NEW AND NOVEL LABORATORY AND PILOT TECHNIQUES FOR PYROMETALLURGY

New Laboratory and Pilot Techniques Applied at Mintek in Aid of Furnace Containment System Designs

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The Pyrometallurgy Division at Mintek is known internationally for the development of applications of direct current (DC) arc furnace technology in smelting applications, more specifically in the smelting of primary resources, i.e., chromite, ilmenite, titanomagnetite, nickel laterite, and ores containing precious group metals, and secondary resources, i.e., furnace slag or dust. From a furnace containment perspective, either an insulating or a conductive design philosophy can be applied, irrespective of the raw material being processed. In the initial stages of a project, desktop studies are typically conducted which include the selection of a furnace containment design philosophy, specific to the application. To lower the risk associated with incorrect selection of a design philosophy and/or furnace containment system components, it is prudent to conduct tests on laboratory and pilot scale and to transfer the knowledge gained to industrial applications. The paper presents examples of the laboratory and pilot techniques utilized.

INTRODUCTION

In pyrometallurgy, the two main purposes of the furnace containment system are (1) to manage containment and flow of process materials in a controlled manner and (2) to minimize energy losses to atmosphere. Two basic philosophies apply: (1) an insulating or (2) conductive lining design.1 In both design philosophies, refractory materials form an integral part of the design. In an insulating lining design, fluid process materials are in direct contact with the hot face of the refractory lining. Chemical compatibility between the fluid process materials and the refractory lining is therefore of utmost importance to ensure the integrity of the furnace containment system. The chemical compatibility is driven by both the system temperature (which can also consider the so-called superheat in the case of slag or alloy) and compositions of the fluid process materials and refractory materials under consideration. In a conductive lining design, a layer of process materials freezes onto the surface of the hot face of the refractory lining. This layer forms a barrier between the refractory lining and the fluid process materials and therefore protects the refractory from chemical wear.

BACKGROUND

In a paper presented at the TMS Annual General Meeting and Exhibition in 2017,2 the modern tools utilized at Mintek, in aid of furnace containment design philosophy selection on desktop study level, were described within the context of ferrochromium (FeCr) production. These tools can be summarized as follows. For details on the numbers applied, the reader is referred to the original paper:

1. Steady-state, one-dimensional (1D) heat transfer calculations, conducted in Microsoft Excel, are utilized in estimations of the temperatures of refractory materials and the steel shell in both insulating and conductive lining designs and estimations of the thickness of the layer of frozen process material in conductive lining designs.
2. Thermochemistry calculations, conducted in
3. Viscosity calculations are conducted partially in FactSage™ and partially in Microsoft Excel to confirm that the slag will remain liquid enough to be tapped from the furnace with ease, irrespective of the lining design philosophy applied.

Steady-state models and equilibrium calculations tell only part of the story. Insulating or conductive linings have very different transient responses to disturbances in process conditions, which can affect the operability of the furnace significantly. Therefore, the importance of laboratory and pilot-scale test work to validate the selection of lining philosophy and design, including the materials selected, cannot be overstated. By not doing these tests on laboratory- and pilot-scale, the industrial-scale furnace essentially becomes a pilot furnace.

**ILLUSTRATIVE CASE STUDY**

An extreme case in point is ilmenite smelting. It is well known that ilmenite smelters can only operate with a freeze lining, as no refractory material can withstand the aggressive slag formed. Yet, if one has to base the selection of a refractory design philosophy on FactSage™ calculations only, one would consider an insulating lining design, based on MgO-refractory as hot face material, suitable. This is illustrated for a slag containing 84.2% TiO₂, 9.9% FeO, 2.0% SiO₂, 1.8% MnO, 1.2% Al₂O₃, 0.6% MgO and 0.2% CaO. For the calculation, it is assumed that the refractory material consists of 100% MgO. The Equilib module in version 8.0 of FactSage™ was applied. The FactPS and FToxid databases were selected. For pure species, the formation of all gas and pure solids was allowed. The formation of all solution phases was allowed, except in cases where there was more than one option, in which case the A option was selected. The pressure was fixed at 1 atmosphere, and both normal and transitional equilibria were calculated.

Figure 1a presents the equilibrium amount of liquid phase formed, as a function of temperature, for two cases: (1) 100 g slag only and (2) 100 g slag reacted with 100 g MgO. If the slag dissolves the refractory material in Case 2, the amount of liquid phase formed will exceed that of the amount of liquid phase formed in Case 1. This is not the case, as the amount of liquid phase formed in Case 2 is significantly less than for Case 1 (Fig. 1a). If the decision is based on comparing the amount of liquid formed in the two cases and, as a first assumption, that the solid phases formed will protect the hot face of the refractory, the slag and refractory are deemed compatible. In Fig. 1b, the equilibrium phase distribution of Case 2, as a function of temperature, is presented. The major equilibrium phase formed is titanium spinel (Mg₂TiO₄) with very little monoxide (MgO with some substitution of Mg by Fe and Mn) remaining. The liquid slag phase that forms is enriched with MgO and contains 31.8% MgO at 1700°C, which is alarming.

Another approach is to fix the temperature at 1700°C and the amount of refractory material at 100 g and to add slag at increments of 50 g. With initial additions of slag, the MgO reacts with the slag to form titanium spinel (Fig 2a). Once the MgO depleted at 133 g slag addition, only the titanium spinel remains, which then dissolves into the slag until it depleted at 201 g slag addition. When one considers the MgO content of the slag formed (in g), the increase is 0.1 g MgO/g slag added when the titanium spinel forms but 1.2 g MgO/g slag added when it dissolves. The MgO distribution per phase as a function of the slag added is presented in Fig. 2b.

This explains why high-TiO₂ slag is not compatible with MgO refractory, but the deeper investigation was driven by the prior knowledge gained experimentally through laboratory-, pilot-, or industrial-scale tests. Were the results of the latter not available and a decision made to build an industrial-scale furnace based on the results presented in Fig. 1a, the refractory lining would have been destroyed within days of start-up if not hours, with dire consequences. Through a number of examples, the article illustrates the laboratory and pilot techniques utilized at Mintek in aid of furnace containment system designs.

**LABORATORY-SCALE INVESTIGATIONS**

The advantages of laboratory-scale investigations include tight control of conditions of interest, i.e., temperature, atmosphere, composition of materials and time. They require small amounts of material and are inexpensive compared to larger, pilot-scale investigations. The disadvantages include the fact that refractory materials are only exposed to small amounts of process materials and for much shorter time periods compared to the industrial applications. Variability in operational conditions, typical of industrial applications, are also not mimicked. Laboratory investigations are therefore more suitable for studies that require very controlled conditions, i.e., cup tests used to validate the results of thermodynamic calculations, rotary finger tests that investigate the effect of liquid flow on refractory wear and water-cooled finger tests used to study
freeze-lining formation in slags. Practical challenges include atmosphere control, especially where the equilibrium partial pressure of the alloy components of interest are high and many fumes are formed, i.e., lead smelting, and containment of the liquid process materials, in the case of the rotary finger and water-cooled finger tests. When selecting crucibles for the latter applications, the slag under consideration has to be saturated or chemically inert to the material of choice, which can be determined by FactSage calculations. The cup test and rotary finger examples presented below are for processes near carbon saturation. More development work will be required, when applications more oxidizing than carbon saturation are to be considered.

**Cup Test**

The cup test, also called crucible, cavity or brick test, is an example of a stationary corrosion test.
and applicable to insulating lining designs. Corrosion tests are applied in cases where the potential for chemical wear, identified through equilibrium thermodynamic calculations, is validated.

As an example, in an investigation into the wear mechanisms of carbon-based refractory materials in silico-manganese (SiMn) tap holes, equilibrium calculations conducted in FactSage™ indicated the potential for SiO₂ and MnO in the slag to react with carbon refractory material to produce SiC, an alloy phase containing Mn, Si and C and a gas phase containing CO, Mn and Si. The results of the calculations were validated using the cup test method. Two different types of refractory materials were considered: a carbon block and a carbon-based ramming paste. A crucible, which also served as a susceptor, was cut from the carbon block. The typical dimensions of the crucible were as follows: inner diameter of 25 mm, outer diameter of 65 mm, total height of 70 mm and crucible depth of 45 mm. The carbon-based ramming paste was rammed into a graphite crucible by hand and pre-baked at 950°C. The cavity in each crucible was filled with 40–45 g of slag. Experiments were conducted in an induction furnace, in an argon atmosphere for 1 h, where the holding temperature was 1400°C, 1500°C and 1600°C respectively. Upon cooling, the cavity was filled with resin and the sample sectioned. The wear and infiltration profiles were determined by hand, although in this example both were insignificant. Samples were subsequently sectioned and prepared for investigation by scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS). SEM-EDS analysis confirmed the presence of the reaction products (alloy and SiC) predicted by the FactSage™ calculations.

It is thus the combination of cup tests and post-mortem investigations that improved the confidence in the FactSage™ results. These tests are typically run when the choice of refractory material needs to be narrowed down from several types to one or two types or from several suppliers to one or two suppliers before evaluation on pilot scale.

Rotary Finger Test

The rotary finger test is applied in refractory applications where the potential for both chemical wear and erosion exist, i.e., the furnace tap hole in either an insulating or conductive lining design.

The experimental set-up is presented in Fig. 3. The refractory cylinder (“finger”) is attached to a cylindrical graphite rod using graphite glue. The graphite rod is mounted onto an extension arm, which is connected to the rotating motor. The graphite crucible is placed inside the 25-kW induction furnace to facilitate heat transfer, and alumina bubbles are added between the crucible and the furnace chamber to achieve the required height and minimize energy losses to the environment.

Thermodynamic calculations in FactSage™ are conducted to predict the chemical wear when the refractory is in contact with the slag/alloy, and the experimental tests are then used to validate the thermodynamic calculations. An initial experiment is conducted in which the finger is kept stationary to allow for a comparison between the results of the thermodynamic calculations and chemical wear only. The static test results also form the baseline against which the results of the dynamic tests, which allow for the contribution of erosion due to hydrodynamic effects to be quantified, are compared.

As an example, in an investigation into the wear mechanisms of carbon (C) and silicon carbide (SiC) based refractory materials by SiMn alloy, equilibrium calculations were conducted in FactSage™ at temperatures of 1550°C, 1600°C and 1650°C. The alloys contained 15, 17 and 18 mass% Si and were put in contact with carbon-based refractory material (Type K) or a SiC based refractory material (Type SiC). The calculations indicated that the SiMn alloy was not saturated in either C or SiC, that the C solubility in the alloy decreased with increasing Si content and that the alloy was saturated with SiC at 17 mass% Si.

FactSage™ calculations further predicted that the Type SiC refractory material would experience the most wear when the temperature was varied and the alloy composition was fixed at a Si content of 15 mass%. When the temperature was fixed at 1600°C and the Si content varied from 15 to 18 mass%, the calculations predicted that Type K refractory material will experience the most wear. The results of the calculations were validated using the rotary finger test. The changes in the dimensions of the refractory cylinders were measured by hand before and after the experiments, and the
results were visually presented using Autodesk Fusion 360 3D CAD/CAM design software.

For Mn-Fe-Si-C alloys with a Mn:Fe mass ratio of 4.4, it was found that at various temperatures and constant Si content at 15 mass%, Type SiC refractory material experienced more wear than Type K. This was explained by the FactSage calculations, which indicated that increasing the temperature at constant Si content would result in the alloy becoming C saturated and SiC unsaturated. Varying the Si content at a constant temperature of 1600°C, Type K refractory material was expected to experience more wear, and when the alloy was SiC-unsaturated Type SiC was expected to experience more wear. Experimentally, the behavior of Type K refractory material was contradictory as the alloy experienced less wear, which could be attributed to the SiC product forming a protective layer that inhibited further refractory wear. The behavior of Type SiC was also contradictory, and this was due to physical properties such as porosity, which allowed the alloy to preferentially attack the carbon-based binder, thus promoting refractory disintegration.

In this case, the combination of rotary finger tests and post-mortem investigations improved the confidence in the FactSage results. It was also found that the physical properties of refractory material can create deviations from thermodynamic calculations and thus illustrate the importance of doing both the calculations and experimental work.

**Water-Cooled Finger Test**

As mentioned in the introduction, conductive lining (also known as freeze lining) designs are obtained by maintaining a layer of frozen process material on the hot face of the refractory lining. This is achieved by manipulating the furnace conditions in such a way that heat is efficiently removed to keep refractory temperatures low enough to enable the formation of freeze linings. Efficient heat transfer can be achieved by internally cooled panels or externally cooling the shell of the furnace. In recent years, conductive linings have proven to increase productivity by reducing refractory wear, but by using internal and external coolants a malfunction or a burn-through can result in catastrophic failures. A proper understanding of the mechanisms behind the formation of freeze linings and their thermal properties is required, which is why Mintek established a "cold-finger"/"cold-probe" experimental test facility. The cold-finger facility aims at simulating the formation of freeze linings under real furnace conditions.

The experimental set-up (Fig. 4) is based around a 30-kW induction furnace, allowing temperatures up to 1700°C to be simulated. The furnace cavity can fit a crucible capable of handling 3500 cm³ of process material. Once the process material is in a molten state, a forced cooled probe is submerged into the molten bath, onto which a freeze lining then starts to form. The probe is assembled in a counter-flow arrangement with an outer diameter of 16 mm, fabricated from 316 stainless steel. By having the probe assembled in a counter-flow arrangement, heat transfer between the probe and the process material is optimized. The probe is designed to be used with a gas or a liquid as a coolant. A K-type thermocouple is fitted to the probe-coolant inlet and outlet, to be used for determining the rate of heat transfer. The probe is fitted to an actuating platform that allows the exact position of the tip of the probe, relative to the molten bath, to be controlled and maintained. By making use of the actuating platform, the depth and size of the freeze-lining sample can be controlled, as it is also used to extract the sample from the molten bath. The freeze-lining sample, frozen onto the probe, can then be removed from the furnace and externally cooled, or it can be retracted to just above the molten bath and cool with the furnace in a purged environment. The whole assembly is enclosed in a safety enclosure and all control operations can be done remotely, ensuring the safety of the operator in case of a probe malfunction.

It is foreseen that the samples prepared in this experimental set-up will provide more information on the extent of the phases, predicted from equilibrium calculations in FactSage form. The installation is new. Further development work will include developing methods to determine how the data from the cold finger tests can be utilized in the design of freeze linings, evaluating the effect of changes in slag composition on freeze lining formation (or wear) and the effect of utilization different cooling media on freeze lining formation.

**PILOT-SCALE INVESTIGATIONS**

The main advantage of pilot-scale investigations is the fact that the variability in operational conditions, typical of industrial applications, can be mimicked to some extent. The tests are typically
run over a number of days, or even weeks, which allows for the containment system design in totality (refractory material, steel shell and cooling system) to be evaluated under various conditions. The main disadvantage of pilot-scale investigations is the costs involved in running a campaign, and a campaign is seldom executed for the sole purpose of evaluating the containment design, especially on the larger-scale facility. Having said this, small-scale pilot tests are conducted for a fairly short period of time to evaluate the performance of refractory bricks from a number of suppliers under similar conditions.

100 kVA Test

An example of small-scale pilot-scale test work in which the performance of different types of refractory bricks is evaluated under similar conditions is the pot test conducted in the 100 kVA DC arc furnace. The furnace is made from a mild-steel shell, and the furnace roof refractory is usually lined with a casting compatible with the test refractory bricks. Typically an alumina-based casting is used. The furnace roof has three ports, one for the electrode, the second for the electrode and the last one as the feeding port. A single tap hole is used to tap the furnace by tilting the molten material out. The furnace has dimensions of 650 mm ID, 570 mm deep. In Fig. 5a, the schematic diagram of a typical brick set-up can be observed. Six different types of brick can be accommodated if they can be ground to have a taper; otherwise, five types of brick would be the maximum. In Fig. 5b, three different refractory bricks were used in the refractory evaluation test, where two bricks were of experimental interest and the other was used as a filler brick.

Due to operational challenges and downtime, deviations from the plan may occur but the overall campaign is representative of what would take place in industry. The furnace is operated for 12 h continuously per test and allowed to cool at the end of the last tap, after which it is dismantled and prepared for the next test. Macroscopic investigations are conducted to quantify the amount of wear of each brick and determine which type of brick wore the most. The wear is quantified by measuring the changes in the dimension of the bricks by hand followed by SEM-EDS analyses confirming any chemical or phase changes in the brick. The wear profile of the refractory bricks, based on the measured dimensions, are presented using Autodesk Fusion 360 3D CAD/CAM design software.

200/300 kVA Campaign

The 200 kVA DC arc or 300 kVA AC arc furnaces are the smallest scale furnace applied in continuous pilot-scale operation where a campaign lasts over a number of days. Because of the low thermal efficiency associated with the operation of these smaller furnaces, the campaigns are typically focused on exploratory test work for the evaluation of new flowsheet designs. When conducting test work on the effect of preheating of ore on furnace performance, in terms of CO₂ emitted and energy consumed in the case of high-carbon ferromanganese (HCFeMn) production, a radically different design approach had to be followed.

The conventional insulating designs for the pilot furnace are based on high MgO castable refractory material. In the new design, a high alumina castable refractory material, which has a thermal conductivity of 2.4 W/mK (according to the data-sheet received from the supplier), was selected for the roof and hearth. The working lining consisted of carbon brick (thermal conductivity of 10 W/mK), which has been found in industry to be most compatible with the slag associated with HCFeMn production even in insulating designs. For the backlining, slip cast bubble alumina will be installed. Many applications of bubble alumina, in non-slip cast form, can be found in the laboratories at Mintek but this will be the first time the material is used in slip-cast form on a pilot scale. This ensured not only compatibility with the refractory
material in the hearth and the roof but, with a thermal conductivity of 0.5 W/mK, also significantly improved the minimization of energy losses. To accommodate the design changes, the diameter of the furnace had to be increased significantly (from 960 mm to 1560 mm). The design is presented in Fig. 6. Installation of the lining and evaluation of its performance will take place during an upcoming campaign in the next few months.

3 MVA Campaign

The large-scale pilot furnace (3 MVA DC arc) is typically applied for 2–3-week technology demonstration campaigns where the main focus of the campaign is not necessarily evaluating the furnace containment design, but rather to generate sufficient process information for furnace scale-up, as would be required for commercialization of the process. In other words, such a campaign would produce the design criteria for the industrial-scale plant for a specific resource, operating regime and commodity produced. It is important though to realize that a secondary benefit of such a campaign is to evaluate the furnace containment design philosophy selected as well as the components it is based on, i.e., refractory materials, cooling system and metal structural components as well as instrumentation used for monitoring and control.

An example of such a pilot-scale campaign was the evaluation of dual-electrode technology for application in the production of HCFeMn. Although the main purpose of the campaign was to develop the electrode control philosophy associated with the dual-electrode configuration, the fact that HCFeMn was selected as commodity allowed for a number of interesting techniques to be developed in terms of furnace containment.

The conventional lining design applied in the operation of the 3-MVA furnace is a conductive design and consist of a magnesia-based rammable in the hearth, magnesia bricks on the sidewalls and alumina castable in the roof. Both the sidewalls and the roof are typically water-cooled, the roof by forced pipe cooling and the sidewalls either by thin-film cooling or forced pipe cooling. Bi-level tap holes are installed at 60° and typically 300 mm apart, radially and vertically, respectively, for slag and alloy tapping. The furnace and lining design has developed over the years, starting with ferrochromium production, but was also applied to the campaigns associated with development of the applications of DC furnace technology in ilmenite and nickel laterite smelting.

For the dual-electrode campaign, the roof design was modified to allow for manual rabbling but the refractory design was maintained. The sidewall and hearth lining designs were adapted to include a carbon rammable as working lining with the magnesia bricks as back-lining. Water cooling was maintained on the sidewall (in hindsight it probably contributed to tapping problems experienced during the campaign). A graphite tapblock was installed to form a single, single-level tap hole through which slag and alloy were tapped during the same tap. The modified design is presented in Fig. 7. In hindsight, the design suffered from the hybrid-design problems that result from installing different linings in existing equipment. Fortunately, the matter could be rectified when the furnace in Fig. 6 was designed from scratch.

The graphite tapblock also had a custom design, which included 30 1.5-mm K-type thermocouples installed in such a fashion to measure the heat losses radially at 90-mm intervals from the cold face of the tapblock in the direction of the hot face. Although the original intention, which was for the
thermocouple readings to validate a heat transfer model of the tapblock, did not materialize, the thermocouple readings were very useful in monitoring the tapping intervals and tap durations during the campaign.

During the furnace excavation, after the campaign, useful information is gathered in terms of the wear profile of the lining, and samples of the refractory material are taken for phase chemical analyses. The wear profile is used to identify areas of high wear which require design modifications. The conventional method of determining the wear profile is based on a stick held as a reference in the center of the furnace and manual measurements done from the hot face of the lining to this stick. The manual method allows for point measurements from which a wear pattern is inferred. For the new method, a 3D scan (Fig. 8a) was prepared by an external company using a Leica HDS6100 ultra-high-speed laser scanner. The 3D scan was converted into 2D drawings of 12 sections to allow for a thorough investigation of the wear profile. Figure 8b presents the cross-section through the tap hole where the 2D drawing of the wear profile was superimposed unto the initial refractory design (an improvement will be to scan the refractory lining after installation, before use) to identify areas of high wear and build-up, or lifting, of the refractory material. After scanning, samples of the refractory material were taken for so-called post-mortem analyses. The conventional method is based on removing the refractory material with jackhammers and selecting samples by hand. For the new method, samples are core-drilled from the lining just after
being scanned but before the jack-hammers are introduced.

CONCLUSION

The design of a new furnace containment system can be based on an insulating\textsuperscript{14} or conductive\textsuperscript{15–17} philosophy. The selection of refractory materials plays an important role in the system design irrespective of the philosophy applied.\textsuperscript{18} Heat transfer and thermodynamic calculations are useful on desktop study level when alternatives are considered, but validation of the results by experimental work on laboratory- or pilot-scale is important\textsuperscript{19} lest the industrial-scale furnace becomes the pilot furnace. Laboratory-scale investigations are more suitable to studies requiring very controlled conditions\textsuperscript{20,21} to validate the results of thermodynamic calculations. Pilot-scale investigations can better mimic industrial conditions but are expensive. Pilot-scale investigations are seldom executed for the sole purpose of evaluating the containment design, especially on the larger-scale facility. Evaluation of the containment design is rather a consequence of work done in preparation of the design criteria for the industrial-scale plant for a specific resource, operating regime and commodity produced.\textsuperscript{10,22} Mintek is in the unique position that it not only has laboratory- and pilot-scale facilities available\textsuperscript{23} in which research in terms of furnace containment designs can be conducted, but also capabilities in FactSage\textsuperscript{TM} calculations\textsuperscript{24} and facilities in which phase chemical analyses are conducted on as-received and post-mortem refractory samples.\textsuperscript{25} It is the combination of these facilities and techniques that makes the research conducted on furnace containment designs so powerful.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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