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# Research in Pyrometallurgy at the University of Pretoria, 1980-2005

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**Abstract** – Substantial research in pyrometallurgy at the University of Pretoria started in 1980 with the appointment of Rian Dippenaar to the Iscor Chair, a position that he held until 1996, when Chris Pistorius became the leader of this pyrometallurgy group. This paper reviews the main research themes over the quarter-century from 1980 to 2005, as well as some of the industrial developments that have shaped the research approach. The main topics have been the use of electrochemical sensors for equilibrium measurements, ilmenite smelting, steel processing, and reduction.

## INTRODUCTION

This paper gives a brief overview of the pyrometallurgical activities in the Department of Materials Science and Metallurgical Engineering of the University of Pretoria, from 1980 to the present. While 1980 does not represent the start of pyrometallurgy at the University of Pretoria – before 1980 Prof. D.B. le Roux held the position of Professor in the Iscor Chair – the period since 1980 has been well documented (both in an internal report[1] and many scientific publications), and I also have personal experience of much of this period. Rian Dippenaar held the position as Professor in the Iscor Chair from 1980 to 1996, and since 1996 I have been in charge of the pyrometallurgical activities in this department. I give below my perspective on the major activities over the quarter-century since 1980.[*]

This is not a scientific paper, in that it offers no hypothesis that can be tested by experiment. However, it is based on the premise that successful academic activities in pyrometallurgy require the three major elements of, first, training, teaching, and development of students; second, industrially relevant research and; third, strong contact with industry. This review addresses the ways in which these three elements were developed. I submit that neglect of any one of

[*] This paper also departs from the conventions of a scientific paper, in that the first person is used frequently. Referring to my own points of view while avoiding the words 'I' or 'me' appears rather artificial in a ruminative paper such as this. The convention of a long reference list is retained, though – all papers from this group, which have been published in refereed journals since 1980 are listed. While this makes for a disproportionately long reference list, the papers constitute a useful record of the research activities of this group.

these three would cause academic pyrometallurgy to wither and disappear. To a large extent, this threefold approach results from the unique position in South Africa, with its very large and sophisticated metallurgical (and specifically pyrometallurgical) industry, compared with a small academic community in this field. This unusual ratio makes for a wealth of opportunities for teaching and research; also, it is clearly a challenge for any one department to cover the rich spectrum of South African pyrometallurgy.

## TODAY

Three academic staff – Andrie Garbers-Craig, Johan de Villiers, and I – lead projects in pyrometallurgy. The undergraduate programme in Metallurgical Engineering includes a module on metallurgical thermodynamics, one on refractory materials, one on reaction equilibria of pyrometallurgical reactions, and one on kinetics of pyrometallurgy. The postgraduate coursework programme has a module on each of refractory materials and pyrometallurgy. A bridging module on pyrometallurgy and refractory materials forms part of an honours programme for BSc and BTech graduates. Short courses on relevant instrumental techniques, refractory materials, and analysis of pyrometallurgical processes are offered to industry. At any time, between ten and twenty postgraduate research students are engaged in research on topics which include ilmenite smelting and the phases in titania slag, sinter production and properties, mould flux, scale growth during reheating, clean steel practice, metal flow in continuous casting, reduction of iron oxide, the behaviour of coal in pyrometallurgy and gasification, and immobilisation of hexavalent chromium. (Numbers at the start of 2006 are thirteen master's degree students and six doctoral students.) Final-year undergraduate students in Metallurgical Engineering complete projects that are aligned with these industrially sponsored research programmes, with a typical number of five such projects annually.

All research topics are planned annually with the relevant industrial partners – for the current projects; these are Mittal Steel, Kumba Resources, Columbus Stainless, and Sasol. Further input from industry is gained at biannual meetings of the departmental Advisory Board, which consists of senior engineers that represent the entire local metallurgical industry.

## 1980 - 1996

Rian Dippenaar occupied the Iscor chair from 1980 to 1996. When Rian Dippenaar took up the Iscor chair, Dirk Kotze (then also on the academic staff of the department) played a large role in the initial development of the activities and direction of the pyrometallurgy group. Notable features of Rian Dippenaar's tenure included the establishment of his strong personal contacts with noted experts in ferrous pyrometallurgy overseas, many of whom visited South Africa to present lectures and short courses – these included Richard Fruehan, Alan Cramb, Geoff Belton, Keith Brimacombe and Indira Samarasekera. Also, through cooperation with Mintek, a research group on ironmaking and steelmaking was established, with full-time master's degree and doctoral graduates who are now playing – or played – notable roles locally and overseas. The doctoral graduates include Martin van Wijngaarden, Ockert Pauw, Piet Botha, Willem van Niekerk, Kobus Geldenhuis, John Niayesh, Lourens Erasmus, and Andrie Garbers-Craig.

This was a period of innovation and change at Iscor itself, including its transformation from a state-owned to a private company, the construction of the first Corex plant at Pretoria Works, and the short-lived conversion of that works to stainless steel production. Research themes during this period focused on industrial issues of importance to Iscor. Slag-metal equilibrium was a prominent theme, with electrochemical sensors the tool of choice to quantify activities – of iron oxide and manganese oxide,[2,3,4,5] and of alloys in the metal phase.[6,7] The fundamentals of zirconia-based probes were elucidated.[8] The major effect of sodium oxide on dephosphorisation was clearly established.[9,10,11]

The shortage of coking coal in South Africa was then, as now, the spur behind much research activity, and this was also true of the Dippenaar group, with work on interaction with alkalis, optimal coal blending,[12] and DRI production (in a fluidized bed).[13]

The first number of projects on ilmenite smelting started during this period; detail on their outcomes is given in the next section.

This period saw the installation of several vertical tube furnaces in the university laboratory; these remain the core of the laboratory equipment.

In a 1990 review of his first ten years in the Iscor chair, Rian Dippenaar wrote: "I am grateful for the great understanding at Iscor for the role and function of a university department in the industrial development of this country, and especially because the university is increasingly seen as a partner."

## SINCE 1996

Several changes that took place around the time that I inherited the pyrometallurgy group (in 1996) have shaped the group's activities over the past ten years. Discussions with several leaders of research groups elsewhere – Richard Fruehan, Alan Cramb, Geoff Belton, Ken Mills and Seshadri Seetharaman – helped to choose the research direction and to decide on a list of required equipment. As a result, grants (from the NRF, THRIP, and the Innovation Fund) were used to add a high-temperature viscometer, mass spectrometer, gas mixing units, and a radio-frequency induction furnace. With Kobus Geldenhuis, then a colleague at the University of Pretoria, we developed a strongly computer-based short course on steelmaking – first presented at Saldanha Steel in 1998, and repeated many times since, at several plants (including the then BHP in Australia). The approach is to make available equilibrium data in a convenient form that supports quantitative process analysis. Through Andrie Garbers-Craig and – since 2005 – Johan de Villiers joining the group, a much broader research programme has become possible. The main outcomes of this are the following:

**Research in ilmenite processing and smelting**

This research programme started when Iscor acquired ilmenite deposits, and started to develop a process to upgrade this to titanium slag. All the development work was undertaken by Iscor, later Iscor Heavy Minerals, and then Kumba Technology and Ticor. The role of the university programme was to establish some of the fundamental knowledge. The secrecy around the details of ilmenite smelting had the result that, at the inception of the programme, little detail was known on ilmenite smelting – the available information was in papers published by Quebec Iron and Titanium (QIT) until the 1970s, and equilibrium work performed by Josef Pesl and Hürman Eric (of the University of the Witwatersrand) in the early 1990s. This meant that much fundamental work could and had to be done; understanding of the smelting process developed through three essential elements: pilot plant measurements by Iscor Heavy Minerals (later Kumba Technology), laboratory projects by research students, and the availability of thermodynamic solution models for relevant liquid and solid phases in the FactSage software package. Laboratory-scale experimental study of smelting is difficult (because of the high temperatures involved, and the aggressive nature of the slag towards possible containment materials), and hence the access to the pilot plant samples and results has been crucial to the programme.

The main questions that have been addressed concerned smelting itself, pre-processing of ilmenite, and oxidation of solidified slag.

*a) Smelting reactions*

The overall reaction in ilmenite smelting is not particularly complex, involving reduction of most of the iron oxide in the feed to liquid iron, and of some of the tetravalent titanium to trivalent titanium (which remains in the slag phase). However, a major question is why the very specific relationship between the extents of these two reactions is found. This question is important both for practical reasons (since the formation of trivalent titanium affects the melting point of the slag, smelting energy requirement, and oxidation reactions of the solidified slag after smelting) and for its link to fundamental smelting reactions. The origin of this relationship has been teased out through the following research questions:

- Is reaction equilibrium responsible for the reduction relationship? No![14,15]
- Is the relationship consistent between furnaces of different size and type, and with different ilmenite purities? Yes![15]
- Does the relationship originate from the solidification equilibrium between the liquid slag and the solidified slag that forms the freeze lining in the furnace? No![16,17,18]

It has been concluded that the combination of cycling of material and partial solidification of the slag in lower temperature regions (the metal bath is 100-150°C colder than the liquid slag) is the most likely driver of the observed slag composition relationship.[19]

Earlier in this programme, the behaviour of vanadium in the slag was studied.[14,20] Vanadium is regarded as an impurity in the titania slag product, and the redox equilibria of vanadium could possibly be used as an indicator of the extent of reduction. The conclusion was that the activity coefficient of trivalent vanadium in the slag is similar to that of trivalent titanium.
The process understanding which has resulted from this programme is best summarised in two recent papers.[17,19]

*b) Processing of solidified slag*
The fundamental issue is the relationship between slag composition (mainly FeO and trivalent titanium contents are important) and phase changes during oxidation, whether in air or during the initial stages of carbochlorination. The projects confirmed the importance of trivalent titanium, through the phase changes that are triggered by its oxidation to the tetravalent form.[21] This has implications for slag upgrading processes,[22] decrepitation of the slag by low-temperature oxidation,[23,24] and exothermicity and phase changes during chlorination.[25]

*c) Processing of ilmenite before smelting*
Crude ilmenite (as obtained from minerals processing by density and electrostatic separation) often contains chromite as an impurity. Oxidative roasting serves to increase the magnetic susceptibility of ilmenite, allowing magnetic separation from chromite. The assumption that chromite does not change during oxidative roasting was tested[26] – the chromite in fact also magnetises to some extent, with implications for separation efficiency. An alternative to smelting is hydrometallurgical processing of ilmenite by leaching; hydrolysis of dissolved titanium was found to play a major role in leaching,[27] and to be the likely cause of the apparently contradictory leaching results in the literature.

**Research in steel processing**
The specific issues addressed by this group in the very wide and well-researched field of steel processing have all dealt with the link between processing conditions and steel quality. The projects have used a combination of plant samples and laboratory measurements, to study the following:
a) How do the composition and viscosity of the mould flux (lubricant for continuous casting) change during casting?[28,29]
b) What is the behaviour – kinetics and equilibrium – of nitrogen and sulphur during processing of liquid steel in a ladle and in the weld pool?[30,31,32,33]
c) What is the link between steel composition and inclusion formation and modification?[34,35]
d) What factors govern the de-scaling behaviour of reheated stainless steel slabs?[36]

**Research in reduction**

The overall aim of these projects is to establish the link between process conditions (such as temperature, reductant type, and heat transfer) and the rate and efficiency of reduction of metals from their oxides.

a) The reduction rates of lump ore[37] and self-reducing pellets[38] have been studied experimentally, with modelling predictions of the link between heat transfer and fundamental reaction kinetics;[39] the possibility of increasing sintering rate without losing sinter quality was examined.[40]

b) The relationship between vanadium oxide activity and the basicity of alumina-rich lime aluminate slags was determined, to optimise vanadium recovery in ferrovanadium production.[41]

**Research in process control**

In process control, my inputs have been in support of projects led by Ian Craig, of the Department of Electrical, Electronic and Computer Engineering at the University of Pretoria. The aim is similar in all cases, to develop a fundamental, dynamic process model that is then linked to the controller, to obtain improved process control. This approach has been applied to electric furnace steelmaking,[42,43,44] hot rolling,[45,46] and continuous casting.[47,48,49]

**Changes in industry**

The changes in the pyrometallurgical industry during this period are well-known, but worth recording here because of their profound effect on the activities in the group, and of the graduates from the group. These changes included the unbundling of Iscor into Kumba and Iscor, and Iscor then becoming part of the world's largest steelmaker, Mittal Steel; Columbus Stainless becoming part of Acerinox, large increases in ferrochromium production, and shifts in ownership, and the large expansion programmes in the platinum industry. The historical lack of work on platinum smelting in this group is now being remedied; stronger activity in ferro-alloys is envisaged in the near future.

## THE NEAR FUTURE

The main criteria for selection of research topics in this group are their importance to local industry (all the research is planned in conjunction with the relevant industries, who support the work financially), scientific interest (essential to postgraduate training), and feasibility (each project must be formulated in such a way that it can be completed with the equipment, data, and computer models which are available, within the allocated time of a postgraduate project).

Core themes for the next few years are likely to be as follows; some work is underway in all of these already:

*Clean steel:* The overall aim is to understand the constraints to and possibilities of local production of steel with controlled inclusion content, size, and composition. Much is known about clean steel already, but production of higher-value steels (such as those used in roller and ball bearings) require better and more consistent cleanliness levels than those generally achieved by the local steel producers.

*Reductant behaviour in pyrometallurgy:* Reductants (coal, coke, and char) are a major input cost in the production of ferro-alloys, yet there are not clear guidelines (in the open literature) for their selection, especially with regard to reactivity and dissolution behaviour in the metal. Reductant selection criteria can only be formulated if reaction mechanisms and electrical effects in submerged-arc furnaces are well understood. Current work on reduction under heat transfer control is the first step, focusing on reduction of iron oxide, and radiative heat transfer. In the production of ferro-alloys, electrical effects and slag melting behaviour need to be considered as well. The aim is hence to combine modelling with laboratory-scale experimentation (*e.g.* measurement of reductant dissolution and reductant-slag reaction rates).

*Process modelling:* Johan Zietsman's PhD project dealt with the modelling of ilmenite smelting.[19,50] The model combines reaction kinetics with equilibrium calculations (using the FactSage / ChemSage approach to free energy minimization and solution modelling). The model was developed as a generic framework, and in current projects it is being applied to zinc sulphide roasting (with the addition of population balancing), and ilmenite smelting (extended version of original), in a joint project with Johan Zietsman and his consulting company (Ex Mente).

*Equilibria and kinetics of high-temperature phase changes:* The main projects in this area deal with iron sinter production, refractory materials, and coal ash. Substantial experience has been built up on the phases in iron sinter, and their formation and effect on sinter behaviour during reduction. The next phase will relate the laboratory-scale results to in-plant behaviour. The recent addition of a high-temperature X-ray diffraction facility allows changes during oxidation of titanium slag and sintering to be followed in much more detail. An important additional theme is the formation of hexavalent chromium in plant by-products, and the stabilisation of the chromium by subsequent thermal treatment.

*Possibilities:*
Current exploratory projects that may lead to longer-term core topics are as follows:
- Electrochemistry in pyrometallurgy: A project on oxygen activity measurements during conversion of platinum-bearing matte is currently being completed. A short sabbatical in Derek Fray's group served to complete a short research project related to the FFC process for electrochemical production of metals.[51] This process is potentially important to South Africa as a producer of titanium feedstocks, and has the scientific attraction of combining pyrometallurgy with electrochemistry.
- Reactions in smelting of platinum-bearing concentrates: A part-time project is currently underway on base metals losses to slag; initial results have shown the

usefulness of FactSage for equilibrium predictions on matte and slag solidification.

Finally, the aim is to achieve closer cooperation with other research groups in pyrometallurgy in South Africa, possibly through the formation of a combined research group, with joint planning and reporting of research.

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