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

Doctoral thesis

by

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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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*The use of coated pellets in optimising the blast furnace operation*
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Contributions to the supplements by the author:

**Supplements I and II:**
- Laboratory experiments: 100%
- Evaluation of experiments: 80%
- Writing: 90%

**Supplement III:**
- Exp. blast furnace trials: Participation
- Commercial scale trials: Participation
- Evaluation of trials: 80%
- Writing: 60%

**Supplement IV:**
- EBF trials: Participation
- Evaluation of trials: 80%
- Writing: 80%

**Supplement V:**
- Laboratory tests: Ordering and observing
- EBF trials: Project leader
- Evaluation: 80%
- Writing: 75%

The author also contributed to the following works:

Lawrence Hooey, Jerker Sterneeland and Mats Hallin:
*Evaluation of operational data from the LKAB experimental blast furnace*
Presented at the 60th Ironmaking Conference, Baltimore, MD, USA, March 2001.

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*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 Sterneeland and Dag Thulin:
*LKAB:s experimental blast furnace and pellet development*
Submitted for publication in La revue de Métallurgie, Association Technique de la Siderurgie Francaise (ATS), February 2002.

Jerker Sterneeland and Magnus Andersson:
*Evaluation of reducibility and reduction strength of burden materials taken from the LKAB experimental blast furnace*
Presented at the 61st Ironmaking Conference, Nashville, TN, USA, March 2002.
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Stockholm, May 2002

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  Jerker Sterneland and Mats Hallin

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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.

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.
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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
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.\[3\]

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.\[4\] 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.\[5\]
- Clixby defined a critical zone of temperature and reduction in which a rapid rise in differential pressure is experienced by the sample.\[6\]
- Yamaoka et al defined an average permeability resistance coefficient over the softening and melting temperature range.\[7\]
- Gudenau et al calculated a dimensionless pressure parameter to represent the differential pressure versus temperature curve during the entire test.\[6\]

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\[2,5,6,8-15\] are listed.
Table 2.1. Specifications of test procedures found in literature.

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

<table>
<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 pattern</th>
<th>% CO</th>
<th>% CO₂</th>
<th>% H₂ (Nm/min)</th>
<th>Flow rate (Nm/min)</th>
<th>Load (kg/cm²)</th>
</tr>
</thead>
<tbody>
<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>Constant</td>
<td>30</td>
<td>-</td>
<td>-</td>
<td>15</td>
<td>1.4</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>Constant</td>
<td>30</td>
<td>-</td>
<td>-</td>
<td>7.2</td>
<td>1</td>
</tr>
<tr>
<td>British Steel, Teesside, Gr. Britain [6,10,15]</td>
<td>800</td>
<td>90</td>
<td>66</td>
<td></td>
<td>Constant</td>
<td>3,3,5,6</td>
<td>Constant</td>
<td>40</td>
<td>-</td>
<td>-</td>
<td>60</td>
<td>0.5</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>100</td>
<td>42</td>
<td>35</td>
<td>Yes</td>
<td>Constant</td>
<td>10</td>
<td>Constant</td>
<td>30</td>
<td>-</td>
<td>-</td>
<td>5.4</td>
<td>0.5</td>
</tr>
<tr>
<td>Kobe, Japan [6]</td>
<td>500</td>
<td>75</td>
<td>50</td>
<td></td>
<td>Constant</td>
<td>10</td>
<td>Constant</td>
<td>30</td>
<td>-</td>
<td>-</td>
<td>15</td>
<td>2</td>
</tr>
<tr>
<td>MEFOS, Luleå, Sweden [2,6]</td>
<td>500</td>
<td>80</td>
<td>70</td>
<td></td>
<td>Constant</td>
<td>3</td>
<td>Constant</td>
<td>30</td>
<td>-</td>
<td>-</td>
<td>300</td>
<td>0.5</td>
</tr>
<tr>
<td>Nippon Kokan, Japan [6]</td>
<td>350</td>
<td>70</td>
<td>40</td>
<td></td>
<td>Constant</td>
<td>5</td>
<td>Constant</td>
<td>30</td>
<td>-</td>
<td>-</td>
<td>15</td>
<td>0.5</td>
</tr>
<tr>
<td>Nippon Steel, Hirohata, Japan [5,6]</td>
<td>105</td>
<td>70</td>
<td>70</td>
<td></td>
<td>Constant</td>
<td>5, 5,6 or 2.8</td>
<td>Constant</td>
<td>30</td>
<td>-</td>
<td>-</td>
<td>30</td>
<td>0.9</td>
</tr>
<tr>
<td>CSM, Italy [6]</td>
<td>500</td>
<td>70</td>
<td></td>
<td></td>
<td>Constant</td>
<td>3</td>
<td>Only N₂, pre-reduced sample</td>
<td></td>
<td>50</td>
<td>Constant 0.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>USSR [13]</td>
<td>30</td>
<td>60</td>
<td></td>
<td></td>
<td>Constant</td>
<td>10</td>
<td>Only Ar, pre-reduced sample</td>
<td></td>
<td>low</td>
<td>Constant 2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Burghardt, Germany [6]</td>
<td>1200</td>
<td>125</td>
<td></td>
<td></td>
<td>Isothermal</td>
<td>-</td>
<td>Constant</td>
<td>40</td>
<td>-</td>
<td>-</td>
<td>83</td>
<td>0.5</td>
</tr>
<tr>
<td>Hoogovens [6]</td>
<td>500</td>
<td>71</td>
<td>88</td>
<td></td>
<td>Isothermal</td>
<td>-</td>
<td>Constant</td>
<td>40</td>
<td>-</td>
<td>-</td>
<td>30</td>
<td>0.8</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 pattern</th>
<th>% CO</th>
<th>% CO₂</th>
<th>% H₂ (Nm/min)</th>
<th>Flow rate (Nm/min)</th>
<th>Load (kg/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Centre de Res. Minerales, Canada [8]</td>
<td>150</td>
<td>42</td>
<td>60</td>
<td>?</td>
<td>Constant</td>
<td>6,7</td>
<td>Var. 4 steps 18-40</td>
<td>0-22</td>
<td>-</td>
<td>-</td>
<td>9</td>
<td>0.8</td>
</tr>
<tr>
<td>Dofasco Hamilton, Ontario, Canada [8]</td>
<td>500</td>
<td>75</td>
<td>32/32</td>
<td></td>
<td>Constant</td>
<td>5</td>
<td>Var. 4 steps 18-40</td>
<td>0-22</td>
<td>-</td>
<td>-</td>
<td>20</td>
<td>0.5/1</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)</th>
<th>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 30</td>
<td>15</td>
</tr>
<tr>
<td>Centro Guilluppo 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 40</td>
<td>0,5</td>
</tr>
<tr>
<td>CNRM [6]</td>
<td></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 30</td>
<td>2</td>
</tr>
<tr>
<td>Kobe, Japan [6]</td>
<td>3 particles</td>
<td>42</td>
<td>mono layer</td>
<td>Varying</td>
<td>8.8/0/6.6</td>
<td>1500</td>
<td>Constant 30</td>
<td>-</td>
<td>2</td>
</tr>
<tr>
<td>Nippon Steel, Japan [6]</td>
<td>500</td>
<td>70</td>
<td>60</td>
<td>Varying</td>
<td>5/3/5</td>
<td>1400</td>
<td>Constant 30</td>
<td>-</td>
<td>20</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>-</td>
<td>33</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>-</td>
<td>33</td>
</tr>
<tr>
<td>Tamman, Brazil [8]</td>
<td>500</td>
<td>60</td>
<td>120</td>
<td>20/20</td>
<td>Varying</td>
<td>10/4,3/2</td>
<td>1600</td>
<td>Constant 30</td>
<td>15</td>
</tr>
<tr>
<td>Usiminas 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 30</td>
<td>15</td>
</tr>
</tbody>
</table>

#### Group 4: 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)</th>
<th>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>mono layer</td>
<td>Varying</td>
<td>6/4/3/2</td>
<td>1320</td>
<td>Var. 8 steps</td>
<td>26-36</td>
<td>14</td>
</tr>
<tr>
<td>British Steel, Teeside, Gr. Britain [6,11]</td>
<td>350</td>
<td>60</td>
<td>60</td>
<td>Varying</td>
<td>3/4/6</td>
<td>1600</td>
<td>Var. 4 steps</td>
<td>25-45</td>
<td>15</td>
</tr>
<tr>
<td>ISCOR Research and proc. developm. [8]</td>
<td>400-750</td>
<td>80</td>
<td>60</td>
<td>25/25</td>
<td>Varying</td>
<td>5/7/1,3/5</td>
<td>Var. 3 steps</td>
<td>22-41</td>
<td>40</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</td>
<td>Var. steps</td>
<td>25-50</td>
<td>38</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>2000</td>
<td>100</td>
<td>230</td>
<td>Yes</td>
<td>Varying BF sim.</td>
<td>5/7/1,3/5</td>
<td>Var. steps</td>
<td>25-50</td>
<td>38</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>1000</td>
<td>100</td>
<td>90</td>
<td>Yes</td>
<td>Varying BF sim.</td>
<td>5/7/1,3/5</td>
<td>Var. steps</td>
<td>25-50</td>
<td>38</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>600-700</td>
<td>75</td>
<td>70</td>
<td>Yes</td>
<td>Varying BF sim.</td>
<td>5/7/1,3/5</td>
<td>Var. steps</td>
<td>25-50</td>
<td>38</td>
</tr>
<tr>
<td>Japanese laboratory [8]</td>
<td>500</td>
<td>70</td>
<td>70</td>
<td>Yes</td>
<td>Varying BF sim.</td>
<td>5/7/1,3/5</td>
<td>Var. steps</td>
<td>25-50</td>
<td>38</td>
</tr>
<tr>
<td>LKAB Malmberget, Sweden [2,8,14]</td>
<td>300</td>
<td>50</td>
<td>50</td>
<td>No</td>
<td>Varying Lin. BF sim.</td>
<td>1300</td>
<td>Var. line</td>
<td>24-40</td>
<td>25</td>
</tr>
<tr>
<td>Nippon Kokan, Fukuyama, Japan [6,8]</td>
<td>1400</td>
<td>120</td>
<td>65</td>
<td>Varying</td>
<td>9/0/8/5</td>
<td>1700</td>
<td>Var. steps</td>
<td>23-45</td>
<td>90</td>
</tr>
<tr>
<td>Sumitomo, Japan [6,8]</td>
<td>700</td>
<td>70</td>
<td>100</td>
<td>Varying</td>
<td>12,5/2,2/5</td>
<td>1500</td>
<td>Var. steps</td>
<td>20-45</td>
<td>50</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,\[^{16}\] 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.\[^{6,10,17}\]

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\textsubscript{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
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>
<thead>
<tr>
<th>British Steel, Great Britain</th>
<th>CRM, Belgium</th>
<th>US Bureau of Mines, USA</th>
<th>US Steel, USA</th>
<th>ICM, China</th>
<th>ICM, China</th>
<th>Tokyo University, Japan</th>
<th>NKK, Japan</th>
<th>Sumitomo, Japan</th>
<th>Nippon Steel, Japan</th>
<th>Kawasaki, Japan</th>
<th>LKAB, Sweden</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Working volume (m\textsuperscript{3})</strong></td>
<td>8.6</td>
<td>8.6</td>
<td>9.6</td>
<td>17.5</td>
<td>0.8</td>
<td>0.8</td>
<td>3.9</td>
<td>0.9</td>
<td>0.3</td>
<td>8.2</td>
<td></td>
</tr>
<tr>
<td><strong>Hearth diameter (m)</strong></td>
<td>0.5</td>
<td>1.4</td>
<td>1.4</td>
<td>1.2</td>
<td>1.5</td>
<td>0.6</td>
<td>1.0</td>
<td>0.9</td>
<td>R = 0.6\textsuperscript{2}</td>
<td>0.4</td>
<td>1.2</td>
</tr>
<tr>
<td><strong>Tuyeres</strong></td>
<td>2</td>
<td>5</td>
<td>3</td>
<td>3</td>
<td>4</td>
<td>4</td>
<td>3</td>
<td>3</td>
<td>1</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
<td><strong>Blast temp. (\textdegree C)</strong></td>
<td>800</td>
<td>1000</td>
<td>1360</td>
<td>980</td>
<td>1250</td>
<td>800</td>
<td>850</td>
<td>890</td>
<td>1050</td>
<td>1300</td>
<td>800</td>
</tr>
<tr>
<td><strong>Top pressure (bar, gauge)</strong></td>
<td>-</td>
<td>1.5</td>
<td>0.2</td>
<td>-</td>
<td>2.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.5</td>
</tr>
<tr>
<td><strong>Productivity (tHM/m\textsuperscript{3} x 24h)</strong></td>
<td>1.7</td>
<td>4.3</td>
<td>5</td>
<td>6.4</td>
<td>4.0</td>
<td>2.5</td>
<td>7.4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Fuel rate (kg/tHM)</strong></td>
<td>800</td>
<td>560</td>
<td>580</td>
<td>600</td>
<td>600</td>
<td>700</td>
<td>680</td>
<td>670</td>
<td>670</td>
<td>620</td>
<td>530</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]}
CRM, Ougrée, Belgium
An oval shaped furnace built in 1953 was in 1965 rebuilt to an 8.6 m³ 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]\n
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]\n
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]\n
ICM, Beijing, China
A 17.5 m³ 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]\n
Tokyo University, Tokyo, Japan
The working volume of the furnace was originally 0.5 m³, but after rebuilding in 1970 it was enlarged to 0.8 m³. 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]\n
NKK, Kawasaki, Japan
A small furnace of 0.6 m³ working volume was first installed in 1967. In 1970 the furnace was rebuilt and enlarged to 0.8 m hearth diameter and 3.2 m³ working volume, and has after that been further enlarged to 1.0 m and 3.9 m³ 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]\n
Sumitomo, Kokura, Japan
Originally, this was an experimental smelting furnace. It was rebuilt to an experimental blast furnace
Some aspects on the reduction of olivine pellets in laboratory scale and in 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.\[25,26\]

_Nippon Steel, Kimitsu, Japan_

The experimental blast furnace of Nippon Steel is a 90° 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.\[22\]

_Kawasaki, Chiba, Japan_

A small, single tuyere, experimental blast furnace of 0.3 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 down sizing 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.
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Figure 3.3. The NKK experimental blast furnace, before and after enlargement in 1970.\textsuperscript{[23]}

Figure 3.4. The CRM experimental blast furnace, 1965-72.\textsuperscript{[18]}
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:

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

(4.1)

where \( m_{\text{Acc Rem O}, \text{Rem}} \) = accumulated mass of oxygen removed from sample (g); and \( m_{\text{O}, \text{Ori}} \) = 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 metallographical 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 \( i \), \( R_i \):
\[ R_i (\%) = v_{\text{Magnetite}} \times R_{\text{Magnetite}} + v_{\text{Wüstite}} \times R_{\text{Wüstite}} + v_{\text{Iron}} \times R_{\text{Iron}} \] (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 CO, CO\(_2\) and N\(_2\) is, for simplicity, first dealt with. After that, the presence of 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 CO/CO\(_2\)/N\(_2\) gas mixture is at any time given by:

\[ \dot{m}_o = M_o \times \nu_{\text{CO}} + 2 \times M_o \times \nu_{\text{CO}_2} = (\nu_{\text{CO}} + 2 \times \nu_{\text{CO}_2}) \times M_o \] (4.3)

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

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

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

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

\[ \nu_i = \frac{\theta_i}{V_o} \] (4.4)

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

\[ \dot{m}_o = \left( \frac{\theta_{\text{CO}}}{V_o} + 2 \times \frac{\theta_{\text{CO}_2}}{V_o} \right) \times M_o = (\theta_{\text{CO}} + 2 \times \theta_{\text{CO}_2}) \times \frac{M_o}{V_o} \] (4.5)

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

\[ \theta_i = \frac{[i] \%}{100} \times \theta \] (4.6)
where $[\%i]$ is the volume percentage of species $i$ and $\theta$ is the total volume flow of gas. This gives:

$$\dot{m}_o = \left( \frac{[\%CO]}{100} \times \theta + 2 \times \frac{[\%CO_2]}{100} \times \theta \right) \frac{M_o}{V_o} = \left( \frac{[\%CO] + 2 \times [\%CO_2]}{100} \right) \frac{\theta \times M_o}{100 \times V_o}$$

(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):

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

(4.8)

This relation applied to equation (4.7) gives:

$$\dot{m}_o = \left( \frac{[\%CO] + 2 \times [\%CO_2]}{[\% N_2]} \right) \frac{\theta_{N_2} \times M_o}{[\% N_2] \times V_o}$$

(4.9)

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

$$\dot{m}_{O, Rem} = \dot{m}_{O, Outlet} - \dot{m}_{O, Inlet}$$

$$= \left( [\%CO]_{Outlet} + 2 \times [\%CO_2]_{Outlet} \right) \frac{\theta_{N_2} \times M_o}{[\% N_2]_{Outlet} \times V_o}$$

$$- \left( [\%CO]_{Inlet} + 2 \times [\%CO_2]_{Inlet} \right) \frac{\theta_{N_2} \times M_o}{[\% N_2]_{Inlet} \times V_o}$$

$$= \left( \frac{[\%CO]_{Outlet} + 2 \times [\%CO_2]_{Outlet}}{[\% N_2]_{Outlet}} - \frac{[\%CO]_{Inlet} + 2 \times [\%CO_2]_{Inlet}}{[\% N_2]_{Inlet}} \right) \frac{\theta_{N_2} \times M_o}{V_o}$$

(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

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

\[
- \left( \frac{[\%CO]_{\text{Inlet}} + 2 \times [\%CO_2]_{\text{Inlet}} + [\%H_2O]_{\text{Inlet}}}{[\%N_2]_{\text{Inlet}}} \right) \times \theta_{N_2} \times M_O \div V_o
\]

(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\(_2O\) by H\(_2\) in equation (4.11):

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

\[
\theta_{H_2}^{\text{Inlet}} + \theta_{H_2O}^{\text{Inlet}} = \theta_{H_2}^{\text{Outlet}} + \theta_{H_2O}^{\text{Outlet}}
\]

(4.12)

The inlet flow of H\(_2O\), \(\theta_{H_2O}^{\text{Inlet}}\), is assumed to be zero. Using equations (4.6) and (4.8) together with (4.12) a suitable expression for the H\(_2O\) content of the outlet gas is obtained:

\[
\frac{[\%H_2O]_{\text{Outlet}}}{[\%N_2]_{\text{Outlet}}} = \frac{[\%H_2]_{\text{Outlet}}}{[\%N_2]_{\text{Outlet}}} = \frac{[\%H_2]_{\text{Inlet}}}{[\%N_2]_{\text{Inlet}}}
\]

(4.13)

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

\[
\dot{m}_{O,\text{Rem}} = \left( \frac{[\%CO]_{\text{Outlet}} + 2 \times [\%CO_2]_{\text{Outlet}} + [\%H_2O]_{\text{Outlet}}}{\text{Outlet}} - \frac{[\%H_2]_{\text{Outlet}}}{[\%N_2]_{\text{Outlet}}} \right)
\]

\[
- \left( \frac{[\%CO]_{\text{Inlet}} + 2 \times [\%CO_2]_{\text{Inlet}} + [\%H_2O]_{\text{Inlet}}}{[\%N_2]_{\text{Inlet}}} \right) \times \theta_{N_2} \times M_O \div V_o
\]

(4.14)

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

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

(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, \(\dot{m}_{O,\text{Rem}}\), and the time, \(t\), of the increment
under consideration:

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

(4.16)

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

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

(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:

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

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

(4.18)

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

\[ m_{C, Dep} = \int_0^t (\dot{m}_{C, Dep}) \times dt \]  

(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:

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

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

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.
4.5 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>SiO₂</th>
<th>CaO</th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>TiO₂</th>
<th>MnO</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>V₂O₅</th>
<th>P₂O₅</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
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

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.

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.
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.[16] 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.[38] 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.[7] 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.[6] 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.[38] and all carbon dioxide formed by reduction of iron oxides will be reduced back to CO by carbon according to the Boudouard reaction (CO₂ + C → 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.
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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\textsubscript{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)
Some aspects on the reduction of olivine pellets in laboratory scale and in an experimental blast furnace

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.
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 I11-17, 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.

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.

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.

![Figure 6.8](image)

Figure 6.8. Bed contraction schematically shown in three separate stages.

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

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.

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>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>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.\cite{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. Internal denomination (LKAB)</th>
<th>Campaign A PMK1</th>
<th>Campaign B PMK4</th>
<th>Campaign C PMK5</th>
<th>Campaign D PMK8</th>
<th>Campaign E PMK9</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Productivity (tHM/m²/24h)</strong> 1)</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/m²/24h)</strong> 2)</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.
Chapter Eight

Comparison of reduction between the experimental blast furnace and laboratory experiments

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](image-url)

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.

![Diagram showing comparison of reduction degree](image)

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
iron bearing burden (in 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 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 CO\textsubscript{2} contents were also lower, resulting in a higher CO/CO\textsubscript{2} ratio. This is demonstrated in Figure 8.3, where the gas ratios (CO/[CO+CO\textsubscript{2}] \times 100) in the laboratory tests and experimental blast furnace have been inserted into the Fe-C-O equilibrium diagram.\[^{38}\] 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 CO\textsubscript{2}, and almost all CO\textsubscript{2} 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

Figure 8.4. Extent of different iron oxide/iron phases in the experimental blast furnace, mid-radius position.

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 8.5. Sample bed of a laboratory reduction experiment interrupted at 1430 °C, 69 per cent bed contraction.
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 analysis 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>SiO$_2$ (%)</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>Al$_2$O$_3$ (%)</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>TiO$_2$ (%)</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>K$_2$O (%)</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>V$_2$O$_5$ (%)</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>P$_2$O$_5$ (%)</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>SiO$_2$ (%)</th>
<th>MgO (%)</th>
<th>CaO (%)</th>
<th>Fe (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPBO-2</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>

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.
9.3 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,[42] 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% H\textsubscript{2}, 21% CO, 14% CO\textsubscript{2} and 10% N\textsubscript{2}) was changed to a carbon monoxide/nitrogen based gas mixture (2% H\textsubscript{2}, 40% CO and 58% N\textsubscript{2}) 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.
Table 9.3. 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 (^{11})</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 (^{21})</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).
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.

![SEM image of surface layer of reduced olivine coated pellet, collected by a shaft probe, 3.4 m below stockline.](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 9.6. 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 9.5. 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 9.7. 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.
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.
References


2. Tottie, M. and Hahlin, P.
   “Testing high temperature properties of burden material for blast furnace use”

3. Papacek, H.G.
   “Quality aspects in pelletising of iron ores”

4. Mu, J., Bi, X. and Xi, G.
   “Indices for evaluating results of melting down test”

5. Tayama, A., Shimomura, Y., Kushima, K., Nakata, T. and Fujita, K.
   “Production and use of high quality pellets”

6. Ranade, M.G.
   “Testing of softening and melting characteristics of iron-bearing materials: A critical review of procedures and applications”

7. Yamaoka, Y., Hotta, H. and Kajikawa, S.
   “Testing method of high-temperature properties of blast furnace burdens”

8. “ISO TC102/SC3 (PHYSICAL TESTING)”
   - Convenors report on softening/melting test procedures, August 1990.

   “Quality requirements for burden materials and testing methods used in Germany”
10. Clixby, G.  
   “Softening and melting of superfluxed sinters and acid pellets”  

11. Clixby, G.  
   “Influence of softening and melting properties of burden materials on blast furnace operation”  

12. Kortmann, H.A. and Ritz, V.J.  
   “Quality aspects of blast furnace burden material at elevated temperatures”  

13. Scheel, J.H.  
   “Development and application of high temperature softening testing at ARMCO”  

14. Sterneland, J.  
   “Förbättring av högtemperaturregenskaper hos järnmalmsagglomerat”  

15. Clixby, G.  
   “Simulated blast furnace reduction of acid pellets in temperature range 950 °C-1350 °C”  

16. Biswas, A.K.  
   “Principles of blast furnace ironmaking”  

17. Takahashi, R., Omori, Y., Takahashi, Y. and Yagi, J.  
   “Effect of gaseous components circulating within blast furnace on softening-melting behaviour of agglomerates”  

18. Wikström, J.O.  
   “Delrapport för projektet Förstudie pilotmasugn”  
   - Internal Mefos-report (the metallurgical research foundation) no. MF95022K (in Swedish), Luleå, Sweden, 1995.
REFERENCES

19. Wikström, J.O.
“Förstudie pilotmasugn – Seminarium i Malmberget 15-16 december 1994”

20. Sköld, B.E.
“Minnesanteckningar från besök vid USS Technical Center, Monroeville den 5 maj 1995”

21. Wikström, J.O.
“Möte i Hamilton, 1995-05-08, angående pilotmasugn – Minnesanteckningar”

22. Yamaguchi, K., Ueno, H. and Tamura, K.
“Maximum injection rate of pulverized coal into blast furnace through tuyeres with consideration of unburnt char”

23. Ando, R., Miyashita, T., Shimotsuma, T., Kaburagi, T. and Yamada, Y.
“Research and development of ironmaking techniques in experimental blast furnace”

“Development of the oxygen blast furnace”

“Development of ultra combined blasting for blast furnace”

26. Yamaoka, H. and Kamei, Y.
“Experimental study on an oxygen blast furnace using a small test plant”

27. Morris, J.P. and Woolf, P.L.
“Examination of an experimental iron blast furnace after quenching with nitrogen”
1963.

28. Tate, M., Suzuki, K., Chang, T.S., Kuwano, Y., Go, H., Honda, K., Matsuzaki, M., Tsuji, E. and Nakamura, S.
   “Effects of low-grade coke on the operation of an experimental blast furnace”

29. Tate, M., Kuwano, Y., Suzuki, K., Matsuzaki, M., Tsuji, E., Chang, T.S., Go, H. and Honda, K.
   “Observation of high-temperature region in an experimental blast furnace by means of fibre- and bore-scope”

30. Matthews, T.
    - Private communications, Corus, Teesside, Great Britain, May 2002.

31. Ma, J.

32. “LKAB 1996 - The products”
    - LKAB product handbook, web.info@lkab.com

33. The Iron and Steel Institute of Japan
    “Blast furnace phenomena and modelling”

34. Kärsrud, K.

35. Solbrand, K.
    - Private communications, SSAB Oxelösund AB, Oxelösund, Sweden, June 1997.

36. Turkdogan, E.T.
    “Blast furnace reactions”

37. Turkdogan, E.T. and Vinters, J.V.
    “Gaseous reduction of iron oxides: Part I. Reduction of hematite in hydrogen”
38. Edström, J.O.
   “The mechanism of reduction of iron oxides”

39. Berglund, G. and Virtala, J.

40. Kanayama, H., Yamaguchi, H., Maekawa, M., Narita, K. and Uenaka, T.
   “Properties of pellets, sinter and crushed pellets taken from the cohesive zone in the blast furnace”

41. Hooey, L., Sterneland, J. and Hallin, M.
   “Evaluation of Operational Data from the LKAB Experimental Blast Furnace”

42. The iron & steel society, Edt. Feinman, J. and Mac Rae, D.R.
   “Direct reduced iron: Technology and economics of production and use”