to 1100°C. Therefore, the maximum value of swelling is believed to be more affected by the temperature profile and gas comp-
Shrinking

After the first stage without contraction the specimen bed starts to contract at an apparently constant rate (Fig. 9). The contraction of specimens treated without mechanical load applied follows the manner of that of specimens reduced under load up to a temperature of ~1100°C for the midradius and centre profiles, whereas the deviation starts at about 1200°C for the wall profile (Fig. 13).

Figure 15 shows photographs of pellets reduced without load according to the midradius profile, taken out of the furnace at different temperatures. As can be seen from Fig. 15, there is increased crack propagation with increased temperature. At higher temperatures, though, small cracks seem to be eliminated while bigger cracks expand (cf. pellets 7 with 8 and 9). The volume change of the first five pellets shown roughly follows the contraction behaviour of the corresponding experiment with load applied (cf. Fig. 13b). For pellets 8 and 9, as mentioned, some of the cracks have started to sinter together, while other cracks have expanded widely. The contraction behaviour of these pellets therefore starts to deviate from the results of contraction under load. It is natural to assume that cracks in specimens treated under load are prevented from expanding by the applied stress, and thus continue to contract owing to sintering.

From measurements of pellet particle dimensions before and after reduction it was observed that the onset of stage 2 of the contraction process is caused by shrinkage of individual particles of the specimen bed. As this behaviour has been observed during reduction without load, this implies that the start of shrinkage is independent of the applied load. At the start of the shrinkage stage the temperature is in a region where reduction reactions are fast and the metallic iron formed can start to coalesce and sinter together. This makes shrinkage possible, and the driving force for shrinkage behaviour of the particles is the energy minimisation that is caused by minimising the total surface energy of the material during elimination of the pore structure. When a 64–66% degree of reduction is reached, the contraction for specimens treated under load increases more than for specimens treated without load. The continued contraction in the case of applied load is thought to be due to a combination of the thermally increased mobility of the iron produced, resulting in sintering of the particles, and the continuously increasing mechanical load applied to the specimen bed, causing continued shrinkage of the particles. The increasing mechanical load possibly also assists sintering between the individual particles.

Carbonisation and melting

Stage 3 and part of stage 2 of the contraction process are generally referred to in the literature as softening and melting. In this investigation, stage 2 has been shown to be caused by shrinkage due to sintering. Stage 3 is believed to be caused by a carbonisation/melting process proceeding over a temperature range starting at 1420–1450°C and 55–60% contraction.

During metallographic investigations of specimens treated without load, the process of reduction was found to proceed topochemically. This has also been found in previous reduction under load tests of the investigated pellet material, MPBO, under similar reducing conditions as those applied in this investigation.8 The topochemical reduction of each iron oxide particle thus results in a pellet with a shrinking core of iron oxide surrounded by a metallic shell increasing in thickness.

During reduction the metallic iron shell formed around each particle can be carbonised with carbon either from the gas phase, in the form of carbon monoxide, or from the coke layers or the graphite crucible. This carbonisation lowers the melting point of the iron. The melting temperature or rather the temperature range, of the oxide core also depends on its composition. Therefore, to be able to discuss the mechanism of rapid contraction, the carbon content of the melted iron and the composition of the remaining slag phases after reduction were analysed. The results of the analyses of carbon contents of the metallic iron are shown in Table 3, along with roughly estimated melting points.8

The carbon contents of the melt collected are considered high. Between meltdown and leaving the specimen crucible, the liquid iron penetrates through the coke layer and then through the perforated crucible bottom. It is assumed that, owing to the restricted kinetics of solid–solid reactions, as compared with liquid–solid reactions, most of the carbonisation of the iron takes place in the liquid phase. Thus, the carbon contents at meltdown should have been much lower.

![Graph showing bed contraction as a function of degree of reduction](image)

14 Specimen bed contraction as function of degree of reduction

Unreduced

830°C

872°C

980°C

1070°C

1160°C

1256°C

1332°C

1413°C

15 Photographs of pellets reduced without load according to midradius profile, using strong gas, taken out of furnace at different temperatures
than Table 3 shows, possibly 1.2–1.7% according to the registered melting temperatures. This will be examined in
the later investigation, where reduction under load tests will be interrupted before meltdown.

The slag phases analysed (12–14Al₂O₃, 5–6CaO, 35–36MgO, and 46–47SiO₂ (wt-%)) all have high melting temperatures, roughly in the range 1600–1650 °C. They do not, however, represent the compositions of the oxide phases entrapped inside the metallic shell of the pellet before meltdown. After meltdown these oxide phases come into contact with the carbon in the crucible and coke and, thus, the iron oxides possibly constituting a large part of the slag phases are reduced. As the degree of reduction, in any case, does not reach 100% before meltdown, it can be assumed that the oxide phase in the pellet core consists of a large part of ferrous oxide FeO. The general effect of FeO in a slag phase mainly consisting of MgO and SiO₂ is gradually to lower the melting point, or the temperature interval of melting. The amount of FeO in the oxide core before meltdown can be calculated from the degree of reduction at that time. As the original contents of MgO and SiO₂ are known, the slag composition before meltdown can be estimated. The general effect of small amounts of other constituents of the slag phases, i.e. Al₂O₃, CaO, etc., is to lower the respective melting points. The actual analyses together with the calculated contents of FeO in the slag phase before meltdown of the pellet led to the conclusion that the slag phases inside the pellet core are in the liquid state before meltdown occurs. This conclusion is consistent with previous reports that the slag phase of an olivine pellet is, despite a high MgO content, molten at 1400 °C (Ref. 8). The third contraction stage with rapid contraction starts with carbonisation of the metallic shell. Consequently, rapid contraction can start either by melting of the metallic shells or by collapse of the shells, when the molten core is ejected owing to the applied stress.

The start of rapid contraction takes place at roughly the same temperature (1420–1450 °C) and degree of contraction (55–60%), as shown in Fig. 9. The applied load on the specimens is at that time largest for the wall profile and smallest for the centre profile. The difference in load hardly affects the start of rapid contraction. As shown in Fig. 14, the degree of reduction at the start of rapid contraction is in a slightly wider range (75–85%), indicating that the thickness of the metallic shell, and consequently the core size, is of less importance to when rapid contraction starts. Therefore, the rapid contraction is not caused by collapse of pellets but by melting.

Table 3 Carbon contents of metallic iron after meltdown (average values of several specimens) and corresponding melting points*

<table>
<thead>
<tr>
<th>Wall profile</th>
<th>Midradius profile</th>
<th>Centre profile</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strong red. gas</td>
<td>Weak red. gas</td>
<td>Strong red. gas</td>
</tr>
<tr>
<td>%C</td>
<td>4.09</td>
<td>3.58</td>
</tr>
<tr>
<td>Melting point, °C</td>
<td>1180</td>
<td>1240</td>
</tr>
</tbody>
</table>

Another observation that can be made from Fig. 9 is that, when rapid contraction starts, the rate of contraction is higher, at all temperature profiles, for the specimens treated with the weaker reducing gas, but the temperature of the start of contraction is the same for both strong and weak reducing gas. This could be due to the difference in degree of reduction. The process of carbonisation/melting is faster for the material with the lower degree of reduction, i.e. with the thinnest metallic shell, because of the smaller amount of metal to be carbonised and melted.

Based on the observations given above, the following mechanism of rapid contraction is proposed. At temperatures above 1400 °C, carbonisation of the metallic iron is fast. As a result of the increased carbon content of the metal, the particles in contact with the graphite crucible, or coke, eventually melt. At the start of melting only very small parts of the particles are in contact with solid carbon and the melting rate is low. The melting rate will then accelerate owing to increasing iron/carbon contact surfaces as the melted parts drip away from the interface. Possibly, the liquid metal penetrating down through the specimen bed also helps in accelerating the process by carbonising the metal not in direct contact with carbon. Finally, the outer metallic shell of the particle becomes very thin and breaks. The core of liquid slag retained inside the shell is released and can penetrate down to the coke layers where the iron oxide of the slag phase will be quickly reduced owing to the high temperature and the favourable kinetics of the liquid–solid phase reaction. This process of carbonisation, melting, and collapse will be repeated for the next layer of particles now exposed to the carbon surfaces of the coke layers. Thus, owing to the continuously increasing temperature, the process of meltdown proceeds over a temperature range instead of instantaneously at a specific temperature.

Thus, the start of rapid contraction occurs at a certain temperature, independent of the thickness of the metallic shell, but the rate of rapid contraction is dependent on the thickness of the metallic shell. With a thinner metallic shell, each particle collapses after less carbonisation and complete meltdown occurs earlier than for particles with a thicker shell.

CONCLUSIONS

Under simulated blast furnace conditions, significant carbon deposition takes place in the temperature range 600–850 °C. The reaction rate has a maximum at 750–800 °C and depends on the ratio of carbon monoxide to carbon dioxide.

With roughly the same temperature profiles as have been used in this investigation, and charged with the olivine pellet MPBO, the formation of a W shaped cohesive zone would occur in any blast furnace.

The contraction of the investigated material has been found to proceed in three different stages, referred to as swelling, shrinkage, and rapid contraction, respectively. The swelling period represents an expansion of the pellets. Shrinkage takes place at almost constant rate up to a contraction of 55–60%. Rapid contraction starts at 1420–1450 °C.

Swelling depends, apart from reduction temperature and reducing gas composition, on the temperature profile and the gas composition cycles used.

The start of shrinkage during reduction is not induced by the applied load but is a consequence of the continuing reduction and increasing sintering of metallic iron at higher temperatures.

Contraction above 1100–1200 °C is caused by an increased mobility of the reduced metallic iron in combination with the applied mechanical stress, resulting in sintering of the iron shell in individual particles as well as between the particles.

Rapid contraction starts with carbonisation of the metallic iron shells of the reduced pellets. As melted metal drips away, more iron is carbonised and melted. When the metallic outer shell becomes very thin, the particle collapses and the molten slag present in the core is released. A new pellet layer comes into contact with the carbon surface and the carbonisation/melting process is repeated. The rapid contraction therefore proceeds over a temperature range rather than at a specific temperature.
REFERENCES
SUPPLEMENT II

Jerker Sterneland and Ashok Lahiri

“New equipment for simulation of blast furnace reduction”

Presented at the 58th Ironmaking Conference, Chicago, IL, USA, March 1999.

NB: In order to get highest quality copies, reprint was made from author originals, not from the conference proceedings.
A New Equipment for Simulation of Blast Furnace Reduction

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INTRODUCTION

Iron bearing materials in the blast furnace are supposed to experience different reducing conditions depending on to which position in the furnace that they are charged. The reducing conditions will of course also change drastically as the materials descend through the furnace. From the stock line down to the hearth the temperature of solids will increase from ambient temperature up to 1500°C or more, while the encountering reducing gas will give up its heat content to the solids while ascending through the furnace from the raceway flame temperature of above 2000°C to the exhaust gas temperature of roughly 150 to 300°C. While the temperature of the gas decreases the gas composition will also change from a highly reducing gas composition at high temperature to the more moderate reducing power at lower temperatures, where CO₂ is more stable than CO.

In addition to the changing reducing conditions in the vertical direction, the prevailing reducing conditions in the blast furnace are also changing in the radial direction. Many blast furnace managers apply the strategy of central gas flow in the furnace to get a high gas velocity from the raceway, increasing the productivity and giving rise to the favourable inverted V shape of the cohesive zone of the solid materials. This is accomplished mainly by a careful charging of raw materials. A high proportion of coke charged to the centre of the furnace, consequently with an increasing ore to coke ratio against the furnace walls, will direct a large part of the blast gas to flow through the centre of the furnace, since the permeability is much higher in the coke than in the pellet layers.

Because of different temperature profiles, gas compositions and gas flows in the radial direction of the furnace, the reducing conditions are expected to be largely different at different radial positions of the furnace.

In a previous investigation[1] the contraction behaviour of the olivine iron ore pellet MPBO during reduction under load has been roughly divided into three different stages of contraction. The three stages include initial expansion, with virtually no contraction, followed by contraction at a certain rate and eventually a third stage with contraction at a higher rate. This generalised behaviour is schematically depicted in Fig. 1.

Stage one is defined as the swelling period, whereas stages two and three are called the shrinking and carbonization/melting stages respectively. This contraction/melt down phenomenon will be further outlined in the discussion section of this paper.
Fig. 1. Bed contraction of MPBO pellets during reduction under load, schematically shown.

PELLET TESTING

Improved testing techniques are continually being developed, with current emphasis on better simulation of blast furnace conditions in the low temperature lumpy upper zone and in the high temperature lower cohesive zone. However, in the present state of the art, there is no one single laboratory test which can approximate the very complex successive processes to which the burden is exposed in its descent from the distributor to the melting zone. A true simulation in laboratory scale appears to be impossible; final optimization of blast furnace smelting is still achieved only in extended commercial scale operation. Consequently, the investigator is obliged to consider a number of chemical, physical and metallurgical tests in conjunction with each other. Such combination of test results might then be correlated, usually satisfactorily, with actual performance data for a given furnace with a given burden composition.

STANDARDISED METHODS OF TESTING IRON ORE PELLETS

Standardisation has been done regarding several test methods to describe the quality of a pellet[2-5]:

- Crushing strength (also known as cold compression strength, CCS), measuring the average force required to crush a pellet particle - ISO 4700.
- Tumbler strength, measuring abrasion resistance during handling (and transport) - ISO 3271.
- Swelling, measuring swelling index (during isothermal reduction) - ISO/CD 4698.
- Low temperature breakdown (LTB), measuring LTB strength and abrasion (+6,3 mm and -0,5 mm respectively), aiming to simulate pellet behaviour in the upper shaft of the blast furnace - ISO/DIS 46997.
- Reducibility test, measuring isothermal reducibility at 900°C - ISO 7215.
- Reduction under load (RUL), measuring reducibility, resistance to gas flow and contraction, aiming to simulate pellet behaviour at higher temperatures (up to 1050°C) - ISO 7992.

Because of the complexity of the reduction, softening and melting tests, standardisation of these test methods is not likely to be considered in the nearest future[2], though.

LIMITATIONS OF REDUCTION TESTS

The majority of the reduction/softening-melting test methods that have been developed are directed to simulation of blast furnace conditions completely or to some extent. Although the softening and melting tests attempt to meticulously reproduce the time-temperature-gas composition-stress cycles experienced by the iron bearing materials in the blast furnace, they do have some limitations. Heat transfer and oxygen transfer reactions in the blast furnace are interrelated[6]. Both gases and solids are heated externally in the softening/melting tests. Consequently, heat transfer in the furnace and its possible effect on the kinetics of iron oxide
reduction cannot be accurately simulated. Further, the operating conditions found in the blast furnace are so complex that they can only be obtained during operation of a blast furnace. In the attempts to simulate those conditions some limitations therefore have to be accepted. A further drawback of the reduction/softening/melting test methods is that, as mentioned earlier, there is yet no standardised method to predict the blast furnace performance based on the test results.

Laboratory tests are often conducted at approximately atmospheric pressure, while the gas pressure inside the blast furnace can be two to four times higher. Also alkali and sulphur have been reported to influence the softening and melting behaviour\cite{7-9} of burden material. Among the reduction/softening/melting test methods found in literature, the following limitations have been found:

- Constraints in sample or crucible size.
- Constant instead of varying rate of temperature increase.
- Restrictions in maximum temperature attainable.
- Constant, or stepwise changing, instead of a continuously varying reducing gas composition.
- Only nitrogen and carbon monoxide, and no carbon dioxide or hydrogen, considered in the reducing gas.
- Too low gas flow rates.
- No, too small, or constant instead of varying, mechanical load.

## SELECTION OF TEST CONDITIONS

The selection of test conditions will be a function of the intended application of the test results, and depends on whether a qualitative simulation of the softening and melting phenomena is sufficient, or a more quantitative simulation for obtaining accurate data regarding softening and melting temperatures, degree of reduction, and permeability is required. Test precision, equipment and operating costs, and ease of operation may be other considerations.

## EXPERIMENTS

### Materials

The iron bearing material examined in this study is the olivine iron ore pellet MPBO. The MPBO pellet is commercially produced by LKAB and is the dominating blast furnace feed at the Swedish iron plants. Chemically it consists of 94,7 % Fe$_2$O$_3$ and 0,7 % FeO, which corresponds to a total Fe content of 66,8 %\cite{10}. The olivine part of the pellet consists of 1,40 % MgO and 2,0 % SiO$_2$. Besides these oxides the pellet also contains small amounts of Al$_2$O$_3$ (0,42 %), CaO (0,37 %), TiO$_2$ (0,30 %) and V$_2$O$_5$ (0,21 %), the last two significant of the Malmberget ore. The particle size is generally between 11 and 13 mm.

The coke, placed in layers above and under the pellet bed, is produced by SSAB Oxlöösund AB. Originally the coke is of "fist" size, but the coke used in experiments has been crushed to sizes of 10 to 15 mm to make up a suitable bed layer in the sample crucible.

### Equipment

Reduction under load experiments have been conducted in a newly developed reduction equipment. The features of the equipment are the following. To be able to closely simulate the reducing conditions as experienced by iron bearing materials in the blast furnace, it is during reduction possible to govern the furnace temperature, gas composition and flow rates, and the applied load over the sample. A variety of parameters are registered during the test, including bed shrinkage and weight loss. Each parameter is registered ten
times per second, and averaged minute values are produced. The size of the sample crucible (graphite, with perforated bottom and lid) is 80 mm in diameter and 125 mm in height. The sample bed size is 500 g, which corresponds to a bed height of roughly 55 mm. Above and below the pellet bed 30 mm layers of coke are placed. In addition to external preheating of the reducing gas a gas preheating chamber is placed below the sample crucible, where also a melt collector is placed. A ceramic tube surrounds the crucible and the preheating chamber, which all are placed on a balance, for continuous measurement of the weight loss during reduction. A 15 kW tube furnace consisting of six Kanthal Super elements and an isolation of ceramic fibre materials are used for heating. The furnace is placed around the ceramic tube and a loading ram above the sample crucible applies the intended load onto the sample bed. The physical outlook of the equipment is schematically shown in Fig. 2.

![Figure 2. A schematic view of the reduction under load equipment.](image)

**Experimental conditions**

Simulation of the blast furnace process was made for a blast furnace of intermediate size, with central flow of reducing gas and, consequently, an inverted V shape of the cohesive zone. The parameters that are decided to be the most important with respect to the behaviour of iron bearing materials during reduction are the following:

- rate of temperature increase,
- rate of reducing gas flow,
- change in reducing gas composition; and
- change in mechanical stress experienced by a sample.

These parameters were interpreted for three different radial positions in the blast furnace; the extremes of centre and wall, and the intermediate middle (radius ratios of 0.1, 0.9 and 0.5 respectively).

Under the conditions prevalent in the blast furnace stack, though, the reduction kinetics are expected to be independent of the prevailing gas velocity. For laboratory purposes Edström found that a relatively low critical gas velocity exists, above which a further rise has very little influence on the rate of reduction, given as a linear gas velocity of 0.12 m/s. Yamaoka et al and Yoshinaga et al have independently obtained similar values of the critical gas velocity in laboratory scale by using a gas velocity in excess of 0.1 to 0.13 m/s. Gas velocity is defined as gas flow rate per unit cross sectional area of the sample bed, and hence, the larger the bed diameter, the greater the gas flow needed to achieve the specified gas velocity. Since high gas flow rates can cause longitudinal temperature gradients and increased operating costs, it is desirable to use the minimum gas flow rate necessary to achieve the specified gas velocity. The gas flow rates used in this work, therefore, has been minimized to the critical gas velocity of 0.1 m/s.
Experimental plan

A number of experiments were performed to establish the dependence of temperature, load and reducing gas composition profiles on the behaviour of an olivine pellet during reduction. The experiments are really of the reduction/softening/melting type but will, for simplicity, be referred to as reduction under load tests. They are, thus, not to be mixed up with the standardised RUL test (ISO 7992) which ends at a temperature of 1050°C.

After complete melt down experiments were completed several experiments were interrupted at various temperatures, contraction stages and reduction degrees.

Experimental conditions

From the literature concerning various measurements in blast furnaces during operation, dissection of blast furnaces and modelling of the conditions in the blast furnace based on results of dissections etc[6,8,13,14], the parameters of rate of temperature increase, reducing gas composition and mechanical stress at different radial positions in the blast furnace can be considered as given in Table I.

<table>
<thead>
<tr>
<th>Radial position</th>
<th>Wall</th>
<th>Mid-radius</th>
<th>Centre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rate of temperature increase</td>
<td>Low</td>
<td>Medium</td>
<td>High</td>
</tr>
<tr>
<td>Reducing gas potential</td>
<td>Medium</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Mechanical load</td>
<td>High</td>
<td>Medium</td>
<td>Low</td>
</tr>
</tbody>
</table>

Table I  A rough characterisation of some conditions at different radial positions in the blast furnace.

Fig. 3. Temperature profiles used in reduction under load experiments.

Reducing gas composition - Reduction experiments were conducted by applying two different reducing gas potentials for each temperature profile. The gas composition profiles for carbon monoxide are shown in Fig. 4. The hydrogen content of the reducing gas has been coupled to the carbon monoxide content by using a hydrogen content of one tenth of the carbon monoxide content. The two reducing gases have been balanced by carbon dioxide to 40 volume per
cent, the rest being nitrogen. These proportions of the reducing gas have been arrived at after studying operating data from several blast furnaces\[^{17,18}\].

**Table II  Rate of temperature increase and time for completion of the reduction under load tests for the different temperature profiles used.**

<table>
<thead>
<tr>
<th>Temperature interval (°C)</th>
<th>Temperature profile 1</th>
<th>Temperature profile 2</th>
<th>Temperature profile 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>400 - 800</td>
<td>4</td>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>800 - 1000</td>
<td>5</td>
<td>1.2</td>
<td>5</td>
</tr>
<tr>
<td>1000 - 1600</td>
<td>5</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Time for complete test (min)</td>
<td>260</td>
<td>320</td>
<td>193</td>
</tr>
</tbody>
</table>

**Fig. 4.** Six different composition profiles of carbon monoxide corresponding to the three temperature profiles.

**Mechanical load** - In Fig. 5 the different loads applied onto the sample during reduction are shown. The magnitude as well as the increase rate of load are interpreted from literature\[^{14}\].

**Fig. 5.** Profiles of the mechanical load applied for the different temperature profiles.

**RESULTS**

**Sample bed contraction**

In Fig. 6 the sample bed contraction of the seven samples reduced under temperature profile 1 is shown together with previous results of contraction for the same material under identical reducing conditions\[^{1}\].

As can be seen in Fig. 6 the contraction behaviour of the interrupted tests closely resembles the behaviour previously obtained for reduction under identical conditions. The reduction experiments have been interrupted before and after the transitions between the different stages of contraction, i.e. from swelling to shrinking and from shrinking to carbonization/melting.
Investigation of samples after interruption

After interruption the samples were cooled down under nitrogen atmosphere. The photograph presented in Fig. 7 shows the appearance of samples taken from experiments I11-14 compared with an unreduced pellet.

The size of the samples, as shown in Fig. 7, is increased for I11, whereas the size decreases for samples I12 to I14. This is in agreement with the registered contraction behaviour. The fine cracks seen in I11 and I12 seem to have been sintered together in I13 and I14, while the larger cracks have grown. This behaviour was also found during reduction without applied load in a previous investigation[1]. Noticeable from the interrupted experiment is that even at 1360°C (I14) the shape of each individual particle is not deformed, but retains their original spherical shape, see Fig. 7. In Fig. 8 a photograph of the sintered sample bed of experiment I15 is shown.
Some particles of the sample bed shown in Fig. 8 have been mechanically deformed, but most of them show little, or no, tendency of deformation. The particles around the periphery are partially melted. The main reason for the start of melting at the periphery is the carbonization taking place in contact with the crucible wall. The increased carbon content of the metallic iron decreases its melting point leading to partial melting.

The carbon content of the iron shell has been analysed for several of the samples. In Table III the values of these analyses are given together with analyses of the carbon content of metal which had already melted before interruption, in the case of experiments interrupted at high temperature.

Table III Specification of carbon contents in samples from some of the experiments.

<table>
<thead>
<tr>
<th>Exp.no.</th>
<th>Temp. at interruption (°C)</th>
<th>Range of C content of metallic shell (%)</th>
<th>Carbon content of melted iron (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I11</td>
<td>1000</td>
<td>0.01 - 0.02</td>
<td>-</td>
</tr>
<tr>
<td>I12</td>
<td>1145</td>
<td>0.02 - 0.03</td>
<td>-</td>
</tr>
<tr>
<td>I13</td>
<td>1255</td>
<td>0.02 - 0.05</td>
<td>-</td>
</tr>
<tr>
<td>I14</td>
<td>1360</td>
<td>0.02 - 0.05</td>
<td>4.6</td>
</tr>
<tr>
<td>I15</td>
<td>1430</td>
<td>0.02 - 0.06</td>
<td>4.4</td>
</tr>
<tr>
<td>I22</td>
<td>1420</td>
<td>0.02 - 0.04</td>
<td>4.3</td>
</tr>
<tr>
<td>I32</td>
<td>1090</td>
<td>0.02 - 0.03</td>
<td>-</td>
</tr>
</tbody>
</table>

In the ranges of carbon content of the metallic shell given in Table III the higher values were obtained in shells in contact with carbon, while the lower values were found in pellets without contact with carbon. As the samples removed for analysing reasonably cannot be taken infinitely small, the compositions given by the analyses do not represent the composition of one spot but is an average value of a limited volume element (with the size of the sample taken for analysis). Therefore it can be assumed that for the partly melted iron shells where higher carbon contents were expected, a composition gradient exists within the volume analysed such that a low average value of an element can be obtained in spite that one part of the sample has a considerably higher concentration of the element in question.

Also the slag phases entrapped in the pellet cores were removed and analysed after interruption of the reduction, Table IV.

Table IV Chemistry of slag phases entrapped in the pellet cores (normalised for FeO, MgO and SiO<sub>2</sub>) and roughly estimated melting ranges.<sup>[19]</sup>

<table>
<thead>
<tr>
<th>Sample</th>
<th>% FeO</th>
<th>% MgO</th>
<th>% SiO&lt;sub&gt;2&lt;/sub&gt;</th>
<th>Melt. range (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I14</td>
<td>89,5</td>
<td>6,8</td>
<td>3,7</td>
<td>1500 - 1525</td>
</tr>
<tr>
<td>I15a</td>
<td>88,7</td>
<td>6,0</td>
<td>5,3</td>
<td>1450 - 1475</td>
</tr>
<tr>
<td>I15b</td>
<td>88,0</td>
<td>6,5</td>
<td>5,5</td>
<td>1450 - 1475</td>
</tr>
<tr>
<td>I22</td>
<td>82,9</td>
<td>11,1</td>
<td>6,0</td>
<td>1600 - 1625</td>
</tr>
<tr>
<td>I32</td>
<td>95,6</td>
<td>3,5</td>
<td>0,9</td>
<td>1450 - 1475</td>
</tr>
</tbody>
</table>

The melting ranges, given in Table IV are for the normalised content of FeO, MgO and SiO<sub>2</sub>, which were representing 94 to 98 per cent of the analysis, the rest being mainly Al<sub>2</sub>O<sub>3</sub>, CaO, TiO<sub>2</sub> and V<sub>2</sub>O<sub>5</sub>. The true melting temperatures for the slag phases are difficult to interpret, due to the complexity of the slag compositions, but it is believed that most of the slag components neglected in Table IV have a fluxing effect on the slag, lowering the melting temperatures.

Survey of microstructures

To get a clear picture of the reduction process, the structure of the unreduced pellet and the
structure at low degrees of reduction were studied. The iron bearing raw material of the MPBO pellet is magnetite ore concentrate. During production the pellet is indurated under oxidising atmosphere. The pellet is then oxidised topochemically from the surface towards the core. The microstructure of the unreduced MPBO pellet is therefore mainly consisting of hematite, Fig. 9, occasionally with some rest-magnetite left in the core due to incomplete oxidising, Fig. 10.

Figs. 9 and 10. Surface layer and centre of unreduced pellet respectively. Magnification 100 times.

The magnetite stage during reduction corresponds to roughly 11 per cent degree of reduction. Figs. 11 and 12 show a pellet reduced to 9 per cent reduction degree (590°C). Most of the pellet is reduced to magnetite, except for inside larger grains and in the centre of the pellet where some hematite remain unreduced.

Figs. 11 and 12. Surface layer and centre of a pellet reduced to 9 per cent degree of reduction (590°C). Magnification 100 times.

The wüstite stage of reduction represents a degree of reduction of 33 per cent. Figs. 13 and 14 show a pellet reduced to 29 per cent reduction degree (850°C). This sample was etched in 4 per cent HCl in alcohol, to differentiate between magnetite and wüstite. Most of the pellet is reduced to wüstite, except for in the centre of the pellet where some magnetite remain.

The continuation of the reduction follows in Figs. 15 and 16 (1000°C), where sample I11 reduced to 44 per cent is shown. The surface layer consists mainly of wüstite, but some iron reduced from the wüstite can be seen inside the wüstite grains. The structure is an inheritance of the original structure of the hematite pellet, i.e. irregular. Some of the magnesia in the olivine grains have started to diffuse to the surrounding wüstite resulting in a border of magnesioferrite. The magnesia content of
the olivine is replaced by wüstite. Except for some larger pores the porosity is evenly spread throughout the pellet. The interior of the pellet, as shown in Fig. 16, has a very similar microstructure except that it does not contain any iron and there is a large crack.

Figs. 13 and 14. 29 per cent degree of reduction (850°C). Magnification 100 times – etched in 4% HCl.

In sample I12, Fig. 17 (1145°C), at a reduction degree of 59 per cent, the reduction is observed to proceed in a topochemical mode, thus producing a border of iron around the periphery of the pellet. The edges of the iron has started to round off, which indicates that sintering has started. Also the wüstite phase is more rounded and the pore structure is coarser. In the interior of the pellet, Fig. 18, the sintering has not proceeded to the same extent and the pore structure, except for the elongated pores, or cracks, throughout the sample, is more widely spread.

Figs. 15 and 16. Surface layer and centre at 44 per cent degree of reduction, 1000°C. Magnification 100 times.
Figs. 17 and 18. Surface layer and centre at 59 per cent degree of reduction, 1145°C. Magnification 100 times.

Figs. 19 and 20. 1255°C. Magnification 100 times.

The reduction degree for sample I13, Figs. 19 and 20, (1255°C) is somewhat higher, 70 per cent, and the iron structure has sintered into a skeleton around the pellet. In the interior, Fig. 20, the wüstite phase has grown considerably in size. The pore structure is coarser both in the interior and in the shell.

At 1360°C the sintering has continued even further, Figs. 21 and 22. Three different microstructures can be seen in sample I14 - the core of wüstite is surrounded by a transition layer of both iron and wüstite and, outside that, a layer of sintered iron. The pore structure is very coarse in the interior, whereas the porosity of the sintered surface layer has decreased due to pore diffusion and the surface is so dense that the access of the reducing gases to the interior of the particle is probably substantially decreased.
Figs. 23 and 24 show sample I15 (1430°C). Due to sintering of the iron a completely dense metallic shell has formed around the particle. In the interior the iron is very coarse and so are the oxide phases, which now form a rim inside the iron shell resulting in a large cavity in the pellet core.

Further samples of interrupted reductions verified the observations made, but did not provide any new information.

**DISCUSSION**

Sample bed contraction results of interrupted reduction under load experiments, as shown in Fig. 7, show that the reproducibility of the equipment and experimental technique used is good. This is also verified by the values of degree of reduction obtained, which are close to previous results[^1].

**Mode of reduction**

From metallographical studies it was confirmed that the reduction process proceeds topochemically, from the pellet surface to the centre, and for higher oxides each grain is reduced topochemically, Fig. 11.

The absence of higher oxides in the pellets reduced more than 40 per cent, Figs. 15-16, and metallic iron for the pellets reduced less than 30 per cent, Figs. 13-14, clearly indicates that the reduction of hematite via magnetite and wüstite to iron proceeds stepwise. The reason to the stepwise behaviour of the reduction is believed to be the following. The driving force for the reduction of the less stable phases is the highest. When the gaseous product of reduction reactions of higher oxides in the pellet core diffuses out through the particle, the local partial pressure of carbon dioxide in the outer layers of the particle gets too high for further reduction of the outer layers.

In the initial stage the reduction of wüstite was observed to proceed in an internal mode inside the wüstite grains, instead of starting at the surface, Fig. 15. The same phenomenon has been reported in other investigations[^11]. This phenomenon is believed to be a result of nucleation taking place at sites in the grains where for example a crystal defect makes nucleation more favourable than at the outer surface of the grain.

The main role of olivine in the olivine pellet is to improve the high temperature properties of the pellet. Magnesia is known to raise the melting point of wüstite[^19] and forms a solid solution with wüstite during reduction of iron ore pellets[^20]. From each olivine grain magnesia diffuses to the surrounding
wüstite phases, whereas the magnesia in the olivine is replaced by wüstite. This initially forms a border of magnesioferrite around the olivine, which can be seen in Fig. 16. When the magnesioferrite gets reduced the magnesia is "released" and diffuses very fast to the surrounding wüstite phase forming a completely homogeneous solid solution[21]. Without the magnesia, the silica content of the pellet would have formed low melting fayalite slags with the wüstite, lowering the melting point from that of the pure wüstite, 1371°C, down to 1177°C[19] at the lowest. The homogenous slag phase with magnesia has a higher melting point, but has been found to be in the liquid state at 1430°C, Fig. 24.

Start of contraction

In a previous investigation it was shown that the contraction of the MPBO pellet during reduction under load proceeds over three separate stages, namely swelling, shrinking and rapid contraction[1]. The first stage, swelling, is a widely investigated phenomenon, which is believed to be caused by the growth of fibrous iron, also known as whiskers. The start of shrinking was found to be independent of the applied load, and was started even without load applied. Therefore it was concluded that the shrinking was a result of sintering of the metallic shell, decreasing the porosity and thus also the volume of the sample.

In the metallographical survey of this investigation sintering of the iron phase was observed to start, Fig. 17, at approximately the same temperature, between 1000 and 1145°C, as the previously observed start of contraction. The sintering thereafter continues, resulting in a denser product. During sintering the pores not only get coarser but is also eliminated from the material, Figs. 19-24. The pores of the oxide phase trapped by the dense metallic shell diffuse towards the core of the pellet, forming a cavity, while the pores in the metallic shell diffuse out of the pellet and, thus, the particle shrinks. The driving force for the shrinking is the minimisation of surface energy. In experiments without applied load[1] it was found that cracks in the particle surface develops when temperature is increased, resulting in very large cracks at temperatures over 1100-1200°C. The cracks found at lower temperatures in this investigation was observed to sinter as temperature was increased. The effect of an applied load on the sample during reduction is therefore believed to be to force the pore diffusion to the pellet surface instead of to the cracks.

Thus, after the metallographical review, it is considered established that the start of contraction origins from the shrinking of the individual particles caused by sintering of the metallic iron shell.

Shrinking due to sintering

After initial contraction due to sintering of the iron shells of the individual particles the sintering will also take place between the particles. This was observed under the examination of the samples from the interrupted experiments. The samples of the first interrupted experiments were easy to take apart because the sintering had only started at the contact points between the individual samples. As sintering continues between the particles, gradually the connection between them becomes more extended and, thus, stronger. Shrinking due to sintering of iron shells and between particles continues until the next stage of contraction, carbonization/melt down, starts.

Carbonization and melt down

The last stage of contraction, at a high rate, is normally referred to as softening and melting. In this investigation it has been observed that no actual softening occurs, but the particles continues to sinter until melt down starts. This is an important observation for the blast furnace operation. The softening behaviour normally described in literature
results in a sharp decrease in permeability of the particle bed. During sintering of particles in this investigation the voidance of the bed decreases but there is still channels for the gas to pass through the sample bed, and thus some permeability remain. Even at the interruptions after melting and rapid contraction had started very few particles were deformed, compare Fig. 9. The permeability of the sample bed is therefore not considerably affected by deformation, but a decrease in permeability is induced by the sintering between the particle decreasing the voidance of the sample bed. At the interruption at 1485°C (87 per cent bed contraction) the individual particles were still possible to discern, and were only slightly deformed, but the sintering between individual particles had decreased the voidance strongly - and thereby impaired the permeability of the sample bed. Therefore it can be concluded that considerable softening does not occur during carbonization/melt down, and the permeability is therefore not strongly decreased until the sintering between particles eliminates the voidance of the sample bed. Thus the permeability of the iron bearing layers of the cohesive zone in the blast furnace might not be so seriously impaired as is implied by the softening process theory.

The carbonization/melt down stage is initiated by carbonization and partial melting at interfaces between iron and carbon, e.g. at the crucible wall or at the coke layers above and below the sample. The contact surfaces, though, are expected to be small, in the extreme case more of a contact point than a surface, since the original geometry of the particles is spherical. As the contact points of the particles of the sample bed partially start to melt and the metal flows away, larger contact surfaces between iron and carbon arise. The extent of carbonization and subsequent melting of carbonized iron is then increased. As more iron melts and penetrates out of the sample bed "fresh" iron is exposed to the carbon interfaces and the process of carbonization and melting is repeated, and thus, in combination with the continuously increasing temperature, the melting process will proceed over a temperature range rather than at a fixed temperature.

The carbon contents found in the metallic shell of the pellets examined after interrupted reduction, as were given in Table III, are so small that the decrease of the melting point of the iron is very small. However, the values found for the iron that had melted before the experiments were terminated are in the vicinity of the eutectic, at 4,3% C in the iron-carbon phase diagram, which represents a melting point of 1147°C[22]. From this, two important conclusions can be drawn. First, at 1430°C a carbon content of 1,5 per cent is required to melt the iron. Thus carbonization from 1,5 per cent up to 4,3 per cent takes place in the liquid phase as the liquid iron penetrates through the coke bed. This implies that carbonization is fast in the liquid state. Secondly, as the carbon content of the partly melted metal on the outside of the sintered sample bed was found to be very low, diffusion of carbon in the solid metal is considered slow, i.e. slower than the melting and separation of a portion of already carbonized iron. This is in agreement with investigations of the blast furnace process, where also a large part of the carbonization has been observed to take place in the liquid state, while the liquid iron penetrates down through the coke layers of the lower part of the blast furnace and in the metal pool[6,13].

The final stage of contraction was found to start at roughly the same temperature, 1420°C to 1450°C, for all reducing conditions of the previous investigation[1]. As the carbon contents of the metallic shells examined after reduction are so low, due to comparatively slow diffusion of carbon in the solid state, there is not expected to be any significant difference between the particles treated under various reducing conditions. This was confirmed by analyses of metallic shells of samples treated under different reducing conditions, Table III. Therefore all samples starts to melt at roughly the same temperature. Neither the difference in achieved degree of reduction prior to start of melt
down was found to affect the starting temperature of melt down, which also can be explained by the conclusion that the melting start temperature is only depending on the properties of the iron shell and the kinetics of carbonization.

The proposed process of carbonization and melt down during rapid contraction is schematically shown in Fig. 25.

![Diagram](image)

Fig. 25. Schematic picture of the continuous process of carbonization and melt down.

When carbonization of the metal shells starts (a), the particles in contact with the graphite crucible, or coke, will eventually melt (b). The melting rate will accelerate due to increasing contact surfaces iron/carbon (c) as the melted parts drips away from the interface. Possibly also the liquid metal penetrating down through the sample bed helps in accelerating the process by carbonizing the metal not in direct contact with carbon. Finally the outer metallic shell of the particle becomes very thin and breaks (d). The core of liquid slag retained inside the shell is released and penetrates down through the sample bed (e). In the coke layer below the sample the iron oxide of the slag phase will be quickly reduced due to the high temperature and the favourable kinetics of the liquid/solid phase reaction. This process of carbonization, melting and collapsing will repeat for the next layer of particles now exposed to the carbon surfaces of the coke layers or crucible wall (f).

As could be seen from Table III small amounts of melt were obtained even at 1360°C. This was found in contact with the graphite crucible wall, but not at the coke interfaces, which possibly is a result of the graphite being more reactive than the coke.

Rapid contraction occurs at a certain temperature, independent of the thickness of the metallic shell, but the rate of rapid contraction is depending of the thickness of the metallic shell. With a thinner metalic shell each particle collapses after less carbonization and complete melt down occurs earlier than for particles with thicker shell.

As shown by Figs. 21 and 23 the sintering of iron makes the shell very dense. This prevents the reducing gas from reaching the oxides in the core. Therefore the reduction gets retarded at high temperatures and complete metallisation is not attained before melt down. This phenomenon was observed in the previous investigation[1] where a tendency to a decreasing rate of reduction at high temperatures could be noticed. During the later stages of the process of carbonization and melt down an increase in the rate of reduction was again observed, which is here taken as a verification of the fast reduction of the liquid oxide phase taking place after melting of the iron shell of the pellet.

**CONCLUSIONS**

The reduction process of the olivine MPBO pellet proceeds topochemically, from the pellet surface to the centre, and for higher oxides each grain is reduced topochemically. The reduction takes place in stages, in spite of a reducing gas
composition with a high enough reducing power to reduce lower oxides. An internal mode of reduction was observed in the initial stage of reduction of wüstite.

During reduction, the olivine phase of the MPBO forms a solid solution with wüstite resulting in a homogeneous oxide phase with a melting point above the melting point of fayalite, or even pure wüstite, thus improving the high temperature properties of the pellet.

Sintering of metallic iron shells, with accompanying pore diffusion, resulting in shrinking of the individual particles is the cause of contraction start. Continued sintering of both the metallic iron shells and between individual particles results in shrinking of the individual particles and contraction of the sample bed.

Carbonization and partial melting of iron in contact with coke initiates rapid contraction. Due to melting of the outer iron shell, the pellet eventually collapses and the slag entrapped in the core is released and penetrates down through the sample bed. Then new iron surfaces are exposed to the carbon interface and carbonization again takes place. This continues layer by layer and results in rapid contraction.

During this investigation the phenomenon normally referred to as softening was not found to occur. Except for the sintering between particles the particles retain their original spherical shape until melting starts, and even after that. For the blast furnace process this observation is very important, since it implies that the permeability of the iron bearing layers of the cohesive zone in the blast furnace might not be so impaired as is implied by the softening process theory.

REFERENCES


10. “LKAB 1996 - The products”


SUPPLEMENT III

Jerker Sterneland and Mats Hallin

“The use of an experimental blast furnace for raw material evaluation and process simulation”

Presented at the 6th Japan-Nordic Countries Joint Symposium, Nagoya, Japan, November 2000.

NB: In order to get highest quality copies, reprint was made from author originals, not from the conference proceedings.
1. Introduction

The need for a step in between laboratory scale metallurgical testing and full-scale tests of new blast furnace pellets has been more and more evident during the last 10-15 years. An experimental blast furnace is a way to reduce the risks before full-scale tests are performed. The possibilities for operating an experimental blast furnace in a broad range of process concepts can also allow for faster progress in pellet development and for testing concepts too risky to test directly in a full-scale blast furnace.

In 1994 a feasibility study was initiated to evaluate the possibilities to build and design an experimental blast furnace. This was done by Mefos (the metallurgical research foundation in Luleå, Sweden) and LKAB in co-operation and discussions were made with experts from all over the world. In October of 1996 the LKAB board of directors decided to build the furnace and also to run five campaigns in 1997-1999. Hot commissioning started in 1997 and in 1997-2000 six test campaigns were carried out.

The experimental blast furnace in Luleå is a strategic investment that enables faster, assured product development. The blast furnace is a cornerstone of LKAB's research and development efforts. By using an experimental blast furnace in the process of product development, LKAB expects to strengthen its position as a leading supplier of blast furnace pellets, in terms of product quality and performance. The goal is to design and produce pellets that create added value in the customer's process, compared to other burden materials.

The present blast furnace pellet of LKAB was first introduced in 1982. It is produced from magnetite ores, mined in Kiruna or Malmberget. The concentrate has a very low gangue content. Bentonite is added as a binder and olivine to adjust the composition before pellets are formed and fired. During almost twenty years, outstanding operational results have been achieved in Nordic blast furnaces, using 100% olivine pellets.

2. The experimental blast furnace

The experimental blast furnace was fully financed by LKAB, who also owns the furnace. It is situated at the Mefos premises, next to the Luleå plant of SSAB. During operation SSAB supplies a lot of the raw materials, including gases, and carries out analyses on hot metal and slag, etc. This has had a great influence on the costs of building and operating the experimental blast furnace. LKAB and Mefos are operating the blast furnace in co-operation.

2.1. Description of the furnace

The experimental blast furnace is shown in Figure 1. It has a working volume of 8.2 m³ and a hearth diameter of 1.2 m. From tuyere level to stock line the height is 6 m, and there are three tuyeres placed with 120 degrees separation. The tuyeres have a diameter of 54 mm, resulting in a blast velocity of 150 m/s at normal...
blast volume. The furnace is equipped with systems for injecting pulverised coal, oil and other injection materials.

![LKAB's experimental Blast Furnace in Luleå](image)

**Figure 1. Layout of the experimental blast furnace**

Great effort has been taken to keep heat losses to a minimum and therefore insulating refractories were chosen. Only the bosh and tuyeres are water-cooled. The blast is normally preheated to 1170-1250°C in pebble bed heaters. There are two pebble bed heaters, working in cycles.

The raw materials system consists of four bins for pellets or sinter, one bin for coke and two small bins for slag formers. Each material is weighed separately according to the actual recipe. The material is transported to the furnace top by a skip and to a receiving hopper. Below the receiving hopper, there is a pressure equalising lock hopper. Furnace top pressure can be controlled up to 150 kPa overpressure.

The experimental blast furnace is equipped with a bell top. There is a moveable armour for burden distribution control. Two mechanical stock rods are used to monitor the burden descent and to control the charging into the furnace. The top gas is transported through the uptakes and down-comer to a dust catcher. The gas is further cleaned in a venturi scrubber and a wet electrostatic precipitator. Finally, the top gas is flared in a torch.

The furnace has one tap hole. It is opened with a pneumatic drill. After each tap, the tap hole is closed with a hydraulic mud gun. The hot metal and the slag are tapped into a ladle, transported to the SSAB steel plant, and charged as scrap to the BOF.

Burden probes are installed at three different levels. There are two horizontal probes, one at tuyere level and one in the shaft, and one inclined probe to sample in the bosh. The shaft probe and the inclined probe are equipped with two different heads. One is used to collect material samples from the furnace, while the other is used to collect furnace gas for analysis; and to measure the temperature. The tuyere probe is equipped with a gas collecting and temperature-measuring head.

To make dissection and repair easy the hearth is detachable and can be separated from the furnace in one to two hours.

### 2.2. Raw Material preparation and dust treatment

The raw materials for the experimental blast furnace are normally produced before each campaign. The test pellets are produced either in the Steel Belt Plant located in Malmberget, or in one of the grate kiln plants located in the Kiruna area. Pellets are made using normal pellet feed concentrate. The balling and induration is done at standard plant practise, which means that the test pellets have normal size distribution.
Test pellets are transported to the experimental blast furnace and stored in a stock house. During the campaigns the pellets are screened at 6 mm and sampled before they are stored in day bins.

Sinter is transported from the actual steel plant to the stock house at the experimental furnace. It is normally standard sinter as produced at different steel plants. The sinter is sampled and tested in the same way as for the pellets.

Coke is produced at SSAB coke plant in Luleå, close to the blast furnace. Before each campaign production coke is screened in the fraction 15-30 mm. The coke is transported to the experimental blast furnace for storage in the stock house. During campaigns, coke is transported to the experimental blast furnace with a front loader, screened at 6 mm and sampled before storing in the day bin for coke.

fluxes are screened to the fraction 10-20 mm and stored in big-bags. The big-bags are transported to the day bins, no additional screening of fines is done.

Dry flue dust is collected in a storage container, sampled twice a day and analysed for the chemical composition. The wet flue dust (sludge) is collected in a thickener and sampled when the thickener is emptied by a suction truck.

2.3. Operating the experimental blast furnace

The operation of the blast furnace is very similar to a commercial blast furnace. At blow-in a small amount of charcoal in front of the tuyeres is used as start up burden. Within the first hour, full wind is reached and the blast temperature is in the range of 800-850°C. The blast temperature is increased to the desired set point in the first 24 hours of operation. The amount of reducing agents is slowly decreased during the first 72 hours, to a level corresponding to about 110%, compared to normal operation. After this period injection of coal or oil is started. Operation is stabilised during the next 48 to 72 hours.

The furnace is operated at a productivity ranging from 3.4 to 4.0 t/m³day. Hot metal and slag composition is kept at set points decided before the campaign. Normal tap-to-tap time is 60-80 minutes, depending on actual production rate; and the normal tap duration is five to fifteen minutes. Drill diameter varies between 25 and 28 mm, depending on tapping conditions in previous tap. The hot metal temperature is measured with a temperature probe. Hot metal and slag are sampled and analysed after every tap.

The test periods normally starts with a transition period where the test material is charged to the furnace for a period of 24-36 hours, corresponding to six to nine throughputs. After that period the actual test starts. The test time depends on what objectives that are to be met for the test. A typical test period range from two days up to six days. During this time operational data is logged and monitored closely. Process data are logged every second and stored in a database, as ten-second and minute averages. These data are transferred at regular intervals to another database where process data calculations are carried out. Data in this database is used for reports, trend charts and mass- and heat balance calculations. Chemical analyse for raw materials, hot metal and slag are also stored in this database.

Reference material is charged to the furnace at regular intervals to check if there has been any change in the furnace or auxiliary equipment that influence the operation.

Sampling of burden materials, using the burden probes, is done once or twice every shift. The material retrieved by the probe is divided into samples representing different positions on the radius of the furnace. All material taken out of the furnace is screened and the fraction >6.3 mm is sorted, so that pellets, coke and slag formers are separated. Breakdown and strength of materials are tested and samples are prepared for microscopy investigations. Chemical analysis is also done, primarily to determine the overall reduction degree.

The efforts to keep heat losses to a minimum have proven sufficient. Because of this the consumption of reducing agents are similar to many full-scale blast furnaces. The chemical and thermal treatment the pellets undergo in the experimental blast furnace can therefor be said to simulate a full-scale blast furnace. As to now the experience is that the experimental blast furnace is a very sensitive tool for detecting differences in properties for different pellets. The response time is much shorter for the experimental furnace compared to a commercial furnace. One example is the time for the blast furnace to go from normal heat levels to cold conditions, it could take less than six hours. On the other hand, the blast furnace also recovers quickly to normal conditions, after corrective actions are made.
2.4. Campaigns 1 - 6

During the campaigns performed so far, many different process concepts have been tested in the experimental blast furnace. Examples are:

- 100 % pellets as ferrous burden with injection of PCI in the range of 0 – 150 kg/tHM.
- Simultaneous injection of PCI slag formers and oil injection up to 65 kg/tHM. The slag rate has varied between 85 kg/tHM and 190 kg/tHM.
- Sinter/pellets as ferrous burden with pellet ratio from 20 % to 60 %. Oil injection up to 65 kg/tHM. Slag rates between 170 kg/tHM to 220 kg/tHM.

These various operation modes have given different results not only in consumption figures but also in process characteristics. For example, it is interesting to see the difference between 100 % pellet operation and operation with high amounts of sinter. The big difference is in the rate of change in operating condition, meaning that when operating with high amounts of sinter a slower response in the operational parameters can be seen, when changes are made. This is probably due to harder burden structures (less moveability) in the case of sinter burdens.

3. Test procedures and results from iron ore pellet evaluations

The blast furnace process is, as well known, complex. Although the fundamentals are the same for all operations, even two different blast furnaces at the same plant behave differently. This means that the evaluation of actual operation data with the goal to rank different raw materials and their associated properties is difficult. In general, there is an agreement of the paramount importance of raw materials quality to blast furnace operation results. Some estimates have been made that 80% of the overall result is dependent of the raw materials used. This is true, not only for the quality of the ferrous burden components, but also for coke, fluxes and injection materials such as pulverised coal.

The issue about ranking different pellets, e.g. what are the characteristics of a good pellet, have been under discussion during many years. When it comes to judge and discriminate between different pellets tested in the experimental blast furnace, the following criteria are used:

1. The overall permeability and its variation during operation, measured as the Burden Resistance Index (BRI). This index is calculated based on the pressure drop from tuyere level to top and the bosh gas volume ((blast pressure^2-top pressure^2)/(bosh gas volume)^1.7 * k1).
2. The gas utilisation and its stability, measured as ηCO ((%CO_2) / (%CO + %CO_2)), calculated from top gas analysis.
3. The burden descent rate, and its stability, calculated from stock rod information.

4. Results from dissections

After each experimental blast furnace campaign the blast furnace has been quenched and dissected. When furnace operation is shut down, nitrogen is flushed from the furnace top down through the blast furnace burden. This action prevents a heat wave moving upwards through the furnace, and causes the blast furnace reduction to “freeze”, i.e. stopping all further chemical reactions. After two to three weeks of cooling down the burden by nitrogen the furnace top is removed and dissection can commence. Dissections are carried out by carefully removing the burden material layer by layer, starting from the top of the blast furnace. As long as possible, the original pellet and coke layers are followed. The work is concentrated on the pellet layers.

The appearance and nature of each burden layer uncovered is documented in the form of photographs and video shots. After that material samples are taken from several points of the burden surface, for chemical analysis, physical testing and microscopic characterisation. Figure 2 shows an example of the development in reduction degree in samples taken out at one specific point in each ferrous burden layer down through the furnace, while Figure 3 shows the microscopic structures observed at the same sample point.
Figure 2. Development in reduction degree in each ferrous burden layer down through the furnace.

Figure 3. Example of microscopic structures at the same sample point as in Figure 2 above.
Figure 2 and 3 above are from dissection after Campaign 1, where the blast furnace was charged with an olivine iron ore pellet, MPBO, table 1. In Figures 4 and 5 below, a comparison of differences in reduction disintegration between two pellet types and at different locations in the furnace is made. Figures 4.A and B are from Campaign 1 while Figures 5.A and B are from Campaign 4, where the furnace burden was a fluxed pellet, FP20, table 1. The A samples are taken out close to the furnace wall, while the B samples are from the furnace centre. The per cent of reduction disintegration is calculated from screening results after mechanical tumbling of the samples. The reduction disintegration is divided into two groups, <0.5 mm and 0.5-6.3 mm.

Table 1. Compositions of pellets in the dissections after Campaign 1 and 4 respectively.

<table>
<thead>
<tr>
<th>Pellet</th>
<th>Type</th>
<th>Fe (%)</th>
<th>SiO₂ (%)</th>
<th>CaO (%)</th>
<th>MgO (%)</th>
<th>Dissection</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPBO</td>
<td>Olivine</td>
<td>66.8</td>
<td>2.0</td>
<td>0.4</td>
<td>1.5</td>
<td>Campaign 1</td>
</tr>
<tr>
<td>FP20</td>
<td>Fluxed</td>
<td>66.9</td>
<td>1.6</td>
<td>1.6</td>
<td>0.3</td>
<td>Campaign 4</td>
</tr>
</tbody>
</table>

From Figures 4 and 5 it can be seen that the reduction disintegration into 0.5-6.3 mm is in the beginning larger for the fluxed pellet, while further down the blast furnace shaft the olivine pellet has the higher values. A critical point is observed, especially for the fluxed pellet, where the reduction disintegration has a maximum and then decreases further down in the furnace. The dust formation, <0.5 mm, on the other hand, has a tendency of increasing down through the furnace.
Before quenching the blast furnace several basket samples are introduced into the burden layers. The individual baskets contain about 600 grams of raw material, and provide the opportunity to study the behaviour of several different burden materials after each dissection. The material from each basket sample is also examined chemically, physically and by microscope; and is compared to the “bulk” material next to the individual baskets.

To be able to evaluate and present the vast amount of data that is produced during the dissections a visualisation model has been developed, displaying data in a three dimensional mode.

5. Results from simulation of a commercial blast furnace

Some burden mixtures tested in LKAB's experimental blast furnace have been tested also in commercial blast furnaces. In table 2 an example of a burden mixture tested at the steel plant Stahlwerke Bremen in Germany is shown. The no 2 blast furnace in Bremen has a diameter of 12 m, and is charged with a mixture of sinter and pellets (together with a small addition of lump ore).

Table 2. Comparison of some results from experimental and commercial size furnaces.

<table>
<thead>
<tr>
<th></th>
<th>Experimental blast furnace</th>
<th>Bremen no 2</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Burden A</td>
<td>Burden B</td>
</tr>
<tr>
<td>ηCO (%)</td>
<td>45.7</td>
<td>47.2</td>
</tr>
<tr>
<td>Std dev, ηCO</td>
<td>2.9</td>
<td>0.7</td>
</tr>
<tr>
<td>Permeability resistance, BRI</td>
<td>6.6</td>
<td>6.7</td>
</tr>
<tr>
<td>Std dev, BRI</td>
<td>0.25</td>
<td>0.17</td>
</tr>
</tbody>
</table>

Burden A is a mixture of 54% sinter, 23% fluxed pellet and 23% olivine pellet, while in burden B the olivine pellet is changed to an acid pellet. This example shows that even a small change of the blast furnace burden (23% in this case) alters the behaviour of the furnace operation. The results from the experimental blast furnace show how the burden will behave in a commercial blast furnace.

6. Conclusions

The experimental blast furnace is a unique test facility for blast furnace burden materials. It gives big opportunities for product development and process research. Many of the risks involved in full-scale production trials can be avoided. The furnace was primarily built for the purpose of product development, but is also used in other blast furnace related research projects.

As burden material can be sampled from the furnace during operation or after quenching, investigation of reduction mechanisms for various materials and process conditions can be done. This will aid future design of blast furnace pellets.

Several dissections of the experimental blast furnace have provided many interesting research results. The change in properties during reduction in a blast furnace can be studied in detail. An example is the reduction disintegration, which has been found to vary between different pellet types as well as along the radius and height of the furnace.

Results from the experimental blast furnace have been verified by full-scale trials in the Bremen no 2 furnace. This shows that results from the experimental blast furnace can predict how a burden material, or burden mixture, will behave in a commercial blast furnace. Possible effects of inferior raw material quality are more pronounced in the experimental furnace compared to when the same raw material is tested in a full-scale blast furnace.

Used to its full potential, the experimental blast furnace will serve as a valuable tool for blast furnace development and to improve process efficiency. The experimental blast furnace strengthens Luleå's position as an internationally significant centre of metallurgical development.
SUPPLEMENT IV

Jerker Sterneland, Margareta Andersson and Pär Jönsson

“Comparison of iron ore reduction in an experimental blast furnace and a laboratory scale simulation of the blast furnace process”

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COMPARISON OF IRON ORE REDUCTION IN AN EXPERIMENTAL BLAST FURNACE AND A LABORATORY SCALE SIMULATION OF THE BLAST FURNACE PROCESS

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ABSTRACT

The reduction of iron ore pellets in an experimental blast furnace has been surveyed by a dissection of the blast furnace. Results of the dissection, as well as some results of measurements of the blast furnace interior during operation, have been compared to previously conducted laboratory reduction, softening and melting tests with the same type of pellets. The aim of the work was to compare laboratory testing with results from an experimental blast furnace. The high temperature phenomena occurring when reducing the olivine iron ore pellet MPBO, with limited softening and a short temperature range of the melting process, resulting in a thin cohesive zone, were found to be the same in laboratory tests and in the experimental blast furnace. The reduction down through the burden of the experimental blast furnace was similar, but not identical to the results of the reduction, softening and melting experiments. The differences were found to be due to different reducing conditions. Therefore, it was concluded that a simulation of the reduction occurring in the blast furnace can be performed in laboratory scale, provided that the experimental conditions are correctly chosen. Information of the internal state of the blast furnace can be obtained from the experimental blast furnace.
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1 INTRODUCTION

In order to improve the performance of the blast furnace it is of interest to increase the knowledge of how iron oxide is reduced in the furnace for different types of iron-bearing materials. In the blast furnaces in Sweden only pellets are used as iron-bearing materials. If the knowledge of how different types of pellets perform with respect to the reduction of iron oxide can be developed, it would be possible to optimise the properties of pellets to give the optimum performance for the blast furnace.

There is not much recent information reported on reduction degree of iron oxides within commercial blast furnaces. The reason is of course that this is difficult to measure during operation. Since the blast furnace quenching period of the nineteen seventies and early eighties, when many blast furnaces were quenched and dissected (mostly in Japan, but also in USA, former Soviet and Europe)\textsuperscript{[1]}, the opportunity to examine the internal state of a commercial size blast furnace has been limited. The information from blast furnace dissections in the past can be difficult to interpret, since quenching in most cases were performed with water. Water quenching may alter the oxidation state of the blast furnace burden, for instance by the reaction $\text{Fe} + \text{H}_2\text{O} \rightleftharpoons \text{FeO} + \text{H}_2$ and also a temperature increase in the shaft has been observed during water quenching.\textsuperscript{[1]} In addition, the blast furnace process has developed considerably since the nineteen seventies, and the dissection information is therefore not up-to-date.

Information of the reduction behaviour of iron oxide can also be obtained by using an experimental pilot plant blast furnace. The size of an experimental blast furnace should be large enough to enable to simulate the work as done in a commercial size blast furnace, and at the same time small enough to be economical to operate for research purposes. The comparatively small size of the furnace provides opportunities for quenching and dissecting the furnace, so that up-dated information on the reduction degree of iron oxide in different positions can be obtained. However, since experiments in an experimental blast furnace still can be quite costly it would also be useful to carry out trials in a small laboratory furnace to determine what trials that are worthwhile to perform in an experimental blast furnace.

In the literature, a large number of researchers have studied the reduction behaviour of iron oxide in laboratory experiments.\textsuperscript{[2-10]} In a previous work at KTH (the Royal Institute of Technology, Stockholm, Sweden) a reduction, softening and melting, also called reduction-under-load, equipment was used to characterise the behaviour of olivine iron ore pellets during reduction.\textsuperscript{[11,12]} Since then, trials have been conducted the LKAB experimental blast furnace using the same type of material as burden feed, the olivine pellet MPBO. Part of the results from these trials will be presented in this work.

Earlier publications from the LKAB experimental blast furnace have dealt with the design, instrumentation and operation of the furnace, results from trials with different iron bearing materials
(especially different pellet types, but also sinter and lump ores), and comparisons with commercial size blast furnaces.\textsuperscript{[13-16]} Some results from dissecting the experimental blast furnace have also been published.\textsuperscript{[17]} The aim of the present work is to compare results from the experimental blast furnace with results from laboratory testing, with the focus on reduction behaviour. To the authors knowledge a similar attempt to compare data on reduction behaviour of iron ore obtained from an experimental blast furnace with data from laboratory trials has not been presented in the published literature. To be able to make the comparison, the operation of the experimental blast furnace and some of the measurements made have to be explained. Therefore, the first part of the paper will describe how the experiments were carried out in the experimental blast furnace, and also the procedure of the laboratory scale tests. Thereafter, the results of the comparison of the data from the experimental and laboratory furnaces will be presented and discussed. Results that will be treated are the temperature and gas profiles obtained, the progress of reduction and phase distribution during reduction, and finally qualitative information on softening and melting is given.
2 EXPERIMENTAL BLAST FURNACE EXPERIMENTS

2.1 GENERAL

When LKAB decided to build the experimental blast furnace in 1997, one of the goals was to provide the best tool for blast furnace research. All typical equipment required for a blast furnace has therefore been installed in the experimental blast furnace, and the monitoring and measuring means are even much more extensive. The general specifications and operating parameters of the experimental blast furnace are shown in Table 1.

Compared to a commercial blast furnace, the experimental blast furnace has less compression of the burden, shorter gas and iron ore residence time, higher heat losses (per tonne of hot metal), lower hot metal temperature and higher hot metal silicon content.[16] Despite these differences, the experimental blast furnace proceeds a complete iron making process as done in the commercial blast furnace, all the functions proceeded including reduction, softening, melting, desulphurising, alkali re-circulation and so on, are the same. In addition, some abnormal operations such as scaffolding, hanging, channelling and other typical disturbances may also occur in the experimental blast furnace, depending on operation conditions and burden quality. The main difference is perhaps higher fuel rate for the smaller furnace due to its high heat losses, thus giving a higher rate of reducing gas to iron ore. In order to get a fuel rate close to that reached in a commercial scale furnace, the experimental blast furnace has used higher blast temperature and higher intensity of the operation than that used in a normal commercial scale furnace. To further decrease the heat losses, ceramic refractory insulation of the furnace has been used where possible, and only the tuyeres and the lower bosh region immediately above the tuyeres are water-cooled. Thereby the heat loss is minimised and consequently the reductant rate is kept relatively low, 510 to 540 kg/tHM, which is comparable to production blast furnaces.

The experimental blast furnace is regularly operated in campaigns of four to ten weeks, twice a year. So far there have been nine campaigns conducted in the furnace, in total over 430 days of operation. Over 40 different pellet types (commercial as well as trial pellets), several sinter grades and lump ores have been tested – as constituting 100 % of the iron bearing burden or in burden mixtures (i.e. different pellet mixtures, pellet/sinter mixtures, pellet/sinter/lump ore mixtures etc).

Results from the experimental blast furnace presented in this paper are all from trials using the standard grade olivine pellet MPBO. This pellet type is regularly used as a reference burden, since its behaviour is well recognised after over twenty years of operation in Swedish blast furnaces. The composition of the pellet MPBO is shown in Table 2. Experimental conditions for the different trials are given in the results section.
2.2 MEASUREMENTS AND SAMPLING DURING FURNACE OPERATION

In the experimental blast furnace horizontal probes have been installed at three different levels of the blast furnace shaft, as shown in Fig. 1, to take out material samples and to measure temperature and gas composition. The vertical positions of these three probes, with respect to the burden surface of stockline, are given in Table 3. When using the horizontal gas- and temperature probes, measurements can be made at several positions of the furnace diameter. In general, measurements are performed at five different radial positions; close to the walls, in the centre and on both sides of the centre (between centre and the two opposite walls).

By using a vertical temperature probe, i.e. letting a thermocouple descend with the burden, an improved picture of the vertical temperature profile within the furnace can be obtained. When inserting the vertical thermocouple into the burden of the experimental blast furnace, the geometry of the furnace top is such that the thermocouple follows the burden close to the wall, thus giving the wall profile of the temperature in the burden column.

2.3 EXCAVATION AND MATERIAL SAMPLING

Shut-down and quenching of the experimental blast furnace is performed by flushing the burden column with nitrogen gas. After reducing the blast flow to zero, cold nitrogen introduced at the furnace top immediately flows down the burden and out through the tuyeres, evacuating the reducing gases and preventing heat from the lower part of the furnace to move upwards. The purpose is to stop all further reactions and chemically “freeze” the blast furnace in the state it was in prior to shut-down. The nitrogen flow during quenching is 300-400 Nm$^3$/h (i.e. 40-50 Nm$^3$/h×m$^3$ of working volume) - high enough to exchange the total gas volume of the burden in less than a minute. After the immediate “freezing” of the furnace, nitrogen is kept flowing through the furnace for a period of two weeks to further cool the burden (and the furnace refractories).

The subsequent dissection of the furnace is carried out by first removing the furnace top, and then carefully removing the burden material layer by layer, from stockline down to the hearth. As far down as possible, the original pellet and coke layers of the burden are removed separately. However, further down the furnace it is often difficult to distinguish between different layers because of more and more mixing of the layers. This is an inevitable consequence of the burden descending movement in combination with an increasing diameter of the furnace shaft and penetrating of pellets into the coke layers. When mixed burden layers are reached, excavation proceeds by removing the burden in uniform layers (a certain distance of mixed material) instead of the previously distinct material defined layers. Each burden layer uncovered is examined, and the appearance and nature of every separate layer are documented by photographs and video shots, in addition to written documentation.

Samples are taken from several radial positions of each burden layer, as shown in Fig. 2. At these positions samples are collected from each pellet layer down through the furnace. Cross sections of the reduced pellet samples are studied in optical microscope and the iron oxides and iron structures
are identified. To distinguish between magnetite and wüstite, samples are etched in 5% HF in an H₂O solution for 20 seconds, giving the wüstite a darker (grey) appearance in the microscope (magnetite and wüstite are difficult to separate in microscope because they both constitute a cubic lattice. However, during etching the wüstite is more readily dissolved, giving it a darker appearance in optic microscope). After manually defining zone boundaries for the different structures, automatic image analysis can be used to calculate the extent of each of the structures.
3 LABORATORY EXPERIMENTS

3.1 REDUCTION-UNDER-LOAD TESTS

In a previous work a Reduction-Under-Load (RUL) equipment was developed at KTH. Several experiments were performed using the olivine pellet MPBO. Some of the results from that work are used in the present investigation for comparison with results from the experimental blast furnace. For a description of the reduction equipment and detailed results, the reader is therefore referred to previous papers.[11,12] In this section only a summary of test conditions will be given. Results of the RUL experiments will be presented in the results section. The experiments performed were really of the reduction/softening/melting type (non-isothermal) but is, for simplicity, referred to as reduction-under-load tests. The test method is, thus, not to be confused with the standardised RUL test (ISO 7992, isothermal at 1050 °C).[18]

Reducing conditions, with special attention to the parameters of rate of temperature increase, reducing gas composition and mechanical stress, at different radial positions in a blast furnace can be roughly comprehended as given in Table 4. Based on literature data, six reducing programs were established for three different temperature and load profiles, with two different gas composition profiles for each temperature profile.[11] In the results section, averages of the two trials at each radial position will be used for comparison with data from the experimental blast furnace. In Table 5, the rates of temperature increase and time for completion of the laboratory experiments are shown. The gas composition was continuously changed as the temperature increased. Table 6 shows the carbon monoxide contents of the reducing gas at various temperatures during reduction. Hydrogen content of the reducing gas was coupled to the carbon monoxide content by using a hydrogen content of one tenth of the carbon monoxide content. These two reducing gases were then balanced by carbon dioxide up to 40 % by volume and the rest of the reducing atmosphere being nitrogen. The load applied to the sample, also shown in Table 6, was different for each of the temperature profiles, and continuously increased during testing to simulate the increasing load of the overlaying burden during the descent in the blast furnace.

Samples of 500 g of oxide material, corresponding to a bed height of roughly 55 mm, were reduced under load in an 80 mm diameter graphite crucible. Below and above the pellet bed 30 mm layers of coke breeze were placed. The weight loss during reduction was continuously measured and in- and outgoing reducing gas compositions were regularly recorded. From this information the reduction degree could be calculated. The following definition of degree of reduction, R, was used:

\[
R = \frac{m_{O,\text{removed}}}{m_{O,\text{original}}} \times 100
\]

(1)

Calculation of the degree of reduction from the registered weight loss during reduction is then a straightforward exercise. When calculating the degree of reduction from gas analysis the oxygen
removal, $m_{O,\text{removed}}$ in equation (1), was arrived at by integrating the oxygen removal rate over time. In the ingoing gas, the analysis of carbon mono- and dioxide, hydrogen and nitrogen was controlled, as well as the total gas flow. In the outgoing gas carbon mono- and dioxide, and hydrogen contents were measured. The $H_2O$ amount in the outgoing gas and the total amount of outgoing gas were calculated by a simultaneous hydrogen and nitrogen balance. The oxygen removal rate was then calculated by an oxygen balance of ingoing oxygen in carbon mono- and dioxide subtracted from outgoing oxygen in water vapour, carbon mono- and carbon dioxide. All calculations above were corrected for carbon deposition (at low temperatures, i.e. below 750 to 800 °C) and carbon gasification (at higher temperatures), which had been registered in reduction programs without an oxide sample. [11]
4 RESULTS
As mentioned earlier, the olivine pellet MPBO is used as a reference material in the experimental blast furnace. Therefore operational data with this pellet type in the furnace are available from all experimental blast furnace campaigns. Table 7 shows the origin of the data used in this investigation, whereas Table 8 specifies the operating parameters in the respective campaigns. The main differences between the operating conditions of the different campaigns were the coal injection (and oxygen enrichment) rates used. While Campaign A was run on all coke operation, Campaigns B and C were run with a moderate coal injection rate and finally Campaigns D and E with comparatively high injection rates. Another operational difference was that during Campaign A the blast furnace was wall-working, while in later campaigns the operation has been centre-working.

4.1 EXPERIMENTAL BLAST FURNACE MEASUREMENTS

Temperature and Gas Distribution in the Blast Furnace Shaft
Results from gas composition and temperature measurements using the horizontal probes in Campaigns D and E are given in Figs. 3 to 5, which show the contents of CO and CO\textsubscript{2} in the gas and the horizontal temperature distribution across the furnace shaft at the three probe levels. As can be seen in these figures, the blast furnace at the time of the measurements was operated slightly centre-working, i.e. the measured temperature was higher in the centre than at the walls, indicating a higher gas flow in the centre. It can be noted that the CO content of the blast furnace gas decreased dramatically while the gas was ascending through the burden column, from 42-45 % via 34-35 % and 28-32 % at the depths of 3.4 m, 2.4 m and 1.0 m respectively, down to approximately 24-26 % at the gas off-takes. Similarly there was an increase in the gas CO\textsubscript{2} content up through the furnace, from approximately 0-2 % via 13-14 % and 17-20 % at the depths of 3.4 m, 2.4 m and 1.0 m respectively, to 23-25 % at the gas off-takes. The gas temperature decreased at the same positions, from 860-980 °C via 850-900 °C and 690-820 °C down to 140-240 °C when leaving the furnace.

The vertical probe, i.e. in-burden thermocouple, was used to measure the wall profile of the temperature in the burden. The reproducibility of this measurement can be observed in Fig. 6, which shows the results of vertical temperature probings on different days during experimental blast furnace Campaign E. For the major part of the blast furnace shaft, from a depth of 1.5 m to 4 m (800 °C to 1100 °C), the difference in the temperature measured at different occasions was less than 50 °C.

Fig. 7 gives a comparison of the in-burden wall temperature profile between different campaigns. The burden composition was almost the same, but different operating parameters have been used in different campaigns, resulting in different in-burden temperature profiles. The differences in temperature between Campaigns B and C and between Campaigns D and E were small, in the range of 50 °C for most of the furnace shaft. However, the difference between Campaigns B and C and
Campaigns D and E was more pronounced, 100-150 °C in a large part of the shaft, which can be explained by the different operating conditions, as previously described (see also Table 8).

4.2 EXPERIMENTAL BLAST FURNACE EXCAVATION

All data presented in this section are from the excavation of the experimental blast furnace after Campaign A. Operating parameters before quenching of the furnace were shown in Table 8. During excavation of the experimental blast furnace samples were collected and afterwards investigated using optical microscopy and image analysis. The following iron oxide/iron structure types were identified:[17]

- Coarse hematite
- Medium coarse hematite
- Fine hematite
- Hematite lamellae
- Old (original) magnetite
- Large grain magnetite
- Mixed size magnetite
- Magnetite matrix
- Wüstite
- Snowflake metallic iron
- Sintered metallic iron
- Melted metallic iron

The previously given definition of the degree of reduction, $R$, can be modified to include the analysed contents of oxygen in the samples (weight per cent):

$$R = \frac{m_{O,removed}}{m_{O,original}} \times 100 = \frac{\%O_{initial} - \%O_{analysed}}{\%O_{initial}} \times 100 \quad (2)$$

The reduction degree of the different iron oxide/iron phases can then be determined as:

- Hematite, Fe$_2$O$_3$  \hspace{1cm} R$_{\text{Hematite}} = 0 \%$
- Magnetite, Fe$_3$O$_4$  \hspace{1cm} R$_{\text{Magnetite}} = 11.1 \%$
- Wüstite, Fe$_x$O  \hspace{1cm} R$_{\text{Wüstite}} = 29.6 \%$
- Iron, Fe  \hspace{1cm} R$_{\text{Iron}} = 100 \%$

For the non-stoichiometrical wüstite phase, Fe$_x$O, the value of x at the lowest temperature of stable existence, 570 °C, has been used, 0.947 (below this temperature FeO decomposes to Fe and Fe$_3$O$_4$, but FeO can easily be undercooled, whereby the decomposition is prevented).[19,20] The difference
in reduction degree calculated with the two different forms, FeO and Fe$_{0.947}$O, is 3.7 percentage units for samples with 100 % wüstite.

The area fractions of each phase determined by image analysis were recalculated to volume fractions, which were used to calculate the reduction degree of each sample from the blast furnace excavation. Equation (3) gives the reduction degree of sample $i$, $R_i$:

$$R_i (\%) = v_{\text{Magnetite}} \times R_{\text{Magnetite}} + v_{\text{Wüstite}} \times R_{\text{Wüstite}} + v_{\text{Iron}} \times R_{\text{Iron}}$$  (3)

where $v_j$ is the volume fraction of iron oxide/iron phase $j$.

The accuracy of this method was verified by comparing calculated reduction degree values with results from the standard procedure of identifying reduction degree, i.e. from chemical analysis, for samples collected at the same position. The comparison can be seen in Fig. 8, which shows the degree of reduction in different layers of the furnace and different radial positions. The values presented in Fig. 8 show that the deviation between the different methods of calculating the degree of reduction is generally less than 8 percentage units.

Depending on how the cross sections of the individual pellets are chosen in the microscopic analysis, there will be some internal variation in phase composition. From experience it is known that most variations within a pellet particle are found in the radial direction, between the surface and the interior of the particle. This is because of the reduction proceeding topochemically from the pellet surface to the centre.$^{[12]}$ Effort is, therefore, put on cutting and polishing the pellet at the largest possible cross section, ensuring that the pellet centre is included in the microscopic examination. By mounting seven pellet particles together and using the average of the calculated reduction degree, the natural fluctuation in attained reduction degree between different pellet particles from the same sample point is reduced. Variations within a collected sample are to be further investigated in a separate paper.

The furnace cross section was divided into three circular segments with equal surface area, as could be observed by the broken lines in Fig. 2. Thereby the sample points were classified into radial positions, representing wall, mid-radius and centre samples respectively. The average of samples 0 and 6 was, therefore, denoted as the wall sample. Similarly, the average of the samples 2, 3 and 4 gave the centre sample and average of samples 1, 5, 7, 8 was defined as the mid-radius sample. From each of the ten sample points in all 24 iron ore layers, seven pellet particles were characterised and the average reduction degree was calculated for each sample point.

Fig. 9 shows the reduction degree as a function of depth in the blast furnace, measured from stockline and downwards, for the three radial positions wall, mid-radius and centre. As can be seen in the figure, the samples from the wall position gained a higher reduction degree compared to the centre and mid-radius samples from a depth of approximately 1.5 m (or from the wüstite stage)
down to 3.5 m depth, where all samples were close to complete reduction. The reason for this is that the blast furnace at that time was wall-working, which influences the reduction behaviour in the lower shaft of the furnace. The wall-working blast furnace process will be discussed more later.

It can also be seen from Fig. 9 that the reduction was rapid in the beginning and up to a reduction degree of approximately 25%. Here, most of the original hematite had turned into wüstite, leaving only small amounts of magnetite in the core of the pellet particles. After that there was only little reduction for a long time until the reduction rate again increased, reducing the wüstite to iron at a rate similar to, or even higher than, the initial hematite and magnetite reduction.

By using vertical probing in the furnace it was possible, in addition to measure the gas temperature, as was shown in Fig. 7, to also measure the burden descent rate at different heights of the burden column. The vertical probe consisted of a thermocouple to which a weight with a barb-like construction was attached. When assessing the probe data it is of course important know the furnace profile, to be able calculate the vertical distance in the furnace (the probe path is assumed to be somewhat inclined, i.e. to follow the furnace wall, an assumption considered reasonable from observations of material movements during dissections of blast furnaces[1]). This procedure assumes that the descent of the burden was not disturbed. By applying the descent rate measured in Campaigns B to E to data from Campaign A (since this measurement was not available at that time), the data in Fig. 9 can be transformed into Fig. 10. This figure shows the reduction degree as a function of elapsed time since charging into the blast furnace. In this figure the fast initial reduction can again be seen; the first 25 % reduction took less than 40 minutes. The pellets then travelled down through the reserve zone, with only little reduction, for almost up to one hour (shorter for the samples close to the wall). Then the reduction rate increased again, going from about a 30 % reduction degree up to a 90 % reduction degree during the next 30 minutes. Finally, the reduction rate was lowered during the period where complete reduction was obtained.

In Figs. 11 to 13 the extent of the different iron oxides/iron phases down through the furnace is shown for the different radial positions of the experimental blast furnace. It can be seen that the hematite was quickly reduced in the upper part of the blast furnace. However, the reduction of magnetite to wüstite was found to start before complete reduction of hematite to magnetite. Similarly iron started to form long before all magnetite was reduced to wüstite. Thus, in the major part of the blast furnace shaft three different iron oxide/iron phases co-existed.

4.3 COMPARISON BETWEEN LABORATORY TESTS AND THE EXPERIMENTAL BLAST FURNACE

Due to the relatively small volume of the experimental blast furnace, the throughput time is normally about 4 to 5 hours, which is shorter than in large-scale production blast furnaces where a throughput time of 6 to 8 hours is often given. The throughput time of iron bearing materials in a blast furnace is mainly a function of the furnace size, the productivity (defined as hot metal produced per time increment) and the volume of the burden per tonne of hot metal charged from the
top. For a specific blast furnace the throughput time is therefore given by the blast volume, amounts of injected reducing agents, oxygen enrichment, top pressure, raw material properties, etc. For most commercial blast furnaces the strategy applied is to maximise the productivity, giving the best overall economy of the iron production (lowest cost per tonne of hot metal produced). In practice, this means blowing as much blast as possible (restrained only by the capacity of the blower or the permeability of the blast furnace burden). In the case of the experimental blast furnace, the strategy is somewhat different, since maximum production and lowering the hot metal cost is not a primary goal, and therefore not demanded. In fact, the productivity can be chosen in quite a broad range, from 25 to 41 tonnes/day (3.0 to 5.0 tonnes/m³/24h or 22.1 to 36.2 tonnes/m²/24h) depending on the goals of the trials. However, there is a practical lower limit of the productivity, given by the amount of blast which still can create a raceway penetration of a suitable size and far enough into the furnace burden to promote a centre-working blast furnace process. Because of the limitation in minimum blast volume, the throughput time of the experimental blast furnace can not be increased much beyond 5 hours. Therefore the throughput time of iron-bearing materials in the experimental blast furnace, and thus the iron oxide reduction rate, is one of the parameters that deviates from the operation in large scale production blast furnaces (as was mentioned earlier).

Parameters for laboratory reduction-under-load experiments, also called RUL-tests, are chosen to simulate the blast furnace process in a specific blast furnace. In the previous work, RUL experiments were performed simulating a commercial blast furnace, thus choosing a reducing program which had longer reduction time compared to the experimental blast furnace. In Fig. 14, where reduction temperature versus time is compared for the experimental blast furnace and the RUL trials, the differences in temperature/time cycles can be seen.

Because of different reduction times, a comparison of the reduction behaviour between the experimental blast furnace and the laboratory RUL experiments based on a time scale is not suitable, but is better performed comparing the reduction degree as a function of a dimensionless distance, representing the vertical position in the blast furnace. Figs. 15 to 17 show comparisons between the reduction degree as a function of the dimensionless distance originated by distance from stockline in the experimental blast furnace or originated by reduction time in RUL experiments, for the wall, mid-radius and centre positions respectively. The dimensionless distance, D, was thus based on the burden movement in the blast furnace and calculated by equation (4):

\[
D = \frac{\text{vertical distance from stockline}}{\text{stockline - dripping zone distance}}
\]  

(4)

For the RUL laboratory experiments equation (4) was modified and the dimensionless distance calculated as a time fraction of the experiment, according to equation (5):

\[
D = \frac{\text{elapsed time of experiment}}{\text{total time of experiment}}
\]  

(5)
The distances for the RUL results have thus been calculated as a fraction of reduction time in the mid-radius experiment. However, as the RUL experiments are separately performed the dimensionless distance could also be individually calculated, on the time basis of each of the experiments. The difference between these procedures is schematically explained in Fig. 18, i.e. reduction results are compared for a certain vertical level of the furnace (left), or for a certain temperature isotherm of the furnace (right). In Figs. 19 and 20, the alternative procedure has been used. The dimensionless distance is here based on the time scale of the individual experiments, and shown for the wall and centre profiles (the mid-radius profile is the same as Fig. 16, since this experiment constituted the time basis in the first calculation).

As can be seen in Figs. 15 and 19, the shape of the reduction curve for the wall position down through the burden of the experimental blast furnace is similar to the results of reduction-under-load laboratory tests. The main difference is that the reduction was somewhat faster in the experimental blast furnace due to the wall-working process. The deviation is at most of the time in the range of maximum 10 percentage units. There is, however, also a principal difference in the shape of the reduction curves. In the reduction values from the experimental blast furnace, a reduction plateau can be noticed at roughly 25 % reduction degree, a result of the restricted reduction in the reserve zone, and above 40 % degree of reduction the reduction rate is much higher than for the laboratory results.

Also for the mid-radius position, Fig. 16, the plateau-like shape of the reduction curve from the experimental blast furnace can be observed, at 25 to 30 % reduction degree, as can be the higher reduction rate at high degrees of reduction. Apart from these deviations, the reduction pattern is similar between the experimental blast furnace and the laboratory results, most of the time with less than 15 percentage units in difference.

The biggest difference between the experimental blast furnace and the RUL experimental results was for the centre radial position, Figs. 17 and 20. In the experimental blast furnace the reduction values at almost all levels are lower than in the laboratory test. This is an effect of the laboratory tests set-up to simulate a centre-working blast furnace, while the experimental blast furnace was wall-working. Therefore, the reduction in the centre was very fast in the first case, while in the later it was considerably slower, and the difference is up to 30 percentage units comparing temperature isotherms, Fig. 20, and even higher when comparing for the vertical level in the furnace, Fig. 17. Also in this case there was restricted reduction in the reserve zone of the experimental blast furnace, while it was not in the laboratory reduction test.
5 DISCUSSION

5.1 EXPERIMENTAL BLAST FURNACE MEASUREMENTS

Horizontal gas and temperature measurements

Horizontal gas composition and temperature profiles measured in the experimental blast furnace (Figs. 3 to 5) corresponded well to gas profiles in commercial blast furnaces reported in literature.\cite{1,21} To some extent the horizontal variations in the blast furnace gas composition separates the experimental blast furnace from commercial blast furnaces. This is explained by the comparatively narrow shaft of the experimental blast furnace, limiting the possible extent of horizontal variations in gas composition. The same explanation can be used for temperature measurements, where horizontal temperature variations were found in the experimental blast furnace, but to a lesser extent compared to commercial scale blast furnaces. Using pellets, with a small response angle, also contributes to giving a flat temperature and gas distribution if the charging distribution is not sufficiently controlled.

In the first trial in the experimental blast furnace (Campaign A), the tuyeres had a diameter that, afterwards, was found to be too big. This resulted in a too low gas velocity through the tuyeres, and the impact of the blast was therefore too small to make a deep raceway penetration into the furnace. Cold model trials had shown that by different speed and lifting levels of the bell-ring (construction was simplified by inserting a movable ring and fixing the bell instead of lowering the bell from a fixed ring) the burden distribution could be controlled. After the first campaign, however, it was found that this strategy did not work sufficiently. Later, therefore, a movable armour was installed for burden distribution purposes, and has worked well since then. However, during experimental blast furnace Campaign A the burden distribution was not sufficiently controlled. This fact, in combination with the short penetration of the blast into the furnace, made the blast furnace become wall-working instead of the preferred centre-working process. Fig. 21, showing the average temperatures above the burden surface for the last 48 hours prior to furnace shut-down of Campaign A, compared to more “normal” conditions, in this case 48 hours of operation during Campaign D, verifies that the blast furnace was wall-working before shut-down in Campaign A.

Vertical temperature measurements

In general, the shape of the temperature profile in most cases followed the pattern given in literature, i.e. fast heating immediately after charging followed by a slower heating rate through the thermal reserve zone, and finally fast heating again in the lower part of the blast furnace.\cite{1,21} However, the vertical temperature profile within a blast furnace can differ a lot depending on operating practise, and of course also depending on what burden materials that are used. Naturally, the results of temperature measurements are also very much depending on the radial position of the measurement. In the experimental blast furnace, vertical measurements of the temperature profile were made close to the furnace wall. The results in Fig. 6 shows that if the vertical temperature is measured in the same positions at different times but for the same operating conditions, the
maximum temperature difference is 50 °C, or less than 5 %, in the temperature interval 800 to 1100 °C. However, as was seen in Fig. 7, the vertical temperature profiles in the experimental blast furnace had different appearances in different campaigns. The reason for the different levels of temperature in the shaft can be found in the different operation of the furnace.

Comparing temperature measurements by horizontal and vertical probes, Figs. 3-5 and 7 (curves 3 and 4), it can be noted that at the level of the upper shaft probe the two measurements are in close agreement. At the middle and lower shaft probes, though, there is a deviation between horizontal and vertical probe temperature measurements. The temperatures measured by the horizontal probes are lower than for the corresponding values from the vertical probe measurement, by 80 °C and 140 °C for the middle and lower shaft probes respectively. The following errors have been identified as possible reasons for the difference:

i. The measurements were, for practical reasons, conducted with a time delay of a few hours.

ii. The angular position of the vertical probe measurement was 90 degrees displaced compared to the position of the entrance of the shaft probes into the shaft, i.e. 90 degrees from as well the inner and the outer end of the horizontal shaft probe measurement across the furnace radius.

iii. Measurement error.

The influence of the time delay, i., is considered as minor, as the measurements were performed during stable blast furnace operation. For error no ii., there is of course some temperature differences at different angular positions of the furnace shaft, but these are believed to be much less than the observed temperature deviation. The remaining, and most likely, possible reason for the difference in measured temperatures in the lower shaft is a measurement error, iii. The probable cause for this is the water-cooling of the horizontal shaft probes influencing the temperature measurements, especially at higher temperatures, i.e. above 800 °C. Contrary, the vertical probe temperature measurement, which is not influenced by any cooling system, is believed to supply more reliable data than the horizontal probes. Consequently the actual temperature in the lower shaft should then be higher than measured by the middle and lower horizontal shaft probes. However, if the cooling effect from the probe cooling is caused by heat conduction from the measuring point to the probe, the error in temperature measurement should become proportional to the temperature difference between the measuring point and the probe. If so, it could be argued that even though the level of temperature measurements at the middle and lower shaft probes is too low, the observed temperature distribution across the furnace diameter should be reliable.

5.2 EXPERIMENTAL BLAST FURNACE EXCAVATION

From studying the above-burden temperature profile shown in Fig. 21, it could be concluded that there was a large portion of peripheral gas flow prior to shut down of the experimental blast furnace in Campaign A. This implies that the working condition of the furnace was a wall-working process. During excavation of the furnace, heavy lining wear in the lower part confirmed that the furnace
really had been wall-working. The reason for this was discussed previously, and the result was that the reduction of the ferrous oxides was faster closer to the wall than in the centre of the furnace, which could be seen in Figs. 9 and 10. Due to a higher gas flow, and potentially also faster heating of the burden, the material close to the wall should start to melt earlier than the central portion. Indications of this was also seen during the excavation of the blast furnace, where the cohesive zone was found to be almost flat, but somewhat inclining upwards to the wall. From the lower part of the shaft (from pellet layer no. 15 of totally 24 layers of iron ore, from stockline to the hearth) the pellet particles close to the wall had started to sinter to large aggregates. Further down the furnace the sintering was even more pronounced, but the original spherical shape of the individual pellet particles was not changed. The melting of the metallised pellets was found to be rapid, occurring only in two consecutive layers of the burden, i.e. only two layers contained semi-melted material, or what is normally referred to as cohesive zone formation.

5.3 COMPARISON BETWEEN LABORATORY TESTS AND THE EXPERIMENTAL BLAST FURNACE

The main purpose of this paper is to study how well laboratory data compare with data from an experimental blast furnace. Below the findings from the study will be discussed.

Temperature and Gas Composition

The heating rate of the centre position of the reduction-under-load tests resembles the vertical temperature profiles at the wall of the experimental blast furnace. This was an effect of the comparatively fast reduction process in the experimental blast furnace as opposed to the conditions used in the reduction-under-load experiments. Also the temperature and gas composition profiles chosen for the RUL experiments resemble the horizontal temperature and gas composition profiles measured in the experimental blast furnace.

Reduction Degree

As shown in Fig. 9, an initial fast reduction in the experimental blast furnace can be observed, followed by a very low reduction rate, and finally again a high reduction rate (disregarding minor reduction probably occurring in the liquid phase). The same observation is often given in literature describing the reduction mechanisms in the blast furnace.\[1,21\] It is stated that the initial reduction of hematite and magnetite to wüstite in the blast furnace shaft is fast due to a reducing gas composition far above the equilibrium of those reduction reactions. In combination with a comparatively small amount of oxygen to remove (compared to the wüstite reduction), i.e. the lower part of the blast furnace produces excessive amounts of gas for the primary reduction steps, the initial reduction becomes fast. For the next reduction step, from wüstite to iron, a gas with a lower oxygen potential is required (compare the equilibrium phase diagram of Fe-O-C).\[20,21\] Therefore, considerable reduction of FeO does not take place until the wüstite enters the middle or lower part of the blast furnace shaft, where the reducing gas is strong enough for further reduction. Consequently, the region including very little reduction is referred to as the chemical reserve zone. The extent of the chemical reserve zone is depending on the geometry of the blast furnace, i.e. the height of the
internal burden column for a specific blast furnace. From the dissection after Campaign A, the chemical reserve zone could be defined as reaching from roughly 1 m to 2 m below the stockline (compare Fig. 9).

Similarly, there is also a thermal reserve zone, where there is only little heat transfer from the ascending blast furnace gas to the descending burden. The thermal reserve zone often includes the chemical reserve zone, but also a part of the region of initial reduction above the chemical reserve zone and the region of indirect reduction, i.e. the gaseous reduction of wüstite starting below the chemical reserve zone and stretching down to the end of the thermal reserve zone. At the end of the thermal reserve zone the temperature of the ascending gas is higher, and consequently the descending material is heated further. In the high temperature region in the lower part of the blast furnace, above 1000 °C, the highly endothermic Boudouard reaction prevails, leading to direct reduction of FeO. The thermal reserve zone of the experimental blast furnace could be studied in Fig. 7, where it typically stretched from 1 to 4 m below the stockline for the wall position of Campaigns B to E.

For the wall and the mid-radius positions, the deviation in reduction between laboratory experiments and the experimental blast furnace was not considerably large. However, the shape of the reduction curves was different. In the experimental blast furnace, the reduction was restricted in the middle part of the furnace, corresponding to the chemical reserve zone as described above. Though a thermal reserve zone was applied in the mid-radius reduction-under-load experiments, a chemical reserve zone behaviour was not observed, as shown in Fig. 16. This was due to the reducing gas potential used in the experiments, which was somewhat too high to restrict the reduction in the thermal reserve zone. The effect of the limited gas flow in the centre of the experimental blast furnace, restricting the reduction rate, in combination with the experimental set-up of RUL-experiments with extremely strong reducing gas and a high heating rate, was a strong deviation in reduction after the wüstite reduction stage for the centre sample.

The differences described above are due to differences in reducing conditions. The gas compositions were somewhat different, when comparing the levels of the different gas components between the laboratory tests and the results of the experimental blast furnace gas measurements (Campaigns D and E). In the laboratory tests the contents of CO in the gas used were lower than that found in the experimental blast furnace. However, the CO₂ contents were also lower, resulting in a higher CO/CO₂ ratio. This is demonstrated in Fig. 22, where the gas ratios (CO/[CO+CO₂] × 100) in the laboratory tests and experimental blast furnace have been inserted into the Fe-C-O equilibrium diagram. It can be seen that the gas ratios below 900 °C were all higher for the laboratory tests, compared to the conditions of experimental blast furnace Campaign D and E.

The lower gas ratios below 900 °C in the experimental blast furnace is caused by the equilibrium between iron and wüstite, governing the gas ratio at low temperatures. At higher temperatures the CO gas is much more stable compared to CO₂, and almost all CO₂ produced is converted to CO,
resulting in a gas ratio of close to 100. The effect of the lower gas ratios at low temperatures was found in the restricted reduction in the middle part of the experimental blast furnace. Similarly, the fast increase in gas ratio at 900 to 1000 °C, for the experimental blast furnace conditions compared to the laboratory tests, was reflected in a faster reduction in the lower part of the furnace.

The differences in reduction time for the laboratory tests compared to the experimental blast furnace is partly compensated for when comparing degree of reduction as a function of distance in the furnace, as shown in Figs 15 to 17 and 19 to 20. The similar gas ratios thereby results in similar reduction for the wall and mid-radius positions, when comparing by the distance in the furnace, except for the absence of a chemical reserve zone in the laboratory tests due to the comparatively strong reducing gas compositions used. For the centre position, on the other hand, the conditions were quite opposite between laboratory and the experimental blast furnace, resulting in large differences in observed reduction degree.

However, from the studies of the microstructures of iron oxides during reduction another difference has been found between the experimental blast furnace and the laboratory test results. In the previous investigation of reduction-under-load samples, the reduction was found to take place in stages, i.e. reduction to a lower oxygen containing iron oxide phase did not occur until the previous reduction step was completed. Therefore, at no time more than two iron oxide (or iron) phases co-existed. In Figs. 11 to 13 it was shown that this was not the case for materials collected in the experimental blast furnace. There was a clear over-lapping in the reduction process resulting in three co-existing phases (i.e. hematite/magnetite/wüstite or magnetite/wüstite/iron) for longer periods. This was probably due to the faster reduction in the experimental blast furnace. However, the stepwise reduction observed in RUL experiments was found to occur in spite of a reducing gas potential high enough for a further reduction to a lower iron oxide phase. This phenomenon was explained by an assumed locally high carbon dioxide potential. Because the driving force for reduction is highest for the less stable phases (i.e. the highest oxides), these were reduced first. When the gaseous product of the reduction reactions of higher oxides in the pellet core (i.e. carbon dioxide) diffused out through the particle, there was a potential for a local partial pressure of carbon dioxide high enough to temporarily prevent further reduction of the outer layers of the particle. In the experimental blast furnace, on the other hand, this “reduction preventing” layer was probably not so significant due to the higher heating rate and the excessive amounts of strongly reducing gas, which forced the reduction of the outer pellet layers to continue.

**Softening**

In the reduction-under-load experiments it was found that the pellet particles studied retained their original spherical shape until melting started, and even after that. During heating the metallised iron in the particles of the pellet bed was found to pick up carbon in contact with coke, and thereby eventually melt because of the lowered melting temperature of the iron phase. There was an increasing, and especially at very high temperatures extensive, sintering between the particles, lowering the void fraction of the pellet bed. There was also sintering within particles, causing a
shrinking of the individual particles and thereby a contraction of the bed. What is normally referred
to in literature as softening, though, was not found to occur.[12] As was described above, even
though the reduction was found to be considerably faster in the experimental blast furnace, the
mechanism of contraction of the pellet bed due to sintering and the melt down process, without
considerable deformation, was found to be the same. This is due to the MgO (originating from the
olivine addition) forming a solid solution with wüstite during reduction, which increases the melting
point far above the melting point of wüstite, or fayalite (2FeO·SiO$_2$). This circumstance contributes
to the advantageous high temperature properties of the olivine pellet.[12] The absence of significant
softening during reduction/melt down is believed to be a special feature of this type of olivine
pellet, since in later experimental blast furnace excavations other burden materials have been found
to behave differently during reduction and melt down. In many cases, using other pellet types, there
has been extensive softening of pellets in the high temperature region of the furnace, resulting in
several (in extreme cases up to 7-8) layers of what was defined as the cohesive zone.

**Summary**

The very narrow range of the melting process in the experimental blast furnace was in agreement
with what was found in the laboratory experiment, and is considered as an important advantage of
the MPBO pellet, together with the start of melting occurring at a high temperature. The narrow
melting range results in a narrow cohesive zone in the blast furnace, which in turn gives a low
pressure drop. The high melting temperature gives a cohesive zone low in the blast furnace, which
provides more space for gaseous reduction. Thus, the high temperature phenomena of softening and
melting were found to be correctly simulated in the laboratory reduction-under-load tests.

Comparing the progress of reduction in laboratory scale with blast furnace reduction can be
appropriately made as long as the reduction parameters for laboratory scale testing are correctly
chosen. The deviation in reduction behaviour observed in this work was mostly due to the
experimental parameters chosen for laboratory testing deviating from the conditions of the
experimental blast furnace. Therefore, if laboratory reduction-under-load tests are considered for
simulation of the blast furnace process, those could certainly be adequately performed by adjusting
the laboratory reduction parameters, i.e. temperature/gas composition/time programs, for the blast
furnace process in question.

In order to simulate the blast furnace operation in laboratory scale it is, of course, important to
know the conditions in the blast furnace process. Although information of temperature and gas
composition at various levels in commercial blast furnaces can be found in the literature, this
information may only be valid for these furnaces measured under their conditions, such as raw
materials used, operational parameters and strategies applied. Problings, excavations and dissections
of the experimental blast furnace can provide more useful information of the state inside the blast
furnace. Using such information to adjust the test parameters used in laboratory tests a much better
simulation of the blast furnace process could be made in laboratory scale.
6 CONCLUSIONS

The high temperature phenomena occurring when reducing the MPBO pellet, including softening and melting, were found to be the same in laboratory tests and in the experimental blast furnace. The progress of reduction down through the burden of the experimental blast furnace was similar, but not identical to the results of the RUL experiments. The differences were found to be due to different reducing conditions. By using the same experimental conditions in laboratory scale reduction-under-load experiments as those obtained in the experimental blast furnace, a better simulation of the reduction in the blast furnace can be performed in laboratory scale, as far as the reduction extent is concerned. Thereby it is concluded that laboratory reduction-under-load, or reduction softening and melting, tests can be appropriately used to simulate the blast furnace process. However, to perform such a simulation it is very important to know the internal state of the blast furnace. Since the blast furnace process has developed much in the last decades (concerning injection of reductants, lowering of coke rates, different charging strategies, increasing productivity etc) up-dated and accurate information of the blast furnace interior is needed to perform correct simulations of the blast furnace process in laboratory scale. Such information can be provided from the experimental blast furnace.

The more specific conclusions from this study can be summarised as follows:

Experimental Blast Furnace Results

- The reduction degree can be as accurately determined based on identification of iron oxide/iron phases in excavated material studied under microscope as from chemical analyses.
- Vertical probes can be used to measure the burden descent rate in the blast furnace shaft, and thereby an assessment of excavation data from vertical level to treatment time can be made, i.e. the reduction degree in an experimental blast furnace can be determined both as a function of depth as well as time from charging.
- The reduction profile in the experimental blast furnace is similar to what is described in literature, i.e. fast initial reduction to wüstite followed by slow reduction through the chemical reserve zone and again fast reduction of wüstite in the lower part of the furnace.
- Vertical temperature probe measurements were found to be very reproducible, and the results of temperature measurements agree with data from commercial furnaces given in literature.
- Temperature measurements by horizontal shaft probes can be unreliable at temperature above 800 °C, where the cooling of the probe influences the measuring results.
- Horizontal temperature probe measurements in the upper shaft or the top gas temperature profile measurements can be used to determine the gas distribution along the radius direction, and if the blast furnace is centre-working or wall-working.
- The wall- or centre-working process can be verified by determination of reduction degree from excavated material.
• The gas profiles measured in the experimental blast furnace shows that below 900 C, the gas composition is governed by the equilibrium between iron and wüstit (which means that the olivine pellet is very reducible).

**Comparison between Data from Experimental Blast Furnace and from RUL Tests**

• The reduction time in the experimental blast furnace was shorter than used in the RUL experiments (and what is given in literature). By applying a dimensionless vertical distance, either based on vertical level or on temperature isotherms, it is possible to compare results from the experimental blast furnace with RUL results (or results from commercial furnaces).

• The reduction down through the burden of the experimental blast furnace was similar to the results of the RUL experiments for the wall and mid-radius profiles, when comparing different levels of the furnace and disregarding the difference in experimental time. The biggest difference was found for the centre profile, caused by the wall-working operation of the experimental blast furnace, while the RUL experiments were set-up to simulate a centre-working operation.

• The deviation in the shape of the reduction curves between laboratory scale and the experimental blast furnace was due to the absence of a chemical reserve zone in the laboratory experiments. This was, in turn, caused by the choice of reducing parameters for the tests.

• In the RUL experiments, at all time during reduction only two phases (iron oxides or iron) co-existed and the reduction was found to take place in stages. In the experimental blast furnace, most of the time during reduction three phases co-existed. The reason given is faster reduction in the experimental furnace in comparison to the laboratory furnace.

• Softening of the MPBO pellets was not found to take place either in the experimental blast furnace or in the RUL furnace.

• Cohesive zone formation (low-permeable, semi-melted material) was found to be very restricted when using the MPBO pellet in the experimental blast furnace. Only two layers of semi-melted material indicates a fast melting of the metallised pellets, which was also observed in the RUL experiments.
FUTURE WORK

Future work should include the design of new RUL experiments to imitate the operation of the experimental blast furnace. The results can again verify the conclusion that it is possible to make a simulation of blast furnace reduction in laboratory scale.

By adding temperature indicators (so called tracers, or markers) to the burden before quenching of the experimental blast furnace it should be possible to establish the temperature profile of the complete cohesive zone region. Thereby, in a future work more comparisons between laboratory tests and the experimental blast furnace can be done, with more emphasis on temperatures of cohesive mass formation, start of melting and final melting, which are valuable results of reduction-under-load (or reduction, softening and melting) tests.

The gas composition profile along the height of the experimental blast furnace should be monitored by vertical gas probings, to provide an increased knowledge of the blast furnace interior, and to establish proper experimental conditions for laboratory simulations of the process.
ACKNOWLEDGEMENTS
Professor Jitang Ma is sincerely acknowledged for critically reviewing the paper. Special thanks also to Mr. Mats Hallin, Ms. Anna Dahlstedt and Dr. Lawrence Hooey of LKAB for valuable suggestions, and to all LKAB personnel involved in the experimental blast furnace trials.
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Table 7. Specification of the use of different data in this investigation.
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### Table 1. Experimental blast furnace specifications.

<table>
<thead>
<tr>
<th>Specification</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Working volume</td>
<td>8.2 m³</td>
</tr>
<tr>
<td>Hearth diameter</td>
<td>1.2 m</td>
</tr>
<tr>
<td>Working height</td>
<td>5.9 m</td>
</tr>
<tr>
<td>Tuyeres</td>
<td>54 mm diameter (x3)</td>
</tr>
<tr>
<td>Top pressure</td>
<td>up to 1.5 bar</td>
</tr>
<tr>
<td>Charging</td>
<td>Modified bell type</td>
</tr>
<tr>
<td>Burden distribution</td>
<td>Movable armour</td>
</tr>
<tr>
<td>Injection</td>
<td>Coal, oil, slag formers</td>
</tr>
<tr>
<td>Blast</td>
<td>up to 2000 Nm³/h</td>
</tr>
<tr>
<td>Blast heating</td>
<td>Pebble heaters</td>
</tr>
<tr>
<td>Max. blast temp.</td>
<td>1300°C</td>
</tr>
<tr>
<td>Furnace crew*</td>
<td>5/shift</td>
</tr>
<tr>
<td>Tapping volume</td>
<td>1.3-1.8 tHM/tap</td>
</tr>
<tr>
<td>Tap time</td>
<td>5-10 min.</td>
</tr>
<tr>
<td>Tapping interval</td>
<td>60 min.</td>
</tr>
<tr>
<td>Reducing agents</td>
<td>510-540 kg/tHM</td>
</tr>
<tr>
<td>Quenching N₂ gas flow</td>
<td>300-400 m³/h</td>
</tr>
</tbody>
</table>

*excluding sampling/research staff

### Table 2. Composition of the olivine iron ore pellet MPBO.

<table>
<thead>
<tr>
<th>Element</th>
<th>Composition (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>66.7</td>
</tr>
<tr>
<td>SiO₂</td>
<td>1.95</td>
</tr>
<tr>
<td>MgO</td>
<td>1.45</td>
</tr>
<tr>
<td>CaO</td>
<td>0.25</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.40</td>
</tr>
<tr>
<td>MnO</td>
<td>0.06</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.35</td>
</tr>
<tr>
<td>V₂O₅</td>
<td>0.24</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.02</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.04</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.02</td>
</tr>
</tbody>
</table>

### Table 3. Position of horizontal shaft probes in the experimental blast furnace.

<table>
<thead>
<tr>
<th>Probe</th>
<th>Distance below burden surface (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upper shaft probe</td>
<td>1.0</td>
</tr>
<tr>
<td>Middle shaft probe</td>
<td>2.4</td>
</tr>
<tr>
<td>Lower shaft probe</td>
<td>3.4</td>
</tr>
</tbody>
</table>

### Table 4. A rough characterisation of reducing conditions at different radial positions in a blast furnace.

<table>
<thead>
<tr>
<th>Radial position</th>
<th>Wall</th>
<th>Mid-radius</th>
<th>Centre</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rate of temperature increase</td>
<td>Low</td>
<td>Medium</td>
<td>High</td>
</tr>
<tr>
<td>Reducing gas potential</td>
<td>Medium</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Mechanical load</td>
<td>High</td>
<td>Medium</td>
<td>Low</td>
</tr>
</tbody>
</table>
Table 5. Rate of temperature increase and time for completion of the reduction-under-load tests for the different temperature profiles used.

<table>
<thead>
<tr>
<th>Temperature interval (°C)</th>
<th>Rate of temperature increase (°C/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wall profile</td>
</tr>
<tr>
<td>400 – 800</td>
<td>4</td>
</tr>
<tr>
<td>800 – 1000</td>
<td>5</td>
</tr>
<tr>
<td>1000 – 1600</td>
<td>5</td>
</tr>
<tr>
<td>Time for complete test (min)</td>
<td>260</td>
</tr>
</tbody>
</table>

Table 6. Reducing gas CO content of RUL-experiments at different temperatures (two reducing potentials, weak and strong, used for each of the radial positions), and corresponding load programs.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Wall profile</th>
<th>Mid-radius</th>
<th>Centre profile</th>
<th>Wall profile</th>
<th>Mid-radius</th>
<th>Centre profile</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>23.5 / 25.0</td>
<td>20.0 / 21.5</td>
<td>26.5 / 28.0</td>
<td>300</td>
<td>200</td>
<td>50</td>
</tr>
<tr>
<td>800</td>
<td>25.8 / 27.3</td>
<td>22.5 / 24.0</td>
<td>29.0 / 30.5</td>
<td>600</td>
<td>400</td>
<td>200</td>
</tr>
<tr>
<td>1050</td>
<td>30.5 / 31.5</td>
<td>28.0 / 29.0</td>
<td>33.0 / 34.0</td>
<td>662</td>
<td>612</td>
<td>412</td>
</tr>
<tr>
<td>1150</td>
<td>34.5 / 34.5</td>
<td>34.5 / 34.5</td>
<td>34.5 / 34.5</td>
<td>688</td>
<td>638</td>
<td>438</td>
</tr>
<tr>
<td>1300</td>
<td>36.0 / 36.0</td>
<td>36.0 / 36.0</td>
<td>36.0 / 36.0</td>
<td>725</td>
<td>675</td>
<td>475</td>
</tr>
<tr>
<td>1600</td>
<td>36.0 / 36.0</td>
<td>36.0 / 36.0</td>
<td>36.0 / 36.0</td>
<td>800</td>
<td>750</td>
<td>550</td>
</tr>
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</table>

Table 7. Specification of the use of different data in this investigation.

<table>
<thead>
<tr>
<th>Measurement</th>
<th>Campaigns used</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dissection data</td>
<td>A</td>
</tr>
<tr>
<td>Vertical probe (temperature)</td>
<td>B C D E</td>
</tr>
<tr>
<td>Upper shaft probe (gas analysis and temperature)</td>
<td>D</td>
</tr>
<tr>
<td>Middle shaft probe (gas analysis and temperature)</td>
<td>D</td>
</tr>
<tr>
<td>Lower shaft probe (gas analysis and temperature)</td>
<td>E</td>
</tr>
</tbody>
</table>
Table 8. Blast furnace operating parameters prior to shut-down of Campaign A, and during Campaigns B, C, D and E respectively – averages of 24 hours stable (representative) operation.

<table>
<thead>
<tr>
<th></th>
<th>Campaign A</th>
<th>Campaign B</th>
<th>Campaign C</th>
<th>Campaign D</th>
<th>Campaign E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Productivity (tHM/m³/24h)</td>
<td>4.9</td>
<td>4.3</td>
<td>4.1</td>
<td>4.5</td>
<td>4.3</td>
</tr>
<tr>
<td>Productivity (tHM/m²/24h)</td>
<td>35.5</td>
<td>30.9</td>
<td>29.8</td>
<td>32.6</td>
<td>31.2</td>
</tr>
<tr>
<td>Burden</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>MPBO pellets (kg/tHM)</td>
<td>1396</td>
<td>1381</td>
<td>1389</td>
<td>1384</td>
<td>1387</td>
</tr>
<tr>
<td>Quartzite (kg/tHM)</td>
<td>10</td>
<td>7</td>
<td>7</td>
<td>15</td>
<td>18</td>
</tr>
<tr>
<td>Limestone (kg/tHM)</td>
<td>50</td>
<td>33</td>
<td>42</td>
<td>53</td>
<td>48</td>
</tr>
<tr>
<td>BOF slag (kg/tHM)</td>
<td>51</td>
<td>52</td>
<td>54</td>
<td>36</td>
<td>46</td>
</tr>
<tr>
<td>Coke (kg/tHM)</td>
<td>515</td>
<td>442</td>
<td>439</td>
<td>407</td>
<td>403</td>
</tr>
<tr>
<td>Blast parameters</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>1208</td>
<td>1178</td>
<td>1171</td>
<td>1169</td>
<td>1198</td>
</tr>
<tr>
<td>Coal injection, PCI (kg/tHM)</td>
<td>-</td>
<td>74</td>
<td>87</td>
<td>121</td>
<td>132</td>
</tr>
<tr>
<td>Oxygen enrichment (%)</td>
<td>-</td>
<td>1.9</td>
<td>1.5</td>
<td>3.6</td>
<td>3.5</td>
</tr>
<tr>
<td>Moisture (g/Nm³)</td>
<td>40</td>
<td>26</td>
<td>27</td>
<td>23</td>
<td>23</td>
</tr>
<tr>
<td>Top gas</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>121</td>
<td>202</td>
<td>210</td>
<td>209</td>
<td>199</td>
</tr>
<tr>
<td>Pressure (bar, gauge)</td>
<td>0.7</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>ηCO (%)</td>
<td>48.7</td>
<td>48.8</td>
<td>47.9</td>
<td>46.9</td>
<td>46.8</td>
</tr>
<tr>
<td>CO (%)</td>
<td>23.0</td>
<td>22.6</td>
<td>22.5</td>
<td>25.4</td>
<td>25.2</td>
</tr>
<tr>
<td>CO₂ (%)</td>
<td>21.8</td>
<td>21.6</td>
<td>20.7</td>
<td>22.5</td>
<td>22.2</td>
</tr>
<tr>
<td>H₂ (%)</td>
<td>2.4</td>
<td>2.6</td>
<td>2.8</td>
<td>3.6</td>
<td>3.8</td>
</tr>
<tr>
<td>Hot metal</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>1416</td>
<td>1460</td>
<td>1435</td>
<td>1468</td>
<td>1408</td>
</tr>
<tr>
<td>C (%)</td>
<td>4.5</td>
<td>4.6</td>
<td>4.4</td>
<td>4.5</td>
<td>4.4</td>
</tr>
<tr>
<td>Si (%)</td>
<td>0.66</td>
<td>1.17</td>
<td>1.54</td>
<td>1.71</td>
<td>1.23</td>
</tr>
<tr>
<td>S (%)</td>
<td>0.065</td>
<td>0.032</td>
<td>0.030</td>
<td>0.032</td>
<td>0.067</td>
</tr>
<tr>
<td>Slag</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rate (kg/tHM)</td>
<td>146</td>
<td>136</td>
<td>137</td>
<td>140</td>
<td>148</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>30.5</td>
<td>30.9</td>
<td>34.1</td>
<td>32.6</td>
<td>33.1</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>34.5</td>
<td>34.0</td>
<td>32.8</td>
<td>32.9</td>
<td>35.7</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>17.0</td>
<td>18.7</td>
<td>17.3</td>
<td>18.2</td>
<td>17.3</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>12.8</td>
<td>14.6</td>
<td>14.5</td>
<td>15.2</td>
<td>13.1</td>
</tr>
</tbody>
</table>

1) Utilisation of furnace volume (working volume from stockline to tuyere level).
2) Output per unit hearth area.
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Fig. 19. Comparison of reduction degree in the experimental blast furnace and RUL experiments, as a function of distance from stockline, wall position. RUL results based on reduction time.

Fig. 20. Comparison of reduction degree in the experimental blast furnace and RUL experiments, as a function of distance from stockline, centre position. RUL results based on reduction time.

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SUPPLEMENT V

Jerker Sterneland and Pär Jönsson

“The use of coated pellets in optimising the blast furnace operation”

ISRN KTH/MSE--02/15--SE+METU/ART,
Division of Metallurgy, Royal Institute of Technology, Stockholm, Sweden, May 2002.
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by

Jerker Sterneland
Pär Jönsson

Stockholm Division of Metallurgy
May 2002
Department of Material Science and Engineering
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S-100 44 Stockholm, Sweden
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Division of Metallurgy
Department of Material Science and Engineering
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S-100 44 Stockholm, Sweden

Stockholm 2002

ISRN KTH/MSE--02/15--SE+METU/ART
THE USE OF COATED PELLETS IN OPTIMISING THE BLAST FURNACE OPERATION

J. Sterneland* and P.G. Jönsson**

*LKAB R&D, Box 952, SE-971 28 Luleå, Sweden and also a graduate student at KTH**
**Div. of Metallurgy, Dept. of Materials Science and Engineering, KTH, SE-100 44 Stockholm, Sweden.

ABSTRACT

With the aim to improve the blast furnace process, coating of blast furnace pellets was investigated in laboratory scale as well as in the LKAB experimental blast furnace. Olivine, dolomite and quartzite respectively were applied as coating agents onto the regular LKAB olivine pellet MPBO. Testing of the coated pellets revealed the following: i) Dust generation was significantly decreased when using coated pellets in the blast furnace, ii) Sticking was prevented by the coating material. This was verified in different laboratory tests, as well as by studying probe samples from the blast furnace, and iii) Gas utilisation was higher for all coated pellets, with a lower variation, indicating a smoother blast furnace operation. Coating of pellets is considered as a significant improvement in optimising the blast furnace operation.
1 INTRODUCTION

The LKAB experimental blast has been run in nine campaigns since it was built in 1997. Seven of the campaigns were ended by a quenching of the furnace, followed by an excavation. While dissecting the furnace a sticking, or clustering, phenomenon of pellets was observed. The ferrous burden was found to start sticking together in the middle and lower part of the shaft, before the material entered the cohesive zone. In the middle shaft the pellet aggregates formed could be separated by hand, but lower in the furnace the sticking was more pronounced.

If clustering of the particles in a pellet bed is pronounced, the smooth descending movement of the ferrous burden in the blast furnace can be disturbed. In the worst case the burden descent can be held up, resulting in hanging, followed by a slip when the burden suddenly again moves downwards. Another consequence of clustering in the pellet layers is the forming of gas channels. Gas channels represent short cuts for the reducing gas through the ferrous burden, resulting in bad utilisation of the gas and thereby the fuel rate increases. By using coated pellets, i.e. covering the pellet surface with a thin layer of non-sticking material, it should be possible to decrease the clustering tendency, at least above the cohesive zone of the blast furnace. In addition, several other advantages can also be expected.

The technique of coating of the direct reduction (DR) iron ore pellets is well known since a few years, and it has been widely used in the DR process to reduce the clustering of direct reduced iron, DRI, in DR shaft furnaces. In the DR processes the product is freshly reduced iron in a solid state. It is, therefore, crucial for the material flow in the reducing module that the solid product does not form aggregates, blocking the material flow within and out of the reactor. In the blast furnace process, on the other hand, the reduced iron is melted in the lower part of the furnace and comes out in liquid form. Therefore, until now clustering and sticking of iron bearing materials have not been considered as a problem. However, as the blast furnace process continuously improves and the fuel rates are minimised, the need for stability of the operation in the blast furnace process has increased. One essential way of improving the process stability is to improve the flows of solids and gases through the blast furnace shaft, resulting in an improved and more uniform gas-solid contact.

The first part of this paper describes the possible effects of using coated pellets in the blast furnace, and some aspects on the method to apply the coating materials. In the experimental part the coating procedure and results obtained by laboratory testing of the coated pellets, as well as testing in an experimental blast furnace, are presented. In the discussion part the main issues covered are the effects on sticking prevention and the influences on gas utilisation and decreased dust generation in the blast furnace.

Applications for patent concerning the use of coated pellets in a blast furnace have been filed by LKAB.
2 POSSIBLE EFFECTS OF THE USE OF COATED PELLETS IN THE BLAST FURNACE

In Figure 1, the possible effects of using coated pellets in the blast furnace are schematically depicted. In the following, the different issues will be further explained.

Low generation of flue dust in the blast furnace
A coating material with a strong binding force should be able to suppress the generation of dust from the pellets which otherwise would have been either carried out of the furnace by the top gas or remained in the furnace, damaging the permeability of the burden. Thereby the utilisation of both the pellets charged and the gas generated in the furnace is increased, resulting in a lower cost of hot metal.

Improved blast furnace process stability
By lowering the clustering tendency of the pellets the descending movement of the burden becomes more regular, and the formation of gas channelling is minimised. This leads to a better gas utilisation resulting in a lower fuel rate and more stable hot metal chemistry, which is very advantageous for the steel shop.

Improved slag formation
Adding fluxes as coating materials, i.e. well distributed on the surface of the pellets, will result in a more homogeneous slag formation, compared with the top charged lumpy slag formers, which can result in locally very basic (or acid) conditions. Thus, a more uniform cohesive zone formation can be expected, which possibly increases the permeability of the fused cohesive layers.

Improved alkali-yield by slag phase
Another improvement of process stability can be achieved by an improved alkali removal from the furnace. By coating pellets with a suitable slag former additive, especially acid coating material, possibly more alkali can be retained in the slag phase due to an increased alkali pick up by the coating material. A higher output of alkali in the blast furnace slag will reduce the circulating load of alkali in the furnace, leading to less alkali-induced degradation of pellets as well as coke, less scaffolding etc. An improved alkali removal in the slag phase can also be utilised to increase the slag basicity, thereby increasing the sulphur capacity of the slag.

In addition to the advantageous of using coated pellets in the blast furnace process, the following effects can also be attained by the coating of pellets:

Adjusting pellet chemistry after production
Using different types of coating material the pellet chemistry, i.e. the composition of non-ferrous oxides, can be slightly modified after pellet production. Thereby the chemistry of pellets produced can be further adjusted to fit individual demands of different customers. Depending on the amount of coating material used, the increase of slag formers in the coated pellets can be up to 0.4 percentage units, as in this investigation (4 kg of coating material per ton of pellets), or even higher.
if a larger amount of coating is used. The result is a decreased demand for lumpy slag formers charged into the blast furnace, about 5 to 6 kg/tHM, or more if larger amounts of coating were applied.

**Decreased dusting during raw material transport and handling**
In accordance with the flue dust suppression, the dusting during handling of pellets will probably also decrease by the applied coating agent. Since coating of DR pellets was introduced in the 1990’s, the dust generated during handling and reloading at the LKAB shipping port in Narvik, Norway, has significantly decreased.\(^2\)
3 THEORY OF COATING

The mechanism of sticking during reduction of iron oxides is mainly due to the freshly reduced iron on the pellet surfaces sintering together with the neighbouring pellets at the contact points. One way of preventing the sintering between pellets is therefore to keep the iron surfaces of individual pellets apart. In order to prevent sticking of pellets a coating material, basically inactive under the reducing conditions prevailing in the shaft part of the blast furnace, should be applied to cover the outer layer of the pellets.

Choice of coating materials

A variety of materials can be chosen as coating agents for blast furnace pellets. The amount applied to the pellets is small, but still very effective in preventing sticking. The coating material has to meet the following demands:

- Chemically and physically inactive (more or less) in the shaft of the furnace where sticking prevention is desired
- Strong adhesive force
- Consist of fine particles with a certain particle size distribution
- Low amount of unwanted impurities
- Available locally at a low price

In the production of coated DR pellets, coating is performed already at the pellet plant (sometimes also at the customer site) – normally using pellet slag former additives mixed with a pellet binder as the coating agent. A similar solution for coating of blast furnace pellets seems very likely.

Binders

A part of the coating mixture should be a binder material, such as a clay, or cement type of materials, which can harden onto the particles holding the coating mixture in place on the surface.

Amount of coating agent

The best case of coating is of course if a thin coating layer covers the entire pellet surface. However, even if the surface area of the pellets is not completely covered, the coating can still result in a remarkable effect in preventing sticking, since the contact surfaces between pellets in a pellet bed are very small, and only a covering of those contact areas is needed. In fact, probably only one of two surfaces in contact covered with coating might be enough. For DR pellets, the LKAB experience is that an amount of 2 to 2.5 kg/ton pellets is sufficient to prevent sticking.

Particle size of coating agent

In the production of DR pellets, the size range of the coating agent is the same as the pelletising slag former additives used, since the coating material is taken from the slag former material preparation. It might not be the optimum size range for coating, but since these slag former additives are readily available and have been found to work satisfactorily, the choice for blast furnace pellets will
probably be the same. Generally, the slag former additives used in pelletising have 65-75 % of the material less than 45 μm in size.
4 EXPERIMENTAL PROCEDURES AND RESULTS

4.1 COATING OF PELLETS

Coating of pellets was performed on the production site in Malmberget. The experimental tests of coated pellets were conducted in laboratory scale at the LKAB metallurgical lab in Malmberget and in pilot plant scale in the LKAB experimental blast furnace in Luleå.

The coated pellets were made by coating regular MPBO (LKAB olivine pellets) with different types of coating materials. The chemical analyses of the pellets are shown in Table 1. MPBO-2 and MPBO-3 are basically the same type of pellets, both are olivine pellets with addition of olivine and a small amount of limestone, and in the MPBO-3 pellet also a small amount of silica is added. The MPBO-3 pellet was used as the base pellet for the coating experiments, while both uncoated MPBO-2 and MPBO-3 were used as reference materials in the experimental blast furnace.

Coating materials

Three types of coating materials were used in this investigation; olivine, quartzite and dolomite. All of them were mixed with 9% of bentonite as a binding phase. Chemical analyses of the coating materials were also shown in Table 1, whilst the size distributions of the coating materials are shown in Table 2, as fractions in different size ranges. All materials used are very similar in size, with most part < 45 μm (65-70%) and only small amounts > 0.125 mm (1-6%), which should result in similar kinetic conditions.

Coating technique

The coating method used is schematically depicted in Figure 2. During the coating procedure, pellets were removed from the pellet bin on a conveyor belt. At the transfer point to a second conveyor belt, pre-mixed coating slurry was sprayed through two nozzles onto the stream of pellets. The coating slurry constituted the coating agent mixed with bentonite as described above, and water added to arrive at a solid content of 25%. The flows of coating slurry and pellets were adjusted to apply an amount of 4 kg of solid coating materials per ton of pellet product. This amount, which is roughly twice the amount used on LKAB DR pellets, was chosen to assure the effect of the coating addition. For DR pellets there is a strong desire to decrease the coating additions to a low level, since even small amounts of acid oxides from the coating are very disadvantageous in the DRI used in steelmaking. For the blast furnace process, on the other hand, this is not a necessary requirement, but rather the opposite. Presently most LKAB customers would welcome a higher slag former addition in the pellets.

Investigation of the coating layer under microscope

The theoretical thickness of the coating layer, when using an amount of coating material of 4 kg/ton of pellets, has been calculated to be 0.010 mm (0.008 mm for olivine due to its higher density), assuming a uniform layer on an ideal sphere with a diameter of 10.5 mm. However, the intention of obtaining an even distribution of coating material on the pellet surfaces was not so easy to reach in practice. Due to the difficulty of evenly spraying a relatively small amount of coating material onto
a large amount of pellets, one can assume a more or less irregular distribution of the coating on the pellet surfaces. Also, the pellet surfaces are not completely smooth, some cracks or other irregularities may exist, where part of the coating slurry can penetrate. Finally, one of the assumptions in the calculation of the average coating layer thickness of 0.010 mm, was a particle size less than 0.010 mm, which in fact is the case only for a part of the different coating materials used. For the above reasons the practical layer thickness could be expected to vary from zero (areas not covered by coating) up to tenths of mm.

The actual coating layers were investigated by scanning electron microscope (SEM). In Figures 3 to 5 examples of the outer part of the olivine coated pellets are shown. The samples were collected prior to charging into the experimental blast furnace, i.e. after long time outside storing, handling, transport (250 km by truck) and on-size screening. The coating layer thickness observed, shown in Figure 3, is in the range from 0.010 to 0.030 mm and in Figure 4 from zero up to 0.100 mm. As expected, there were also some areas of the pellet surfaces examined not covered by coating at all, as shown by the right particle in Figure 4. It was also confirmed by the SEM study that the coating occasionally fills up cracks and cavities, as shown in Figure 5.

4.2 LABORATORY TESTING OF COATED PELLETS

Samples

Samples of 15 kg were taken for every 12 tons of coated pellets produced during the coating process. For each of the coated pellet types, the samples were homogenised and used for chemical analysis as well as mechanical and metallurgical tests. Chemical analysis of the base pellets and the coated pellets are given in Table 3, where chemical analyses of the pellets sampled at the blast furnace site are also given. As can be seen from the data the amount of coating materials could be accurately controlled, and the coating materials were found to well stay on the pellet surfaces even after storage, transport, handling and screening (undersize <6 mm screened off before charging to the blast furnace).

Sticking tests

To investigate the sticking behaviour of the coated pellets, different types of reduction/sticking tests were carried out, of which three are presented here:

1. HYL Standard test
2. BFS (Blast Furnace Sticking) test
3. Modified ISO 7992 (reduction under load) test

1. HYL Standard test

The first test method used was the HYL Standard test,[1] normally performed on all DR pellet deliveries. This test method was developed to describe the sticking tendency of reduced material in a DR shaft furnace. After isothermal reduction at 950 °C to 90 % reduction degree, a load of 1500 g/cm² is applied to the sample bed for 30 minutes, still at 950 °C but in a nitrogen atmosphere. The sample is then cooled in nitrogen and the clustered part of the sample is treated in a 1.0 meter drop
test, for up to 20 drops. The result of the test is a sticking index value describing the tendency for sticking, SI from 0 (no agglomerated particles before commencing the drop test) to 100 (all particles agglomerated even after 20 drops). The company Hylsa, designer of the HYL direct reduction processes and also the designer of the HYL Standard test, has recommended a sticking index below 30, which has been generally adopted at DR plants as the upper limit. For LKAB DR pellets the sticking index is normally in the range of 15 to 25 (presently all pellet deliveries are coated pellets), whereas uncoated pellets have been found to experience values of 75 to 95.

2. BFS (Blast Furnace Sticking) test
Since the HYL Standard test is well-known and has been widely accepted as a testing method for DR pellets, it constituted the basis for the development of a modified test for the coated blast furnace pellets at the beginning of this study. Except for the gas composition all test conditions of the HYL Standard test were used in the modified test, BFS (Blast Furnace Sticking) test, since the conditions in a DR shaft furnace can be considered similar to the shaft part of the blast furnace, with the exception of gas composition. The hydrogen-based gas mixture in the HYL Standard test (55% H₂, 21% CO, 14% CO₂ and 10% N₂) was changed to a carbon monoxide/nitrogen based gas mixture (2% H₂, 40% CO and 58% N₂) in the BFS test. The results of the sticking tests are given in Table 4. A significant effect of the coating materials on the sticking behaviour can be seen, when comparing with the results for the uncoated pellet, which experiences a sticking index in the range of 45 to 50. Olivine and dolomite coatings seem to be the best, SI around 3, while quartzite is a somewhat less effective coating material, with an SI value of 8. These results clearly indicate that all of the three types of coated pellets have a very low sticking tendency.

3. Modified ISO 7992 (reduction under load) test
To examine if a standardised reduction test under load, ISO 7992[3], which is commonly used for testing reduction behaviour of blast furnace materials, could also be used for sticking tests, the ISO 7992 test was appended with a drop test for measuring sticking after reduction. In the ISO 7992 test, 1200 g of pellets are reduced isothermally at 1050 °C to 80 % reduction degree, with a load of 500 g/cm² on the sample bed during reduction in an atmosphere of 2% H₂, 40% CO and 58% N₂. The main differences from the BFS test described above are thus 100 °C higher testing temperature and less load, and the application of the load during the reduction instead of after. After the reduction test the sticking behaviour was analysed by conducting the same drop test as previously described. The results of this test are shown in Table 5. As can be seen in the table, the 100 °C higher temperature of this test, although with lower load applied, cause a significantly increased sticking of all materials.

Influence of temperature on the sticking behaviour
In the modified ISO 7992 test, the use of a load applied during reduction, instead of after, can be considered to better imitate the conditions in the blast furnace shaft. However, the temperature seems a little too high, as the sticking behaviour in the shaft part of the blast furnace, where most part is so-called heat reserve zone, normally at 950 °C to 1000 °C, is mainly concerned. Therefore,
this test was conducted at three different levels of temperature, 1050 °C, 1000 °C and 950 °C. Results of these tests are also given in Table 5, and a graphical description of the change in sticking index numbers at different temperatures is given in Figure 6. As can be seen in the figure, the influence of reduction temperature on the sticking behaviour is distinct in the temperature range from 950 °C to 1050 °C, both for uncoated and coated pellets. The ranking of the coating materials is in line with the findings of the previous tests, i.e. olivine and dolomite are the best materials to prevent sticking, while quartzite is less effective. At 950 °C all the coated pellets have SI values lower than 8, while the uncoated has a value of 22. At 1000 °C the values are in the range of 17 to 40 for coated pellets, compared to 71 for uncoated pellets. Finally, at 1050 °C both the uncoated and the quartzite coated pellets became severely sintered (95 in sticking index), while a sticking preventing effect was still achieved by the olivine and dolomite coatings, resulting in SI values of 47 and 35 respectively.

Mechanical and metallurgical testing
Results of mechanical and metallurgical tests are shown in Table 6. Most parameters related to pellet quality are little or not at all affected by the use of coating. A decrease in the Cold Compression Strength (CCS) is obtained, by 13 to 29 daN/pellet or 6 to 12 %, and in the Low Temperature Disintegration value (LTD), up to 18 percentage units in the >6.3 mm fraction. Both of these changes were actually caused by well-known effects of adding water to oxide pellets, not caused by the coating materials.\cite{4,5}

4.3 EXPERIMENTAL BLAST FURNACE TRIALS WITH COATED PELLETS
Blast furnace testing of the coated pellets was conducted in the LKAB 1.2 m hearth diameter experimental blast furnace, of which a cross-section is shown in Figure 7. Most of the operating parameters of the blast furnace, shown in Table 7, were chosen to resemble the operation of the SSAB Tunnplåt no. 3 blast furnace in Luleå, and were virtually unchanged during testing. The intention was to keep the operating parameters on the same levels during all periods of the trial.

The trial was divided into five different periods:

1. MPBO-2 Reference period using pellets without coating
2. MPBO-O Olivine coated MPBO-3 pellets
3. MPBO-D Dolomite coated MPBO-3 pellets
4. MPBO-Q Quartzite coated MPBO-3 pellets
5. MPBO-3 Reference period using pellets without coating

As mentioned earlier, the MPBO-2 and MPBO-3 are the same type of olivine pellets, only with a small difference of silica addition to the latter. Both pellets have been operated at SSAB Tunnplåt (Luleå) and SSAB Oxelösund in Sweden, and at Fundia Koverhar in Finland, without showing any significant difference in blast furnace operation.
Table 8 shows the moisture contents of the pellets and the amounts of lumpy slag formers charged to the blast furnace for each of the trial periods. The MPBO-2 pellets were taken from a pellet storage bin and were thus dry (less than 0.1 % moisture), while the MPBO-3 pellets were taken from an outside storage and, therefore, had picked up some moisture during storage and transportation, to a level of 2.2 %. The amount of moisture added to the pellets during the coating procedure corresponded to about 1.5 %, indicating that some snowfall during the coating procedure and outside storing (though covered from rain and snow), made the pellets pick up another 0.6 to 0.8 % of moisture before charging to the furnace.

As can also be seen from Table 8, the amount of limestone charged in the burden was kept at an almost constant level in all periods. In order to keep the target slag basicity (\(\text{CaO}/\text{SiO}_2 = 0.9\)) the amount of basic BOF-slag addition was adjusted to compensate for the different chemistry of the different coating materials used, except for the quartzite coating period, in which the lumpy quartzite added from the top was decreased.

*Process responses of the blast furnace*

When changing from one type of burden material to another, normally data from a transition time period representing two to four throughputs of the burden in the experimental blast furnace should be excluded. Since in this case the difference in burden composition was very small between the different periods and the primary interest of the tests was the conditions of the shaft part of the blast furnace, only one to two throughputs of data needed to be excluded. In fact, a quick stabilisation of the gas utilisation was achieved when changing from uncoated to olivine coated pellets, as shown in Figure 8. Thus, the duration of the trial periods could be considered sufficient for studying the effects of the coated pellets, at least for the first four trial periods of 48 to 85 hours, shown in Table 7.

Average values and standard deviations of some main process parameters of the blast furnace are shown in Table 9. The gas utilisation, \(\eta_{\text{CO}}\), is at the highest levels, also with the lowest standard deviation, when using coated pellets. This is more clearly demonstrated in Figure 8, which shows the gas utilisation and Burden Resistance Index (BRI) for one-hour average values throughout the trial. The BRI is a measure of the pressure drop over the burden column (pressure drop corrected for the gas flow through the bosh, and also known as the PV bosh, or permeability resistance measurement). Since modifications of the blast distribution were made at a few occasions during the trial, resulting in a discontinuity in the pressure drop, the BRI values have been compensated in the figure. Therefore, the actual BRI levels are less comparable, but the short-time variations still have a high significance for making comparisons. The variations in the BRI were much lower for the periods using dolomite and quartzite coated pellets.

The effect of the higher moisture content of coated pellets was reflected in the lower top gas temperatures, in comparison with the periods using dry pellets, as shown in Table 9. Burden descent rate and its variation were similar with all types of pellets. The productivity was slightly higher for the period using dolomite coated pellets (yet with somewhat higher oxygen enrichment, as seen in
Table 7), and also the fuel rate was lower, though not very significant. Minimising the fuel rate was not a primary goal of the trials, though, and the testing times were somewhat too short in this particular respect.

Table 10 shows the hot metal quality and the slag composition. For the hot metal temperature the MPBO-3 period deviated with a higher average temperature. The reason was that the fuel rate was kept somewhat higher during this period. This can also be seen in the C and Si contents of the hot metal produced, which were both somewhat higher than that for the other periods. The hot metal C/temperature ratio was almost constant throughout the trial, at 3.1, which indicates similar hot metal carburising conditions, i.e. a well-drained hearth.

Flue dust generation

One of the main interesting things found during the test was a substantially decreased generation of blast furnace flue dust. Table 11 shows the amounts of flue dust collected, and its composition. The amounts of dry flue dust, removed by a dust catcher cyclone, were significantly lower for the three periods of using coated pellets than that for the periods using uncoated pellets. An average size distribution of the collected flue dust was shown in Table 2. It can be seen that the flue dust was considerably coarser than the materials used for coating. The finer part of the flue dust passes through the dust catcher cyclone and is collected by a subsequent wet electrostatic precipitator, in the form of sludge. Table 12 shows the composition of the blast furnace sludge from the different periods.

Blast furnace top gas flue dusts and sludges are normally partly generated inside the furnace, and partly originate from fines brought into the furnace by the burden (iron oxides, coke and, to some extent, also from slag former additives). The contribution of these different materials can be estimated by making mass balances of the different components of the dust and sludge. For instance, coke is the main source of carbon and iron oxide fines is the main source of iron. Other oxide components of the dust and sludge analyses can then be balanced by the contribution of the different slag former additives. Literature data of an investigation of blast furnaces operated with 100 % pellets has shown that the iron in the flue dust can exist in various forms; 1.5 to 3.8 % of the iron was in the investigation found in the metallic form, 13-21 % in the form of Fe₂O₃, the rest of the iron being in the form of Fe₃O₄. This indicates that some of the iron originates from slag former additives, such as BOF slag, or from pellet fines generated inside the furnace during reduction. Therefore, an oxygen to iron ratio of 1.46 for the iron oxides in the dust was used in the calculations. In Table 11 also the estimated contributions of different sources to the flue dust were shown. A more detailed discussion will follow later.

Examination of probe material

Probe samples were taken out of the blast furnace during operation. The lower shaft probe, i.e. the third probe from the top, shown in the left part of Figure 7, was used to take samples at a level of 3.4 m below stockline, where the temperature was measured to be 1000 to 1050 °C. SEM images of two samples of olivine coated pellets, reduced to 70 % reduction degree, are shown in Figures 9 and
10. A layer of the coating material can be observed on the surfaces of the pellets. Beneath the coating layer, the outer layers of the pellets are completely reduced to iron, whereas in the interior, not shown in the figures, there were still some amount of wüstite. This means that the coating materials remains on the pellet surfaces through the thermal reserve zone, where the sticking prevention is considered most important, and that the gaseous reduction takes place without significant obstruction.

An inclined probe, shown in the right part of Figure 7, was used to collect samples from the cohesive zone in the lower part of the blast furnace. The temperature in this region was, from vertical temperature probings, estimated to be in the range of 1200 to 1300 °C. Even at these high temperature levels, the sticking phenomenon had been successfully prevented by the use of coating of the pellets, as shown by the pellet samples in Figures 11 to 14. Inversely, the sticking observed is significantly worse for the uncoated pellets than for the coated ones. The sticking when using quartzite coated pellets is more pronounced than for olivine or dolomite coated pellets.

The probe samples were analysed chemically. The samples from the cohesive zone were found to contain 0.5-2.0 % K$_2$O and 0.1-0.5 % Na$_2$O in the upper samples of the cohesive zone (67-95 % reduction degree), and 2.4-4.0 % K$_2$O and 0.4-0.7 % Na$_2$O in the lower samples of the cohesive zone (96-98 % reduction degree).
5 DISCUSSION

5.1 BLAST FURNACE FLUE DUST GENERATION

A significant decrease in blast furnace flue dust collected in the dry dust catcher cyclone was observed during the trials with coated pellets, shown in Table 11, from 5.4 kg/tHM for the MPBO-2 and 4.4 kg/tHM for the MPBO-3 pellets down to below 3 kg/tHM for the coated pellets. Although the amount of flue dust collected by the cyclone is only part of the dust generated, it could still be considered as a good indicator of dust formation in the blast furnace. The mass balances based on chemical analyses of the flue dust showed that less pellet fines accounted for 0.8 to 1.0 kg/tHM of the decrease in flue dust amounts when using coated pellets compared to uncoated, or inversely expressed: pellet material as the flue dust leaving the furnace decreased by about two thirds. These observations were further confirmed by the fact that in the wet part of the flue dust, i.e. the sludge, the content of iron was also decreased when using coated pellets, as can be seen in Table 12. The causes for the improvement in flue dust generation are believed to be 1) the binding phase of the coating, and 2) better gas distribution.

The coating layer covering the surface of the pellets has a strong adhesive force, thus preventing dust formation of pellets. One of the evidences is the low content of coating material found in the dry dust, as well as in the sludge. The mass balances of the dry flue dust, showed that in the case of using olivine coated pellets, the coating itself contributed by less than 0.2 kg/tHM, compared to the 5 kg/tHM used for coating. Although it is not possible to distinguish whether the SiO$_2$ in the flue dust came from lumpy quartzite or from the quartzite coating during the period of using quartzite coated pellets, the quartzite coating material in the flue dust was believed to be below 0.2 kg/tHM, based on the data of the contribution of the lumpy quartzite to the flue dust in the other periods. In the sludge, there was no indication of significant amounts of the coating materials either, as shown in Table 12. These observations clearly indicate that the coating layer can be kept quite well on the pellets, which was also verified by the examination of probe samples of olivine coated pellet from the lower shaft probe. The SEM images of the samples, as were shown in Figures 9 and 10, clearly shows that the coating layer is still present on the pellet surfaces, even after reduction of the outer layer to iron. It can, therefore, be assumed that the flue dust generation should be decreased when using coated pellets. For uncoated pellets, already clustered masses are probably de-clustered, as the burden descends in the furnace, which possibly generates significant amounts of dust. Contrary, for coated pellets, the sticking, and thereby also the dust generation inside the burden column, is prevented by the coating.

It should also be noted that the amounts of fine particles formed by coke fines as well as the lumpy slag formers charged were all lower for the periods with coated pellets and with the wet MPBO-3 pellet than for the period with dry MPBO-2 pellet. The cause is believed to be the combined effects of the two following factors:
1) The improved gas flow in the shaft of the furnace when using coated pellets. Less clustering as well as less channelling certainly gave a more uniform gas flow with a lower gas velocity locally. Therefore, less fine particles were picked up by the gas.

2) High moisture contents of the pellets used in all periods except for the MPBO-2 period. The higher moisture content of the coated pellets resulted in a lower top gas temperature, due to the energy demanded for vaporisation of the water. Assuming that the gas phase behaves ideally, the lowering of the top gas temperature by 10 °C represents a decrease of 2 % of the top gas velocity (and 4 % for a 20 °C decrease). The lowered top gas velocity possibly resulted in less fine particles blown out of the furnace.

5.2 STICKING PREVENTION
The results of sticking tests have clearly shown that the sticking phenomenon has been prevented significantly by the use of coating, as shown in Table 4. Sticking indexes between 3 and 8 should be considered extremely good, compared to 15-25 for the DR coated pellets after several years of optimising the coating technique. The meaning of the two sticking values of 3 and 8 is that most of the pellets are non-sticking after the reduction even before the drop test, and the few remaining clustered pellets fall apart in the first two or three of the twenty possible drops. The reasons for the better results obtained with coated blast furnace pellets, compared to coated DR pellets, were probably partly due to the use of double the amount of coating material as that used for DR pellets, and partly because of the lower iron content, 66.6 % compared to 67.6-68.1 % Fe (or, on the contrary, the lower amount of slag former oxides in the DR pellets). In addition, the mechanism of metallic iron growth could also be different in the different reducing gases, in terms of H₂ based or CO based gases.

Influence of reduction time
Comparison of the HYL Standard sticking test results with those from the BFS test, shown in Table 4, gives the following interesting conclusion. Despite two to three times longer reduction time in the blast furnace gas composition compared to DR gas composition, the resulting sticking index values were almost identical. These results indicate that the sticking index is probably independent of the reduction velocity.

Influence of reduction temperature
The increase in sticking index with increased reduction temperature from 950 °C to 1050 °C is significant for both coated and uncoated pellets, as shown in Table 5 and Figure 6. At the higher temperature, 1050 °C, there is no effect of the quartzite coating as compared to the uncoated pellets. However, since the heat reserve zone, which occupies most of the shaft of the blast furnace, is normally at 950 °C to 1000 °C, good sticking indexes for the coated pellets obtained at these two temperatures indicate that the formation of clusters in most part of the blast furnace shaft can be largely prevented.

Samples taken by an inclined probe penetrating the cohesive region of the blast furnace verified the sticking prevention effect of the coating. Even at very high temperatures, estimated above 1200 °C,
the coating was still efficiently preventing sticking. As was shown in Figures 11 to 14, the sintering between individual pellet particles was more pronounced for the uncoated pellets compared to the coated. The difference between the coating materials was also seen, especially at high temperatures, with more sintering of the quartzite coated pellets compared to the olivine and dolomite coated pellets, in full agreement with laboratory sticking test results at higher temperatures, Table 5.

**New test method developed for testing sticking of blast furnace pellets**

The Blast Furnace Sticking test, BFS, developed in this study could become a very useful tool for testing the sticking tendency of blast furnace pellets. The fact that most of the test parameters are identical with those used in the HYL Standard sticking test is considered as an advantage, since this test has been regularly used and has been found to be decisive, repeatable and accurate. Also, the modification using a gas composition resembling blast furnace reducing gas can be easily implemented in the established sticking test procedure for DR pellets.

On the other hand, from viewpoint of simulating the conditions in the blast furnace shaft, the modified ISO 7992 test is a better alternative as a sticking test for blast furnace pellets. The application of the load during reduction, instead of after, resembles the situation in the blast furnace better. In this case the choice of test temperature should be 950 °C or 1000 °C, as this is generally considered as the temperature range of the thermal reserve zone, occupying a major part of the blast furnace shaft, where prevention of sticking is the most important. Further investigations need to be conducted to decide whether to use the higher or the lower temperature, or even both. It could also be of interest to examine the influence of temperature on the sticking tendency for other types of pellets, before deciding what test temperature to use.

5.3 GAS UTILISATION

As shown in Table 9, the gas utilisation, $\eta_{CO}$, was higher when using coated pellets, compared to the periods using uncoated pellets. A high value of $\eta_{CO}$ means that more CO is oxidised to CO$_2$ in the furnace shaft. Thereby more heat released by oxidising CO is utilised in the process and the fuel demand is decreased by the increased indirect reduction.

Of course, the measured value of $\eta_{CO}$ can also be influenced by some other factors, for instance CO$_2$ released by calcination in the furnace shaft, and water gas shift due to more steam in the gas, brought by higher content of moisture in the coated pellets. However, the limestone amounts charged to the furnace during the different trials were essentially the same and the amount of dolomite as the coating material was very small, so the influence of calcinations of carbonates on the gas utilisation could be neglected. Also the extent of the water gas shift reaction, caused by the increased moisture input when using coated pellets, is believed to be minor.

The increase in gas utilisation when using coated pellets can be considered as a result of improved reduction conditions by the use of coated pellets, achieved by a smoother burden descent and less channelling in the blast furnace shaft, in turn caused by a decreased sticking in the pellet layers. It also indicates that the covering by a thin layer of coating material on the surface of the pellets do
not seem to result in any negative effect on the reduction of the pellets in the furnace. The lowered variations in gas utilisation could also be taken as evidence of the improved gas-solid contact, with less channelling and smoother burden descent.

5.4 SOME OTHER ISSUES CONCERNED

Economical aspect of decreased dust generation in handling, transport and in the blast furnace

The dust generated during handling and transportation and the flue dust generated in the blast furnace consist of blast furnace feed material, but in an undesirable size range. The normal practice is to return the material to the furnace in an agglomerated form, either by sintering or briquetting. Decreasing the dust formation means increasing the utilisation of the pellets produced, and thus, decreasing the cost for the further treatment.

Influence on pellet chemistry

The influence of coating on the pellet chemistry was exemplified by the different coating materials used in this investigation. The adjustment of the non-ferrous oxides in the pellets can be up to 0.4 % in weight when using a coating amount of 4 kg/ton pellets, as shown in Table 3 (+0.4 % of SiO\textsubscript{2} in the case of quartzite coating and +0.4 % of MgO + SiO\textsubscript{2} in the case of olivine coating). Of course the chemistry adjustment will be even higher if the amount of coating used is increased further. A chemistry modification in this range can be very useful for the flexibility of using different burden mixtures in a blast furnace and utilising available burden materials.

An increased amount of slag formers in the pellets can of course affect the slag volume in the blast furnace process. At a plant where the desire is to reduce the blast furnace slag volume, the use of coated pellets could be a disadvantage. However, for most blast furnaces additional slag formers are presently charged into the furnace from the top. In this case, if coated pellets were used, the target slag volume could still be reached by decreasing the additional charging of lumpy slag formers.

Improved slag formation in the blast furnace

Obviously, the coating materials as fluxes well distributed on the surface of the pellets will give a more homogeneous slag formation, compared with the top charged lumpy slag formers, which results in locally very basic (or acid) conditions. A more homogeneous slag formation is advantageous for the liquid slag flowing from the cohesive zone down to the hearth, thus having a positive effect on the permeability of the lower part of the blast furnace. The decreased variations in BRI, as shown in Table 9 and Figure 8, for the dolomite and quartzite periods, in combination with less clustering formation and less dust generation resulting in a better gas distribution, can be seen as an indication of improved slag formation in the cohesive zone. As for the olivine coated pellet, the slag chemistry in the pellet was less changed by the coating, since most of the pellet addition is also olivine, thereby an improvement in BRI stability was not obtained.

Alkali removal

It was expected that the use of an acid coating material should give a better alkali removal by the slag during the blast furnace operation. However, this expected effect was not verified during the
tests, neither by chemical analyses of probe sample, nor by the alkali content in the blast furnace slag. According to the slag composition, the yield of K\textsubscript{2}O in the slag was generally at the same level for the four first periods, while it was considerably lower for the MPBO-3 period due to higher hot metal and slag temperatures. The alkali contents in the examined probe samples were not significantly different between the coated and uncoated pellets either. The possible reasons are that the alkali load to the furnace was on a comparably low level, only 1.2 and 0.7 kg/tHM of K\textsubscript{2}O and Na\textsubscript{2}O respectively, and that the MPBO pellet in itself already has a high ability to pick up alkali. The difference of some changeable parameters among these trial periods, which are closely related to the removal of K\textsubscript{2}O by the slag, for instance the slag temperature, also brought difficulty to give a clear picture on the issue.

However, another investigation, not yet published, indicated that under certain operating conditions an improved alkali removal could be obtained. In a test carried out in the experiment blast furnace, burdened with a high alkali containing lumpy ore together with uncoated and coated pellets respectively, the use of coated pellets did improve the alkali removal and gave a better operation, resulting in lower fuel rate and higher productivity.

Moisture content of the coated pellets
As mentioned earlier, the use of coating could increase the moisture content of pellets about 1.5%, which would decrease the top gas temperature to a certain extent. For some blast furnaces already operating with a low top gas temperature, a further lowered top gas temperature might be a concerned issue. However, in reality the moisture content in the coated pellets might not be higher, possibly even lower, than that in the uncoated pellets at the customer site. As an example, the coated LKAB DR pellets generally experience a moisture content of 1-1.5 % at the loading port. Before coating was introduced water spraying was regularly used during ship loading in the harbour. Since then, there has been no need of water spraying of coated direct reduction pellets. Therefore, the moisture content in the coated pellets may even be lower than that in uncoated pellets.

Influence on mechanical and metallurgical properties
As was seen in Table 6, most mechanical and metallurgical properties were unaffected by the addition of coating. The LTD values were impaired, which was actually an expected effect of adding water to dry pellets.\textsuperscript{[7]} The reduction in LTD values by water addition has in internal investigations been found to be the worst for fresh pellets, whereas the deteriorating effect decreases with time after production. This is believed to be the reason why the LTD values of the dolomite coated pellets was not decreased as much as the other coated pellets, since the dolomite coating was not performed on newly produced pellets. The decrease in CCS was also an expected effect of water addition to dry pellets. For DR pellets the decreased CCS, caused by coating, has been documented.\textsuperscript{[8]} However, these decreases in CCS and LTD values are not considered crucial for the performance in the blast furnace. Optimising the amounts of coating material and water addition can possibly reduce the deterioration. In fact, the degenerations in CCS and LTD did not
destruct the blast furnace operation in this study. Contrary, as discussed earlier, the dust formation during the test was even much lower.

5.5 SUMMARY OF DISCUSSION
The advantages of using coated blast furnace pellets, as sketched in Figure 1, were exposed during the laboratory and experimental blast furnace trials. All coated blast furnace pellets generated less blast furnace flue dust, and resulted in a higher gas utilisation and a smoother blast furnace operation. The decreased sticking tendency, as shown by laboratory as well as experimental blast furnace test results, was attributed the improved furnace performance, possibly in combination with less dust generation inside the blast furnace.

The increased flexibility in pellet composition changes after production was also verified. The possible improvement in alkali removal was, however, not found in this investigation, but has been observed under other operating conditions. The possible decrease in dusting during handling and transportation of coated blast furnace pellets remains to be verified.
6 CONCLUSIONS

Based on the results of the investigation of the effects of coating of blast furnace pellets the following overall conclusions have been made:

1. The amount of dry flue dust was decreased by 32 up to 50 % when using coated pellets in the blast furnace. The amount of pellet fines in the flue dust were only one third compared to the operation with uncoated pellets.
2. Sticking was largely prevented by the use of coating of pellets. This was verified in different types of laboratory tests, as well as by studying probe samples taken from the experimental blast furnace.
3. Gas utilisation was higher for all coated pellets, 47 to 48 % compared to 46.4 to 46.8 %. The variations in gas utilisation were lower when using coated pellets.

Additional, and more specific, conclusions from this work are:

Coating of blast furnace pellets
- The amount of coating applied to blast furnace pellets can be accurately controlled, within 0.05 weight percent in the slag former oxides of the pellets.
- The coating was found to stay on the pellets well, even after storing, transport, handling and on-size screening. This was verified by chemical analyses as well as by microscopic studies.
- The layer thickness of the coating was determined to be in the range of a few micrometer up to tenths of a millimetre for 4 kg/t pellet of solid coating material. Some areas of the pellet surfaces were not covered.

Laboratory testing of coated pellets
- A new sticking test method, corresponding to blast furnace conditions, has been developed on the basis of the HYL Standard test. The Blast Furnace Sticking test, BFS, included all test parameters of the HYL Standard test except for the reducing gas composition, which was changed from the hydrogen-based mixture to a carbon monoxide/nitrogen mixture with a low hydrogen content, resembling the gas composition in the shaft of the blast furnace.
- A considerable decrease in sticking was achieved by the use of all types of coating materials, in both the BFS and the HYL Standard sticking tests. Sticking index values of 3 to 8 were obtained, which is much below the recommended maximum of 30 for DR pellets and the values of 45 to 50 obtained for uncoated blast furnace pellets.
- The results of the sticking tests were identical with DR and blast furnace gas mixtures, indicating that the sticking index is probably independent of reduction time.
- The coatings of olivine and dolomite gave lower sticking values, compared to quartzite coating.
- Modification of the ISO 7992 reduction-under-load test was used to establish the influence of temperature on the sticking behaviour.
- The tendency of sticking was increased by increasing the test temperature, from 950 °C to 1050 °C. The largest increase was achieved for the uncoated and quartzite coated pellets.
Most mechanical and metallurgical properties given by standardised tests were unaffected by the coating additions. Only the cold compression strength and the low temperature disintegration were worsened to a certain extent, as an effect of water addition to dry pellets. However, both of them are not considered to be detrimental for the operation of the blast furnace.

Coated blast furnace pellets in the blast furnace process

- Mass balances based on chemical analyses of the flue dust and raw materials showed that the coating materials were basically not blown out of the furnace with the flue dust, where less than 4% of the coating materials were found.
- Analyses of the blast furnace sludge indicated that the coating materials did not end up in the sludge either, but stayed on the pellets inside the furnace.
- SEM investigations of probe samples taken from the lower part of the shaft verified that the coating stayed on the pellet surfaces also during and after reduction.
- Three to five times lower contents of iron in the sludge confirmed the conclusion that the dust generation of the pellets was decreased when the coated pellets was used.
- The amounts of coke fines and slag formers were also lower in the flue dust, by 30 to 50%, when using coated pellets. This was attributed to a combined effect of a lower gas velocity, due to lower top gas temperature, and to a better gas distribution with less channelling.
- Probe samples taken from the cohesive region of the furnace proved that sticking was significantly decreased for the coated pellets. The sticking decreased more for the olivine and dolomite coatings than for the quartzite.
- The gas utilisation was better for the coated pellets, also with a lower standard deviation, indicating a smoother blast furnace operation and a potential of getting a lower fuel rate.
- The variation in burden resistance to gas flow was lower, for the dolomite and quartzite coated pellets, indicating an improved slag formation in the cohesive zone, in combination with less sticking and gas channel formation.
- An expected improved alkali yield with an acid coating was not verified during this test.
ACKNOWLEDGEMENTS

Much effort was put into reviewing and commenting of this investigation by Professor emeritus Jitang Ma, which definitely helped in finalising the paper. Dr. Lawrence Hooey, Mr. Lars Norrman, Mr. Mats Hallin, Ms. Anna Dahlstedt and Mr. Dag Thulin of LKAB are sincerely acknowledged for valuable suggestions during this work. Special thanks to Mr. Dan Hallberg for planning and surveying laboratory test work and to Mr. Magnus Andersson for producing the SEM images. Thanks also to the personnel at the metallurgical and chemical laboratories in Malmberget, involved in the laboratory tests and the coating activities, and all LKAB personnel taking part of the experimental blast furnace trials in Luleå.
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Table 5. Sticking indexes of uncoated and coated pellets, after reduction-under-load tests at different temperatures.
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Table 9. Some main process parameters in the experimental blast furnace trials.
Table 10. Hot metal quality (weight per cent), slag rate and composition (weight per cent) in experimental blast furnace trials.
Table 11. Flue dust amounts, composition (weight per cent) and estimated origin.
Table 12. Chemical analyses (weight per cent) of the sludge, collected by a wet electrostatic precipitator, in the experimental blast furnace trials.
### TABLES

**Table 1. Chemical analysis of oxide pellets and coating materials (weight per cent).**

<table>
<thead>
<tr>
<th>Material</th>
<th>MPBO-2</th>
<th>MPBO-3</th>
<th>Olivine</th>
<th>Quartzite</th>
<th>Dolomite</th>
<th>Bentonite</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe (%)</td>
<td>66.6</td>
<td>66.6</td>
<td>5.0</td>
<td>0.3</td>
<td>1.0</td>
<td>3.8</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>1.78</td>
<td>2.00</td>
<td>42.20</td>
<td>98.00</td>
<td>2.00</td>
<td>56.30</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>0.32</td>
<td>0.22</td>
<td>0.80</td>
<td>0.02</td>
<td>29.50</td>
<td>2.83</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>1.48</td>
<td>1.42</td>
<td>49.50</td>
<td>0.09</td>
<td>21.00</td>
<td>3.73</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>0.29</td>
<td>0.29</td>
<td>0.44</td>
<td>1.00</td>
<td>0.37</td>
<td>18.60</td>
</tr>
<tr>
<td>TiO₂ (%)</td>
<td>0.39</td>
<td>0.37</td>
<td>0.03</td>
<td>0.03</td>
<td>0.00</td>
<td>0.83</td>
</tr>
<tr>
<td>MnO (%)</td>
<td>0.06</td>
<td>0.05</td>
<td>0.00</td>
<td>0.01</td>
<td>0.10</td>
<td>0.06</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.02</td>
<td>0.02</td>
<td>0.02</td>
<td>0.29</td>
<td>0.09</td>
<td>0.57</td>
</tr>
<tr>
<td>V₂O₅ (%)</td>
<td>0.26</td>
<td>0.25</td>
<td>0.02</td>
<td>0.01</td>
<td>0.00</td>
<td>0.05</td>
</tr>
<tr>
<td>P₂O₅ (%)</td>
<td>0.017</td>
<td>0.017</td>
<td>0.030</td>
<td>0.011</td>
<td>0.050</td>
<td>0.160</td>
</tr>
</tbody>
</table>

**Table 2. Size distribution of the materials used as coating materials, and of the flue dust from the experimental blast furnace (weight per cent).**

<table>
<thead>
<tr>
<th>Size ranges (mm)</th>
<th>&lt; 0.045</th>
<th>0.045 – 0.063</th>
<th>0.063 – 0.075</th>
<th>0.075 – 0.125</th>
<th>0.125 – 0.250</th>
<th>0.250 – 0.500</th>
<th>&gt; 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Olivine (%)</td>
<td>68</td>
<td>11</td>
<td>5</td>
<td>13</td>
<td>2</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Dolomite (%)</td>
<td>67</td>
<td>13</td>
<td>7</td>
<td>11</td>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Quartzite (%)</td>
<td>70</td>
<td>9</td>
<td>4</td>
<td>10</td>
<td>6</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>Bentonite (%)</td>
<td>65</td>
<td>21</td>
<td>10</td>
<td>3</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Flue dust (%)</td>
<td>9</td>
<td>11</td>
<td>8</td>
<td>24</td>
<td>35</td>
<td>12</td>
<td>1</td>
</tr>
</tbody>
</table>

**Table 3. Compositions of pellets before and after coating (weight per cent). Results shown are a) chemical analysis before coating, b) expected analysis after coating (calculated), c) chemical analysis of pellets after coating, and d) chemical analysis of samples taken at the blast furnace site, i.e. after storing (outside 4 to 6 weeks), transport, handling and on-size screening (+6 mm).**

<table>
<thead>
<tr>
<th>Material</th>
<th>Sample</th>
<th>Coating</th>
<th>SiO₂ (%)</th>
<th>MgO (%)</th>
<th>CaO (%)</th>
<th>Fe (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPBO-3</td>
<td>a) Base material</td>
<td>None</td>
<td>2.00</td>
<td>1.42</td>
<td>0.22</td>
<td>66.60</td>
</tr>
<tr>
<td></td>
<td>b) Theoretical</td>
<td>Olivine</td>
<td>2.16</td>
<td>1.60</td>
<td>0.22</td>
<td>66.33</td>
</tr>
<tr>
<td></td>
<td>c) At pellet plant</td>
<td>Olivine</td>
<td>2.16</td>
<td>1.65</td>
<td>0.26</td>
<td>66.39</td>
</tr>
<tr>
<td></td>
<td>d) At BF site</td>
<td>Olivine</td>
<td>2.15</td>
<td>1.64</td>
<td>0.20</td>
<td>66.44</td>
</tr>
<tr>
<td>MPBO-O</td>
<td>b) Theoretical</td>
<td>Dolomite</td>
<td>2.01</td>
<td>1.50</td>
<td>0.31</td>
<td>66.33</td>
</tr>
<tr>
<td></td>
<td>c) At pellet plant</td>
<td>Dolomite</td>
<td>2.01</td>
<td>1.50</td>
<td>0.38</td>
<td>66.49</td>
</tr>
<tr>
<td></td>
<td>d) At BF site</td>
<td>Dolomite</td>
<td>1.98</td>
<td>1.50</td>
<td>0.29</td>
<td>66.55</td>
</tr>
<tr>
<td>MPBO-D</td>
<td>b) Theoretical</td>
<td>Quartzite</td>
<td>2.37</td>
<td>1.42</td>
<td>0.22</td>
<td>66.33</td>
</tr>
<tr>
<td></td>
<td>c) At pellet plant</td>
<td>Quartzite</td>
<td>2.42</td>
<td>1.40</td>
<td>0.20</td>
<td>66.24</td>
</tr>
<tr>
<td></td>
<td>d) At BF site</td>
<td>Quartzite</td>
<td>2.50</td>
<td>1.44</td>
<td>0.19</td>
<td>66.24</td>
</tr>
</tbody>
</table>

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### Table 4. Results of sticking tests of uncoated and coated pellet samples (* = average of two tests).  

<table>
<thead>
<tr>
<th>Test method</th>
<th>Measured properties</th>
<th>MPBO-3</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>HYL Standard</td>
<td>Sticking index, SI</td>
<td>45</td>
<td>3</td>
<td>3</td>
<td>8*</td>
</tr>
<tr>
<td></td>
<td>Reduction time (min)</td>
<td>60</td>
<td>61</td>
<td>70</td>
<td>66*</td>
</tr>
<tr>
<td>BFS</td>
<td>Sticking index, SI</td>
<td>50*</td>
<td>3*</td>
<td>4*</td>
<td>8*</td>
</tr>
<tr>
<td></td>
<td>Reduction time (min)</td>
<td>160*</td>
<td>163*</td>
<td>147*</td>
<td>169*</td>
</tr>
</tbody>
</table>

1) Test designed for the HYL-processes. Isothermal reduction of 1200 g sample at 950°C to 90 % reduction degree in a gas mixture of 21/14/55/10 % (CO/CO$_2$/H$_2$/N$_2$). After subsequent loading of 1500 g/cm$^2$ for 30 minutes in N$_2$ atmosphere still at 950°C, reduced pellets are cooled down and a drop test performed resulting in a sticking index value (0-100).  

2) Blast Furnace Sticking test, comprising the test parameters of the HYL Standard test modified to blast furnace reducing gas conditions; with a 40/2/58 % (CO/H$_2$/N$_2$) gas mixture.

### Table 5. Sticking indexes of uncoated and coated pellets, after reduction-under-load tests at different temperatures (* = average of two tests).  

<table>
<thead>
<tr>
<th>Test method</th>
<th>Temperature</th>
<th>Measured properties</th>
<th>MPBO-3</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO 7992</td>
<td>1050°C</td>
<td>Sticking index, SI</td>
<td>95*</td>
<td>47*</td>
<td>35*</td>
<td>95*</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Reduction time (min)</td>
<td>73*</td>
<td>75*</td>
<td>75*</td>
<td>83*</td>
</tr>
<tr>
<td>Modified 7992</td>
<td>1000°C</td>
<td>Sticking index, SI</td>
<td>71</td>
<td>17</td>
<td>28</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Reduction time (min)</td>
<td>83</td>
<td>93</td>
<td>92</td>
<td>78</td>
</tr>
<tr>
<td>Modified 7992</td>
<td>950°C</td>
<td>Sticking index, SI</td>
<td>22</td>
<td>3</td>
<td>5</td>
<td>8*</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Reduction time (min)</td>
<td>89</td>
<td>111</td>
<td>109</td>
<td>100*</td>
</tr>
</tbody>
</table>

1) Standardised reduction-under-load test.[3] Isothermal reduction of 1200 g sample at 1050°C to 80 % reduction degree in a gas mixture of 40/2/58 % (CO/H$_2$/N$_2$), with a load of 500 g/cm$^2$ applied. The standardised test was followed by a drop test to get a sticking index value.  

2) Modification of test temperature to examine influence of temperature on sticking values.

### Table 6. Mechanical and metallurgical test results of oxide pellets and coated pellets.  

<table>
<thead>
<tr>
<th>ISO no.</th>
<th>Cold compression strength (daN/pellet)</th>
<th>Tumble strength (% +6.3 mm)</th>
<th>Abrasion (% -0.5 mm)</th>
<th>Low Temp Disintegration (% +6.3 mm)</th>
<th>Reducibility, R40 (%O/min)</th>
<th>ITH (% +6.3 mm)</th>
<th>Pressure drop, Dp (mmH$_2$O)</th>
<th>Bed shrinkage (%)</th>
<th>MPBO-3</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ISO 4700</td>
<td></td>
<td></td>
<td>ISO 3271</td>
<td></td>
<td>ISO 4695</td>
<td></td>
<td>ISO 7992</td>
<td>232</td>
<td>203</td>
<td>215</td>
<td>219</td>
</tr>
<tr>
<td></td>
<td>95.0</td>
<td></td>
<td>4.5</td>
<td>67.7</td>
<td>0.52</td>
<td>71.8</td>
<td>12.9</td>
<td>6.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>95.2</td>
<td></td>
<td>4.4</td>
<td>96.9</td>
<td>0.53</td>
<td>74.8</td>
<td>9.7</td>
<td>3.6</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>95.0</td>
<td></td>
<td>4.4</td>
<td>67.3</td>
<td>0.56</td>
<td>68.4</td>
<td>12.2</td>
<td>6.2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>94.6</td>
<td></td>
<td>4.8</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1) 3 kg sample (less than ISO 3271, where 15 kg samples are tested).  

2) Strength after reduction (reduced material from ISO 4695 is mechanically treated and sieved).

### Table 7. Blast furnace operating parameters during the trials.  

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Duration (h)</td>
<td>85</td>
<td>83</td>
<td>48</td>
<td>68</td>
<td>27</td>
</tr>
<tr>
<td>Blast temperature (°C)</td>
<td>1198</td>
<td>1197</td>
<td>1198</td>
<td>1197</td>
<td>1197</td>
</tr>
<tr>
<td>Wind rate (Nm$^3$/h)</td>
<td>1590</td>
<td>1589</td>
<td>1591</td>
<td>1590</td>
<td>1570</td>
</tr>
<tr>
<td>Coal injection, PCI (kg/tHM)</td>
<td>133</td>
<td>131</td>
<td>123</td>
<td>127</td>
<td>122</td>
</tr>
<tr>
<td>Oxygen enrichment (%)</td>
<td>3.3</td>
<td>3.4</td>
<td>3.5</td>
<td>3.4</td>
<td>3.4</td>
</tr>
<tr>
<td>Blast moisture (g/Nm$^3$)</td>
<td>26</td>
<td>26</td>
<td>27</td>
<td>27</td>
<td>27</td>
</tr>
<tr>
<td>Flame temp. (calculated, °C)</td>
<td>2188</td>
<td>2195</td>
<td>2201</td>
<td>2201</td>
<td>2204</td>
</tr>
<tr>
<td>Top pressure (bar, gauge)</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
</tbody>
</table>
### Table 8. Moisture contents of pellets and amounts of slag formers charged in experimental blast furnace trials.

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pellet moisture (%)</td>
<td>0.1</td>
<td>2.1</td>
<td>2.2</td>
<td>2.3</td>
<td>2.2</td>
</tr>
<tr>
<td>Limestone (kg/tHM)</td>
<td>48</td>
<td>48</td>
<td>49</td>
<td>49</td>
<td>49</td>
</tr>
<tr>
<td>BOF-slag (kg/tHM)</td>
<td>45</td>
<td>41</td>
<td>42</td>
<td>48</td>
<td>48</td>
</tr>
<tr>
<td>Quartzite (kg/tHM)</td>
<td>17</td>
<td>15</td>
<td>17</td>
<td>11</td>
<td>17</td>
</tr>
<tr>
<td>Coke rate (kg/tHM)</td>
<td>408</td>
<td>410</td>
<td>414</td>
<td>421</td>
<td>430</td>
</tr>
</tbody>
</table>

### Table 9. Some main process parameters in the experimental blast furnace trials.

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas utilisation, $\eta_{CO}$ (%)</td>
<td>46.4</td>
<td>47.2</td>
<td>48.2</td>
<td>47.0</td>
<td>46.8</td>
</tr>
<tr>
<td>St.dev. $\eta_{CO}$</td>
<td>1.5</td>
<td>0.9</td>
<td>0.9</td>
<td>0.7</td>
<td>0.8</td>
</tr>
<tr>
<td>Utilisation of hydrogen, $\eta_{H2}$ (%)</td>
<td>54.7</td>
<td>56.2</td>
<td>55.8</td>
<td>56.6</td>
<td>56.1</td>
</tr>
<tr>
<td>Burden resistance index, BRI 2)</td>
<td>8.7</td>
<td>9.0</td>
<td>8.3</td>
<td>8.3</td>
<td>8.9</td>
</tr>
<tr>
<td>St.dev. BRI</td>
<td>0.5</td>
<td>0.5</td>
<td>0.2</td>
<td>0.2</td>
<td>0.3</td>
</tr>
<tr>
<td>Descent rate (cm/min)</td>
<td>5.6</td>
<td>5.6</td>
<td>5.7</td>
<td>5.4</td>
<td>5.9</td>
</tr>
<tr>
<td>St.dev. Descent rate</td>
<td>0.6</td>
<td>0.4</td>
<td>0.5</td>
<td>0.5</td>
<td>0.4</td>
</tr>
<tr>
<td>Top gas temperature (°C)</td>
<td>200</td>
<td>183</td>
<td>177</td>
<td>191</td>
<td>191</td>
</tr>
<tr>
<td>Production (tHM/h)</td>
<td>1.45</td>
<td>1.45</td>
<td>1.49</td>
<td>1.42</td>
<td>1.43</td>
</tr>
<tr>
<td>Productivity (tHM/m²/24h) 3)</td>
<td>30.7</td>
<td>30.8</td>
<td>31.7</td>
<td>30.2</td>
<td>30.3</td>
</tr>
<tr>
<td>Fuel rate (kg/tHM)</td>
<td>541</td>
<td>541</td>
<td>537</td>
<td>549</td>
<td>552</td>
</tr>
</tbody>
</table>

1) Standard deviation $\sigma = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n(n-1)}}$

2) Equals PV bosh $= \frac{(\text{blast pressure})^2 - (\text{top pressure})^2}{(\text{bosh gas volume})^{1.7} \times \text{constant}}$

3) Utilisation of furnace volume (working volume from stockline to tuyere level).

4) Output per unit hearth area.

### Table 10. Hot metal quality (weight per cent), slag rate and composition (weight per cent) in experimental blast furnace trials.

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>No. of tappings</td>
<td>67</td>
<td>77</td>
<td>44</td>
<td>60</td>
<td>25</td>
</tr>
<tr>
<td>Hot metal temp (°C)</td>
<td>1413</td>
<td>1422</td>
<td>1400</td>
<td>1398</td>
<td>1446</td>
</tr>
<tr>
<td>C (%)</td>
<td>4.39</td>
<td>4.41</td>
<td>4.32</td>
<td>4.35</td>
<td>4.48</td>
</tr>
<tr>
<td>Si (%)</td>
<td>1.20</td>
<td>1.55</td>
<td>1.32</td>
<td>1.43</td>
<td>1.73</td>
</tr>
<tr>
<td>C/T (×1000)</td>
<td>3.11</td>
<td>3.10</td>
<td>3.08</td>
<td>3.11</td>
<td>3.10</td>
</tr>
<tr>
<td>Si/T (×1000)</td>
<td>0.85</td>
<td>1.09</td>
<td>0.94</td>
<td>1.02</td>
<td>1.20</td>
</tr>
<tr>
<td>Slag rate (kg/tHM)</td>
<td>151</td>
<td>147</td>
<td>150</td>
<td>152</td>
<td>147</td>
</tr>
<tr>
<td>Basicity, B2 (CaO/SiO₂)</td>
<td>0.90</td>
<td>0.88</td>
<td>0.84</td>
<td>0.87</td>
<td>0.97</td>
</tr>
<tr>
<td>Optical basicity, $\Lambda$ 1)</td>
<td>0.664</td>
<td>0.664</td>
<td>0.660</td>
<td>0.661</td>
<td>0.670</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>36</td>
<td>36</td>
<td>37</td>
<td>36</td>
<td>34</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>32</td>
<td>31</td>
<td>31</td>
<td>31</td>
<td>33</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>17</td>
<td>19</td>
<td>18</td>
<td>17</td>
<td>18</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>13</td>
<td>13</td>
<td>13</td>
<td>13</td>
<td>14</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.47</td>
<td>0.40</td>
<td>0.52</td>
<td>0.48</td>
<td>0.29</td>
</tr>
</tbody>
</table>

1) Optical basicity, $\Lambda$ 6)

$$\Lambda = \frac{\sum x_i n_i \Lambda_{i01} + x_i n_2 \Lambda_{i02} + \ldots}{\sum x_i n_1 + x_i n_2 + \ldots}$$

where

$x_i$ = mole ratio of oxide $i$

$n_i$ = number of oxygen atoms in oxide molecule $i$

$\Lambda_{i0j} = $ optical basicity of oxide $i$
Table 11. Flue dust amounts, composition (weight per cent) and estimated origin.

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flue dust, dry (kg/tHM)</td>
<td>5.4</td>
<td>2.9</td>
<td>2.7</td>
<td>3.0</td>
<td>4.4</td>
</tr>
<tr>
<td>Fe (%)</td>
<td>21.6</td>
<td>13.8</td>
<td>n.a.</td>
<td>13.3</td>
<td>21.8</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>11.1</td>
<td>15.9</td>
<td>n.a.</td>
<td>20.8</td>
<td>17.7</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>16.2</td>
<td>14.1</td>
<td>n.a.</td>
<td>12.1</td>
<td>14.2</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>4.3</td>
<td>9.2</td>
<td>n.a.</td>
<td>6.3</td>
<td>6.8</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>3.0</td>
<td>4.2</td>
<td>n.a.</td>
<td>4.0</td>
<td>4.0</td>
</tr>
<tr>
<td>TiO₂ (%)</td>
<td>0.2</td>
<td>0.2</td>
<td>n.a.</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>MnO (%)</td>
<td>0.3</td>
<td>0.4</td>
<td>n.a.</td>
<td>0.4</td>
<td>0.3</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.3</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.4</td>
<td>0.6</td>
</tr>
<tr>
<td>C (%)</td>
<td>20.4</td>
<td>26.0</td>
<td>n.a.</td>
<td>31.2</td>
<td>16.5</td>
</tr>
<tr>
<td>From pellets (kg/tHM)</td>
<td>1.5</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.5</td>
<td>1.3</td>
</tr>
<tr>
<td>From coke (kg/tHM)</td>
<td>1.4</td>
<td>0.9</td>
<td>n.a.</td>
<td>1.1</td>
<td>0.9</td>
</tr>
<tr>
<td>From limestone (kg/tHM)</td>
<td>1.0</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.4</td>
<td>0.8</td>
</tr>
<tr>
<td>From BOF-slag (kg/tHM)</td>
<td>1.0</td>
<td>0.5</td>
<td>n.a.</td>
<td>0.5</td>
<td>0.7</td>
</tr>
<tr>
<td>From quartzite (kg/tHM)</td>
<td>0.5</td>
<td>0.3</td>
<td>n.a.</td>
<td>0.3</td>
<td>0.7</td>
</tr>
<tr>
<td>From olivine coating (kg/tHM)</td>
<td>-</td>
<td>0.2</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>From quartzite coating (kg/tHM)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.2</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 12. Chemical analyses (weight per cent) of the sludge, collected by a wet electrostatic precipitator, in the experimental blast furnace trials.

<table>
<thead>
<tr>
<th>Period</th>
<th>MPBO-2</th>
<th>MPBO-O</th>
<th>MPBO-D</th>
<th>MPBO-Q</th>
<th>MPBO-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe (%)</td>
<td>6.2</td>
<td>2.4</td>
<td>1.6</td>
<td>1.1</td>
<td>n.a.</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>19.2</td>
<td>20.2</td>
<td>22.6</td>
<td>18.2</td>
<td>n.a.</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>8.8</td>
<td>7.3</td>
<td>8.0</td>
<td>7.4</td>
<td>n.a.</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>8.7</td>
<td>10.3</td>
<td>14.7</td>
<td>10.7</td>
<td>n.a.</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>6.1</td>
<td>6.6</td>
<td>8.4</td>
<td>8.3</td>
<td>n.a.</td>
</tr>
<tr>
<td>TiO₂ (%)</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>0.1</td>
<td>n.a.</td>
</tr>
<tr>
<td>MnO (%)</td>
<td>0.6</td>
<td>0.5</td>
<td>0.7</td>
<td>0.5</td>
<td>n.a.</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>1.2</td>
<td>1.1</td>
<td>1.0</td>
<td>0.7</td>
<td>n.a.</td>
</tr>
<tr>
<td>Na₂O (%)</td>
<td>10.4</td>
<td>9.2</td>
<td>6.5</td>
<td>7.7</td>
<td>n.a.</td>
</tr>
<tr>
<td>V₂O₅ (%)</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
<td>0.1</td>
<td>n.a.</td>
</tr>
<tr>
<td>P₂O₅ (%)</td>
<td>0.1</td>
<td>0.2</td>
<td>0.2</td>
<td>0.1</td>
<td>n.a.</td>
</tr>
<tr>
<td>C (%)</td>
<td>16.0</td>
<td>17.0</td>
<td>11.8</td>
<td>12.3</td>
<td>n.a.</td>
</tr>
<tr>
<td>S (%)</td>
<td>0.3</td>
<td>0.2</td>
<td>0.1</td>
<td>0.2</td>
<td>n.a.</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

Figure 1. Schematic view of possible effects of using coated pellets in the blast furnace.
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Figure 4. SEM image of surface layer of olivine coated pellet. On left particle the coating layer (dark grey) is from zero up to 0.100 mm in thickness, the right particle surface not covered by coating. Scale mark is 600 μm.
Figure 5. SEM image of surface layer of olivine coated pellet. Coating layer (dark grey) has been collected in a cavity of roughly 0.5 mm in depth. Scale mark is 600 μm.
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Figure 10. SEM image of surface layer of reduced olivine coated pellet, collected by a shaft probe, 3.4 m below stockline. Inside the coating layer (dark grey) is reduced iron (reduction degree 70 %). Scale mark is 100 μm.
Figure 11. Probe samples taken from the cohesive region of the blast furnace, uncoated pellets.
Figure 12. Probe samples taken from the cohesive region of the blast furnace, olivine coated pellets.
Figure 13. Probe samples taken from the cohesive region of the blast furnace, dolomite coated pellets.
Figure 14. Probe samples taken from the cohesive region of the blast furnace, quartzite coated pellets.
Lower dust generation
Higher gas utilisation

Less clustering
Less gas channel formation
Better slag formation

Improved alkali removal

Lower pressure drop
Higher productivity
Lower fuel rate

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Upper sample: 95% reduction degree, 0.4% C. 
Lower sample: 98% reduction degree, 1.9% C.

Figure 12. Probe samples taken from the cohesive region of the blast furnace, olivine coated pellets. 
Upper sample: 94% reduction degree, 0.6% C. 
Lower sample: 97% reduction degree, 1.9% C.
Figure 13. Probe samples taken from the cohesive region of the blast furnace, dolomite coated pellets.
Upper sample: 91 % reduction degree, 0.3 % C.
Lower sample: 98 % reduction degree, 1.7 % C.

Figure 14. Probe samples taken from the cohesive region of the blast furnace, quartzite coated pellets.
Upper sample: 95 % reduction degree, 0.2 % C.
Lower sample: 96 % reduction degree, 0.8 % C.