Kinetic Study on Smelting Reduction of Chromite Ore
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ABSTRACT
Smelting reduction of chromite ore was studied in systems and conditions related to ferrochromium production in submerged arc furnace. Dissolution of chromite pellets or lumpy ore as well as reduction of iron, chromium and silicon oxide were studied by immersing chromite samples in a slag melt in a graphite crucible. Experiments were performed at temperatures from 1650°C to 1750°C.

Kinetics of dissolution and reduction were followed by continuous gas analysis. After the experiments the crucibles were submitted to optical microscopy and microanalysis to detect the prevailing phases and their compositions.

The results showed that dissolution rate of chromite into a slag is strongly temperature-dependent, but also the chromite and gangue composition as well as slag composition had a great influence.

Chromite reduction seemed to be a quite complicated process. Metallic product formed and observed in different regions of the reaction system, inside or on the surface of the chromite phase, inside the slag phase or on the crucible bottom were quite different in composition and structure. Metallic particles found on the surface of the graphite crucible had mostly high chromium and silicon content. The high silicon content must be resulted from reduction of SiO₂ at the three phase contact between the slag, the graphite crucible and a metal droplet, eventually (Cr,Fe)₂₃C₆ as an intermediate reaction product. Metal particles formed inside the undissolved sample were rich in iron, low in chromium and very low in silicon. Metal particles observed on the sample surface or inside the slag had an intermediary composition.

Based on the results the mechanisms of smelting reduction of chromite are discussed with contributions to industrial submerged arc furnace process.

1 INTRODUCTION
Ferrochrome, as a central component in stainless steel production, is one of the most rapidly growing primary metallic products. Production of ferrochromium and stainless steel in Finland is based on the Kemi chrome ore deposit, which was discovered in 1959. Mining was started in 1966 and ferrochrome production in Tornio at 1968. Since then, the ferrochromium production has increased gradually and was about 260 000 t in 2000.

At Outokumpu’s Tornio works ferrochrome is produced in two closed submerged arc furnaces with 40 MAV and 75 MAV, respectively. The Cr-bearing charge is partly upgraded lumpy ore and partly sintered pellets made of fine concentrate. For thermodynamic reasons, the smelting of ferrochromium is an energy intensive process. The process has been intensively developed over the years but despite the improvements in submerged arc furnace operations, there is still a need to reduce the energy consumption and to improve the chromium yield in the process.

In the electric submerged-arc process, reduction proceeds via different mechanisms in different reaction zones, due to large temperature gradient. Knowledge of the reaction mechanisms in each zone can provide guidance for improving the reduction efficiency, achieving better chromium yield and thus decreasing the energy consumption.

The aim of this study was to investigate the dissolution of chromite in the slag and its reduction by solid carbon in conditions related to ferrochromium production in submerged arc furnace.
2 MATERIALS AND EXPERIMENTAL PROCEDURE

2.1 MATERIALS

Experiments were performed by using sintered pellet, prereduced pellet and lumpy ore. Table 1 shows the chemical composition of sintered pellet and lumpy ore. Prereduced pellets were prepared by reducing the sintered pellets in CO-atmosphere with rising temperature from 700°C to 1520°C at the heating rate of 2.5°C/min and then maintained at 1520°C for 1 hour. The reduction degree after prereduction was about 35%. It was calculated according to the following equation:

\[ \text{Reduction degree, } \% = \left( W_0 - W_t \right) \times 100 / W_0 \]  

where

- \( W_0 \) = weight of the sample at the beginning of reduction after the moisture removal
- \( W_t \) = observed weight of the sample at time \( t \) during the reduction
- \( W_0 \) = amount of total initial removable oxygen (in the calculations only the chromium and iron oxides were regarded as reducible components)

Slags used in the experiments were prepared from chemically pure components. \( \text{SiO}_2, \text{Al}_2\text{O}_3, \text{MgO} \) and \( \text{CaO} \) powders were mixed in appropriate proportions, the homogeneous mixtures were then melted in a graphite crucible in an induction furnace and finally the cooled solid slag samples were ground to powder. Table 2 shows the slag compositions and estimated liquidus temperatures.

<table>
<thead>
<tr>
<th>Slag type</th>
<th>( \text{SiO}_2 )</th>
<th>( \text{Al}_2\text{O}_3 )</th>
<th>( \text{MgO} )</th>
<th>( \text{CaO} )</th>
<th>Estimated liquidus, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>39.8</td>
<td>31.5</td>
<td>17.8</td>
<td>10.9</td>
<td>1575</td>
</tr>
<tr>
<td>2</td>
<td>40.9</td>
<td>24.6</td>
<td>24.9</td>
<td>9.6</td>
<td>1580</td>
</tr>
<tr>
<td>3</td>
<td>37.6</td>
<td>25.9</td>
<td>34.4</td>
<td>2.1</td>
<td>1660</td>
</tr>
<tr>
<td>4</td>
<td>37.0</td>
<td>22.2</td>
<td>38.6</td>
<td>2.2</td>
<td>1660</td>
</tr>
<tr>
<td>5</td>
<td>34.6</td>
<td>25.5</td>
<td>30.2</td>
<td>9.7</td>
<td>1670</td>
</tr>
<tr>
<td>6</td>
<td>35.1</td>
<td>30.3</td>
<td>25.0</td>
<td>9.6</td>
<td>1660</td>
</tr>
</tbody>
</table>

Table 2 - Chemical analyses and melting points of the slag types used in the experiments.

In a typical experiment, 40 g of slag was charged into the graphite crucible. The chromite sample was located about 5 cm above the crucible top during the heating. As the furnace temperature was raised gradually, the chromite sample became preheated. When the desired temperature was reached, the sample was slowly pushed down using Mo-rod and immersed in the liquid slag. A PtRh30-PtRh6 thermocouple was installed just below the crucible to record the temperature of the reaction zone. The reduction kinetics were investigated by continuously measuring the amount of CO and \( \text{CO}_2 \) gas generation during the experiments. Kinetics and mechanisms were also observed by optical microscopy and microanalyses of the samples/crucibles after the experiments. The experiments were carried out in Ar atmosphere with the flow rate of 1 l/min. Table 3 shows the experimental programme.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Slag type</th>
<th>Experimental temperature, °C</th>
<th>Experimental method</th>
</tr>
</thead>
<tbody>
<tr>
<td>lumpy ore</td>
<td>slag 1</td>
<td>1650, 1700</td>
<td>optical microscopy &amp; microanalyses</td>
</tr>
<tr>
<td>prered. pellet</td>
<td>slag 2</td>
<td>1650, 1700</td>
<td>measurement of CO and ( \text{CO}_2 ) generation</td>
</tr>
<tr>
<td></td>
<td>slag 3</td>
<td>1650, 1700</td>
<td></td>
</tr>
<tr>
<td></td>
<td>slag 4</td>
<td>1700</td>
<td></td>
</tr>
<tr>
<td>pellet</td>
<td>slag 5</td>
<td>1700</td>
<td></td>
</tr>
<tr>
<td></td>
<td>slag 6</td>
<td>1750</td>
<td></td>
</tr>
</tbody>
</table>

Table 3 - Experimental programme for slag experiments.

2.2 METHOD AND PROCEDURE

Experiments were carried out in a vertical alumina-tube furnace with molybdenum disilicide heating elements. The temperature was measured by Pt20Rh-Pt40Rh thermocouple and controlled by Eurotherm 903P controller. Fig. 1 shows the experimental arrangement.
3 EXPERIMENTAL RESULTS

3.1 EXPERIMENTS WITH GAS ANALYSIS

Gas analysis was used to follow the reduction kinetics when studying the effect of different chromite samples, slag composition and temperature on dissolution and reduction processes. The CO and CO₂ contents of the gas were analysed by using Hartmann & Braun’s Uras 14 infrared analyzer. The amount of CO gas, formed due to reduction of SiO₂ with the graphite crucible at the slag/graphite interface, was measured in every experiment before immersing the sample in the slag and then subtracted from the CO evolution curves shown in Figs. 2, 4, 5 and 8.

**Effect of different chromite samples**

The effect of different chromite samples on dissolution and reduction processes was studied by using lumpy ore, sintered and prereduced pellets. Fig. 2 shows CO-curves from the experiments with slag type 4 at 1700°C. Only very little CO₂ was detected in the outlet gas.

![Figure 2 - CO evolution curves for experiments where prereduced pellet, lumpy ore and sintered pellet were immersed in slag type 4 at 1700°C.](image)

The amount of CO gas, formed due to reduction of SiO₂ with the graphite crucible at the slag/graphite interface, was measured in every experiment before immersing the sample in the slag and then subtracted from the CO evolution curves shown in Figs. 2, 4, 5 and 8.

Utilizing the measured CO content (subtracted the amount of CO gas, formed due to reduction of SiO₂ with the graphite crucible at the slag/graphite interface) the reduction rate and the reduction degree of the samples were calculated from the following equations:

\[
N_i = 0.5 \times (CO_{i-1} + CO_i) \times F_i / 2240
\]

\[
R_i = \sum_i N_i / O_t \times 100
\]

where

- \(N_i\) = reduction rate of sample at time \(i\) (mol-O/min)
- \(CO_i\) = content of CO in outlet gas at time \(i\) (%)
- \(CO_{i-1}\) = content of CO in outlet gas at time \(i-1\) (%)
- \(F_i\) = flow rate of gas (l/min)
- \(R_i\) = reduction degree of sample at time \(i\) (%/min)
- \(O_t\) = amount of total initial removable oxygen (mol-O)

In the calculations only chromium and iron oxides were regarded as reducible components. However, in the experimental temperatures also some amount of SiO₂ from the chromite samples was reduced.

Fig. 3 shows the reduction degree curves of lumpy ore, sintered and prereduced pellets. In order to facilitate comparing the effect of different chromite samples on dissolution and reduction processes, the zero level was chosen so that it corresponds to the amount of reducible oxygen in sintered pellet. This means that the starting level for lumpy ore is above the zero level due to lower reducible oxygen content compared to sintered pellet and the starting level for the prereduced pellet is the prereduction degree (35%).

![Figure 3 - Reduction degree curves for prereduced pellet, lumpy ore and sintered pellet immersed in slag 4 at 1700°C.](image)

As can be seen, the smelting reduction of prereduced pellet resulted in the highest reduction degree (~75%) after one hour experiment. This figure as well as Fig. 2 shows that prereduction does not decelerate the initial rate of smelting reduction.
**Effect of slag type**

The effect of slag type on dissolution and reduction processes was investigated by using prereduced pellets. Two series of experiments were carried out and the CO-curves are shown in Figs. 4 and 5.

![Figure 4 - CO evolution curves of experiments where prereduced pellet was immersed in slag types 3, 4 and 5 at 1700°C, series 1.](image)

![Figure 5 - CO evolution curves for experiments where prereduced pellet was immersed in slag types 3, 4 and 5 at 1700°C, series 2.](image)

As can be seen, the experiment with slag type 4 resulted in the highest reduction degree in both series. In the experiment with slags 3 and 5, the order changes between the series. The reduction degree curve in the experiment with slag 5 in series 1 (Fig. 6) starts at a lower level than the others due to a little lower prereduction degree.

**Experiment at 1750°C**

Fig. 8 shows the CO-curve from the experiment of sintered pellet with slag type 6 at 1750°C.

![Figure 8 - CO evolution curve for experiment where sintered pellet was immersed in slag type 6 at 1750°C.](image)
As can be seen, the CO generation was very fast after the immersion and the amount was much higher than in earlier experiments. The final reduction degree was found to be over 80% as can be seen from Fig. 9. No pellet pieces were found in the slag phase but the high experimental temperature caused slag to foam out of the crucible possibly along with dissolved chromite particles.

In the case where the samples dissolved weakly in the slag, reduction took place mainly at the slag/graphite crucible interface forming metal on the walls and bottom of the crucible as can be seen from Fig. 10. Metallic particles found on the surface of the graphite crucible had mostly high chromium and silicon content (Table 4).

### Table 4 - Microanalyses of metallic particles on the bottom of the crucible after the experiment with slag 1 at 1700°C.

<table>
<thead>
<tr>
<th>Cr</th>
<th>Fe</th>
<th>Si</th>
<th>place</th>
</tr>
</thead>
<tbody>
<tr>
<td>76.3</td>
<td>7.0</td>
<td>15.9</td>
<td>small particles</td>
</tr>
<tr>
<td>63.5</td>
<td>17.3</td>
<td>18.4</td>
<td></td>
</tr>
<tr>
<td>78.2</td>
<td>6.4</td>
<td>14.7</td>
<td></td>
</tr>
<tr>
<td>70.9</td>
<td>11.6</td>
<td>16.9</td>
<td></td>
</tr>
<tr>
<td>74.2</td>
<td>7.5</td>
<td>17.3</td>
<td></td>
</tr>
<tr>
<td>76.5</td>
<td>6.3</td>
<td>16.2</td>
<td></td>
</tr>
<tr>
<td>72.5</td>
<td>10.7</td>
<td>15.9</td>
<td></td>
</tr>
<tr>
<td>67.7</td>
<td>11.0</td>
<td>20.6</td>
<td></td>
</tr>
<tr>
<td>77.7</td>
<td>10.5</td>
<td>11.2</td>
<td>lens</td>
</tr>
</tbody>
</table>

In the case where the samples dissolved well in the slag, reduction took place mainly at the slag/graphite crucible interface forming metal on the walls and bottom of the crucible as can be seen from Fig. 10. Metallic particles found on the surface of the graphite crucible had mostly high chromium and silicon content (Table 4).
Table 5 - Microanalyses of metallic particles in different regions after the experiment with slag 2 at 1650°C.

<table>
<thead>
<tr>
<th>Element</th>
<th>Graphite Surface</th>
<th>Sample Surface</th>
<th>Inside Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>40.2 42.3 17.5</td>
<td>53.6 58.6 3.3</td>
<td>7.9 90.7 0.1</td>
</tr>
<tr>
<td>Fe</td>
<td>19.2 0.18</td>
<td>2.4 2.7</td>
<td>0.1 0.1</td>
</tr>
<tr>
<td>Si</td>
<td>16.5 0.25</td>
<td>3.3</td>
<td>0.1</td>
</tr>
<tr>
<td>Ti</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

As can be seen, metallic particles formed in different regions of the reaction system are quite different in composition. The chromium and silicon contents of metallic particles in contact with the graphite crucible are high. Metal particles formed inside the undissolved sample are rich in iron, low in chromium and very low in silicon. Metal particles observed on the sample surface have an intermediary composition.

The chromite particles in undissolved samples seem to have two different zones - a darker outer zone and a lighter inner zone (Fig. 12). The analyses of “chromite” particles from the surface towards the centre are shown in Table 6.

Table 6 - Microanalyses of “chromite” particles in different regions after the experiment with slag 3 at 1650°C.

<table>
<thead>
<tr>
<th>Element</th>
<th>Sample Surface</th>
<th>Outer Zone</th>
<th>Transition Zone</th>
<th>Inner Zone</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>16.1 4.9 26.8</td>
<td>0.0 16.1</td>
<td>14.5 4.0 26.8</td>
<td>0.0 39.2</td>
</tr>
<tr>
<td>Mg</td>
<td>11.0 10.4 8.4</td>
<td>5.6 11.0</td>
<td>12.0 10.4 8.4</td>
<td>5.6 12.0</td>
</tr>
<tr>
<td>Al</td>
<td>15.9 14.9 5.6</td>
<td>12.0 15.9</td>
<td>12.0 15.9 5.6</td>
<td>12.0 15.9</td>
</tr>
<tr>
<td>O</td>
<td>50.1 45.2 26.8</td>
<td>0.0 39.2</td>
<td>0.0 39.2 14.5</td>
<td>0.0 39.2</td>
</tr>
</tbody>
</table>

As can be seen from Fig. 12, in the area of effective reduction, the chromite grains started to disintegrate due to reduction and dissolution. Chromite particles in the surface of the sample and inside the sample have two distinct zones, in some particles a very narrow transition zone between the outer and inner zones can also be seen. The iron content of chromite particles increases when moving from the outer zone to the transition zone and further to the inner zone of the particles as can be seen from Table 6.

As mentioned earlier prereduced pellets were used to investigate the effect of slag types on dissolution and reduction processes. The pellets did not dissolve in the slags completely. Fig. 13 shows the structure of the prereduced pellet embedded in slag type 3 and Fig. 14 the marked area (in Fig. 13) of the prereduced pellet after the experiment.

Figure 12 - Structure of the chromite particles in lumpy ore from the surface towards the centre after the experiment with slag 3 at 1650°C for 1 hour.

Figure 13 - Structure of the prereduced pellet embedded in slag 3 after the experiment at 1700°C for 1 hour.
4 DISCUSSION

In the following, different factors affecting the dissolution and reduction processes, and also the dissolution and reduction mechanisms, are discussed based on the experimental results.

4.1 VISCOSITY OF SLAGS

The effect of viscosity on dissolution of the chromite samples in the slag was studied by using measured and calculated data. The compositions of slags N:o 1, 2, 3, 5 and 6 in this study were the same as those used in Forsbacka’s study. Calculations were performed by using a simple regression model based on the measurements by Forsbacka. The viscosity values for slag 4 at 1700°C and for slag 6 at 1750°C were calculated using the following equation:

\[ \ln \eta = \ln A + B/T \]  
(4)

where

\[ \ln A = 3.902N_{SiO_2} + 3.168N_{Al_2O_3} - 0.654N_{CaO} - 0.039N_{MgO} - 6.682 \]

\[ B = -18.06N_{SiO_2} + 7.448N_{Al_2O_3} + 4370N_{CaO} - 1684N_{MgO} + 20206 \]

Table 8 shows the measured (bold) and calculated viscosity values at experimental temperatures.
The lumpy ore experiments with slags 1, 2 and 3 at 1650°C and 1700°C show that the samples dissolved completely in slag 1 at both temperatures and in slag 2 at 1700°C but quite weakly in slag 3 at both temperatures. Comparison of experimental results and viscosity values indicates that the viscosity does not play such an important role in the dissolution of lumpy ore in the slag.

In the case of prereduced pellets, experiments were done with slag types 3, 4 and 5 at 1700°C. Experiments show that pellets did not dissolve in the slags completely but in the case of slag 4 the dissolution and reduction were faster than with the other slag types. Comparison of experimental results and viscosity values indicates that low viscosity has a positive effect on the dissolution and reduction processes in the case of pellets.

### 4.2 SLAG COMPOSITION VERSUS GANGUE

As mentioned before, lumpy ore samples dissolved completely in slag 1 at both 1650°C and 1700°C and in slag 2 at 1700°C but quite weakly in slag 3. In the case of chromite particles, a MgO*(Al,Cr)2O3 zone with a high melting point was formed around the unreacted core. The thickness of the zone increases and the chromium oxide content decreases when moving from the centre to the surface of the samples. Along with the dissolution of chromium oxide into the liquid slag, the outer layer of the particle becomes rich in gangue components MgO and Al2O3. These components must be dissolved and transported to the bulk slag before the inner part of the particle can be dissolved. The liquidus temperature of this system is strongly affected by the weight percentage of SiO2, so dissolution of MgO*Al2O3 spinel in the slag, and dispersion and dissolution of chromite in the slag at the same time, should be promoted by high SiO2 in the slag, which just concerns slags 1 and 2. From Table 2 it can be seen that slags 1 and 2 have the lowest melting points, 1575°C and 1580°C, respectively. In contrast, the other slags melt at temperatures of approximately 1660-1670°C.

As to the dissolving of chromite into the slag solubility of Cr-oxides might have an essential role. The solubility of CrO has been found to increase with decreasing basicity and increasing temperature. The increase in solubility can be interpreted by the decrease in the activity coefficient of (CrO). The basicity of the slags (calculated by (CaO+MgO)/SiO2 ratio) used in the experiments increases in the order 1<2<3<6<4<5. So slag 1, where the lumpy ore samples dissolved completely at both experimental temperatures, seems to have the highest solubility of Cr-oxides.

### 4.3 REDUCTION AND DISSOLUTION MECHANISMS

Microanalysis of the metallic particles showed that metallic product formed in different regions of the reaction system, inside or on the surface of the chromite phase, inside the slag phase or on the crucible bottom were quite different in composition. Particles which were embedded in the slag phase had very high chromium and very low iron content. This and the iron poor zone in the surface of the chromite particles indicates that iron oxide dissolves first from the surface of the chromite particles and transfers into the slag together with precipitated iron-rich metal particles formed by CO reduction or before the smelting reduction as in the case of prereduced pellets (Fig. 15, step 1) leaving in the surface of the sample spinel particles containing chromium and chromite particles with an iron-free MgO*(Cr,Al)2O3 zone in the surface. Also inside the sample, an iron-free zone in the surface of the particles was observed. After that, chromium oxide starts to dissolve in the slag phase from MgO*(Cr,Al)2O3 spinel and chromium oxide starts to reduce from MgO*(Cr,Al)2O3 by CO resulting in small chromium-rich metal particles, which then precipitate from the surface of the sample to the slag phase together with MgO*Al2O3 spinel (step 2). Finally, Fe-Cr-C-Si metal alloy will be formed at the graphite/slag interface (step 3).
The aim of this study was to investigate the dissolution of chromite in the slag and its reduction by solid carbon in conditions related to ferrochromium production in the submerged arc furnace.

The reduction experiments in the $\text{SiO}_2-\text{Al}_2\text{O}_3-\text{MgO-CaO}$ slag showed that in the case of lumpy ores, high silica content of the slag seems to promote the dissolution. Also, the solubility of Cr-oxides in the slag might have an essential role. Low viscosity of the slag seems to have a positive effect on the dissolution and reduction processes as was seen in the case of prereduced pellets. When the viscosity of the slag is low enough, the slag can penetrate into the interior of the pellet through the pores and accelerate the dissolution process.

The effect of different chromite samples on the dissolution and reduction processes showed that the experiment with the prereduced pellet resulted in the highest reduction degree (75%). The situation is comparable with the ferrochrome furnace where prereduction of chromite charge material occurs in the upper part of the submerged arc furnace and there continues to be smelting reduction in the lower hearth.

With increasing temperature up to $1750^\circ\text{C}$, the CO evolution was found to increase considerably but it also resulted in slag foaming out of the crucible.

According to the experiments, two reduction mechanisms are believed to occur. In the case where the samples dissolved well in the slag, reduction took place mainly at the slag/graphite crucible interface forming metal on the walls and bottom of the crucible. Whereas in the case where the samples dissolved weakly in the slag, metal particles were found mainly in the surface of samples together with disintegrated spinel particles containing chromium. This indicates that the samples were reduced by CO gas formed due to the reaction between $\text{SiO}_2$ and graphite and also due to the reaction between Fe- and Cr-oxides and graphite at the slag/crucible interface. Both these mechanisms can be assumed also in the real process in which coke acts as the reductant and also plays an essential role in the thermal and electrical characteristics of the furnace.

Microanalysis of the metallic and chromite particles indicated that iron oxide dissolves first in the slag from the surface of chromite particles. The reduction experiments in the $\text{SiO}_2-\text{Al}_2\text{O}_3-\text{MgO-CaO}$ slag showed that in the case of lumpy ores, high silica content of the slag seems to promote the dissolution. Also, the solubility of Cr-oxides in the slag might have an essential role. Low viscosity of the slag seems to have a positive effect on the dissolution and reduction processes as was seen in the case of prereduced pellets. When the viscosity of the slag is low enough, the slag can penetrate into the interior of the pellet through the pores and accelerate the dissolution process.

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Microanalysis of the metallic and chromite particles indicated that iron oxide dissolves first in the slag from the surface of chromite particles. The reduction experiments in the $\text{SiO}_2-\text{Al}_2\text{O}_3-\text{MgO-CaO}$ slag showed that in the case of lumpy ores, high silica content of the slag seems to promote the dissolution. Also, the solubility of Cr-oxides in the slag might have an essential role. Low viscosity of the slag seems to have a positive effect on the dissolution and reduction processes as was seen in the case of prereduced pellets. When the viscosity of the slag is low enough, the slag can penetrate into the interior of the pellet through the pores and accelerate the dissolution process.

The effect of different chromite samples on the dissolution and reduction processes showed that the experiment with the prereduced pellet resulted in the highest reduction degree (75%). The situation is comparable with the ferrochrome furnace where prereduction of chromite charge material occurs in the upper part of the submerged arc furnace and there continues to be smelting reduction in the lower hearth.

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