Pellet Reduction Properties under Different Blast Furnace Operating Conditions

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Licentiate Thesis

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2006
ACKNOWLEDGEMENTS

First and foremost, I wish to express my gratitude to my supervisors, Professor Bo Björkman and Dr. Lena Sundqvist Ökvist, for all help, support and supervision during my work.

Financial support from the Swedish National Energy Administration (STEM) and the Agricola Research Centre is gratefully acknowledged.

Thanks to the members of the Swedish Steel Producers’ Association JK21059 project, “Closed loop styrning av masugn”, for many discussions about the blast furnace and valuable feedback on my work.

I wish to thank LKAB and SSAB Tunnplåt AB for providing materials, tests, analyses and for supporting the work done. A special thanks to LKAB for providing the opportunity to carry out tests in the LKAB Experimental Blast Furnace.

I am most grateful to Johan Folkesson for his voluntary work in designing the laboratory furnace control system at LTU and for his continuous support whenever hardware or software has caused trouble. This work would not have been as successful without his help.

I would like to thank Anita Wedholm for all her assistance in the lab and for her positive attitude. Thanks to my former office mate Fredrik Engström and Daniel Adolfsson for many enjoyable conversations over coffee breaks and lunches.

Computers seem to live a life of their own, and sometimes unexpectedly refuse to cooperate. Thanks to Bertil Pålsson for always putting them on the right track.

I wish to thank my friends and colleagues at the department of Chemical Engineering and Geosciences. Thanks to my friends outside the academic world for all your support. A special thanks to all of you in the south who, despite the northerly latitude and sometimes cold climate, came to visit in Luleå!

Finally, I would like to thank my parents, my sister and my grandmother.
ABSTRACT

One of the aims of modern blast furnace (BF) ironmaking is to reduce coke consumption. One way is to increase the injection of reduction agents, such as pulverized coal. An increase in pulverized coal injection rate (PCR) will affect the blast furnace process and the conditions for iron oxide reduction. Changes in PCR influence the composition of the ascending gases and the in-furnace temperature isotherms.

The performed tests involve full-scale, pilot and laboratory investigations.

Raw material sampling of, among other things, pellets was carried out during a period of fluctuations in the hot metal Si content at the SSAB BF No. 3 at Luleå. Although differences in pellet low-temperature reduction disintegration and the high-temperature breakdown were observed, the reduction behaviours during blast furnace simulation tests were almost identical. Differences in the hot metal Si content in a production blast furnace were difficult to correlate to raw material properties, since the process conditions were changed in order to control the heat level of the blast furnace.

Tests in the LKAB Experimental Blast Furnace (EBF) were carried out under different pre-set process conditions. Injection of high-volatile (HV) coal resulted in a higher reduction potential in the ascending gas due to a higher H₂ content and an increased shaft temperature compared to operation with low-volatile (LV) coal. A higher pellet reduction degree was attained in pellets taken out with the upper shaft probe during operation with the HV coal compared to injection of the LV coal. The differences receded through the shaft and no differences in pellet reduction degree that can be correlated to the pre-set process conditions were observed in samples taken out with the lower shaft probe. However, differences in the pellet texture were observed. For the HV coal, a higher pellet strength but also an increase in generation of Fe₇₇₇ fines, was observed compared to operation with the LV coal. Different Fe₇₇₇ textures were observed in the pellet depending on the choice of injection coal type. The pore size increased and the Fe₇₇₇ areas became smoother during HV coal operation compared to during LV coal operation. The present results indicate that it was likely that the Fe₇₇₇ texture in the pellet periphery influenced the generation of Fe₇₇₇ fines, which left the EBF with the top gas. Further study on the subject is suggested. The present investigations showed an
increased carburization of Fe$_{\text{met}}$ and an increase in the K content in pellets taken out with the lower shaft probe during injection of the HV coal.

Blast furnace simulation laboratory reduction tests for hypothetical PCR indicated that an increase in hypothetical PCR was necessary to compensate for the decrease in reduction time between a slow and a fast temperature profile. The reduction time influenced the Fe$_{\text{met}}$ texture in the pellet periphery.

Blast furnace simulation laboratory reduction for simulated PCR based on measurements in the EBF showed that larger pores were observed in the Fe$_{\text{met}}$ pellet periphery at high PCR. At simulated low PCR the Fe$_{\text{met}}$ was denser. A grain texture was observed in the pellet core after the simulated low PCR, a phenomenon not found in pellets from the simulated high PCR tests. The present results indicated that the texture differences were introduced in the beginning of the reduction.

Results from the EBF tests and laboratory reduction experiments implied that high H$_2$ levels in the reduction gas, high heating rates and temperature levels were the requirements for formation of a pellet periphery with large pores.
LIST OF PAPERS
This thesis is based on the results reported in the following papers, which are given in Appendix I-III.

AISTech 2006 Proceedings of the Iron & Steel Technology Conference

U. Leimalm has in collaboration with A. Brännmark planned and carried out the experiments related to the EBF test. The major part of the evaluation of the results and laboratory reduction experiments has been carried out by U. Leimalm. Other co-authors have contributed in a supervisory capacity.

II. U. Leimalm, L. Sundqvist Ökvist and B. Björkman. “Effect of Simulated PCI Rate on Olivine Pellet Reduction”
Accepted for publication in proceedings from The 4th International Congress on the Science and Technology of Ironmaking

Co-authors have contributed in a supervisory capacity.

Manuscript

Co-authors have contributed in a supervisory capacity.
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1 INTRODUCTION

1.1 Blast Furnace Ironmaking

The blast furnace (BF) process is still the most common means of producing hot metal. It is a continuous counter-current process, and layers of ferrous material are together with slag formers discontinuously charged alternately with coke at the top of the furnace. Slag formers such as BOF slag, limestone and quartzite form slag together with the gangue materials in the iron-bearing material and coke ash that are insoluble in liquid iron. In the BF the coke has several functions. It acts as a reducing agent, provides the energy required for endothermic reactions and for melting of iron and slag, and it has a mechanical function, in that it provides for passage of liquids and gases in the furnace. Air is supplied in the blast to provide the oxygen necessary to generate adequate amounts of CO(g) and H$_2$(g) and to maintain sufficient temperature for operation. In the water-cooled tuyeres, reducing agents such as pulverized coal are introduced together with the blast air. Coke and pulverized coal, or other reducing agents, injected through the tuyeres are combusted in the raceway, forming CO$_2$. At temperatures above 1000°C, CO$_2$ is unstable in the presence of C and CO is generated according to the highly endothermic Boudouard or solution loss reaction$^{[1]}$

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

(1)

During the descent of material in the blast furnace shaft, the reduced iron picks up carbon from the coke, which lowers the melting temperature. The temperature and CO content in the ascending reducing gas decrease on the way through the blast furnace shaft. Because of the exchange of heat, the burden temperature increases as it moves down through the shaft. Coke remains solid at the tuyere level and in the hearth and provides a mechanical support in the bosh region, where the metal and slag are liquid. The liquid Fe$_\text{net}$ is collected in the hearth. Hot metal and slag are discontinuously tapped. Figure 1 shows a cross-section of a blast furnace.

1.1.1 Reactions in the blast furnace

The blast furnace can be divided into different hypothetical zones.$^{[1]}$ The boundaries between them are diffuse and the reduction reactions that dominate in one of the zones can occur at different positions in the blast furnace.
H₂ also participates in the reduction of hematite to about 800°C. In the upper part of the blast furnace, the burden material temperature rises from 1.1 Upper zone to between 100 and 250°C. Hematite and magnetite are reduced to lower oxides. Indirect reduction of hematite by CO is exothermic.

\[ 3\text{Fe}_2\text{O}_3 + \text{CO} \leftrightarrow 2\text{Fe}_3\text{O}_4 + \text{CO}_2 \] (2)

H₂ also participates in the reduction of hematite

\[ 3\text{Fe}_2\text{O}_3 + \text{H}_2 \leftrightarrow 2\text{Fe}_3\text{O}_4 + \text{H}_2\text{O} \] (3)
CO or H\textsubscript{2} reduces magnetite by the endothermic reactions

\[ \text{Fe}_3\text{O}_4 \text{ CO} \leftrightarrow 3\text{FeO} \text{ CO}_2 \] \hspace{1cm} (4)

\[ \text{Fe}_3\text{O}_4 \text{ H}_2 \leftrightarrow 3\text{FeO} \text{ H}_2\text{O} \] \hspace{1cm} (5)

Carbon decomposition according to the reverse Boudouard reaction is most pronounced at temperatures between 500 and 550°C

\[ 2\text{CO} \leftrightarrow \text{CO}_2 + \text{C} \] \hspace{1cm} (6)

1.1.1.2 Middle zone

In the isothermal or thermal reserve zone, the temperatures of the solids and gas are in the range between 800 and 1000°C\textsuperscript{[1]} In this zone, most of the indirect reduction, especially of wustite, occurs. The indirect reduction of wustite by CO is exothermic.

\[ \text{FeO} \text{ CO} \leftrightarrow \text{Fe}_{\text{met}} \text{ CO}_2 \] \hspace{1cm} (7)

The reduction step from wustite to Fe\textsubscript{met} is by H\textsubscript{2} endothermic, but less endothermic at increased temperatures.

\[ \text{FeO} \text{ H}_2 \leftrightarrow \text{Fe}_{\text{met}} \text{ H}_2\text{O} \] \hspace{1cm} (8)

A chemically inactive zone, which is of major importance for a stable operation of the blast furnace, is found inside the isothermal zone, where the exchange of oxygen between the ore and the gas is minor. Thus, the changes in gas composition are also small.

Generation of H\textsubscript{2} takes place according to the water-gas shift reaction,

\[ \text{CO} \text{ H}_2\text{O} \leftrightarrow \text{CO}_2 \text{ H}_2 \] \hspace{1cm} (9)
1.1.1.3 Lower zone
In the area from the tuyere level to a few meters above, called the bosh zone, the molten material reaches a temperature of 1400-1450°C and the gas is cooled down to 800-1000°C. C directly reduces oxides under the formation of CO.\(^1\) Remaining wustite is directly reduced according to

\[
\text{FeO} + \text{C} \leftrightarrow \text{Fe} + \text{CO}
\]  

(10)

The reaction is endothermic.

Combustion of reduction agents in front of the tuyeres results in an empty space in the hearth periphery, which allows downward flow of the materials. The shape of the combustion zone is of importance for a uniform gas distribution and burden descent in the blast furnace.

1.1.1.4 Carbon deposition
At temperatures below 900°C, carbon deposition can occur in Fe-O-C-H systems due to the decomposition of CO.\(^2\) Addition of H\(_2\) will enhance the carbon deposition. CO content up to 20 percent does not influence the carbon deposition. Once the CO percentage increases beyond this value, the carbon deposition will be rapid. Iron catalyses the decomposition of CO.\(^3\) Presence of C or Fe\(_3\)C is reported to hinder the densification and sintering of iron phase.\(^4\) Carburization of Fe\(_{\text{met}}\) or wustite grains can occur according to

\[
3\text{Fe} + \text{C} \rightarrow \text{Fe}_3\text{C}
\]  

(11)

\[
3\text{Fe} + 2\text{CO} \rightarrow \text{Fe}_3\text{C} + \text{CO}_2
\]  

(12)

\[
3\text{FeO} + 5\text{CO} \rightarrow \text{Fe}_3\text{C} + 4\text{CO}_2
\]  

(13)

At temperatures above 900°C, Fe\(_3\)C reacts with wustite according to\(^5\)

\[
2\text{FeO} + \text{Fe}_3\text{C} \leftrightarrow 5\text{Fe} + \text{CO}_2
\]  

(14)
1.1.2 Si content in hot metal

SiO(g) is the dominant source of Si transfer in the blast furnace. SiO gas is formed in the high-temperature reaction of coke carbon on ash or slag silica. To obtain a low silicon operation, the heat input should be lowered, to suppress the SiO formation. This is accomplished if the height of the dropping zone is lowered and raceway temperature is decreased. The carburized metal reacts with the ascending gases containing SiO. As a result of increased PCR, the Si content in molten metal tends to increase, and it is assumed that the pulverized coal type will have an influence on the Si content.

1.1.3 Pulverized coal injection

In modern blast furnace ironmaking, continuous efforts are made to reduce coke consumption by replacing coke with e.g., an increased amount of injected pulverized coal. Injection of reducing agents is mainly done for economic reasons. Pulverized coal injection (PCI) was taken into operation in Sweden in 1985. Successful operation at pulverized coal injection rate (PCR) exceeding 200 kg/tHM is reported at blast furnaces.

An increase in the PCR will among other things affect the composition of the ascending reduction gases, the in-furnace temperature isotherms and possibly the position of the cohesive zone. As the ore-to-coke ratio increases, so does the load on the charged material. In-furnace isotherms for 1000˚C, determined with a feed-type vertical probe, showed higher temperature levels in the shaft with the increase of PCI. As a consequence, the cohesive zone will move upwards, resulting in an increase in high-temperature furnace volume and a decrease in low-temperature furnace volume, and thus a possible decrease in the amount of indirect reduction of pellets. Due to the amount of volatile matter in the injected coal, the H2 content in the reducing gas will increase as PCR increases. Injection coals with different amounts of volatile matters are used for blast furnace operation. A high-volatile (HV) coal will generate more H2 compared to a low-volatile (LV) coal type. Investigations by Peters et al. show that the amount of dust in the blast furnace gas increases with a higher PCR. The phenomenon is ascribable to higher gas velocities caused by the increase in ore-to-coke ratio and the more fine-grained nature of burden compared with coke. At the same time, an increase in PCR will generate a higher amount of gas in the blast furnace.

According to Peters et al., the limits of PCR, from a process engineering viewpoint, can mostly be expected as a consequence of incomplete combustion of
the coal during injection. Char is non-combusted leftovers of pulverized coal. Incomplete combustion will, however, not be limiting when char is consumed in direct reduction or carburization of iron. To ensure high combustibility at high PCR, the design of the injection lances is an important consideration. An injection method that creates a spatially uniform dispersion of the injected coal particles promotes combustion efficiency, since it favours effective use of the oxygen for combustion near the coal particles. Yamaguchi et al. concluded that a factor controlling the PCR is the discharge of unburnt char from the furnace top.

1.2 Conditions for Iron Oxide Reduction
Reduction of iron oxides can, as shown in section 1.1.1, both take place by CO and H$_2$. In the blast furnace, the CO content in the ascending gas is, due to the Boudouard reaction, dependent on pressure and temperature. Decreasing temperature and increasing pressure reduce the amount of CO in the gas in equilibrium with carbon. Reduction by H$_2$ is independent of pressure. Figure 2 shows the equilibrium conditions for reduction by CO and H$_2$.

![Figure 2](image_url)

*Figure 2* The Fe-O-C (full) and Fe-O-H (dotted) equilibrium curves with the superimposed Boudouard curve.

In the blast furnace, reduction of iron oxides is simultaneously carried out with CO and H$_2$. H$_2$ acts as a stronger reducing agent than CO at sufficiently high
temperatures relevant to the blast furnace process. Studies on reduction of sintered ore have shown that the reduction rates, especially in the temperature range between 700°C and 1000°C, increased due to addition of H2.\textsuperscript{[13]} Reduction of commercial low silica pellets in a CO-CO\textsubscript{2}-N\textsubscript{2} gas mixture over the temperature range 800-1100°C has shown that the rate of reduction increases with a higher partial pressure of CO.\textsuperscript{[14]} The higher the temperature the faster was the rate of reduction. Isothermal reduction of pellets in a bed with CO-H\textsubscript{2} atmosphere showed that the overall reduction rates increased with increasing reduction temperature and decreased with the degree of reduction.\textsuperscript{[15]} At non-isothermal reduction of low silica hematite pellets in a CO-H\textsubscript{2} atmosphere the reduction rate was faster at a higher ratio of H\textsubscript{2} in (CO+H\textsubscript{2}) mixture for a constant heating rate and constant initial temperature.\textsuperscript{[16]} For reduction in H\textsubscript{2} rich gas mixture, the reduction rate for non-isothermal condition was lower than for isothermal reduction at corresponding temperatures.

1.2.1 Textures

Studies by St. John et al.\textsuperscript{[17]} have shown that the conditions for formation of porous iron depended on the gas composition, reaction temperature, oxide stoichiometry and the presence of impurity elements in solid solution with FeO. Reduction tests on hematite pellets to form magnetite in a CO-CO\textsubscript{2} gas mixture were carried out by Bradshaw et al.\textsuperscript{[18]} At 600°C and p\textsubscript{CO}=0.25 atm a random distribution of pores was observed in the magnetite phase. An increase in the reaction temperature to 800°C and p\textsubscript{CO}=0.50 atm resulted in coarser pores and formation of lamella from the hematite/magnetite interface into the unreduced hematite. At 1000°C and p\textsubscript{CO}=0.125, no microporosity was apparent in the magnetite. All the investigated pellets display an outer zone in which all but the largest grains were reduced to magnetite. In the intermediate zone various stages of reduction were attained and in the pellet core the hematite grains remained unattacked. Reduction of granular solids of hematite in CO-CO\textsubscript{2} mixtures at a reaction temperature of 600°C showed random distribution of pores in the formed magnetite, a phenomenon not observed at 1000°C.\textsuperscript{[19]} Wustite formed at reduction of magnetite is dense, while wustite that originates from hematite and oxidized magnetite is porous.\textsuperscript{[20]}

During reduction of iron oxides, the initial reduction temperature is of major importance for the pore structure of the reduced iron.\textsuperscript{[21]} The pore structure of Fe\textsubscript{met} becomes coarser with increasing temperature. A structure that is formed at a low reduction temperature does not readily coarsen at subsequent temperature rise. Olsson et al.\textsuperscript{[22]} showed that the pore structure of Fe\textsubscript{met} formed during reduction of
iron oxides with H\textsubscript{2} was a function of the reaction temperature. At a reduction temperature of 900°C the pore structure was homogenous. At 500°C the pore structure was heterogeneous with large pores, which appeared to follow the initial grain boundaries. Very fine secondary pores were observed through the individual grains. Judging from the photomicrographs by Olsson et al. it can be concluded that the average pore size in the Fe\textsubscript{met} increases with increasing reduction temperature. Wright\cite{15} concluded, based on isothermal reduction of pellets in CO-H\textsubscript{2} mixtures, that the structure of metallized pellets was highly temperature dependent. At low reduction temperatures there was a great deal of internal porosity in the individual Fe\textsubscript{met} particles and the interparticle pore sizes were very small. At increased reduction temperature, the voids progressively grow and coalesce with the particle interstices and larger pores are formed.

In pellets of high original porosity there was a rapid reduction from hematite to wustite without formation of distinct product layers.\cite{21} The following reduction step of wustite to iron was relatively rapid. The greater the driving force for reduction, the more pronounced was the formation of the product layers. In the case of sintered hematite pellets the reduction proceeds topochemically during the formation of product layers.

1.2.2 Effect of impurity elements
Laboratory investigations by Geva et al.\cite{23} have shown that the presence of impurity elements in solid solution in the iron oxide affects the final iron product morphologies. Mg, Ti, Si, Ca, Na and K present together with FeO result in decreases in the CO concentrations necessary to obtain porous iron growth at any reaction temperature relative to reactions on pure wustite. P has a marginal effect on the porous/dense iron transition and Al restricts the range of gas compositions over which porous iron can be obtained. In H\textsubscript{2}/H\textsubscript{2}O mixtures, the presence of Ti, Mg, P, Si, Ca, K and Na favours porous iron formation. Additions of Al make the formation of a porous iron layer difficult. The effects of impurities are additive. Na and K are present in the blast furnace and are generally undesirable, but can on the other hand play a significant role in improving the reducibility of the burden. K functions as a catalyst for the reduction of iron oxide.\cite{24} Since the solubility of K in bulks of iron oxides has been reported not to exceed 0.1 wt%, much of the K is expected to exist on the surface of iron oxides.\cite{25}
1.3 Objectives

The objective of the performed studies was to develop an understanding of how different blast furnace operation conditions will affect the reduction properties of commercial pellets. Due to the increase in PCR, the conditions for pellet reduction change as the ascending gas composition, the in-furnace temperature isotherms and possibly the position of the cohesive zone are affected. Not only the PCR but also the volatile amounts in the injected coal and the method for oxygen supply to the lance may influence these properties in the blast furnace.

To learn more about pellet reduction under different blast furnace operating conditions, laboratory reduction tests under blast furnace simulating conditions were carried out for different PCR. Heating rate and temperature profiles were based on mass and heat balance calculations and measurements in the LKAB Experimental Blast Furnace (EBF). Conditions for formation of different pellet textures were determined. Pellet samples and process data from tests in the EBF during operation at different PCR, injection of a LV and a HV coal and using different methods for oxygen supply were evaluated.

The hot metal Si content fluctuates in a blast furnace. Process data and pellets taken out at the pellet charging at BF No. 3 at SSAB Tunnplåt AB at Luleå were investigated in an attempt to find possible correlations between pellet reduction properties and the hot metal Si.

Table 1 gives an overview of studies presented in Appendix I-III.
Table 1: Overview of studies presented in Appendix I-III.

<table>
<thead>
<tr>
<th>Appendix</th>
<th>Laboratory Furnace</th>
<th>EBF</th>
<th>SSAB BF No. 3</th>
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<tbody>
<tr>
<td>I</td>
<td>Reduction</td>
<td>Effects of PCR and injection coal type on conditions for pellet reduction</td>
<td>Raw material sampling</td>
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<td>- BF simulating</td>
<td>- Properties of pellets reduced during different PCR and injection coal types</td>
<td>Pellet properties</td>
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<tr>
<td></td>
<td>- Hypothetical PCR</td>
<td>- Process data</td>
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<td></td>
<td>- Pellet texture</td>
<td>- Pellet textures</td>
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<tr>
<td>II</td>
<td>Reduction</td>
<td>- Process data</td>
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<tr>
<td></td>
<td>- Simulated PCR</td>
<td>- Pellet textures</td>
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<td>- Evaluation of pellet textures from operation during different PCR, injection coal types and methods for oxygen supply.</td>
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<td>III</td>
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<td>- Simulated PCR</td>
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<td>- Evaluation of pellet textures from operation during different PCR, injection coal types and methods for oxygen supply.</td>
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<td>ments in the EBF</td>
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</table>
2 MATERIALS AND METHODS

2.1 Materials

Commercial olivine pellets were used in the investigations performed. Material samplings for the laboratory investigations were carried out at the LKAB pelletizing plant at Malmberget and at the SSAB BF No. 3 pellet-charging stream. The chemical composition of the olivine pellets MPBO and KPBO produced by LKAB can be seen in Table 2. MPBO was used in the EBF trial.

<table>
<thead>
<tr>
<th>Pellet</th>
<th>Fe</th>
<th>FeO</th>
<th>CaO</th>
<th>SiO₂</th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>CaO/SiO₂</th>
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</thead>
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<tr>
<td>MPBO</td>
<td>66.8</td>
<td>0.5</td>
<td>0.35</td>
<td>1.7</td>
<td>1.5</td>
<td>0.32</td>
<td>0.21</td>
</tr>
<tr>
<td>KPBO</td>
<td>66.6</td>
<td>0.4</td>
<td>0.46</td>
<td>2.0</td>
<td>1.5</td>
<td>0.23</td>
<td>0.23</td>
</tr>
</tbody>
</table>

In the EBF tests two different coal types were used: a LV containing 19.6 percent volatile matters and a HV containing 38.0 percent volatile matters. Other additions corresponded to operation at the SSAB BF No. 3, except for quartzite, which was charged to provide for the SiO₂ in the slag and reach the desired slag volume.

2.2 SSAB Blast Furnace No. 3

SSAB Tunnplåt AB has had blast furnaces operating at the Luleå plant since 1951. In August 2000 the new BF No. 3, which replaced BF No. 1 and BF No. 2, was taken into operation with a hot metal production of 2.2 Mt/year. Design data for the SSAB BF No. 3 are listed in Table 3. The ferrous burden consists entirely of olivine pellets from Malmberget, MPBO, and Kiruna, KPBO, produced by the Swedish iron ore producer LKAB.

2.2.1 Raw material sampling

Raw material samplings were carried out at the pellet feeders close to the blast furnace. Pellets were taken out every second hour. Process data, including the hot metal Si content, were stored at the same time. Low temperature reduction disintegration tests were performed and the reduction strength determined, see paragraph 2.5.1.
| Table 3 Design data, Blast Furnace No. 3 at SSAB Tunnplåt AB, Luleå
<table>
<thead>
<tr>
<th>BF No. 3</th>
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<tbody>
<tr>
<td>Year of erection</td>
</tr>
<tr>
<td>Design</td>
</tr>
<tr>
<td>Start of campaign</td>
</tr>
<tr>
<td>Hearth diameter</td>
</tr>
<tr>
<td>Working volume</td>
</tr>
<tr>
<td>Total volume</td>
</tr>
<tr>
<td>No of tuyeres</td>
</tr>
<tr>
<td>No of tap holes</td>
</tr>
<tr>
<td>Top pressure</td>
</tr>
<tr>
<td>Charging equipment</td>
</tr>
<tr>
<td>Daily output</td>
</tr>
</tbody>
</table>

2.3 LKAB Experimental Blast Furnace

The EBF at MEFOS has a working volume of 8.2 m³, a diameter at tuyere level of 1.2 m and is equipped with a system for injection of reduction agents. The height from tuyere level to stock line is 6 m and there are three tuyeres separated by 120°. After a campaign, the furnace can be N₂ quenched and excavated. The process is interrupted, nitrogen throughput from the top started, followed by a decreased and finally stopped blast volume. Dissection of the EBF can start after at least ten days of cooling and is carried out like an archaeological excavation, where basket-samples introduced in the final hours of operation are recovered. A schematic drawing of the EBF plant including raw material handling, injection system and gas cleaning system can be seen in Figure 3.

During operation, in-burden probes were used for sampling of the burden and for the measurement of the horizontal temperature- and gas profiles. Figure 4 show the positions of the shaft probes in the EBF. The shaft probe material was divided in sub-samples. During ideal conditions, a packed probe will generate 5 sub-samples for the upper probe and 6 for the lower. Usually, only 3-4 sub-samples were generated for each probe and it was assumed that the sub-sample with the highest number was taken close to the wall.

Estimation of the positions of the in-furnace temperature isotherms was done by allowing a thermocouple to descend with the burden.
Figure 3 Drawing of the EBF plant

Level beneath upper flange
Upper flange 0 mm

Burden surface 1834 mm
Upper shaft probe 2735 mm
Lower shaft probe 5205 mm
Centre tuyere 7430 mm

Figure 4 Drawing of the EBF with shaft probes indicated
2.3.1 EBF tests

2.3.1.1 Influence of pre-set process conditions

The influence of PCR, coal type and method for oxygen supply on olivine pellet reduction properties was investigated during an EBF test. The PCI system was an oxy-coal system and the oxygen added to the lance could be replaced by air if desired. In the tables and figures, operation with oxygen added to the lance is termed Oxy Coal and operation with air addition to the lance termed Air Coal. Horizontal gas composition and temperature profiles were measured at the positions of the shaft probes. Pre-set process conditions during the 6 test periods are presented in Table 4.

<table>
<thead>
<tr>
<th>Test Period</th>
<th>Coal type</th>
<th>PCR (kg/tHM)</th>
<th>Method for oxygen supply</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>LV</td>
<td>152</td>
<td>Oxy Coal</td>
</tr>
<tr>
<td>2</td>
<td>LV</td>
<td>146</td>
<td>Air Coal</td>
</tr>
<tr>
<td>3</td>
<td>LV</td>
<td>79</td>
<td>Oxy Coal</td>
</tr>
<tr>
<td>4</td>
<td>HV</td>
<td>94</td>
<td>Oxy Coal</td>
</tr>
<tr>
<td>5</td>
<td>HV</td>
<td>152</td>
<td>Oxy Coal</td>
</tr>
<tr>
<td>6</td>
<td>HV</td>
<td>143</td>
<td>Air Coal</td>
</tr>
</tbody>
</table>

In-burden material was taken out with the shaft probes and the pellet properties were evaluated. During the test periods, process data including burden decent rates were stored. Pellet strength and fines generation were studied based on samples from EBF operation during oxygen addition to the coal lance.

2.3.1.2 Basket samples

Basket samples were introduced into the EBF prior to quenching. About 600 grams of pellet were put into a basket constructed of a high-temperature resistant metallic net. Pellets for the basket samples were taken out during the raw material sampling period at SSAB Tunnplåt AB. For further details on the pellet selection, see section 3.1.

2.4 Laboratory Reduction Furnace

2.4.1 Finishing of laboratory reduction furnace

Prior to the laboratory reduction experiments, considerable improvements of the former reduction furnace system were made. The furnace at Luleå University of Technology (LTU) is a vertical steel tube-type furnace with an inner diameter of about 6 cm that is heated electrically by U-shaped Super-Kanthal elements with a
heating zone of about 8 cm in height. The experimental apparatus is shown in Figure 5. Hardware and software for simultaneous temperature and gas control were developed and put into operation. The design of the Furnace Control program enables use of blast furnace simulating conditions with optional choice of heating rate and gas composition as well as isothermal test programs using a constant or varied reduction gas composition. Test runs were made for adjustment of the PID controller parameters and measurement of the temperature profile inside the vertical steel tube. During the reduction tests, the pellet samples were placed in the 10 cm zone in which the temperature gradient was 2-3°C. The gas supply system is equipped with digital Multi-Bus Flow-Bus regulators for input gas flows of CO, H₂, CO₂ and N₂. The gas is introduced in the bottom of the tube and heated in a bed of Al₂O₃ balls. A thermocouple for temperature measurement is introduced from the bottom of the tube and situated approximately 20 mm below the sample, which is suspended in the balance with metal wires. Test runs were made to investigate the influence of input flow on pellet reduction in blast furnace simulating tests. Input gas composition and temperature are controlled by a computer, which also is used to store data with a frequency chosen for the parameters.

![Figure 5 Schematic view of the experimental apparatus used for laboratory reduction tests](image)

2.4.2 Experimental procedure
At the test starting temperature, a nitrogen flow at 12 l/min was introduced. The temperature and N₂ gas flow were held at constant values for a few minutes before the sample was introduced into the furnace. Each sample consisted of dry pellets with a starting weight of 70 to 85 grams. The sample material was placed in a
basket. As soon as the sample had been introduced into the constant temperature zone, the test was started and the temperature program started at the same time as the gas composition was changed into a reducing atmosphere. After the test, which could be interrupted at any optional point the sample was cooled in pure N₂ in the water-cooled top. Total gas flow was maintained at 12 l/min during the entire test. The reduction degrees attained in the laboratory reduction tests were calculated according to

\[
\text{reduction degree (\%)} = \frac{\text{oxygen removed (g)}}{\text{original oxygen (g)}} \times 100
\]  

(15)

It was assumed that the total weight change during reduction was caused by removal of oxygen.

2.4.3 Laboratory reduction conditions

2.4.3.1 Simulated BF

One blast furnace simulating reduction profile was based on a normal test program used at LKAB. Heating rate and reduction gas composition for this program are presented in Figure 6.

![Figure 6](image)

**Figure 6** Blast furnace simulating reduction profile based on a normal test program at LKAB.

Pellets taken out during the material sampling period at SSAB Tunnplåt AB were reduced in the simulated BF reduction profile. The tests were interrupted at a
temperature of 1025°C. An overview of the selection of pellets for laboratory reduction from the sampling occasions is given in section 3.1.

2.4.3.2 Hypothetical PCR
Two different temperature profiles were used in the study of the influence of hypothetical PCR on pellet reduction properties. The slow heating rate was based on a normal test procedure at LKAB\textsuperscript{[29]} and the fast heating rate was based on a vertical temperature measurement made in the EBF during a PCR of approximately 130 kg/tHM. The gas compositions used for the different hypothetical process conditions with all coke operation and hypothetical PCR were calculated using mass and heat balances for estimation of the gas composition at various levels in the blast furnace. The test conditions for hypothetical All Coke and a hypothetical PCR of 200 kg/tHM are shown in Figure 7 and Figure 8.

**Figure 7** Reduction profiles for calculated hypothetical All Coke and a PCR at 200 kg/tHM. Fast heating rate based on a vertical temperature measurement in the EBF.
Table 5 shows an overview of laboratory reduction experiments with hypothetical PCR. Olivine pellets produced at Malmberget were used in tests 1-6.

**Table 5** Schematic overview of blast-furnace-simulating reduction experiments performed with hypothetical PCR

<table>
<thead>
<tr>
<th>Test</th>
<th>Temperature Profile</th>
<th>Gas Profile</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Slow: X</td>
<td>Fast: X</td>
</tr>
<tr>
<td>1</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>X</td>
<td></td>
</tr>
</tbody>
</table>
2.4.3.3 Simulated PCR

Heating rate and gas profiles for the simulated PCR profiles were estimated from measurements made in the EBF during operation at the high and low PCR in test periods 1 and 3. Oxygen supply was by oxy coal and the LV coal type was injected. The heating rate profiles were estimated from:

- Vertical temperature measurements
- Average burden decent rates
- Horizontal temperature profiles at the position of the shaft probes
- CO/CO₂ ratio at the position of the lower shaft probe for temperature estimation from an oxygen potential diagram

For the high and the low PCR, a fast heating rate was estimated to simulate a centre profile and a slow heating rate was estimated to simulate an intermediate/wall profile. The reduction gas compositions were estimated from the top gas analysis and the gas composition at the position of the upper and lower shaft probes. The gas compositions used together with the fast heating rates were estimated from the centre gas composition in the EBF. For the slow heating rates, the gas compositions at an intermediate position in the EBF were the basis of the reduction gas composition. N₂ was filled up to a gas flow of 100 percent. Test conditions are shown in Figure 9 and Figure 10.

![Figure 9](image_url) Heating rate and gas composition profiles for simulated high PCR. Heating rate and gas profiles estimated from measurements in the EBF
One set of experiments was interrupted at a reduction degree of approximately 40 percent and one set was interrupted at a furnace temperature of 1100˚C. Table 6 gives an overview of the simulated PCR experiments performed. Olivine pellets produced at Malmberget were used in tests 7-14.

**Table 6** Schematic overview of BF PCR simulating experiments performed

<table>
<thead>
<tr>
<th>Test</th>
<th>PCR</th>
<th>Heating Rate</th>
<th>Test End</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>High</td>
<td>Low</td>
<td>Fast High PCR</td>
</tr>
<tr>
<td>7</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>8</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>9</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>10</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>11</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>12</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>13</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>14</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
</tbody>
</table>
2.5 Evaluation of Pellet Properties

2.5.1 Pellet strength

Low-temperature reduction disintegration (LTB) and the high-temperature breakdown (ITH) tests were carried out on a selection of pellets taken out during the raw material sampling period at SSAB Tunnplåt AB. LTB was performed by LKAB according to ISO13930\textsuperscript{[26]}, except for the pellet size fraction, where pellets from the total samples were used instead of the 10-12.5 mm fraction. ITH was performed by LKAB and determined by tumbling in a drum with a length of 700 mm and an inner diameter of 130 mm at 20 rpm for 600 turns after reduction according to ISO 4695\textsuperscript{[30]}. Pellets from the total samples were also used in this case. After tumbling, the sample was sieved for +6.3 mm and –0.5 mm and the ITH result was presented as the percent fraction +6.3 mm and –0.5 mm.

The strength of pellets taken out with the upper shaft probe in the EBF was tested in an I-drum with a diameter of 130 mm and a length of 700 mm. 300 grams of pellets in the size range 10-12.5 mm were rotated 600 times at 20 rpm.

2.5.2 Pellet textures

Pellet textures were investigated by light optical microscopy (LOM) and by scanning electron microscopy (SEM). Prior to observation, pellets were cold mounted in epoxy resin. In order to observe the largest pellet cross section, the mounts were cut at the maximum pellet diameters. The surface of the section was polished. A Nikon E600 POL polarizing microscope was used for LOM. For SEM, a Philips XL 30 equipped with Energy Dispersive X-ray Analysis (EDS) for chemical mapping was used. Prior to SEM investigation, the cross sections of the mounted pellet samples were coated with a thin layer of gold using a Bal-Tec MCS 010 sputter coater.

2.5.3 Identification of trace elements in pellets

In order to identify trace elements in the pellet periphery and core, pellets were rotated in an I-drum at 600 revolutions for 30 minutes. After I-drum treatment, pellet fragments in the size range 212\(\mu\)m – 1,5 mm and >10 mm were separately ground in a ring mill and X-Ray diffraction was carried out on the ground materials. A Siemens X-ray diffractometer was used to record scattering intensities of samples by using a Copper Ka radiation (40 kV, 40mA) as the X-ray source. The samples were continuously scanned over an angular range of \(\sin20\ 10-90^\circ\) by
using a step size of 0.020° and over sin20 35-55° with a step size of 0.020° and a step time of 6 seconds at each step.

Identification of trace elements in pellets was performed on sub-samples 3 taken out with the lower shaft probe during the EBF test. Investigations on the pellet core, >10 mm fragments, were carried out for the low PCR periods.

2.6 Chemical Analysis
Chemical analyses were provided by SSAB Tunnplåt AB and LKAB. X-ray fluorescence and LECO combustion analysis were used at SSAB Tunnplåt AB and wet chemical titration at LKAB.
3 RESULTS

3.1 Evaluation of Process Data and Pellet Samples from SSAB Blast Furnace No. 3, Appendix I

3.1.1 Process data and selection of pellets for further testing
During the raw material sampling period, the Si content in the hot metal reached between 0.15 and 0.54 percent and legible decreases were observed between observations 19 to 28 and 38 to 49, see Figure 11. The 74 Si observations corresponded to measurements during 48 hours. Samples of MPBO and KPBO for further testing were chosen during a period of increase and decrease of the Si content in hot metal. Sampling occasions A-H and the relation to the hot metal Si content are marked in Figure 11. Figure 12 shows the PCR in relation to the hot metal Si during the period in question. The time delay between pellet charging and hot metal tapping was estimated to approximately 8 hours. Pellets charged in connection to pellet sampling occasion A influenced the hot metal Si content 8 hours later.

LTB and ITH tests were performed on MPBO and KPBO samples A-H. Samples with the lowest share, highest share, and median share of the >6.3 mm fraction were chosen for further investigation. MPBO samples B, E and G, respectively, and KPBO samples B, F, and G, respectively, were chosen.

![Figure 11](image-url) Si content in hot metal during the raw material sampling period and A-H samples used for metallurgical testing.
3.1.2 Laboratory reduction under simulated BF conditions

Reduction profiles of the MPBO samples B, E and G and KPBO samples B, F and G indicated almost the same reduction behaviour, see Figure 13.

3.1.2.1 Pellet textures attained

After reduction, Fe$_{\text{met}}$ dominated in the pellet peripheries, while a few small areas were found in the pellets core. Wustite dominated the cores and some magnetite was observed. Differences in Fe$_{\text{met}}$ texture were observed between the pellet types. Compared to the KPBO, the MPBO samples showed larger areas of coherent Fe$_{\text{met}}$.
in the pellet peripheries samples. Figure 14 shows typical textures of the MPBO and KPBO pellets.

\[ \text{Figure 14} \] Typical pellet textures of MPBO (upper) and KPBO (lower) observed in LOM after blast furnace reduction simulation tests. Pellet core (left) and periphery (right). Magnetite = Smooth grey, Wustite = Broken grey, Fe\text{met} = White, Pores = Black

3.1.3 Basket samples

From an excavation of the EBF, basket samples containing material from the same samples as MPBO sampling B, E and G and KPBO sampling B, F and G were recovered directly below the position of the upper and lower shaft probes. All of the basket samples containing corresponding material as in the simulated blast furnace tests showed similar iron oxide and Fe\text{met} textures after reduction in the EBF. The reduction degrees attained were higher in basket samples recovered further down in the EBF. The size and number of Fe\text{met} areas increased with increasing reduction degree.
3.2 Laboratory Reduction for Hypothetical PCR, Appendix I

Figure 15 shows that an increase in the hypothetical PCR increased the degree of reduction at any temperature once the reduction degree had reached about 10 percent; a pattern that was independent of temperature profile. The most apparent difference in reduction behaviour was observed between a hypothetical All Coke case, tests 1 and 4, and a hypothetical PCI at 146 kg/tHM or 200 kg/tHM, tests 2 or 3 and 5 or 6. A clear difference between the hypothetical PCR of 146 and 200 kg/tHM, tests 2 and 3, was observed for the slow temperature profile at temperatures above 750°C.

![Graph showing temperature and reduction profiles](image)

**Figure 15** Temperature- and reduction profiles for laboratory reduction tests simulating hypothetical All Coke and different PCR

Typical textures from the study of pellets reduced in laboratory tests based on hypothetical All Coke and hypothetical PCR tests are presented in Figure 16. Significant differences in the iron oxide and Fe\textsubscript{met} textures were not observed when pellets from tests with hypothetical PCR of 146 and 200 kg/tHM and corresponding temperature profiles were compared. An increase of the hypothetical PCR for the slow temperature profile increased the amount of Fe\textsubscript{met} in the pellet core. Wustite dominated in the pellet cores with few Fe\textsubscript{met} areas. For the hypothetical All Coke, magnetite dominated the pellet core and wustite and Fe\textsubscript{met} the periphery. Magnetite was observed through the entire cross section of pellets at the same time as Fe\textsubscript{met} and wustite dominated in the pellet periphery for pellets at hypothetical PCR and a fast temperature profile. For the hypothetical All Coke, magnetite dominated in the core. Fe\textsubscript{met} was observed together with wustite and some magnetite in the pellet periphery.
Figure 16 Pellet textures observed in LOM after hypothetical All Coke (1, slow temperature profile and 4, fast temperature profile) and PCR of 200kg/SHM (3, slow temperature profile and 6, fast temperature profile). Pellet core (left) and periphery (right). Magnetite = Smooth grey, Wustite = Broken grey, Fe$_{\text{net}}$ = White, Pores = Black
3.3 EBF Tests, Appendix I-III

3.3.1 Ascending gas compositions, Appendix I and III

The H₂-content in the reduction gases was observed to increase with increasing PCR. Oxygen supply to the coal lance as well as operation using the HV coal resulted in a higher H₂ amount as compared to when air was added to the lance and with operation using the LV coal. The H₂ content increased during operation with the HV coal, except during test period 4, when lower temperatures were observed in the EBF. The H₂ content was at a generally higher level at the lower shaft probe in the EBF, due to the higher temperature.

\[ \eta_{CO} = \frac{100\%CO_2}{(\%CO + \%CO_2)} \quad (16) \]

was observed to reach a lower level at the position of the upper shaft probe during operation using the LV coal compared to operation using the HV coal. During the tests, the reduction potential of the gas was higher in the centre of the furnace and decreased towards the periphery.

3.3.2 Influence of coal type and PCR, Appendix I

The particle size distribution of material taken out with the upper shaft probe during high and low PCR of the HV and LV coal type can be seen in Figure 17. Figure 18 shows the content of Fe and C in the <0.5 mm fraction. The fine fractions, 0-0.5 mm, 0.5-3.3 mm and 3.3-6.3 mm, constituted a larger part of the material using the HV coal compared to operation using the LV coal. The Fe content in the <0.5 mm fraction increased at the same time as the C content decreased when the HV coal was injected at the high PCR.
The total amount of fines leaving the furnace with the top gas reached the highest level when the HV coal was injected. Figure 19 shows the amount of Fe and C in flue dust under different process conditions. Injection of the HV coal increased the Fe content in the flue dust at the same time as the amount of C decreased.
3.3.3 Pellet properties, Appendix I and III

3.3.3.1 Pellet reduction and disintegration

Injection of the HV coal resulted in the highest reduction degree of pellets taken out with the upper shaft probe. Correlations between operation parameters and the pellet reduction degree were not observed at the level of the lower shaft probe. The pellet reduction degrees reached in sub-samples 1-3 during the test periods are presented in Figure 20 and Figure 21.
The result from the I-drum tests on pellets taken out with the upper shaft probe during oxygen supply to the coal lance showed that the highest amount of the fraction >6.3 mm were found at an increased reduction degree. This result indicated low disintegration, but at the same time the highest amount of abrasion in the <0.5 mm fraction was observed. As can be seen in Figure 22, a linear relation is obtained between the pellet reduction degrees and the disintegration. The highest pellet strength and the highest reduction degree were observed in samples taken during injection of the HV coal.
3.3.3.2 Pellet textures

Magnetite dominated the cores of pellets taken out with the upper shaft probe during injection of the LV coal in test periods 1-3. Some hematite and wustite were also observed in the pellet cores. The pellet peripheries were mainly made up of wustite. Wustite and magnetite dominated in the pellet cores in pellets taken out with the upper shaft probe during injection of the HV coal in test periods 4-6. A few areas of \( \text{Fe}_\text{met} \) observed in the pellet core increased in size and number towards the periphery. Wustite and \( \text{Fe}_\text{met} \) dominated the periphery. The transition between the texture in the pellet core and periphery was blurry and any distinct product layers were not formed. Typical textures of pellet cores and peripheries obtained in pellets taken out with the upper shaft probe during test periods 1-6 are shown in Figure 23 and Figure 24.

In pellets taken out with the lower shaft probe, \( \text{Fe}_\text{met} \) dominated the texture. Injection of the HV coal type increased the pore size in the periphery. Comparing the pellet cores, the size of the continuous \( \text{Fe}_\text{met} \) areas increased during injection of the HV coal compared to injection of the LV coal at high PCR and independent of oxygen supply method. In the pellet core from period 1, softened wustite dominated. Typical textures of pellet cores and peripheries obtained in pellets taken out with the lower shaft probe are shown in Figure 25 and Figure 26.

EDS mapping of pellet core, intermediate area and pellet periphery taken out during operation with the LV and HV coal type, test periods 2 and 5, are presented in Figure 27 and Figure 28. Slag areas were found next to or enclosed in the \( \text{Fe}_\text{met} \) in the entire cross section in pellets taken out with the lower shaft probe during test periods 1-6. The size and distribution of the slag areas and areas with increased K content varied between the test periods. The tendency for liquid formation, as can be seen when Figure 27 and Figure 28 are compared, was more pronounced in pellets taken out during injection of the HV coal type, as can be seen by the smooth slag areas compared to the diffuse slag areas during LV coal operation. Sometimes, the pellet periphery was covered by a layer consisting of mainly Mg, Si, K, Ca and Al.

K was observed in pellets taken out with the lower shaft probe, independent of preset process conditions. Areas of increased K content were in the core of the pellets to be found next to or surrounded by \( \text{Fe}_\text{met} \). In pellets taken out during injection of the LV coal type, K was located next to \( \text{Fe}_\text{met} \), while \( \text{Fe}_\text{met} \) surrounds K after injection of the HV coal type. In the pellet periphery, K was found between \( \text{Fe}_\text{met} \) as
well as in the surface cover of pellets, independent of the pre-set process conditions
during sampling. K formed compounds with Si, Al, Mg, and in a few cases K and
Ti coexist. The coexistence of K and the slag elements was more frequent in the
pellet periphery, and especially in the surface cover, compared to the core of the
pellets. Ca-Si slag was observed in the pellet cores and coexistence between the
elements was observed in the pellet periphery as well as in the surface cover. Al
was mainly observed in the surface cover during injection of the LV coal type,
though throughout the entire pellets during HV coal injection. In the surface cover
the most prominent areas of coexistence of K, Si, Mg and Ca were observed.
**Figure 23** Typical textures, observed in LOM, of pellets taken out with the upper shaft probe in the EBF during test period 1-3. Pellet core (left) and pellet periphery (right). Hematite = Light grey, Magnetite = Smooth grey, Wustite = Broken grey, Pores = Black.
Figure 24 Typical textures, observed in LOM, of pellets taken out with the upper shaft probe in the EBF during test period 4-6. Pellet core (left) and pellet periphery (right). Hematite = Light grey, Magnetite = Smooth grey, Wustite = Broken grey, $\text{Fe}_{\text{net}}$ = White, Pores = Black
Figure 25 Typical textures, observed in LOM, of pellets taken out with the lower shaft probe in the EBF during test period 1-3. Pellet core (left) and pellet periphery (right). Wustite = Grey, $\text{Fe}_{\text{met}}$ = White (lightest), Pores = Black
Figure 26 Typical textures, observed in LOM, of pellets taken out with the lower shaft probe in the EBF during test period 4-6. Pellet core (left) and pellet periphery (right). Wustite = Grey, Fe_{net} = White (lightest), Pores = Black
Figure 27 EDS mapping of core, intermediate area and periphery of pellet taken out with the lower shaft probe during operation with the LV coal type in test period 2
Figure 28 EDS mapping of core, intermediate area and periphery of pellet taken out with the lower shaft probe during operation with the HV coal type in test period 5
3.3.3.3 Pellet fragments

Chemical analysis of the pellet fragments in the size range 212μm – 1.5 mm showed an increased carbon content after operation with injected HV coal. Increased contents of K and Ca, see Figure 29, are also observed. In the XRD-analysis Fe₃C was observed in samples from test periods 2-6.

![Figure 29 Element distributions in the fraction 212μm – 1.5 mm after l-drum treatment of pellets from sub-sample 3 taken out with the lower shaft probe during operation in the EBF.](image)

For the low PCR, the chemical analysis of the >10 mm pellet fragments showed an increased amount of carbon after operation with the injected HV coal type. In the diffractogram, Fe₃C was observed after operation with the injected HV coal type and FeO was observed after injection of the LV coal type. The pellet fragments were mainly made up of Fe₄met.

3.3.4 Laboratory reduction with simulated PCR, Appendix II and III

Figure 30 shows the reduction profiles for tests 7-14. For the same level of simulated PCR, at the test end temperature of 1100°C, a higher reduction degree was attained for the slow heating rates when tests 10 and 8 and tests 14 and 12, were compared. After reduction simulating the fast heating rate profiles, tests 8 and 12, approximately equal reduction degrees were attained independent of simulated PCR. A higher reduction degree was attained for the slow heating rates at the end of test 10, simulated high PCR, compared to test 14, simulated low PCR. In tests 7, 9, 11 and 13, the reduction was interrupted at a reduction degree of approximately 40 percent.
3.3.4.1 Pellet textures

Significant differences in the iron oxide textures are observed in the pellet core when pellets from the reduction tests simulating the high PCR (see Figure 31) and low PCR (see Figure 32) are compared. A grain texture was observed in the pellet core of samples after tests 11-14, simulating the low PCR, an observation that was not discernible after tests 7-10, simulating the high PCR. Textures from the pellet core and periphery from tests 7-14 are presented in Figure 31 and Figure 32. Together with the areas of Fe\textsubscript{met} in the pellet periphery, a few areas of iron oxide were present after tests 8 and 10, which were full-length tests simulating the high PCR. After full-length tests simulating the low PCR, tests 12 and 14, the corresponding areas in the pellet periphery showed noticeable areas of iron oxide surrounded by Fe\textsubscript{met} within the texture dominated by Fe\textsubscript{met}. A few areas of Fe\textsubscript{met} were observed in the pellet core from test samples interrupted at the test end temperature of 1100°C. Fe\textsubscript{met} was not observed in pellets reduced to a reduction degree of approximately 40 percent. Significant differences in the Fe\textsubscript{met} texture of the pellet periphery, independent of final reduction degree, were not observed between pellets reduced according to similar reduction profile. The distribution between the pellet core, mainly made up of iron oxide, and the pellet periphery dominated by Fe\textsubscript{met} varied, however, in accordance with the reduction degrees attained.
Figure 31 Pellet textures observed in LOM after reduction tests 7-10. Pellet core (left) and pellet periphery (right). Iron oxides = Grey, Fe_{met} = White, Pores = Black
Figure 32 Pellet textures observed in LOM after reduction tests 11-14. Pellet core (left) and pellet periphery (right). Iron oxides = Grey, Fe_{met} = White, Pores = Black
4 Discussion

4.1 Reduction

Laboratory reduction experiments at LTU were carried out in three different ways. Conditions for blast furnace simulating tests were based on a normal test program at LKAB. For the hypothetical PCR tests the reduction gas composition at various temperatures was calculated using heat and mass balances. The test start temperature was independent of temperature profile and hypothetical PCR. The simulated PCR tests were based on measurements in the EBF during different PCR. The test start temperature was not constant for all of the simulated PCR tests.

An increase in PCR, resulting in an increased reduction potential and increase in the temperature level, compensated for the loss in reduction time between the slow and the fast heating rates. Generally, a higher PCR resulted in a higher pellet reduction degree at an equal reduction time. However, the initial reduction temperature and heating rate were of importance for the reduction degrees attained. A higher start reduction temperature, as in the simulated high PCR, resulted in a faster initial reduction compared to a lower start temperature. On the other hand, a faster heating rate compensated for a lower start temperature and lower simulated PCR. Although there were differences in PCR, approximately equal reduction degrees were attained for the simulated low PCR and fast heating rate and simulated high PCR and slow heating rate at a reduction time exceeding 35 minutes. In general, an increased reduction time increases the final reduction degree at the end of the test. A small addition of H₂ to the reduction gas, compared to reduction without H₂, had a more prominent effect on the pellet reduction than an increase in the H₂ content.

In the EBF, pellets in samples taken out with the upper shaft probe have, as was confirmed by the chemical analysis as well as by LOM, during injection of the HV coal reached a higher reduction degree compared to during operation with the LV coal. In pellets taken out with the lower shaft probe significant differences in average reduction degree were not found. The results indicated that the test conditions in terms of the reduction potential of the gas, temperature profile and time for all cases resulted in a thermal reserve zone that was sufficient for reduction of pellets under all the investigated process conditions.

Pellet samples, which showed differences in LTB and ITH, indicated almost the same reduction behaviour. In the simulated blast furnace laboratory tests the
conditions were equal and differences in LTB and ITH obviously did not correlate to the reduction behaviour.

4.2 Texture

The initial reduction conditions, in terms of temperature and gas composition, were conclusive for the pellet texture formed. In the simulated PCR tests, differences in the pellet textures that were observed in samples after reduction to 1100°C were already found at a reduction degree of 40 percent. The thickness of the peripheral zone, in which $\text{Fe}_{\text{met}}$ dominates, increased with increasing reduction degree but distinct boundaries between the pellet core and periphery were not observed. The tests with simulated high PCR and slow heating rate and simulated low PCR and fast heating rate showed, despite similar reduction behaviour above a reduction degree of 25 percent, differences in pellet texture. These results support the claim that the texture differences were most likely to occur in the beginning of the reduction. A simulated low PCR resulted in a grain texture in the pellet core that was not observed at simulated high PCR. In the pellet periphery, iron oxides were surrounded by $\text{Fe}_{\text{met}}$ and the $\text{Fe}_{\text{met}}$ texture was denser at low PCR. The size of the pores in the pellet periphery was larger at high PCR. A higher start reduction temperature and a more rapid temperature increase in combination with an increase in the reduction potential of the gas counteracted the formation of a grain texture in the pellet core. At the same time, a porous $\text{Fe}_{\text{met}}$ texture was formed in the pellet periphery.

The present results indicate that the initial reduction temperature is more important for the pellet texture formed than the gas composition. Significant texture differences, which most likely were absent due to identical test start temperatures, were not observed in the pellet core after hypothetical PCR reduction in the laboratory furnace. The pellet peripheries showed a similar texture, except for the hypothetical PCR of 200 kg/tHM and slow temperature profile, where the areas of $\text{Fe}_{\text{met}}$ became denser. No distinct boundaries between the pellet core and periphery were observed in the pellets from the hypothetical PCR laboratory experiments.

In pellets taken out with the upper shaft probe during the EBF tests, $\text{Fe}_{\text{met}}$ was not observed during injection of the LV coal. During HV coal operation, a few areas of $\text{Fe}_{\text{met}}$ were observed in the pellet core, increasing in size and number towards the periphery. The results from pellets taken out with the lower shaft probe during the EBF tests indicated that a combination of a high temperature level and high $H_2$...
content in the reduction gas were two important parameters for the formation of smooth Fe\textsubscript{met} areas. A straggling Fe\textsubscript{met} texture was formed at lower reduction temperatures and H\textsubscript{2} levels in the reduction gas. The pores of the Fe\textsubscript{met} textures throughout the pellets and the solid Fe\textsubscript{met} areas in the pellet cores during injection of the HV coal were larger compared to operation with LV coal. The marked Fe\textsubscript{met} areas were observed to be more compact during operation with the HV coal type compared to the LV coal type. The straggling Fe\textsubscript{met} texture was, especially in the pellet periphery, identified during operation with the LV coal type, while HV operation resulted in a smooth Fe\textsubscript{met} texture. Examples of typical straggling and smooth Fe\textsubscript{met} textures are presented in Figure 33.

![Figure 33](image)

**Figure 33** Typical examples of a straggling Fe\textsubscript{met} texture (left) and a smooth Fe\textsubscript{met} texture (right).

Rounded and assembled slag areas were identified during HV coal operation, while LV coal injection resulted in scattered pattern of slag. The test conditions in the EBF, in terms of reduction potential of the gas and in-furnace temperature profile, did not result in formation of distinct product layers in the reduced pellets.

In the BF simulation tests the temperature and reduction conditions were the same for all the pellets tested. From this point of view it was reasonable that differences in the pellet texture did not occur between pellets of the same type. It should be noted that the influence of original pellet texture has not been considered in any of the investigations.

### 4.3 Process

Softened areas of slag and smoother Fe\textsubscript{met} texture in the pellets indicated that injection of the HV coal type increased the shaft temperature in the EBF. Due to the relatively high amount of volatile matters in the coal, the H\textsubscript{2} content in the ascending gas was higher during injection of the HV coal compared to during
injection of the LV coal. Increasing the PCR increases the H₂ content of the gas, independent of the coal type. The differences in ηCO were quite small. Consequently, the changes in reduction potential under different process conditions seemed to be more or less dependent on the H₂ content.

Fe₃C was observed in pellets taken out with the lower shaft probe in the EBF. The chemical analyses showed an increase in C content during injection of the HV coal type. It was therefore concluded that the formation of Fe₃C was more frequent during injection of the HV coal type. It was difficult to determine the cause of increased Fe₃C formation during injection of the HV coal, but there are some possible explanations. Fe₃C was formed in the shaft between the positions of the shaft probes. A higher amount of H₂ in the ascending gas facilitated the iron oxide reduction by H₂ and, as a consequence the CO concentration available for carburization increased as the CO to a lesser extent contributed to the reduction. Reaction of Fe₃C with wustite, reaction 14, was more likely to occur during injection of the LV coal type, since the pellet reduction degree was lower. Therefore, a lower carbon content should be expected when using the LV coal which is in agreement with the present results.

The differences in hot metal Si at SSAB BF No. 3 can not be correlated to the iron ore raw materials used during the sampling period. Differences in FeO content influencing the hot metal Si content were not expected in the EBF, since no significant differences in average reduction degrees in pellets taken out with the lower shaft probe were observed. During the EBF test, the trend of the hot metal was decreasing Si content, mainly due to increased CaO/SiO₂ of the slag.

4.4 Generation of Fines
The highest amount of fine material was observed during the HV coal operation during oxygen supply to the coal lance, which could be seen in the 0-6.3 mm material fraction taken out with the upper shaft probe. When the share of the 0-0.5 mm fraction increased, the analysis of corresponding samples showed an increased amount of Fe at the same time as the C content decreased. Injection of the HV coal at the high PCR increased the Fe content in the fines in the material taken out with the upper shaft probe. The generation of fine material leaving the EBF, and Fe content in flue dust, increased when injecting the HV coal. The highest pellet strength was observed at the highest reduction degrees; however, an increased
generation of fines in the 0-0.5 mm fraction was also evident, resulting in higher Fe\textsubscript{met} losses through the top gas.

It is believed that the Fe\textsubscript{met} textures formed in the pellet periphery during reduction will affect the generation of fines. During abrasion, the edges of the straggling Fe\textsubscript{met} areas get stuck to each other, while a smooth Fe\textsubscript{met} surface area does not exhibit this property. Since the Fe\textsubscript{met} areas with smooth Fe\textsubscript{met} surfaces dominated during HV coal injection, the Fe\textsubscript{met} texture is probably one explanation for the increase in fines generation compared to LV coal operation.

4.5 Sources of Error
According to the literature, a random distribution of pores has been observed in magnetite formed during reduction of hematite pellets, but magnetite without internal porosity was also observed\cite{18}. The magnetite textures were obtained at different temperatures and at different reduction gas compositions. In the present investigations it was assumed that no internal porosity existed in the magnetite and that the areas of similar colour, in which internal porosity was observed, consisted of wustite. There is a possibility that magnetite and wustite were not kept separate at all times.

Iron oxide reduction and carbon deposition occur simultaneously\cite{2}. It cannot be concluded for certain that carbon deposition did not occur during the laboratory reduction tests performed and if so was the case, a higher reduction degree compared to the ones presented might have been reached.

4.6 Concluding Discussion
The results from the EBF tests indicated that the test conditions in terms of the reduction potential of the gas, temperature profile and time for all cases resulted in an extension of the thermal reserve zone that was sufficient for reduction of pellets under all the investigated process conditions. The different Fe\textsubscript{met} textures observed in pellets taken out with the lower shaft probe indicated differences in reduction conditions at positions above the lower shaft probe in the EBF. The results indicate that operation with an LV coal was preferred if a high PCR was desired. The generation of Fe\textsubscript{met} fines would most likely have been decreased, thanks to the formation of straggling Fe\textsubscript{met} grains. The initial reduction conditions, in terms of temperature and gas composition, were in the performed investigations conclusive for the pellet texture formed.
5 CONCLUSIONS

In the present investigation, pellet reduction properties and their influence on the blast furnace process have been studied under different blast furnace operating conditions. Laboratory reduction tests as well as tests in the EBF were carried out. The investigation included also data from SSAB BF No. 3. The results lead to the following conclusions:

- Differences in hot metal Si content in a production blast furnace are difficult to correlate to raw material properties, since the process conditions are changed in order to control the heat level of the blast furnace.
- The pellets were well suited for blast furnace operation during different PCR; a conclusion supported by laboratory tests as well as EBF investigations. Results from the laboratory reduction showed that the decrease in reduction time with an increase in simulated or hypothetical PCR were compensated by increased reduction potential of the gas, temperature and porosity in the Fe\textsubscript{met} pellet periphery.
- The initial reduction conditions, in terms of temperature level and reduction gas composition, will have a significant effect on the pellet texture up to a reduction degree of at least 60 percent.
- The choice of injection coal type was conclusive for the reduction degrees attained in the upper shaft of the EBF. Injection of the HV coal generated a higher pellet reduction degree compared to the LV coal type. The differences in pellet reduction degree receded through the shaft.
- The choice of injection coal type was conclusive for the pellet strength, fines generation, the Fe\textsubscript{met} texture formed during reduction, extent of Fe\textsubscript{met} carburization and K distribution in the pellet.
- Injection of the LV coal type generated less fine material leaving the EBF with the top gases compared to operation with the HV coal type. Consequently, with respect to the generation of fines, the LV coal type should be chosen for operation at high PCR.
6 FUTURE WORK

The performed investigation has shown that the initial pellet reduction conditions, in terms of temperature and reduction gas composition, were decisive for the texture in the pellet periphery. An investigation should be carried out in order to determine the relationship between the Fe$_{\text{met}}$ texture in the pellet periphery and the generation of Fe$_{\text{met}}$ fines. An increased understanding in this matter would facilitate the possibility to predict fines generation from pellets as an outcome of, for example, different PCR. Investigations in order to assess whether there is a correlation between top gas temperature and composition and generation of and Fe content in flue dust and sludge should be carried out.

An increased PCR increases the ore-to-coke ratio. Higher demands are placed on pellet strength and ability to withstand generation of Fe$_{\text{met}}$ fines. Investigation to predict the highest PCR at which a normal amount of fines is generated ought to be carried out with different injection coal types or different reducing agents used for injection. At the same time, the pellet strength should be investigated.

Analyses of pellets taken out with the lower shaft probe in the EBF showed contents of Fe$_3$C. Carbon deposition and carburization take place in reduction gas mixtures of H$_2$ and CO$^2$. It is of interest to know more about how the reduction gas composition and temperature, which are affected by among other things the PCR, influence the carburization of pellets and their metallurgical properties. It is also important to learn more about how carburization of pellets influences the pellet strength and formation of Fe$_{\text{met}}$ fines. Reduction experiments, followed by pellet characterization, should be carried out in laboratory scale for different temperatures and reduction gas composition profiles.
7 REFERENCES


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29 LKAB, private conversation

Appendix I

Correlation Between Pellet Reduction and Some Blast Furnace Operation Parameters

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Correlation Between Pellet Reduction and Some Blast Furnace Operation Parameters

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Key words: blast furnace, pellet reduction, pellet properties, PCI, injection coal type, fines generation

INTRODUCTION

Modern blast furnace ironmaking aims at lowering consumption of reduction agents. Coke consumption is decreased while the amount of injected pulverized coal is increased. As the ore-to-coke ratio increases, so does load on the charged materials. An increased pulverized coal injection (PCI) will among other things affect the composition of the ascending reduction gases, the in-furnace temperature isotherms and possibly the position of the cohesive zone; all parameters important for the reduction of iron oxides. The loss of pellet strength depends on the reduction gas composition1.

As a result of increased PCI the Si content in molten metal tends to increase, and it is assumed that the pulverized coal type will have an influence on the Si content under high PCI operation2. In-furnace isotherms for 1000°C, determined with a feed-type vertical probe, go up in the shaft with the increase of PCI3. As a consequence, the cohesive zone will move upwards resulting in an increase in high-temperature furnace volume and a decrease in low-temperature furnace volume, and thus a possible decrease in the amount of indirect reduction of pellets. Due to the amount of volatile matters in the coal, increased PCI increases the H2 content in the reducing gas4. H2 is associated with higher reducing ability compared to CO. Indirect reduction of FeO by CO is exothermic, but endothermic by H25. Direct reduction of FeO by C to Fe is a strong endothermic reaction.

It is of interest to study the effect of high PCI on pellet reduction properties and to determine if the H2 in the reduction gas increases the indirect reduction to compensate for the loss of low-temperature furnace volume. It is also of interest to understand if an increased amount of FeO will reach the high-temperature zone and possibly affect the hot metal Si content. According to Loo et al., low-temperature disintegration increases the longer materials are subjected to low temperatures in the range 300-600°C. From this point of view, the low-temperature disintegration would decrease if the PCI rate were increased.

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The reduction behaviour of olivine pellets and the textures formed during laboratory reduction simulating hypothetical PCI rates are investigated. Reduction degrees and pellet strength attained in the LKAB Experimental Blast Furnace (EBF) using different levels of PCI and types of coal are discussed as well as the reduction gas compositions under the different process conditions. Possible correlations between pellet reduction behaviour and Si content in hot metal are discussed.

**MATERIAL**

Commercial olivine pellets are used in the investigations performed. Material samplings for the laboratory investigations are carried out at the LKAB pelletizing plant in Malmberget and at the SSAB BF No. 3 pellet-charging stream. The chemical composition of the olivine pellets MPBO and KPBO produced by LKAB can be seen in Table I.

<table>
<thead>
<tr>
<th>Pellet</th>
<th>Fe</th>
<th>FeO</th>
<th>CaO</th>
<th>SiO₂</th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>CaO/SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPBO</td>
<td>66.8</td>
<td>0.5</td>
<td>0.35</td>
<td>1.7</td>
<td>1.5</td>
<td>0.32</td>
<td>0.21</td>
</tr>
<tr>
<td>KPBO</td>
<td>66.6</td>
<td>0.4</td>
<td>0.46</td>
<td>2.0</td>
<td>1.5</td>
<td>0.23</td>
<td>0.23</td>
</tr>
</tbody>
</table>

During the EBF campaign MPBO is delivered by lorry from the pelletizing plant. Two different coal types are used in the investigations, a low-volatile coal containing 19.6 percent volatile components and a high-volatile coal containing 38.0 percent volatile components.

**EXPERIMENTS**

The Laboratory Reduction Furnace

The experimental apparatus used for reduction tests I-XII (see Table II), is shown in Figure 1. The furnace is a vertical steel tube-type furnace with an inner diameter of about 60 mm and is heated electrically by U-shaped Super-Kanthal elements with a constant temperature zone of about 80 mm in height. Digital Multi-Bus Flow-Bus regulators control the gas flows of H₂, CO, CO₂ and N₂. The gas is introduced in the bottom of the tube and heated in a bed of crushed ceramics. A thermocouple for temperature measurement and control is introduced from the bottom of the tube and situated approximately 20 mm below the sample, which is suspended in the balance with metal wires. The sample material is placed in a basket with three different levels. A water-cooled top for cooling of the sample after test completion is situated on top of the steel tube. A propane off-gas burner is placed on top of the furnace. Input gas composition and temperature are controlled and measured by a computer storing data with a frequency chosen for each parameter.

The furnace is heated to the start temperature of the test without any input gas. At the test start temperature a nitrogen flow at 12 l/min is introduced. The temperature and gas flow are held at constant values for a few minutes before the sample is introduced into the furnace. Sample start weight is between 70 and 85 grams of pellets and drying of the pellets is carried out outside the furnace prior to reduction. As soon as the sample has been introduced into the constant temperature zone, the test is started and the temperature program starts at the same time as the gas composition is changed into a reducing atmosphere. After the test, which can be interrupted at any point, the gas is changed into pure N₂ and the sample is put into the cooling top. Total gas flow is maintained at 12 l/min during the entire test.

In one laboratory test program the temperature and gas profiles are based on a normal test procedure at LKAB⁸. Two temperature profiles are used in the study. The slow heating rate is based on a normal test procedure at LKAB⁸ and the fast heating rate is based on a vertical temperature measurement made in the EBF during a PCI rate of approximately 130 kg/tHM. The gas compositions used for the different hypothetical process conditions with all-coke operation and hypothetical PCI rates are calculated using mass and heat balances for estimation of the gas composition at various levels in the blast furnace. Changing the hypothetical PCI rate mainly
influences the H₂ content in the gas. Test conditions are shown in Figure 2. Nitrogen is filled up to maintain the total gas flow at 12 l/min during the tests. Table II gives an overview of the experiments performed.

![Figure 2 Reduction profiles for calculated hypothetical all-coke, a PCI rate at 200 kg/tHM and a normal LKAB test. Slow temperature profile (left), and fast temperature profile based on a vertical temperature measurement in the EBF (right).](image)

Table II Schematic overview of blast furnace simulating reduction experiments performed.

<table>
<thead>
<tr>
<th>Test</th>
<th>Material</th>
<th>Temperature Profile</th>
<th>Gas Profile</th>
<th>Slow</th>
<th>Fast</th>
<th>All Coke</th>
<th>PCI 146 kg/tHM</th>
<th>PCI 200 kg/tHM</th>
<th>LKAB normal</th>
</tr>
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<tbody>
<tr>
<td>I</td>
<td></td>
<td>Temperature</td>
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<td>Temperature</td>
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<tr>
<td>III</td>
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<td>Temperature</td>
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<td></td>
<td>Temperature</td>
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<td></td>
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<tr>
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<td></td>
<td>Temperature</td>
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<tr>
<td>VII</td>
<td></td>
<td>Temperature</td>
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<td>Temperature</td>
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</tbody>
</table>

Table II is a schematic overview of blast furnace simulating reduction experiments performed. The temperature and gas profiles are shown for different conditions, including normal, PCI 146 kg/tHM, and PCI 200 kg/tHM. The EBF is quenched after a campaign, with nitrogen flow started to decrease and finally stop. Dissection can start after at least ten days of cooling.

**EBF**

The EBF has a working volume of 8.2 m³ and a diameter at tuyere level of 1.2 m. The height from tuyere level to stock line is 6 m, with three tuyeres separated by 120°. After a campaign, the furnace can be quenched. The process is interrupted, nitrogen throughput from the top is started, followed by a decreased and finally stopped blast. Dissection of the EBF can start after at least ten days of cooling and is carried out like an archaeological excavation. Basket samples introduced in the final hours of operation are recovered.

During operation, in-burden probes are used for sampling of the burden and for the measurement of the horizontal temperature- and gas profiles. The shaft probe material is divided in sub-samples. A packed probe will during ideal conditions generate 5 sub-samples for the higher probe and 6 for the lower. It is common that only 3-4 sub-samples are generated for each probe. A schematic drawing of the EBF can be seen in Figure 3.

![Figure 3 Schematic drawing of the EBF. Positions of probes and basket samples recovered at excavation indicated.](image)
In the EBF the pellet properties attained at the probe positions are investigated during operation with different PCI rates and injection of the high and the low-volatile coal. Using the low-volatile coal the average PCI rates are 152 and 79 kg/tHM, respectively and for the high-volatile coal 152 and 94 kg/tHM. The material sampled from the shaft probes is sieved and the pellet fraction >6.3 mm recovered. The strength of pellets taken out with the upper shaft probe are tested in an I-trommel with a diameter of 130 mm and a length of 700 mm. 300 grams of pellets in the size range 10-12.5 mm are rotated 600 times at a revolution of 20 rpm. Flue dust and sludge are sampled every fourth hour during the test periods with different process conditions.

**SSAB Blast Furnace No. 3**

At SSAB Tunnplåt, BF No. 3 produces about 2.2-2.3 M tonnes of hot metal every year. The ferrous burden consists entirely of olivine pellets. Two thirds of the average burden is MPBO and one third KPBO. The chemical composition can be seen in Table I. The low gangue content and good metallurgical properties of the olivine pellets together with the relatively low ash content of coke at SSAB are the main attributes for the high productivity and low slag volumes. Design data of BF No. 3 are presented in Table III.

### Table III Design data of BF No. 3

<table>
<thead>
<tr>
<th>BF No. 3</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Year of erection</td>
<td>2000</td>
</tr>
<tr>
<td>Design</td>
<td>SSAB/Kvaerner</td>
</tr>
<tr>
<td>Shaft of campaign</td>
<td>Aug 2000</td>
</tr>
<tr>
<td>Hearth diameter, m</td>
<td>11.4</td>
</tr>
<tr>
<td>Working volume, m³</td>
<td>2340</td>
</tr>
<tr>
<td>Hearth volume, m³</td>
<td>2850</td>
</tr>
<tr>
<td>No of tuyeres</td>
<td>32</td>
</tr>
<tr>
<td>No of tap holes</td>
<td>2</td>
</tr>
<tr>
<td>Runners</td>
<td>Replaceable troughs</td>
</tr>
<tr>
<td>Dust pressure, kPa</td>
<td>350</td>
</tr>
<tr>
<td>Molding equipment</td>
<td>Belt / BLT central feed</td>
</tr>
<tr>
<td>Daily output, tonnes</td>
<td>6700</td>
</tr>
</tbody>
</table>

Raw material samplings are carried out over a period of a few days at the pellet feeders close to the blast furnace. Process data, including the hot metal Si content, are stored at the same time. Low-temperature reduction disintegration tests are performed according to ISO13930, except for the pellet size fraction where pellets from the total samples are used instead of the 10-12.5 mm fraction. The reduction strength, ITH, is determined according to the description of Hooey et al. but pellets from the total samples are also used in this case.

**RESULTS**

**Laboratory Reduction for All-Coke and Hypothetical PCI Rates**

Increasing the hypothetical PCI rate increases the degree of reduction at any temperature once the reduction degree has reached about 10 percent; a pattern that is independent of temperature profile, see Figure 4. The most apparent difference in reduction behaviour is observed between a hypothetical all-coke case, tests I and IV, and a hypothetical PCI at 146 kg/tHM or 200 kg/tHM, tests II or III and V or VI. A clear difference between the hypothetical PCI rates of 146 and 200 kg/tHM, tests II and III is observed for the slow temperature profile at temperatures above 750°C.

![Figure 4 Temperature- and reduction profiles for laboratory reduction tests simulating hypothetical all-coke and different PCI.](image)

Significant differences in the iron oxide and Fe₉₈₉₈ textures after reduction are not observed when pellets from tests II and III are compared. Wustite is dominating in the pellet cores with a small number of Fe₉₈₉₈ areas. The size and number of the Fe₉₈₉₈ areas increase in the reaction fronts. After test III, Fe₉₈₉₈ dominates the pellet peripheries and after test II, Fe₉₈₉₈ surrounded by wustite...
dominates. Some areas of magnetite, surrounded by wustite, are found in the pellet cores and reaction fronts. In pellets from test I magnetite dominates the pellet core and wustite and Fe met the periphery. The area between the core and the periphery is made up of wustite. A few Fe met areas are observed inside the pellet periphery and in the pellet cores. Typical textures from test I and III are presented in Figure 5.

Figure 5 Pellet textures observed in light optical microscope after reduction tests I (upper) and III (lower). Pellet core (left), periphery (right) and reaction front (middle). Magnetite = Smooth grey, Wustite = Broken grey, Fe met = White, Pores = Black.

In pellets from reduction tests IV-VI, areas of Fe met are observed in the entire pellet cross section after tests V and VI. In pellets from test IV, Fe met is observed together with wustite and some magnetite in the pellet periphery. Magnetite dominates in the core and wustite in the area between the core and the periphery. In the pellet cores after test V and VI, wustite dominates, which is also the case in the area between the core and the pellet periphery. Magnetite is observed through the entire cross section of the pellets. Fe met and wustite dominate the pellet periphery. Typical textures from test IV and VI are presented in Figure 6.

Figure 6 Pellet textures observed in light optical microscope after reduction tests IV (upper) and VI (lower). Pellet core (left), periphery (right) and reaction front (middle). Magnetite = Smooth grey, Wustite = Broken grey, Fe met = White, Pores = Black.

**EBF Operation using Different PCI Rates and Injection Coal Types**

Figure 7 shows the horizontal profile of the reduction gas composition as percentages H_2 and CO.

\[
\% \text{CO} = \frac{100\% \text{CO}}{(\% \text{CO} + \% \text{CO}_2)},
\]

(1)

at the position of the upper shaft probe during the four test periods. An increase in the H_2 content is observed when the PCI rate is increased and is most distinct when the high-volatile coal is injected. As can be seen from the H_2 and CO profiles, during the tests, the reduction potential of the reducing gas is higher in the centre of the furnace, position 0, and decreases towards the periphery. The high PCI rate generates the highest CO content. The H_2 content during the EBF tests is in general at a higher level at the lower shaft probe due to the difference in temperature at the position of the shaft probes.
Figure 7 $H_2$ and $n_{CO}$ profiles at the position of the upper shaft probe in the EBF during tests with different PCI rates and a low and a high-volatile injection coal type. Position 0 indicates the centre of the EBF and the positive and negative extremes the periphery.

The particle size distribution of material taken out with the upper shaft probe and the content of Fe and C in the <0.5 mm fraction can be seen in Figure 8. The fine fractions, 0-0.5 mm, 0.5-3.3 mm and 3.3-6.3 mm, constitute a larger part of the material using the high-volatile injection coal compared to operation using the low-volatile coal. The Fe content in the <0.5 mm fraction increases at the same time as the C content decreases when the high-volatile coal is injected at the high PCI rates.

Injection of the high-volatile coal results in the highest reduction degree of pellets taken out with the upper shaft probe. Correlations between operation parameters and the pellet reduction degree are not observed at the level of the lower shaft probe. The pellet reduction degrees reached in sub-samples 1-3 during the test periods are presented in Figure 9. The results from the I-trommel tests are presented in Figure 10. The highest amount of the fraction >6.3 mm is found during injection of the high-volatile coal type, indicating low disintegration, but it also shows the highest amount of abrasion in the <0.5 mm fraction. As can be seen in Figure 11, a linear relation is obtained between the pellet reduction degrees and the disintegration. The highest pellet strength and the highest reduction degree are measured in samples taken during injection of the high-volatile coal.
Figure 9 Pellet reduction degrees in sub-samples from samples taken out with the upper (left) and the lower (right) shaft probe in the EBF.

Figure 10 Pellet strength after I-trommel test of sub-samples taken out with the upper shaft probe.

Figure 11 Relation between pellet reduction degree and pellet disintegration in probe samples taken out with the upper shaft probe in the EBF.

The amount of flue dust and sludge generated per tonne of hot metal is shown in Figure 12. The total amount of fines leaving the furnace with the top gas reaches the highest level injecting the high-volatile coal. Figure 13 shows the amount of Fe and C in flue dust and sludge at different process conditions. Injection of the high-volatile coal increases the Fe content in the flue dust at the same time as the amount of C decreases.
Evaluation of Process Data and Pellet Samples from SSAB Blast Furnace No. 3

During the material sampling period, the Si content in the hot metal reaches between 0.15 and 0.54 percent and decreases are observed between observation 19 to 28 and 38 to 49, see Figure 14. The 74 Si observations correspond to measurements during 48 hours. Samples of MPBO and KPBO for further testing are chosen during a period of increase and decrease in the Si content in hot metal. Sampling occasions A-H and the relation to the hot metal Si content are marked in Figure 14, which also shows the PCI rate during the period in question. The particle size distributions are shown in Figure 15. Low-temperature reduction disintegration and the reduction strength for the investigated samples are shown in Figure 16.
Figure 15 Particle size distribution for MPBO (left) and KPBO (right) for the pellet samples A-H.

Figure 16 Low-temperature reduction disintegration (left) and reduction strength (right) presented as fractions >6.3 mm and <0.5 mm for the pellet samples A-H.

Considering the >6.3 mm fraction, the lowest, highest, and a median value correspond for the low-temperature reduction disintegration of MPBO to samples B, G and E, respectively, and for KPBO to samples B, F and G, respectively. The reduction strength in the >6.3 mm fraction reaches the lowest, highest and a median value for MPBO in samples B, E and G, respectively, and for KPBO in samples B, G and F. Reduction profiles of the MPBO samples B, E and G (tests VII-IX, see Table II) and KPBO samples B, F and G (tests X-XII, see Table II) indicate almost the same reduction behaviour, see Figure 17.

Figure 17 Reduction profiles for laboratory reduced MPBO samples B (VII), E (VIII), G (IX) and KPBO B (X), F (XI) and G (XII).

After reduction, $F_{\text{calc}}$ is dominating in the pellet peripheries and a few small areas are found in the pellet core. Wustite dominates the cores and some magnetite is observed. The areas of $F_{\text{calc}}$ grow at the reaction front and the pore sizes are larger compared to the appearance in the cores. Differences in $F_{\text{calc}}$ texture are observed between the pellet types. The MPBO samples show larger areas of coherent $F_{\text{calc}}$ in the pellet peripheries compared to the KPBO samples. Figure 18 shows typical textures of the MPBO and KPBO pellets.

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At the EBF excavation after campaign No. 12, basket samples containing material from the same samples as tests VII-XII are recovered below the position of the upper and lower shaft probes, see Figure 3. The reduction degrees, calculated with respect to the weight loss, are about 30 percent for the upper samples and between 38 and 52 percent for the lower samples. Magnetite and wustite dominate the pellet textures at a reduction degree of 30 percent, although small areas of Fe\textsubscript{met} can be found. The size and number of Fe\textsubscript{met} areas increase with increasing reduction degree. All of the basket samples containing the same material as in tests VII-XII show for the same pellet type similar iron oxide and Fe\textsubscript{met} textures after reduction in the EBF.

**DISCUSSION**

**Effects of PCI Rate and Coal Type**

In the EBF, pellets in samples taken out with the upper shaft probe have during injection of the high-volatile coal reached a higher level of reduction degree compared to pellets in samples taken during injection of the low-volatile coal. The H\textsubscript{2} content at the level of the upper shaft probe is higher during high PCI rate of the high-volatile coal compared to during injection of the low-volatile coal. Increasing the PCI rate increases the H\textsubscript{2} content of the gas independent of the coal type. The differences in H\textsubscript{2} are quite small and consequently, the changes in reduction potential under different process conditions seem to be more or less dependent on the H\textsubscript{2} content. Because of central coke charging, the reduction potential of the gas is higher in the centre. Although there are differences in gas composition, no significant differences in reduction degrees can be correlated to the PCI rates at the position of the upper shaft probe. Temperature measurements indicate that temperature isotherms in the reserve zone move upward in the shaft when the PCI rate is increased. In pellets taken out with the lower shaft probe, significant differences in average reduction degree are not found. The results indicate that the test conditions, in terms of the reduction potential of the gas, temperature profile and time for all cases, result in an extension of the thermal reserve zone that is sufficient for reduction of pellets under all the investigated process conditions.

Since no significant differences in average reduction degrees in pellets taken out with the lower shaft probe are observed, any differences in FeO content influencing the hot metal Si content are not expected in the EBF tests. The trend of the hot metal is decreasing Si content during the EBF test, mainly dependent on increased CaO/SiO\textsubscript{2} of the slag.

During hypothetical reduction of iron oxides, a reduction degree of 13 percent corresponds to complete reduction from hematite to magnetite. Complete reduction to wustite corresponds to a reduction degree of 33 percent. The average reduction degrees attained in pellets taken out with the upper shaft probe when low-volatile coal is injected are 17 and 25 percent. The average reduction steps, from magnetite to wustite, are endothermic at indirect reduction by CO as well as H\textsubscript{2}. When injecting the high-volatile coal the average reduction degrees attained in pellets taken out with the upper shaft probe are 37 and 42 percent. The average indirect reduction steps are from wustite to Fe\textsubscript{met} endothermic by H\textsubscript{2} and exothermic by CO.

Since the indirect reduction of wustite by H\textsubscript{2} is endothermic, but less endothermic at increased temperatures, it is assumed to take place in the lower part of the shaft. A high H\textsubscript{2} content in the reduction gases, as attained at high PCI rate, results in an increased average indirect reduction by H\textsubscript{2} in the lower part of the shaft. Investigations of the pellets taken out with the lower shaft probe do not show whether the indirect reduction at this position is predominantly by CO or H\textsubscript{2}.
Injection of the high-volatile coal generates the highest amount of fine material in the fractions 0-6.3 mm taken out with the upper shaft probe. When the share of the 0-0.5 mm fraction increases in the total material samples, the analysis of corresponding samples shows an increased amount of Fe at the same time as the C content decreases. Injection of the high-volatile coal at the high PCI rate increases the Fe content in the fines in the material taken out with the upper shaft probe. The generation of and Fe content in flue dust increase when injecting the high-volatile coal. The highest pellet strength is observed at the highest reduction degrees attained when high-volatile coal is injected; however, an increased abrasion in the 0-0.5 mm fraction is also evident, resulting in higher Fe losses through the top gas. Comparing temperature profiles in the upper part of the shaft based on vertical temperature measurements, the production rate and rate of descent, it is probable that the dwell time in the upper part of the EBF is approximately the same at different PCI rates, and correlations between the PCI rates and low-temperature disintegration are not found in the tests performed.

Changing the PCI rate affects the temperature and gas profiles in the blast furnace. Adjusting the laboratory conditions to different PCI operations, an increased heating rate is assumed to correspond to an increase in PCI rate. Considering laboratory tests I-VI, an increased hypothetical PCI rate increases the reduction degree attained. At equal hypothetical PCI rate the highest reduction degree is reached for the slow temperature profile. Approximately equal reduction degrees are attained for tests I, slow temperature profile and hypothetical all-coke, and VI, fast temperature profile and a hypothetical PCI rate of 200 kg/tHM. In this case an increase in hypothetical PCI rate is necessary to compensate for the decrease in reduction time between the slow and fast temperature profiles. When the PCI rate in the blast furnace is increased the laboratory investigations indicate that an increased H2 content in the reduction gas can compensate for the possible loss of low-temperature volume, which is confirmed by the EBF test.

The amount of Fe formed in the pellet periphery increases when increasing the hypothetical PCI rate in samples I-VI. The slow heating rate and consequently an increased test time result in peripheries containing a higher amount of Fe compared to pellet peripheries from fast heating rate tests. At a hypothetical PCI rate of 200 kg/tHM the areas of Fe_{eut} in the pellet periphery become denser when increasing the reduction time.

**Process Observations and Pellet Properties at SSAB**

Although differences in low-temperature disintegration and reduction strength are observed in pellet samples taken out at SSAB in connection to increases and decreases in the hot metal Si content, no major differences in reduction behaviour and pellet textures after reduction are observed. The differences in hot metal Si are assumed to be a consequence of different PCI rates used to obtain the best possible process conditions. In the SSAB BF No. 3 the pellets are exposed to different conditions as a result of the process control. Possible differences in the pellet reduction behaviour in the upper part of the shaft are assumed to reappear in the shaft and not affect the hot metal Si content. During the pellet-sampling period the high-volatile coal is injected in the SSAB BF No. 3, which verifies the assumption of equal pellet reduction attained in the lower shaft. In the laboratory tests the conditions are equal for tests VII-XII and, consequently, the absences of differences in pellet reduction behaviour are not surprising.

**CONCLUSIONS**

The reduction properties of MPBO are well suited for stable blast furnace operation under different PCI rates. Injection of the high-volatile coal results in a higher reduction degree of pellets in the upper shaft. The differences in reduction degree recede through the shaft. The pellet strength, measured by I-trommel test on pellets in sub-samples taken out with the upper shaft probe in the EBF, increases when injecting the high-volatile coal. At the same time, the pellet abrasion, considering Fe_{eut} in flue dust and the <0.5 mm fraction after I-trommel test, increases.

At a high laboratory hypothetical PCI rate the Fe_{eut} texture in the pellet periphery becomes more compact with increasing reduction time.

Differences in hot metal Si content in a production blast furnace are difficult to correlate to raw material properties, since the process conditions are constantly changed in order to control the heat level of the blast furnace.

**ACKNOWLEDGEMENTS**

We are grateful to the Swedish National Energy Administration (STEM) and the Agricola Research Centre for financial support. We would also like to thank the members of the Swedish Steel Producers’ Association JK21059 project for fruitful discussions. Thanks to SSAB Tunnplåt AB and LKAB for supporting the work done and providing of materials, analysis, etc. Finally, we thank Mr. Johan Folkesson for designing the laboratory furnace control system at LTU.

**REFERENCES**


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8. LKAB, private conversation


Appendix II

Effect of Simulated PCI Rate on Olivine Pellet Reduction

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Accepted for publication in proceedings from The 4th International Congress on the Science and Technology of Ironmaking, Osaka, Japan, November 2006
EFFECT OF SIMULATED PCI RATE ON OLIVINE PELLET REDUCTION

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ABSTRACT
Reduction behaviour and textures formed during laboratory simulated BF tests with olivine pellets are presented and discussed. Test design is based on gas and temperature profiles during operation at a high and a low pulverized coal injection (PCI) rate with a low-volatile coal in the LKAB experimental blast furnace (EBF). Texture differences, introduced prior to a reduction degree of 40 percent, are observed in the iron oxide in the pellet core and in the Fe met pellet periphery. A simulated high PCI rate decreases the reduction time of the pellets. The olivine pellets investigated are well suited for blast furnace operation at different PCI rates and accordingly different production rates.

1. INTRODUCTION
Modern blast furnace iron making continuously strives at an increase of the amount of injected pulverized coal and a decrease in the coke consumption. The increased PCI will affect parameters important for the reduction of iron oxides as for example the composition of the ascending reduction gases, the in-furnace temperature isotherms and the position of the cohesive zone.

Measurements, of for example temperature profiles and reduction gas compositions, made during operation of the EBF are the basis of laboratory reduction profiles corresponding to differences in a blast furnace operated at various PCI rates. The reduction behaviour of olivine pellets and textures formed during laboratory reduction simulating a high and a low PCI rate at a centre and an intermediate position in the furnace are presented and discussed.

2. MATERIAL
In the investigations performed the commercial olivine pellets MPBO produced by LKAB are used. The chemical composition of the MPBO can be seen in Table 1.

Table 1 Chemical composition of MPBO in percent

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>FeO</th>
<th>CaO</th>
<th>SiO₂</th>
<th>MgO</th>
<th>Al₂O₃</th>
<th>CaO/SiO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>MPBO</td>
<td>66.8</td>
<td>2.5</td>
<td>0.35</td>
<td>1.7</td>
<td>1.5</td>
<td>0.32</td>
<td>0.21</td>
</tr>
</tbody>
</table>

3. EXPERIMENTS
3.1 EBF
The EBF has a working volume of 8.2 m³, a diameter at tuyere level of 1.2 m and is equipped with a system for injection of reduction agents. During operation, in-burden probes are used for the measurement of the horizontal temperature- and gas profiles. Estimation of the positions of the in-furnace temperature isotherms is enabled by a thermocouple which descends with the burden. A drawing of the EBF can be seen in Fig. 1.

Fig. 1 Drawing of the EBF

Measurements in the EBF are carried out during operation at different average PCI rates, 150 and 80 kg/tHM, respectively. A low-volatile coal containing 19.6 percent volatile compounds is used. Process data, including the top gas temperature and composition and burden decent rate, are stored during operation.

3.2 The laboratory reduction furnace
The experimental apparatus used for reduction tests is shown in Fig. 2. The furnace is a vertical steel tube-type furnace with an inner diameter of about 60 mm and is heated electrically by U-shaped Super-Kanthal elements with a constant temperature zone of about 80 mm in height. Digital Multi-Bus Flow-Bus regulators control the gas flows of H₂, CO, CO₂ and N₂. The gas is introduced in the bottom of the tube and heated in a bed of Al₂O₃ balls. A thermocouple for temperature measurement and control is introduced from the bottom of the tube and situated approximately 20 mm below the sample, which is suspended in the balance with metal wires. The sample material is placed in a basket. A water-cooled top for cooling of the sample after test completion is situated on top of the steel tube. Input gas composition and temperature are controlled by a computer that stores data with a frequency chosen for each parameter.

At the test start temperature a nitrogen flow at 12
l/min is introduced. The temperature and gas flow are held at constant values for a few minutes before the sample is introduced into the furnace. Sample start weight is between 80 and 83 grams of pellets. Drying of the pellets is carried out outside the furnace prior to reduction. As soon as the sample has been introduced into the constant temperature zone, the test is started and the temperature program starts at the same time as the gas composition is changed into a reducing atmosphere. After the test, which can be interrupted at any point, the gas is changed into pure \( \text{N}_2 \) and the sample is transferred into the cooling top. Total gas flow is maintained at 12 l/min during the entire test.

### 3.3 Heating rate and gas composition profiles

In the laboratory test programs the heating rate and gas profiles are estimated from measurements made in the EBF during operation at the high and the low PCI rates. The heating rate profiles are estimated from:

- Vertical temperature measurements
- Average burden decent rates
- Horizontal temperature profiles at the position of the shaft probes
- \( \text{CO}/\text{CO}_2 \) ratio at the position of the lower shaft probe

For the high and the low PCI rates, a fast heating rate is estimated to simulate a centre profile and a slow heating rate is estimated to simulate an intermediate/wall profile.

The reduction gas compositions are estimated from the top gas analysis and the gas composition at the position of the upper and lower shaft probes. The gas compositions used together with the fast heating rates are estimated from the centre gas composition in the EBF. For the slow heating rates, the gas compositions at an intermediate position in the EBF are the basis of the reduction gas composition. \( \text{N}_2 \) is filled up to a gas flow of 100 percent. Test conditions are shown in and Fig. 3 and Fig. 4.

One set of experiments is interrupted at a reduction degree of approximately 40 percent and one set is interrupted at a furnace temperature of 1100°C. Table 2 gives an overview of the experiments performed.

### 4. RESULTS

#### 4.1 Reduction profiles

Fig. 5 shows the reduction profiles for tests I-VIII. For the same level of simulated PCI rate, at the test end temperature of 1100°C, a higher reduction degree is attained for the slow heating rates when tests IV and II and tests VIII and VI, are compared. After reduction simulating the fast heating rate profiles, tests II and VI, approximately equal reduction degrees are attained independent of simulated PCI rate. A higher reduction degree is attained for the slow heating rates at the end of test IV, simulated high PCI rate, compared to test VIII, simulated low PCI rate. In tests I, III, V and VII, the reduction is interrupted at a reduction degree of approximately 40 percent.
The rate of reducibility at 40 percent reduction and the average rate of reduction during the total test time to reach 1100˚C for tests II, IV, VI and VIII are presented in Table 3.

<table>
<thead>
<tr>
<th>Test</th>
<th>R40 (%/min)</th>
<th>Average Red. Rate (%/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>II</td>
<td>0.95</td>
<td>0.97</td>
</tr>
<tr>
<td>IV</td>
<td>0.87</td>
<td>0.77</td>
</tr>
<tr>
<td>VI</td>
<td>0.81</td>
<td>0.77</td>
</tr>
<tr>
<td>VIII</td>
<td>0.71</td>
<td>0.59</td>
</tr>
</tbody>
</table>

4.2 Pellet textures

Significant differences in the iron oxide textures are observed in the pellet core when pellets from the reduction tests simulating the high and low PCI rate are compared. A grain texture is observed in the pellet core of samples after tests V-VIII, simulating the low PCI rate, an observation that is not discernible after tests I-IV, simulating the high PCI rate. Textures from the pellet core and periphery from tests I-VIII are presented in Fig. 6.

Together with the areas of Fe\(_{\text{met}}\) in the pellet periphery, a few areas of iron oxide are present after tests II and IV, which are full-length tests simulating the high PCI rate. After full-length tests simulating the low PCI rate, tests VI and VIII, the corresponding areas in the pellet periphery show noticeable areas of iron oxide surrounded by Fe\(_{\text{met}}\) within the texture dominated by Fe\(_{\text{met}}\).

A few areas of Fe\(_{\text{met}}\) are observed in the pellet core from test samples interrupted at the test end temperature of 1100˚C. Fe\(_{\text{met}}\) is not observed in the core of pellets reduced to a reduction degree of approximately 40 percent. Significant differences in the Fe\(_{\text{met}}\) texture of the pellet periphery, independent of final reduction degree, are not observed between pellets reduced according to similar reduction profile. The distribution between the pellet core, mainly made up of iron oxide, and the pellet periphery dominated by Fe\(_{\text{met}}\) varies, however, in accordance with the reduction degrees attained.
5. DISCUSSION

Although the laboratory reduction profiles are estimated from measurements in the EBF during operation using different PCI rates it cannot be determined that they completely correspond to changes in PCI rates. Many factors influence the position of the in-furnace temperature isotherms, the ascending gas composition, the burden descent rate and the entire blast furnace process. However, the characteristics of the laboratory reduction profiles correspond to the different process conditions obtained during operation at a high and a low PCI rate.

The reduction degrees attained at 1100°C are from the fast heating rate profiles, tests II and VI, approximately equal. The extensions of peripherical zones dominated by Fe$_{\text{met}}$ are the same size. Comparing pellets from test VI with pellets from test II, a denser texture, also containing iron oxide in the pellet periphery, is observed in pellets from test VI. A few Fe$_{\text{met}}$ areas are found in the pellet cores. In the cores of pellets from test II no grain texture is observed, which is the case in pellets from test VI. For test II, the R40 is 0.95 %/min and for test VI 0.81 %/min. The average rate of reduction during the total reduction time to reach 1100°C is higher for test II compared to test VI.

A higher reduction degree is at the test end temperature of 1100°C attained for test IV than observed for test VIII when the slow heating rates are compared. The Fe$_{\text{met}}$ texture in the pellet periphery is denser in pellets from test VIII than test IV and also contains iron oxide surrounded by Fe$_{\text{met}}$. A few Fe$_{\text{met}}$ areas are found in the pellet cores. In pellets from test VIII a grain texture is observed in the pellet core, which is not the case in pellets from test IV. The R40 is for test IV 0.87 %/min and for test VIII 0.71 %/min. The average rate of reduction during the total reduction time to reach 1100°C is higher for test IV compared to test VIII.

Comparisons of the results from tests II and VI and IV and VIII, respectively, show an increased reduction rate for the test simulating the high PCI rate. Reduction degrees, at least as high as for the simulated low PCI rates, are attained for the simulated high PCI rates. The increased reduction potential of the gas and the increase in the temperature level compensates for the loss in reduction time between the simulated low and high PCI rates in tests VI and II and IV and VIII. It has been previously observed that an increase in hypothetical PCI rate is necessary to compensate for a decrease in reduction time$^3$.$^4$

According to the results from the laboratory experiments, a sufficient pellet reduction will take place at an increase of the PCI rate and at an increase of the production rate in the blast furnace. Based on the present results it can therefore be concluded that the pellets are well suited for blast furnace reduction at different PCI rates.

Differences in the pellet texture are to be found in pellet cores where a grain texture is observed after test VI, but not after test IV. The Fe$_{\text{met}}$ texture in the pellet periphery is dense and contains some iron oxides in pellets after test VI, whereas the Fe$_{\text{met}}$ texture after test IV shows more pores. The average reduction rates are equal. The R40 of test IV exceeds the R40 of test VI. Since reduction profiles are similar above a reduction degree of 25 percent the texture differences are most likely to occur already in the beginning of the reduction.

Differences in the pellet textures that are observed in samples after reduction to 1100°C are already found at a reduction degree of 40 percent. These observations support the claim that the observed texture differences are introduced at the beginning of reduction. A higher start reduction temperature and a more rapid temperature increase in combination with an increase in the reduction potential of the reduction gas counteract the formation of a grain texture in the pellet core. At the same time, a porous Fe$_{\text{met}}$ texture is formed in the pellet periphery. It should, however, be noted that the influence of the original pellet texture has not been considered in the investigation.

In the blast furnace, elements such as alkalis are present and influence the pellet reduction and most probably the pellet texture. These effects combined with influences of PCI rates are not studied in the present investigation.

6. CONCLUSIONS

The performed laboratory reduction experiments show that the initial reduction conditions, in terms of temperature level and reduction gas composition, will determine the pellet texture up to a reduction degree of at least 60 percent.

Based on the present results it can be concluded that the pellets are well suited for blast furnace reduction at different PCI rates. Although the reduction time decreases with an increase in PCI rate, the present result indicates that this is compensated by increased reduction potential, temperature and porosity in the Fe$_{\text{met}}$ pelletal periphery.

ACKNOWLEDGEMENTS

We are grateful to the Swedish National Energy Administration (STEM) and the Agricola Research Centre for financial support. The members of the Swedish Steel Producers’ Association JK21059 project are acknowledged. Thanks to LKAB and Metlab for support and assistance in the work. Finally, we thank Mr. Johan Folkesson for designing the laboratory furnace control system at LTU.

REFERENCES

Appendix III

Effect of Different PCI Practice on the Texture Obtained During Reduction of Iron Oxide Pellets

U. Leimalm, L. Sundqvist Ökvist and B. Björkman

Manuscript
Effect of Different PCI Practice on the Texture Obtained During Reduction of Iron Oxide Pellets

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Introduction

In modern blast furnace ironmaking, producers continuously strive to reduce coke consumption by replacing coke with e.g., an increased amount of injected pulverized coal. Reducing agents are injected mainly for the sake of economy. As the ore-to-coke ratio increases, so does the load on the charged material and the demands on material strength. An increase in the pulverized coal injection rate (PCR) will affect, among other things, the composition of the ascending gases, the position of the in-furnace temperature isotherms and possibly the position of the cohesive zone. All of these parameters are important for the reduction of iron oxides.1)

As the PCR increases, the H₂ content in the reduction gas will be higher due to an increase in the amount of volatile matters (VM) in the supplied coal.2) It is likely that a high-volatile (HV) coal will generate a higher H₂ content than a low-volatile (LV) coal. In-furnace isotherms for 1000°C, determined with a feed-type vertical probe, move upwards in the shaft with the increase of PCR.3) As a consequence, the cohesive zone will also move upwards, resulting in an increase in high-temperature furnace volume.
A rapid reduction from hematite to wustite without formation of distinct product layers has been reported in pellets of high porosity. The following reduction step of wustite to iron was relatively rapid. The greater the driving force for reduction, the more pronounced was the formation of the product layers. In the case of dense sintered hematite pellets, the reduction proceeded topochemically during the formation of product layers. The initial reduction temperature was of major importance for the pore structure of the reduced iron and it became coarser as the temperature of reduction increased. Subsequent rise in reduction temperature did not readily coarsen the pore structure that was formed at lower temperatures. Studies have shown that the conditions for the formation of porous iron depend on the gas composition, reaction temperature, oxide stoichiometry and the presence of impurity elements in solid solution in the FeO.

Laboratory investigations by Geva et al. have shown that the presence of impurity elements in solid solution in the iron oxide affect the final iron product morphologies. The presence of Mg, Ti, Si, Ca, Na and K together with FeO resulted in a decrease in the CO concentrations necessary to obtain porous iron growth at any reaction temperature relative to reaction with pure wustite. P had a marginal effect on the porous/dense iron transition and Al restricted the range of gas compositions over which porous iron could be obtained. In H₂/H₂O mixtures, the presence of Ti, Mg, P, Si, Ca, K and Na favoured porous iron formation. Additions of Al made the formation of a porous iron layer difficult. The effects of impurities were additive. Na and K are present in the blast furnace and are generally undesirable, but can on the other hand play a significant role in improving the reducibility of the burden. K has been reported to function as a catalyst for the reduction of iron oxide. Since the solubility of K in bulks of iron oxides has been reported not to exceed 0.1 wt%, much of the K is expected to exist on the surface of iron oxides.

In the blast furnace, the accumulated amounts of K, Na and F have been shown to be greater in the lower shaft than in the upper shaft. The gas stream is the transportation medium for these elements. Alkalis are transported from gas to burden in the lower temperature zone and from burden to gas in elevated temperature zones. Laboratory experiments carried out by Lectard et al. showed that an alkaline atmosphere generated with a coke impregnated with potassium carbonate would, especially at low temperatures, have a catalytic effect on the reduction of sinter.
Pellet reduction properties are affected by the reduction gas composition and temperature. As a consequence, differences in reduction behaviour and pellet textures arise. In the blast furnace, a number of factors influence the conditions for pellet reduction. In the present study the effects of PCR, injection coal type and method for oxygen addition on the pellet reduction behaviour were investigated. Tests were carried out in the LKAB Experimental Blast Furnace (EBF) and in laboratory scale. Possible connections to dust forming are also discussed.

Experimental

Materials

Commercial pellets produced by LKAB were used in the investigations performed. Material sampling of pellets for the laboratory investigations was carried out at an LKAB pelletizing plant. The chemical composition of the olivine pellets is presented in Table 1. Two different coal types were used in the EBF trial; an LV coal containing 19.6 percent VM and an HV coal containing 38.0 percent VM. Coke in the 15-30 mm fraction and BOF slag in the 9-15 mm fraction was supplied by SSAB Tunnplåt AB. To obtain a desired slag basicity and slag volume, quartzite, BOF slag and limestone were used as slag formers.

The EBF

The EBF situated at MEFOS has a working volume of 8.2 m³ and a diameter at tuyere level of 1.2 m and is equipped with a system for injection of reduction agents. The pulverized coal injection (PCI) system is an oxy-coal system and the oxygen added to the lance can be replaced by air if desired. The height from tuyere level to stock line is 6 m and there are three tuyeres separated by 120°. During an EBF trial, the PCR, coal type and methods for oxygen supply were changed for the 6 test periods presented in Table 2. In the tables and figures, operation with oxygen added to the lance is termed Oxy Coal and operation with air addition to the lance termed Air Coal. The behaviour of the pellets related to the pre-set process conditions was investigated and evaluated.

During operation, in-burden probes, employed for each parameter setting, were used for sampling of the burden and for measurement of the horizontal gas- and temperature profiles. The material taken out with the horizontal probes was divided in sub-samples. 3-4 sub-samples were generated for each probe. 5 sub-samples are during ideal conditions generated
from the upper shaft probe and 6 from the lower. Estimation of the positions of the in-furnace temperature isotherms was done by allowing a thermocouple to descend with the burden.

**Laboratory Reduction**

*The laboratory reduction furnace*

The experimental apparatus used for reduction tests is shown in Figure 1. The furnace is a vertical steel tube-type furnace with an inner diameter of about 60 mm and is heated electrically by U-shaped Super-Kanthal elements with a constant temperature zone of about 80 mm in height. Input gas composition and temperature are controlled by a computer which also is used to store data with a pre-set frequency chosen for the parameters time, temperature and input gas composition. The gas supply system is equipped with digital Multi-Bus Flow-Bus regulators for input gas flows of CO, H2, CO2 and N2. The gas is introduced in the bottom of the tube and heated in a bed of Al2O3 balls. A thermocouple for temperature measurement and control is introduced from the bottom of the tube and situated approximately 20 mm below the sample, which is suspended in the balance with metal wires. The sample material was placed in a basket. A water-cooled top for cooling of the sample after test completion is situated on top of the steel tube. At the test starting temperature, a nitrogen flow at 12 l/min was introduced. The temperature and gas flow were held at constant values for a few minutes before the sample was introduced into the furnace. Each sample consisted of dry pellets with an initial weight of 80 to 83 grams. As soon as the sample had been introduced into the constant temperature zone, the test was started and the temperature program started at the same time as the gas composition was changed into a reducing atmosphere. After the test, which could be interrupted at any point, the gas was changed to pure N2 and the sample was transferred into the cooling top. Total gas flow was maintained at 12 l/min during the entire test.

**Heating rate and gas composition profiles**

The laboratory test programs were based on measurements in the EBF during test periods 1 and 3. A fast heating rate was estimated to simulate a centre profile and a slow heating rate was estimated to simulate an intermediate/wall profile. The heating rate profiles were estimated from vertical temperature measurements, average burden decent rates, horizontal temperature profiles at the position of the shaft probes and CO/CO2 ratios at the position of the lower shaft probe for temperature estimation from an oxygen potential diagram. The gas composition at different temperatures was based on the top gas compositions and the in-
burden gas analysis made with the upper and lower shaft probes during test periods 1 and 3. The gas compositions used together with the fast heating rates were estimated from the centre gas composition in the EBF. For the slow heating rates, the gas compositions at an intermediate position in the EBF were the basis of the reduction gas composition. Figure 2 and Figure 3 show the gas and temperature profiles used in laboratory reduction tests. \( \text{N}_2 \) was filled up to maintain a total gas flow of 12 l/min during the tests. One set of experiments was interrupted at a reduction degree of approximately 40 percent and one set was interrupted at a furnace temperature of 1100°C. An overview of the experiments performed is given in Table 3.

**Evaluation of pellet properties**

**Pellet textures**

Pellet textures were investigated by light optical microscopy (LOM) and by scanning electron microscopy (SEM). Prior to observation, pellets were cold-mounted in epoxy resin. In order to observe the largest pellet cross section, the mounts were cut at the maximum pellet diameters. The surface of the section was polished. A Nikon E600 POL polarizing microscope was used for LOM. For SEM, a Philips XL 30 equipped with Energy Dispersive X-ray Analysis (EDS) for chemical mapping was used. Prior to SEM investigation, the cross sections of the mounted pellet samples were coated with a thin layer of gold using a Bal-Tec MCS 010 sputter coater.

**Identification of trace elements in pellets**

In order to identify trace elements in the pellet periphery and core, pellets from sub-sample 3 taken out with the lower shaft probe were rotated in an I-drum at 600 revolutions for 30 minutes. After I-drum treatment, pellet fragments in the size range 212\( \mu \)m – 1.5 mm and >10 mm were separately ground in a ring mill and X-Ray diffraction was carried out on the ground materials. A Siemens X-ray diffractometer was used to record scattering intensities of samples by using a Copper K\( \alpha \) radiation (40 kV, 40mA) as the X-ray source. The samples were scanned over an angular range of sin20 10-90’ by using a step size of 0.020’ and over sin20 35-55’ with a step size of 0.020’ and a step time of 6 seconds at each step. For the >10 mm fraction, XRD and chemical analyses were carried out after operation at the low PCR, periods 3 and 4.
Results

EBF
The horizontal H₂ profile at the position of the upper shaft probe during the 6 test periods is shown in Figure 4. The H₂ content in the reduction gases was observed to increase with increasing PCR. Addition of O₂ to the lance as well as operation using the HV coal resulted in a higher H₂ amount as compared to when oxygen was supplied to the blast and air added to the lance and with operation using the LV coal. At the position of the lower shaft probe, the H₂ content in the reduction gas reached a higher level than at the position of the upper shaft probe. Figure 5 and Figure 6 present the horizontal profiles of CO and η_CO at the position of the upper shaft probe. The CO content in the reduction gas was observed to reach a higher level at the position of the upper shaft probe, and η_CO a lower level, during operation using the LV coal compared to operation using the HV coal.

Pellet
Figure 7 shows typical textures of pellet cores and peripheries obtained in pellets taken out with the upper shaft probe during test periods 1-6. Magnetite dominated the cores of pellets taken out with the upper shaft probe during injection of the LV coal in test periods 1-3. Some hematite and wustite were also observed in the pellet cores. The pellet peripheries were made up of wustite. Wustite and magnetite dominated in the pellet cores in pellets taken out with the upper shaft probe during injection of the HV coal in test periods 4-6. A few areas of Fe_melt observed in the pellet core increased in size and number towards the periphery. Wustite and Fe_melt dominated the periphery. The transition between the texture in the pellet core and periphery was blurry and any distinct product layers were not formed.

Figure 8 shows typical textures of pellet cores and peripheries obtained in pellets taken out with the lower shaft probe. In pellets taken out with the lower shaft probe, Fe_melt dominated the texture. Injection of the HV coal type increased the pore size in the periphery when the other test parameters were held constant; a phenomenon noted in comparison of test periods 1 and 5, 2 and 6, and 3 and 4. Comparing the pellet cores, the size of the continuous Fe_melt areas increased during injection of the HV coal compared to injection of the LV coal at high PCR and independent of oxygen supply method. In the pellet core from period 1, softened wustite dominated.
At the position of the upper shaft probe the highest reduction degrees were attained during injection of the HV coal, see Figure 9. As can be seen in Figure 10, no correlation between the pellet reduction degree and pre-set process conditions was observed in sub-samples taken out with the lower shaft probe.

Chemical analysis of the pellet fragments in the size range 212μm – 1.5 mm showed an increased carbon content after operation with injection of the HV coal type, see Figure 11. Increased contents of K and Ca were also observed. Air supply to the lance generated a higher carbon content compared to operation with oxygen addition to the blast air. In the XRD analysis, see Figure 12, Fe₃C was observed in samples from test periods 2-6. Table 4 summarizes the compounds identified in the XRD analysis.

For the low PCR, the chemical analysis of the >10 mm pellet fragments showed an increased amount of carbon after operation with injection of the HV coal type. Fe₃C was observed after operation with injection of the HV coal type and FeO was observed after injection of the LV coal type. The pellet fragments were mainly made up of Fe₃C.

Distribution of slag and trace elements

Slag areas were found next to or enclosed in the Fe₃C areas in the entire cross section in pellets taken out with the lower shaft probe during test periods 1-6. The size and distribution of the slag areas and areas with increased K content varies between the test periods. Sometimes, the pellet periphery was covered by a layer consisting of mainly Mg, Si, K, Ca and Al. EDS mapping of pellet core, intermediate area and pellet periphery taken out during operation with the LV and HV coal type, test periods 2 and 5, are presented in Figure 13 and Figure 14.

K was observed in pellets taken out with the lower shaft probe, independent of pre-set process conditions. Areas of increased K content in the core of the pellets were found to be next to or surrounded by Fe₃C. In pellets taken out during injection of the LV coal type, K was located next to Fe₃C, while Fe₃C surrounded K after injection of the HV coal type. In the pellet periphery, K was found between Fe₃C as well as in the surface cover of pellets, independent of the pre-set process conditions. K formed compounds with Si, Al, Mg, and in a few cases K and Ti coexist. The coexistence of K and the slag elements was more frequent in the pellet periphery, and especially in the surface cover, compared to the core of the pellets. Ca-Si slag
was observed in the pellet cores and coexistence of the elements was observed in the pellet periphery as well as in the surface cover. Mg and Si were more frequently found in the same area in the pellet core and surface cover compared to the pellet cores. Al was mainly observed in the surface cover.

In the area between the pellet core and periphery K was usually observed together with Si and Mg. The tendency for liquid formation, as can be seen when Figure 13 and Figure 14 are compared, was more pronounced in pellets taken out during injection of the HV coal type, as can be seen by the smooth slag areas compared to the diffuse slag areas during LV coal operation.

**Laboratory reduction**

**Reduction profiles**

Figure 15 shows the reduction profiles for tests I-VIII. For the same level of simulated PCR, at the test end temperature of 1100˚C, a higher reduction degree was attained for the slow heating rates when tests IV and II and tests VIII and VI were compared. After reduction simulating the fast heating rate profiles, tests II and VI, approximately equal reduction degrees were attained independent of simulated PCR. A higher reduction degree was attained for the slow heating rates at the end of test IV, simulating high PCR, compared to test VIII, simulating low PCR. In tests I, III, V and VII, the reduction was interrupted at a reduction degree of approximately 40 percent. The rate of reducibility at 40 percent reduction and the average rate of reduction during the total test time to reach 1100˚C are presented in Table 5.

**Pellet textures**

Significant differences in the iron oxide textures were observed in the pellet cores when pellets from the reduction tests simulating the high and low PCR were compared. A grain texture was observed in the pellet core of samples after tests V-VIII, simulating the low PCR, an observation that was not discernible after tests I-IV, simulating the high PCR. Textures from the pellet core and periphery from tests I-VIII are presented in Figure 16. Together with the areas of Fe$_{\text{met}}$ in the pellet periphery, a few areas of iron oxide were present after tests II and IV, which were full-length tests simulating the high PCR. After full-length tests simulating the low PCR, tests VI and VIII, the corresponding areas in the pellet periphery showed noticeable areas of iron oxide surrounded by Fe$_{\text{met}}$ within the texture dominated by Fe$_{\text{met}}$. A few areas of Fe$_{\text{met}}$ were observed in the pellet core from test samples interrupted at
the test end temperature of 1100˚C. Fe\textsubscript{met} was not observed in pellets reduced to a reduction degree of approximately 40 percent. No significant differences in the Fe\textsubscript{met} texture of the pellet periphery, independent of final reduction degree, could be observed between pellets reduced according to similar reduction profile. The extension between the pellet core, mainly made up of iron oxide, and the pellet periphery dominated by Fe\textsubscript{met} varied, however, in accordance with the reduction degrees attained. A summary of the observations is presented in Table 6.
Discussion

Reduction

*EBF*

Changes in the reduction potential of the ascending gas during the EBF tests were mostly a consequence of the differences in H₂ content attained during the pre-set test periods. A higher PCR, comparing test periods 1 and 3 and 4 and 5, increased the H₂ content of the ascending gas. Corresponding results were obtained after switching from operation with injection of LV coal to HV coal at a high PCR. The H₂ content at the level of the upper shaft probe was higher during operation with oxygen enrichment compared to operation with air addition to the coal lance, as can be observed comparing test periods 1 and 2 and 5 and 6, but the result could depend, at least in part, on the higher PCR during operation with oxygen enrichment.

In the investigated cases, a higher temperature level in the EBF was expected during injection of the HV coal. The higher reduction degree observed in samples taken out with the upper shaft probe during injection of the HV coal type, compared to during LV coal operation, was an effect of the higher temperature but also of the differences in H₂ content in the ascending gas. At the level of the lower shaft probe the H₂ content was higher compared to the attained amounts at the level of the upper shaft probe. In pellets taken out with the lower shaft probe, significant differences in average reduction degree were not found and no correlation between pre-set process conditions and reduction degree could be observed. The results indicated that the test conditions in terms of the reduction potential of the gas, temperature profile and time for all cases resulted in a thermal reserve zone that was long enough for reduction of pellets under all the investigated process conditions. Due to the central coke charging, the reduction potential of the gas was higher in the centre of the furnace.

*Laboratory simulated PCR*

Generally, a higher simulated PCR resulted in a higher pellet reduction degree at an equal reduction time. However, the initial reduction temperature and heating rate were of importance for the reduction degrees attained. A higher start reduction temperature resulted in a faster initial reduction. On the other hand, a faster heating rate compensated for a lower start temperature and lower simulated PCR. Despite differences in PCR, an approximately equal reduction degree was attained for the simulated low PCR and fast heating rate and simulated high PCR and slow heating rate at a reduction time exceeding 35 minutes. If these results are applied to the actual BF process, it can be concluded that the increase in reduction potential of
the gas and a higher temperature level compensate for the possible loss in reduction time when the PCR is increased.

**Texture**

**EBF**

Fem areas were observed in pellets taken out with the upper shaft probe for all pre-set process conditions during injection of the HV coal. During injection of the LV coal type, corresponding Fem areas were not observed. During injection of the LV coal type a few areas of hematite were observed in the pellet core, which was not the case in pellets taken out during injection of the HV coal type. The differences in temperature and reduction potential of the gas at high PCR explain the differences in pellet textures under injection of the different coal types. It was likely that the coal type has a corresponding affect on the ascending gas composition at low PCR, but since a period of lower heat level in the EBF occurred during low PCR operation and injection of the HV coal type, the relation could not be proven for the tests performed.

Fem dominated in pellets taken out with the lower shaft probe. The pores of the Fem textures were larger after reduction during injection of the HV coal. In the core of pellets taken out during test period 5, high PCR, HV coal, and oxy coal operation, the largest areas of Fem were found. The marked off areas of Fem were observed to be more compact after operation with the HV coal type compared to the LV coal. A straggling Fem texture was, especially in the pellet periphery, identified after operation with the LV coal type. Previous investigation showed that the generation of fines increases during injection of the HV coal type. During abrasion, the edges of the straggling Fem areas get stuck in each other, while a smooth Fem surface area does not exhibit this property. Since the Fem areas with smooth surfaces dominated during HV coal injection, the Fem texture is assumed to be one explanation for the increase in fines generation compared to LV coal operation. Examples of typical straggling and smooth Fem textures are presented in Figure 17. The test conditions in the EBF, in terms of reduction potential of the gas and temperature profile, do not result in formation of distinct product layers in the reduced pellets. No micro-porosity was observed in the Fem areas in pellets taken out with the shaft probes.

In pellets taken out with the lower shaft probe during injection of the HV coal the slag areas and the surface cover were observed to be softened. Corresponding observations were not
made in samples from injection with the LV coal. No correlation between the pre-set process conditions and elements observed in the slag areas and surface cover could be observed. The formation of softened slag areas indicates exposure to a higher temperature level and it could therefore be concluded that injection of the HV coal type, in the investigated cases, resulted in an increase in the temperature level in the EBF at the position of the lower shaft probe.

Differences in the location of areas with an increased K content were observed between pellets taken out with the lower shaft probe. During injection of the LV coal type, K was found next to Fe₇₇₇₇, while Fe₇₇₇₇ surrounds K during injection of the HV coal type. Since the solubility of K in iron oxides has been reported to be low, it seems probable that K will condense on the iron oxide surfaces. In a porous grain the K might reach the centre of the grain and in this way K could be surrounded by Fe₇₇₇₇ when the iron oxide/Fe₇₇₇₇ becomes more compact as the pellets descend through the EBF. The texture of a wustite grain was more porous in comparison to a magnetite grain. Accordingly, the formation of K surrounded by Fe₇₇₇₇ was reasonably facilitated if K condensed on wustite instead of magnetite. Chemical analysis of the pellets taken out with the shaft probes showed a higher K content in pellets taken out with the lower shaft probe compared to the upper shaft probe. It could be concluded that a major part of the K condensation had taken place at the position between the shaft probes. Since a higher reduction degree was attained in pellets taken out with the upper shaft probe during injection of the HV coal type and the grains were found to be more porous than during injection of the LV coal type, K would most probably condensate inside the porous grain texture and give rise to the texture with K surrounded by Fe₇₇₇₇.

Laboratory reduction

A simulated low PCR resulted in a grain texture in the pellet core that was not observed at simulated high PCR. In the pellet periphery, iron oxides were surrounded by Fe₇₇₇₇ and the Fe₇₇₇₇ texture was denser at low PCR. The pores in the pellet periphery were larger at high PCR. Tests IV and VI showed, despite similar reduction profiles above a reduction degree of 25 percent, differences in pellet texture. The texture differences were most likely to occur in the beginning of the reduction. Differences in the pellet textures that were observed in samples after reduction to 1100°C were already found at a reduction degree of 40 percent. These observations supported the claim that the observed texture differences were introduced at the beginning of reduction. A higher start reduction temperature and a more rapid temperature increase in combination with an increase in the reduction potential of the
reduction gas counteracted the formation of a grain texture in the pellet core. At the same time, a porous Fe\textsubscript{met} texture was formed in the pellet periphery. The results from the present investigations agree with observations made by Turkdogan\textsuperscript{4)}, which state that the initial reduction temperature was of major importance for the pore structure of the reduced iron.

**Sources of error in the texture observations**

According to the literature, a random distribution of pores has been observed in magnetite formed during reduction of hematite pellets, but magnetite without internal porosity was also observed\textsuperscript{11)}. The magnetite textures were obtained at different temperatures and at different reduction gas compositions. In the present investigations it was assumed that no internal porosity existed in the magnetite and that the areas of similar colour, in which internal porosity was observed, consisted of wustite. There is a possibility that magnetite and wustite were not kept separate at all times.

**Carburization of pellets**

Fe\textsubscript{3}C was observed in fractions formed during tumbling of pellets taken out with the lower shaft probe in test periods 2-6. Chemical analysis showed an increase in C content in corresponding samples during injection of the HV coal type. It is concluded that the increase in C content is due to an increase in the amount of Fe\textsubscript{3}C during injection of the HV coal type.

Carburization of reduced iron or wustite forming iron carbide, Fe\textsubscript{3}C, can take place according to\textsuperscript{12)}

\[ 3\text{Fe} + \text{C} \rightarrow \text{Fe}_3\text{C} \quad \text{(I)} \]
\[ 3\text{Fe} + 2\text{CO} \rightarrow \text{Fe}_3\text{C} + \text{CO}_2 \quad \text{(II)} \]
\[ 3\text{FeO} + 5\text{CO} \rightarrow \text{Fe}_3\text{C} + 4\text{CO}_2 \quad \text{(III)} \]

Larger pores facilitated the diffusion of CO in the pellets and Fe\textsubscript{3}C formed according to equation II was facilitated by the increased contact area between Fe\textsubscript{met} and CO. Larger pores, possibly not only formed as a consequence of increased temperature but also as a result of the Fe\textsubscript{3}C content, were observed in the pellets taken out during injection of the HV coal type. The
differences in pellet reduction degree obtained with the different coal types receded through the shaft but most likely remained below the position of the upper shaft probe. Fe$_3$C was formed in the shaft between the positions of the shaft probes. A higher amount of H$_2$ in the ascending gas facilitated the iron oxide reduction by H$_2$ and as a consequence the CO concentration available for carburization increased.

At temperatures above 900°C Fe$_3$C reacts with wustite according to

$$2\text{FeO} + \text{Fe}_3\text{C} \leftrightarrow 5\text{Fe} + \text{CO}_2 \quad \text{(IV)}$$

Reaction of Fe$_3$C with wustite, reaction IV, was more likely to occur during injection of the LV coal type, since the pellet reduction degree was lower. Therefore, a lower carbon content should be expected when using the LV coal which is in agreement with the present results. It cannot be concluded for certain that carbon deposition did not occur during the laboratory reduction tests performed; if so was the case, a higher reduction degree compared to the ones presented might have been reached.

**Concluding discussion**

The results from the EBF tests indicated that the test conditions in terms of the reduction potential of the gas, temperature profile and time for all cases resulted in an extension of the thermal reserve zone that was sufficient for reduction of pellets under all the investigated process conditions. The different Fe$_{\text{met}}$ textures observed in pellets taken out with the lower shaft probe indicated differences in reduction conditions at positions above the lower shaft probe in the EBF. The initial reduction conditions, in terms of temperature and gas composition, were in the performed investigations conclusive for the pellet texture formed.

**Conclusions**

The reduction behaviour of olivine pellets and textures formed during operation in the EBF were investigated. In the investigations performed, an LV and an HV coal type were injected at different PCR while two types of oxygen supply methods were employed. The choice of injection coal type was conclusive for the Fe$_{\text{met}}$ texture formed during reduction, extent of Fe$_{\text{met}}$ carburization and K distribution in the pellet. An HV injection coal resulted in a pellet texture with larger pores and smoother Fe$_{\text{met}}$ areas in the pellet periphery, increased Fe$_3$C
formation and areas of K surrounded by Fe_{net}. The amount of VM in the coal type had a greater effect on the pellet reduction properties than the PCR and oxygen supply methods.

Based on the laboratory experiments for simulated PCR it was concluded that the initial reduction conditions, in terms of temperature level and reduction gas composition, will determine the pellet texture up to a reduction degree of at least 60 percent.

The tests carried out in the EBF showed that the tested pellets were well suited for blast furnace reduction at different PCR, with different injection coal types and for different methods for oxygen supply. The laboratory reduction tests support this conclusion.

**Acknowledgements**

We are grateful to the Swedish National Energy Administration (STEM) and the Agricola Research Centre for financial support. The members of the Swedish Steel Producers’ Association JK21059 project are acknowledged. Thanks to LKAB and SSAB Tunnplåt AB for support and assistance in the work. A special thanks to LKAB for providing the opportunity to carry out tests in the EBF. Finally, we thank Mr. Johan Folkesson for designing the laboratory furnace control system at LTU.
References


### Tables

**Table 1** Chemical composition of the MPBO in percent\(^{10}\)

<table>
<thead>
<tr>
<th>Pellet</th>
<th>Fe</th>
<th>FeO</th>
<th>CaO</th>
<th>SiO(_2)</th>
<th>MgO</th>
<th>Al(_2)O(_3)</th>
<th>CaO/SiO(_2)</th>
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<tr>
<td>MPBO</td>
<td>66.8</td>
<td>0.5</td>
<td>0.35</td>
<td>1.7</td>
<td>1.5</td>
<td>0.32</td>
<td>0.21</td>
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**Table 2** Overview of the pre-set process conditions during the EBF trial

<table>
<thead>
<tr>
<th>Test</th>
<th>Coal type</th>
<th>PCR (kg/tHM)</th>
<th>Method for oxygen supply</th>
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<tr>
<td>1</td>
<td>LV</td>
<td>152</td>
<td>Oxy Coal</td>
</tr>
<tr>
<td>2</td>
<td>LV</td>
<td>146</td>
<td>Air Coal</td>
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<td>3</td>
<td>LV</td>
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<td>Oxy Coal</td>
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<tr>
<td>6</td>
<td>HV</td>
<td>143</td>
<td>Air Coal</td>
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</table>

**Table 3** Schematic overview of blast furnace simulating experiments

<table>
<thead>
<tr>
<th>Test</th>
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<th>Heating Rate</th>
<th>Test End</th>
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<tr>
<td></td>
<td>High</td>
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<td>Slow</td>
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<td>VII</td>
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<td>x</td>
<td>x</td>
</tr>
<tr>
<td>VIII</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
</tbody>
</table>

**Table 4** Compounds identified by XRD investigations in the fraction 212µm – 1.5 mm after I-drum treatment of pellets from sub-sample 3 taken out with the lower shaft probe during operation in the EBF at different PCR, injection coal type and methods for oxygen supply

<table>
<thead>
<tr>
<th>Test period</th>
<th>Compound</th>
<th>Fe</th>
<th>FeO</th>
<th>Fe(_3)C</th>
<th>KAIS(_2)O(_8)</th>
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</thead>
<tbody>
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<td>1</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
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<tr>
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Table 5 Reduction rates for tests II, IV, VI and VIII

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<th>Test</th>
<th>R40</th>
<th>Average Red. Rate</th>
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<tr>
<td>II</td>
<td>0.95</td>
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<tr>
<td>IV</td>
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<td>0.77</td>
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<tr>
<td>VI</td>
<td>0.81</td>
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<td>VIII</td>
<td>0.71</td>
<td>0.59</td>
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</table>

Table 6 Summary of pellet textures and reduction properties attained after simulated PCR in laboratory scale

<table>
<thead>
<tr>
<th>Test</th>
<th>Pellet Core</th>
<th>Pellet Periphery</th>
<th>Reduction properties at 1100 °C</th>
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<tr>
<td></td>
<td>Iron oxide</td>
<td>Fe&lt;sub&gt;int&lt;/sub&gt;</td>
<td>Grain Texture</td>
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<tr>
<td>I</td>
<td>X</td>
<td>X</td>
<td>x</td>
</tr>
<tr>
<td>II</td>
<td>X</td>
<td>X</td>
<td>-</td>
</tr>
<tr>
<td>III</td>
<td>X</td>
<td>X</td>
<td>x</td>
</tr>
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<td>IV</td>
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<td>X</td>
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<tr>
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<td>X</td>
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<td>VI</td>
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<td>VII</td>
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<tr>
<td>VIII</td>
<td>X</td>
<td>X</td>
<td>x</td>
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Figures

**Figure 1** Schematic view of the experimental apparatus used for the laboratory reduction tests

**Figure 2** Heating rate and gas composition profiles for simulated high PCR. Heating rate and gas profiles estimated from measurements in the EBF

**Figure 3** Heating rate and gas composition profiles for simulated low PCR. Heating rate and gas profiles estimated from measurements in the EBF
Figure 4 H₂ at the position of the upper shaft probe in the EBF during test periods 1-6 with different PCR, injection coal types and methods for oxygen supply.

Figure 5 CO at the position of the upper shaft probe in the EBF during test periods 1-6 with different PCR, injection coal types and methods for oxygen supply.

Figure 6 ᵂCO at the position of the upper shaft probe in the EBF during test periods 1-6 with different PCR, injection coal types and methods for oxygen supply.
Figure 7 Typical textures, observed in light optical microscope, of pellets taken out with the upper shaft probe in the EBF during test periods 1-6. Hematite = Light grey, Magnetite = Smooth grey, Wustite = Broken grey, Fe$_{met}$ = White, Pores = Black
Figure 8 Typical textures, observed in light optical microscope, of pellets taken out with the lower shaft probe in the EBF during test periods 1-6. Wustite = Grey, $Fe_{metal}$ = White (lightest), Pores = Black.
Figure 9 Pellet reduction degrees in sub-samples 1-3 taken out with the upper shaft probe in the EBF during tests with different PCR, injection coal type and method for oxygen supply.

Figure 10 Pellet reduction degrees in sub-samples 1 and 3 taken out with the lower shaft probe in the EBF during tests with different PCR, injection coal type and method for oxygen supply.
Figure 11 Element distributions in the fraction 212μm – 1.5 mm after I-drum treatment of pellets from sub-sample 3 taken out with the lower shaft probe during operation in the EBF at different PCR, injection coal type and methods for oxygen supply.

Figure 12 XRD pattern of ground material after I-drum treatment of pellets from sub-sample 3 taken out with the lower shaft probe during operation in the EBF at different PCR, injection coal type and methods for oxygen supply.
Figure 13 EDS mapping of core, intermediate area and periphery of pellet taken out with the lower shaft probe during operation with the LV coal type in test period 2
Figure 14 EDS mapping of core, intermediate area and periphery of pellet taken out with the lower shaft probe during operation with the HV coal type in test period 5.
Figure 15 Reduction profiles for laboratory tests I-VIII. Test II, IV, VI and VIII interrupted at the test end temperature of 1100°C and tests I, III, V and VII at an attained reduction degree of approximately 40 percent.
Figure 16 Pellet textures observed in light optical microscope after reduction tests I-VIII. Pellet core (left) and pellet periphery (right). Iron oxides = Grey, Fe$_{mat}$ = White, Pores = Black
Figure 17 Typical examples of a straggling \( Fe_{\text{str}} \) texture (left) and a smooth \( Fe_{\text{str}} \) texture (right)