ILMENITE SMELTING IN A PILOT SCALE DC FURNACE

S. Pisilä[1], P. Palovaara[2], A. de Jong[3], M. Aula[4]

[1] Outotec (Finland) Oy, Kuparitie 10, PO box 69, 28101 Pori, Finland, sauli.pisila@outotec.com
[2] Outotec (Finland) Oy, Kuparitie 10, PO box 69, 280101 Pori, Finland, petri.palovaara@outotec.com
[3] Outotec Pty Ltd, Unit 6 & 7, West End Corporate Park, 305 Montague Road, West End, QLD 4101, Australia, andredejong@outotec.com
[4] University of Oulu, Process Metallurgy Group, PO Box. 4300, 90014 Finland, matti.aula@oulu.fi

Abstract

Electric arc furnace smelting is one of the main processing routes used to upgrade ilmenite into a suitable titanium feedstock for pigment and sponge producers. During the ilmenite smelting process the iron oxides present in the ilmenite concentrate matrix are reduced to metallic form by using carbon as the reductant. A slag rich in TiO_2 *and low in FeO content remains, together with pig iron as a saleable by-product.*

In the early part of 2014 a pilot scale DC furnace test trial was carried out at Outotec Research Center, Pori, Finland. During this campaign ilmenite concentrate (raw) as well as prereduced ilmenite was used as smelting feed material. The prereduced ilmenite was produced separately and ahead of the smelting campaign using a pilot scale rotary kiln located at the same research facility. In this paper the some of the observations and results from the smelting trial are presented.

Good quality slag and pig iron was produced during the trial. The furnace chemistry was easy to control in both (raw, and prereduced) cases and no major technical difficulties were experienced. Pig iron carbon content was found to be lower using prereduced ilmenite. The energy consumption of the smelting process was 27 % lower using prereduced ilmenite of 58 % Fe-metallization compared to the smelting of raw ilmenite.

The active power distribution between the arc and the molten bath was studied during the trial. The active power can be divided into two sections: firstly the open electric arc and secondly the current flowing through the molten slag bath. Based on the pilot scale tests the power distribution to the arc was 85 % and to the slag 15 %.

Emission spectrometry equipment installed on the furnace roof provided a good on-line prediction of the slag FeO content based on the elemental Ti- and Fe-peaks emitted from the plasma arc. Also slag surface temperature was obtained on-line.

The suitability of the produced slag (slow-cooled and granulated) for both the sulphate and chloride route pigment production was studied with XRD tests and sulphate leaching tests. Iron titanium oxide and anatase were identified as dominant phases in both slow cooled and granulated slags. In the sulphate leaching tests good yields of 90 % TiO_2 *were obtained with both slags.*

1. INTRODUCTION

Electric arc furnace smelting is one of the main processing routes used to upgrade ilmenite into a suitable titanium feedstock for pigment and sponge producers. During the ilmenite smelting process the iron oxides present in the ilmenite concentrate matrix are reduced to metallic form by using carbon as the reductant. A slag rich in TiO_2 and low in FeO content remains together with pig iron as a saleable by-product.

Outotec as a provider of sustainable process technology for the global minerals and metals industry has developed the Energy Optimized Smelting Process (EOSP) concept in order to comply with CO_2 emission targets and to match ever increasing electrical power tariffs. The EOSP consists of a prereduction plant upstream a modern EAF smelter facility.

In the early 2014 a pilot scale DC furnace test trial was carried out at Outotec Research Centre, Pori, Finland. During this campaign raw ilmenite as well as prereduced ilmenite was used as smelting feed material. The prereduced ilmenite was produced separately and ahead of the smelting campaign using a pilot scale rotary kiln located at the same research facility.

In 2012 Outotec successfully conducted its first DC furnace test campaign for the production of ferrochrome. The objective of this follow-up smelting campaign using raw and prereduced ilmenite was to expand the test criteria by investigating the response of the process chemistry, and performance of the furnace equipment and automation system, in a greater level of detail. Newly installed peripheral furnace equipment was also tested, including a high speed camera for filming of the electric arc as well as a newly designed conductive bottom anode. Also, in situ measurement of optical emission spectrum of the electric arc and slag was conducted in order to obtain more on-line data of the furnace conditions.

2. Experimental procedure

2.1 DC furnace

The experiments were conducted by using a pilot scale DC furnace at Outotec Research Centre, located in Pori, Finland (Figure 1).

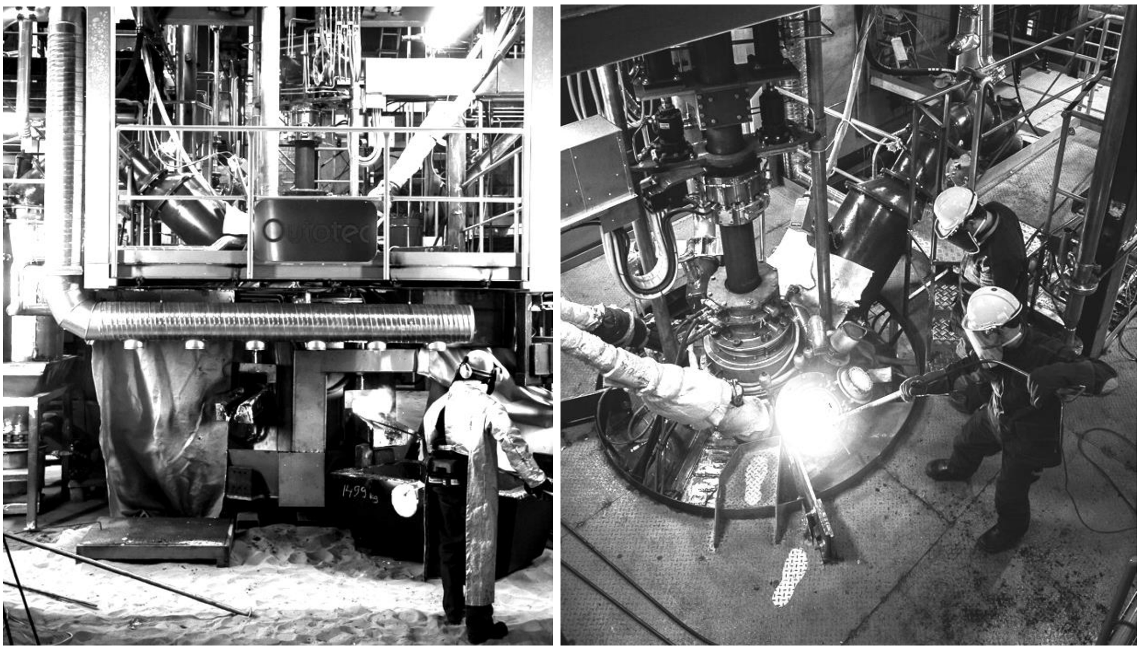

Figure 57. Outotec pilot scale DC furnace

The furnace utilizes a 2 MVA transformer that supplies AC voltage to the rectifier. The rectifier transforms the three phase AC current into DC current. The furnace employs a single graphite electrode as the cathode. The typical active power of the furnace is 400-600 kW. The conductive bottom is the positively charged anode, and the graphite electrode acts as the negatively charged cathode in the electrical circuit. The DC furnace features an in-house developed conductive hearth type bottom anode. A compensation reactor is present in the circuit to smoothen the current flow. A schematic image of the furnace equipment is shown in Figure 2. An open electric arc, shown in Figure 3, is formed between the graphite cathode and the furnace slag. Furnace feed is continuously fed to the molten pool trough feed pipes situated at the furnace roof.

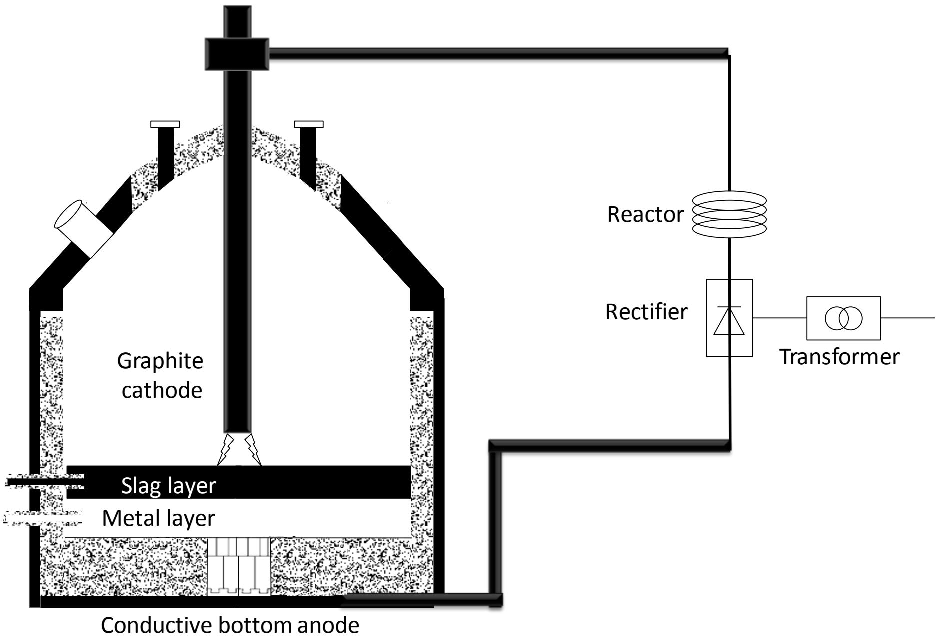

Figure 58. Furnace electrical circuit

Figure 59. Electric arc

The furnace shell is cooled using a spray water system, while the furnace roof and electrode equipment are cooled with a closed water cooling circuit. The off-gases are combusted in the combustion chamber directly after exiting the furnace roof and the gas is consequently scrubbed in a wet scrubber.

In addition to basic furnace equipment a high speed video camera was set up to the side of the furnace allowing the filming of the electric arc. An emission spectrometer was also utilized for the monitoring of the arc and furnace conditions.

2.2 Rotary Kiln

The pilot scale prereduction tests were conducted with the pilot scale rotary kiln (Figure 60) located in Pori Research Centre. The rotary kiln is 11 m long with an inner diameter of 1.2 m at the feed end.

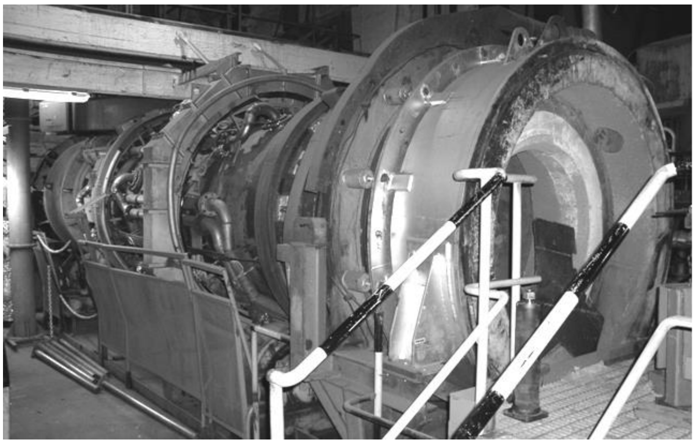

Figure 60. Pilot scale rotary kiln

The feed capacity of the feed mix is typically 300-500 kg/h. The kiln temperature profile is controlled with air blowing. Additional air is blown into the kiln with separate axial fans through air tubes at three different locations along the kiln. The kiln is sealed with water-cooled greased flanges at both ends of the kiln. Kiln pressure is controlled by the position of the flap valve in the off-gas line and with the off-gas fan speed. A slight excess pressure is maintained at the discharge end which eliminates air ingress into the kiln.

The off-gases from the kiln are directed from the feed end of the kiln to the combustion chamber where the hydrogen, carbon monoxide and volatile components are burned with pressurized air. A raw gas stack is connected to the combustion chamber, which can be opened in situations such as off-gas duct blockage, kiln holds, scrubber malfunction, power outage or maintenance.

The burned off-gas is directed to the hot cyclone which separates the coarse dust from the gas. Then the gas is directed to the wet scrubber and then to the main stack. A continuous gas analyzer is installed in the off-gas duct before the combustion chamber.

The reduced rotary kiln product is discharged from the discharge end of the kiln into the rotary cooler with a length of 7 m. The reduced product is cooled indirectly by external spray water on the cooler steel shell. The discharge is then screened and the char is separated magnetically from the prereduced product.

2.3 Test run overview

The smelting campaign was divided into hot commissioning period and 4 balance periods, based on the used feed material mix:

- Hot commissioning
- Raw ilmenite
- Prereduced ilmenite (Low metallization degree ~ 60 %)
- 50% Raw / 50% Prereduced (High metallization degree ~ 85 %)
- Raw ilmenite 2

Furnace feed, consisting of ilmenite concentrate and anthracite (reductant) was continuously fed through the furnace roof into the molten pool at a feed rate of 200 kg /h. The slag temperature was typically between 1650 - 1700^{o}C and metal temperature between 1500 - 1550^{o}C. Slag and metal products were periodically tapped from the bottom of the furnace. Main product target was that the FeO content of the slag is 8-10 w-%.
The campaign was successfully run without any major problems. No stray arcing was experienced during the trial. Also no foaming was observed, even in over reducing conditions, and the process was easy to control. The furnace chemistry responded well to changes in feed mixture and temperature control was easily controlled by the feed/power ratio.

3. Results

3.1 Energy consumption

In Table 17 and Table 18 the power requirements for raw ilmenite and prereduced ilmenite balance periods (and for a steady operation period within that balance period) are presented. The requirement is presented in three ways:

- Power requirement / ton of ilmenite, pilot scale, including heat losses
- Power requirement / ton of ilmenite, projected to industrial scale
- Power requirement / ton of ilmenite, excluding heat losses (Specific Energy Requirement, SER)

Also included in the figures is the SER value calculated with HSC Sim process model for each balance period. HSC Sim is an in-house developed software tool used for process design and scale up purposes.

Table 17. Energy consumption of the smelting process (kWh / t of ilmenite)

Feed ilmenite	Measurement units	Pilot scale*	Industrial scale projection**	SER***	SER HSC projection
Raw ilmenite	kWh / t ilmenite	2654	1531	1194	1050
Prereduced (58 % met. degree)	kWh / t ilmenite	2031	1171	914	730
Difference	%	**24**	**24**	**24**	**30**

*including pilot scale furnace heat losses of 55 %
** assuming heat losses of 22 %
***power consumption, heat loss neglected

It can be seen that the prereduced ilmenite clearly has a lower energy consumption (-24 % at industrial scale compared to raw ilmenite), even at modest 58 % prereduction degree. If the energy consumption is calculated in kWh/t of slag, the benefit is even higher, since more slag is produced from the same amount of ilmenite (Table 2).

Table 18: Energy consumption of the smelting process (kWh/t of slag).

Feed ilmenite	Measurement units	Pilot scale	Industrial scale projection	SER	SER HSC projection
Raw ilmenite	kWh / t slag	3847	2219	1731	1614
Prereduced (58 % metallization degree)	kWh / t slag	2820	1627	1269	1030
Difference	%	**27**	**27**	**27**	**36**

When the experimental SER value for the energy consumption is compared to that given by the HSC process model it can be seen that the experimental values are higher than the value calculated by HSC in both cases (raw and prereduced). HSC Sim calculates the power consumption based on pure ideal elements or phases. It does not take into account any interaction between the given elements or energy needed to decompose the mineral structure. One other reason for the difference could be small errors or deviations in the heat loss that is continuously measured during the trials. When pilot scale heat loss is assumed to be 60 % instead of 55 %, the experimental SER approaches to the value given by HSC.

3.2 Product chemistry

In Figure 61 the slag FeO and metal C contents along with the anthracite and carbon feed rate are presented for each balance period. The slag FeO content was the main parameter, based on which the anthracite feed was adjusted. The aim for the slag analysis was to produce a wide range of slags with different FeO contents. During the main balance periods the slag FeO content ranged from 3 % – 12 %.

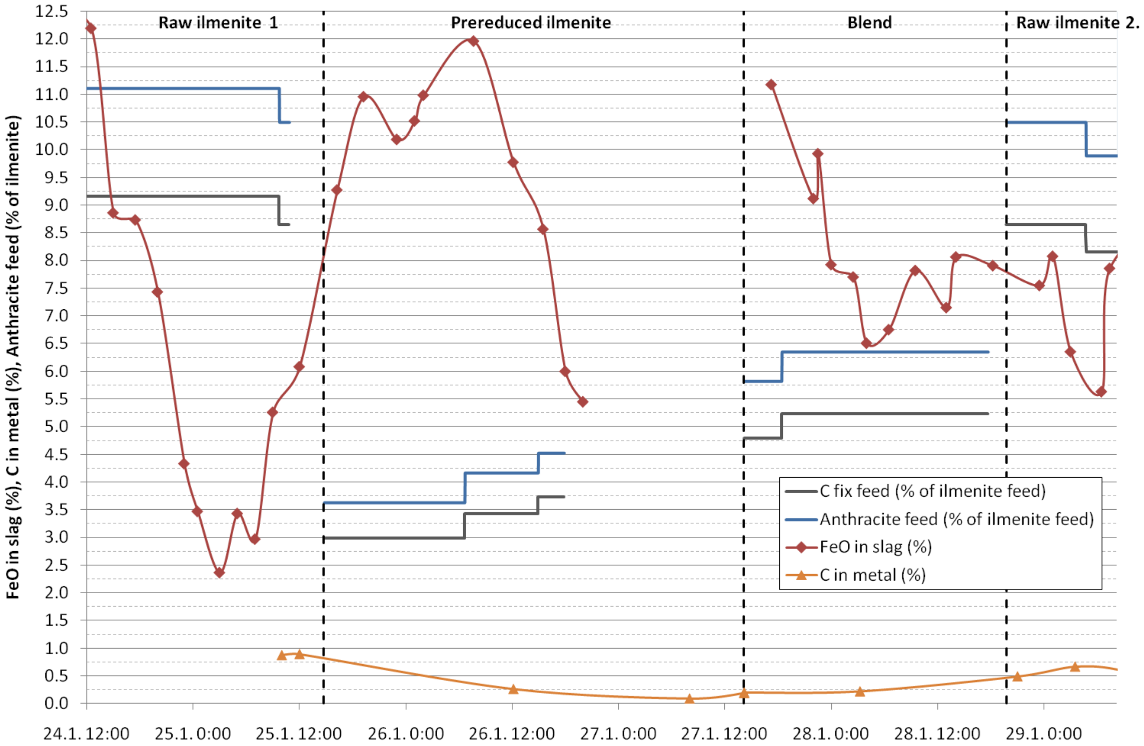

Figure 61. Metal and slag composition for each balance period

Raw ilmenite 1:

The balance period was started with the hot commissioning slag with a considerable content of FeO. During the phase the FeO ranged from 12.3 to 2.4 %. The C_{fix} feed/t of ilmenite was kept at 9.2 % for the most of the phase. This finally resulted in very low FeO content and high viscous slag so the C_{fix} feed was lowered to 8.6 % for the remainder of the phase. Even though the FeO content went as low as 2.4 % the slag viscosity remained low enough for tapping as the high MgO content in the initial slag (initial lining wear) lowered the slag viscosity. At the end of the test phase the furnace was flushed and the MgO rich slag was removed.

The carbon content of the metal was low at 0.9 %C for both metal tappings during the period. The theoretical equilibrium carbon content of pig iron that is in contact with TiO_2 rich slag during ilmenite smelting is calculated at 0.2 %C (dissolved carbon in pig iron has reducing potential to reduce FeO present in slag). However in larger furnaces the dissolved carbon in pig iron is usually ~2% [1]. This indicates that in industrial furnaces the process is further away from equilibrium condition between FeO in slag and carbon in pig iron when compared to the small pilot scale furnace.

Prereduced ilmenite:

The prereduced ilmenite (Low) phase was started with a C_{fix} feed of 3 %. This resulted in an initial FeO content ranging from 9.3 to 12.0. The C_{fix} feed was increased to 3.4 %, which lead to FeO content of 9.7 and 8.5 %. The C_{fix} feed was increased again to 3.7 % and this resulted in slag FeO contents of 6 and 5.4 %. The carbon content in the metal was very low, 0.25 %. This indicates that when prereduced ilmenite is smelted, since there is less total carbon fed into the system, the metal carbon content remains lower than when operating with raw ilmenite. Therefore the carbon content reaches the equilibrium state with the slag phase. This indicates that when smelting pre-reduced ilmenite the process is closer to equilibrium than when smelting raw ilmenite.

Blend:

The blend phase (50 % prereduced High / 50 % raw ilmenite) was started with a C_{fix} feed of 4.8 % which lead to a FeO content of 11.2 %. The C_{fix} feed was increased to 5.3 % for the remainder of the phase and this resulted in a slag FeO content of 8 – 6.5 %. The metal carbon content was very low - 0.2 %

Raw ilmenite 2:

The second raw ilmenite phase was started with a C_{fix} feed of 8.6 %. This resulted in slag FeO of 8.1 – 5.9 %. The metal carbon content was slightly higher than in the prereduced phases at 0.7-0.5 %.

3.3 Power distribution between the electric arc and slag

The active power during DC furnace smelting is generated by the current passing through the bus tubes, the electrodes (top cathode and bottom anode, the furnace gas phase (visual arc), the slag layer and the metal layer that each has a specific resistance. Practically all of the power is generated from the electric arc and the slag layer, as the resistance of the other components is very low. The power distribution between the arc and the slag was studied during the pilot scale test run.

The electric arc power was manually measured by test, whereby the electrode was lowered (while the furnace was switched off) until it touched the slag surface. The electrode cylinder positions were then recorded. The power was then switched back on and once the electrical setpoints were reached and the operation was smooth, the electrode cylinder position was recorded again. The theoretical arc length (straight arc) was calculated based on the cylinder position change. The length was confirmed visually with the high speed video camera feed.

The study of the arc power is based on few assumptions. First of all the arc resistivity is an important factor. In the following calculations a value of 0.0175 Ωcm was used as a basis. The arc resistivity was then calculated based on Bowman´s equations [2]. The arc length also plays an important role in the calculations. An average arc length of 11 cm was measured, when operating with a resistance of 60 mOhms. However, confirmed by the video feed, the arc was usually going around the electrode not straight from tip to slag but about 5-10 cm to the sides of the electrode. This accounts for 1-4 cm extra in arc length. Occasionally the thrust force created by the current formed a valley to the slag. The valley height was calculated to be ~3 cm based on Bowman´s equations for arc depression [2]. Therefore, the total arc length was concluded to be 14 cm on average.

After the arc power was calculated the remaining power of the system can be assumed to be formed in the slag layer so therefore the slag power can be calculated to be $P_{slag} = P_{tot} – P_{arc}$. As a conclusion the following power distributions specified in Table 19 were obtained from two separate measurement campaigns.

Table 19. Power distribution in the pilot scale DC furnace

Measurement campaign no.	-	1	2
Total Power	kW	458	387
Arc Power	kW	387	317
Slag Power	kW	71	70
Arc Power distribution	%	**85**	**82**
Slag Power distribution	%	**15**	**18**
Estimated Arc length	cm	15	13

Based on the calculations the power distribution between the arc and the slag ranged between 82 – 85 % from the arc and 15 - 18 % from the slag. It should be noted that the calculations depend heavily on the assumed arc length and arc resistivity so the margin of error is quite high. However, the results give a clear indication that more power (and resistance) comes from the arc rather than from the slag during ilmenite smelting in a DC furnace.

3.4 Slag mineralogy and suitability for pigment production

There are two main processes to produce TiO_2 pigment from TiO_2 slag: sulphate and chloride process. Feed material specifications vary between the two. Sulphate process utilizes finer slag particles (-100 µm) whereas the chloride process uses coarser slag (100–850 µm) [1]. The chloride process also requires a denser slag with lower level of slag impurities (e.g. MgO, CaO). Regarding the slag mineralogy the chloride process tolerates rutile phase whereas for the sulphate process slag rutile amount should be minimized [3].

Industrial slags are normally cooled by pot cooling or wet granulation. The slag pot cooling produces satisfactory quality for both processes. When wet granulation is used some reoxidation of Ti_2O_3 into TiO_2 may occur but the formation of rutile phase is very low compared to slow slag pot cooling. Totally four slag samples were analysed with XRD. Analysed slags samples consisted of granulated and slow-cooled slag produced in the pilot furnace. Main phases in these slags were the following: anatase (TiO_2), iron titanium oxide ($Fe_3Ti_3O_{10}$) and pseudobrookite (Fe_2TiO_5). Iron titanium oxide and pseudobrookite have similar XRD-intensity spikes; therefore the distribution to these phases can be only estimate from the content of iron in the slag. However, mentioned phases are both suitable for sulphate and chloride processes. Only traces of rutile were found in all of the slag samples.

A phase transformation from anatase to rutile, was observed during calcination test of granulated slag at 1100 °C (rutilization). This demonstrates that the thermodynamic driving force for rutile formation is present in the slow-cooling methods and indicates that slowing the cooling rate increases the amount of rutile in the product. The main phases in calcinated slag were rutile (TiO_2), pseudorutile ($Fe_2Ti_3O_9$) and pseudobrookite (Fe_2TiO_5). The fast cooling of the slag during granulation therefore minimizes rutile formation.

Sulphuric acid leaching tests for both granulated and slow-cooled slags were also conducted to confirm the suitability of the slag for pigment production. In both samples (granulated and slow-cooled) TiO_2 recovery rate of 90% was achieved. These test results for pilot scale test slags were similar to those obtained with commercial slags.

3.5 Emission spectrometry

A measurement system for obtaining the optical emission spectrum of the electric arc was installed to the furnace roof in order to analyse furnace conditions on-line. Optical emission spectrometry is a method rarely used in process control. The light escaping the arc and melt contains abundant information on process conditions. The robust measurement system can be used in real-time process control. The optical emission spectrometer system is under development by Luxmet Oy based in Oulu, Finland.

The measurement campaign conducted during the test run yielded a good on-line prediction of the slag FeO content based on the elemental Ti and Fe peaks emitted from the plasma arc. The correlation is presented in Figure 62. Slag surface temperature was also obtained on-line [4].

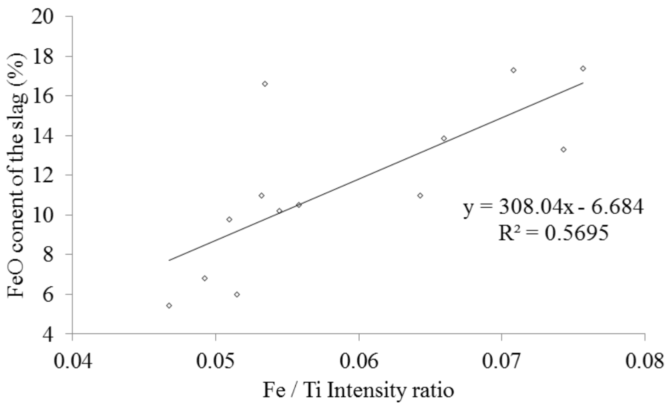

Figure 62. Fe / Ti peak intensities plotted against the slag FeO content. /4/

The downside of the system was however that the measurements could only be obtained ~1 minute after the raw material feed was switched off and the furnace gas space was clear of dust and fumes. This limited the use of the system to feeding pauses such as sampling, temperature measurements, etc.

The development of the system will continue by testing new methods by which the slag composition could be obtained even when fumes and dust are present in the furnace atmosphere. At its current state the equipment provides a great tool for the pilot scale furnace but due to the requirement of feed stoppages the equipment is not yet suitable for full scale implementation.

4. Conclusions

The test campaign was successfully conducted and the technical viability of the Energy Optimized Smelting Process developed by Outotec was demonstrated. Good quality slag and pig iron was produced during the trial. The furnace chemistry and energy balance were easy to control in both (raw and prereduced) cases. The only significant difference between the smelting chemistry between the prereduced and raw ilmenite smelting was that the pig iron carbon content was found to be lower when prereduced ilmenite was used. No significant equipment failures or other technical difficulties were experienced. The energy consumption of the smelting process was by 27 % lower using prereduced ilmenite of 58 % Fe-metallization compared to the smelting of raw ilmenite. The results collected during the project provide a good basis for further development of energy efficient DC furnace technology.

5. References

[1] Gous, M., An Overview of the Namakwa Sands ilmenite smelting operations. The Journal of The South African Institute of Mining and Metallurgy, Vol 106, (2006)

[2] Reynolds, Q.G., Jones, R.T., Semi-empirical modelling of the electrical behaviour of DC-arc smelting furnaces.(2004)

[3] Mackey, T.S., Upgrading Ilmenite into a high-grade Synthetic Rutile. Journal of Metals, April, pp. 59–64. (1994)

[4] Aula, M., Pisilä S., Fabritius T., On-line analysis of process conditions in ilmenite smelting with optical emission spectrometry. European Metallurgical Conference. Dusseldorf. Submitted 2015.