The relationship between FeO and Ti$_2$O$_3$ in ilmenite smelter slags

P. Chris Pistorius

Department of Material Science and Metallurgical Engineering
University of Pretoria

Abstract

Smelting is used to upgrade ilmenite (FeO.TiO$_2$) to high-titania slags which serve as feedstocks for TiO$_2$ pigment manufacture. During smelting partial reduction of FeO to liquid iron, and of TiO$_2$ to Ti$_2$O$_3$, occurs. Lower FeO levels in the solidified slag correlate with higher Ti$_2$O$_3$. Since some pigment manufacturers limit the allowable Ti$_2$O$_3$ level in the slag, the origin of this relationship is of interest. The relationship does not follow equilibrium, nor is there likely to be a simple kinetic cause. The relationship in fact closely follows that required by the stoichiometric M$_3$O$_5$ phase, the major component of solidified slag.
1. Background

The purpose of ilmenite smelting is to yield an upgraded titania slag product, by partial reduction of FeO from ilmenite (FeO.TiO$_2$) with carbon as reductant.$^1$ The upgraded slag, with a typical "equivalent" TiO$_2$ content of 85% by mass or greater, is then used in pigment production (by the chloride or sulphate routes).$^2$

In parallel with reduction of FeO to Fe, some reduction of TiO$_2$ to Ti$_2$O$_3$ also occurs; hence the two main reactions in ilmenite smelting are:

$$\text{FeO} + C = \text{Fe} + \text{CO} \quad (1a)$$
$$2\text{TiO}_2 + C = \text{Ti}_2\text{O}_3 + \text{CO} \quad (1b)$$

The partial reduction of TiO$_2$ (reaction 1b) does not contribute to upgrading of the slag, but consumes electrical energy and carbon. The formation of Ti$_2$O$_3$ does have the beneficial effect of lowering the liquidus temperature of the slag, as indicated by Figure 1.$^3$ However, slags with low FeO and high Ti$_2$O$_3$ are sometimes associated with foaming incidents, perhaps related to a change in the primary phase of the slags from TiO$_2$ (for higher-FeO slags) to M$_2$O$_5$ (for lower-FeO slags).$^4$ (In the Ti-Fe-O system, M$_2$O$_5$ is a solid solution between the end members Ti$_3$O$_5$ and FeTi$_2$O$_5$.) In addition, chloride pigment producers sometimes place a maximum limit on the allowable amount of Ti$_2$O$_3$ in the slag,$^6$ presumably because of the strongly exothermic nature of oxidation of Ti$^{3+}$ to Ti$^{4+}$, which occurs during chlorination.

For these reasons, it is of importance to understand the factors which control the extent of Ti$_2$O$_3$ formation during ilmenite smelting. Previous work indicated that it is not possible to control the relative extents of reactions (1a) and (1b) by manipulating energy and reductant inputs separately.$^3$ In the work presented here, literature data on ilmenite smelter slag compositions were analysed to determine the changes in FeO and Ti$_2$O$_3$ contents in slags after reduction to different degrees; the compositional data were also used to test possible mechanisms which can determine the FeO-Ti$_2$O$_3$ relationship. These mechanisms include reaction equilibrium, kinetic effects, and phase chemistry.

2. The FeO-Ti$_2$O$_3$ relationship in industrial slags

Figure 2 summarises the changes in levels of FeO, Ti$_2$O$_3$ and impurity oxides$^4$ as ilmenite slag is upgraded (by reduction) to different equivalent TiO$_2$ contents. The slag compositions quoted here were taken from those literature data where it appeared that oxidation of the slag during and after tapping was largely avoided.$^6,7,8,9,10$ (Oxidation of the slag converts some of the Ti$_2$O$_3$ in the slag to TiO$_2$, so distorting the FeO-Ti$_2$O$_3$ relationship which had prevailed in the furnace.) Data could be found for two commercial operations in South Africa (at Richards Bay Minerals, which uses six-in-line alternating-current furnaces, and Namakwa Sands, which uses a direct-current hollow-electrode furnace), the Iscor pilot plant operation in South Africa (similar in design to the Namakwa Sands furnace, but smaller by a factor of about 10), and the QIT plant in Canada (which uses similar furnaces to Richards Bay Minerals). The

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* The "equivalent" TiO$_2$ content is the total amount of titanium present as TiO$_2$ and Ti$_2$O$_3$, expressed as an equivalent amount of TiO$_2$.

† Please note that Figure 1 is the corrected version of the diagram presented in previous work,$^3$ which was subsequently found to contain some inaccuracies.

‡ The "impurity oxides" are here defined to be all oxides other than FeO and TiO$_x$, including mainly SiO$_2$, MnO, CaO, MgO, Al$_2$O$_3$, Cr$_2$O$_3$ and V$_2$O$_3$. 
main difference between the South African and Canadian ilmenites is the substantially higher alkali earth content of the latter. The slag composition data are summarised in Table 1. The Ti$_2$O$_3$ content of the slags was stated in the references cited, except for reference 9, where the Ti$_2$O$_3$ content was estimated from the equivalent TiO$_2$ content and a mass balance (assuming the correct Ti$_2$O$_3$ level to be where the slag composition adds up to 100%, with the assumed valencies of impurity oxides as stated below in section 3.3).

The strong increase in Ti$_2$O$_3$ content with increased equivalent TiO$_2$ content (i.e. decreased FeO content) is evident from Figure 2, as is the relatively constant level of impurity oxides (which is as expected, since these oxides are not reduced to the metal bath to any significant extent). Remarkably, the results for the South African furnaces seem to follow a single correlation, despite the very different designs and sizes of the furnaces involved. The solid lines in Figure 2 a) and b) indicate the calculated equilibrium behaviour (as discussed below) for pure TiO$_x$-FeO slags (with zero impurity levels). The striking correspondence between the different operations suggests that some general mechanism fixes the relative degrees of FeO reduction and TiO$_2$ reduction; possible mechanisms are considered below.

3. Possible origins of the FeO-Ti$_2$O$_3$ relationship

3.1 Equilibrium

Equilibrium between the reduced and unreduced forms of iron and titanium in the smelter is expressed by the following reaction:

\[ \text{FeO} + \text{Ti}_2\text{O}_3 = \text{Fe} + 2\text{TiO}_2 \]  

To calculate the slag compositions in equilibrium with metallic iron (assumed to have an activity of 1.0), the parameters for the quasichemical models for the FeO-TiO$_2$ and TiO$_2$-TiO$_{1.5}$ binaries as assessed by Eriksson and Pelton were used, using the procedure proposed by those authors for estimating the activities in the ternary FeO-TiO$_2$-TiO$_{1.5}$ mixture.

The calculated activities in the liquid slag are not far from ideal, as shown by the summary given in Figure 3 (which depicts the calculated activities at 1650°C). The equilibrium relationship between FeO and Ti$_2$O$_3$ which is predicted with these activity data is shown as Figure 4. Clearly, the Ti$_2$O$_3$ content of the slag is higher and the FeO content lower than these would be at equilibrium; reaction (2) is out of equilibrium to the right.

3.2 Kinetic effects

It would seem reasonable to invoke kinetic effects for the departure of reaction (2) from equilibrium – for example, it may be proposed that the reduction of FeO to Fe (reaction [1a]) is more efficient (“faster”) than the reduction of TiO$_2$ to Ti$_2$O$_3$ (reaction [1b]). Given the lack of any kinetic data, it is not possible to test this possibility rigorously. However, some observations serve to cast some doubt on a dominant kinetic effect. Firstly, the FeO-Ti$_2$O$_3$ relationship is remarkably consistent between furnaces of very different sizes and electrical design (see for example Figure 4), despite the very different degree of stirring (mass transfer) in these furnaces. Secondly, given the much higher TiO$_2$ concentration in the slag and the likelihood of slag mass transfer control effects, one would expect TiO$_2$ to be reduced preferentially, followed by reduction of FeO; this is the opposite of the real effect. Lastly, the slags produced from the Canadian ilmenites show a consistently lower Ti$_2$O$_3$ content at a given FeO content (Figure 2) – a difficult effect to explain on the basis of kinetics, given that
the only major difference between the two Canadian and South African ilmenites is the level of alkali earth oxides.

It is hence concluded that – while absence of kinetic data rule out any direct assessment – it appears unlikely that kinetic effects are the primary origin of the observed FeO-Ti₂O₃ relationship.

3.3 Phase chemistry

As observed by others,⁶,⁹,¹² the solidified titania slag consists largely of a single phase – the M₃O₅ solid solution – together with much smaller amounts of rutile, metallic iron, and a silicate glass phase. If this M₃O₅ solid solution is simply a mixture of the stoichiometric end members Ti₃O₅ and FeTi₂O₅, a simple linear relationship between the amounts of FeO and Ti₂O₃ in the solidified slag is expected. This predicted relationship is shown in Figure 5, together with the actual trend. Clearly, the real slag compositions lie well below the composition line of Ti₃O₅–FeTi₂O₅ mixtures.

However, this difference is apparently not due to a departure from stoichiometry in the M₃O₅ phase, but is caused by the impurity oxides which form a significant component of the slag (see Figure 2). Of the impurity oxides, nearly all the SiO₂ and CaO are contained in the glass phase, the amount of Al₂O₃ in the glass phase is about a third (by mass) of the amount of SiO₂, and the other impurity oxides are accommodated in the M₃O₅ solid solution.⁹ Specifically, the divalent impurities (Mg²⁺ and Mn²⁺) substitute for Fe²⁺, and the trivalent impurities (Al³⁺, Cr³⁺ and V³⁺) substitute for Ti³⁺. The higher content of both divalent and trivalent ions in the slag (relative to the tetravalent Ti⁴⁺) than given by only Fe²⁺ and Ti³⁺ implies that the slag composition in fact lies closer to the stoichiometric M₃O₅ line.

To quantify the effects of the impurity oxides, the following procedure was followed:

Given that MnO and MgO are taken to be equivalent to FeO on a molar basis,⁸ the equivalent FeO content is calculated as:

\[(\% \text{FeO})_{eq} = (\% \text{FeO}) + (M_{\text{FeO}}/M_{\text{MgO}})(\% \text{MgO}) + (M_{\text{FeO}}/M_{\text{MnO}})(\% \text{MnO})\]  

where \(M_i\) is the molar mass of oxide \(i\), and the amounts of the oxides are in mass percentages.

Similarly, the equivalent Ti₂O₃ content is calculated as:

\[(\% \text{Ti}_2\text{O}_3)_{eq} = (\% \text{Ti}_2\text{O}_3) + (M_{\text{Ti}_2\text{O}_3}/M_{\text{V}_2\text{O}_5})(\% \text{V}_2\text{O}_5) + (M_{\text{Ti}_2\text{O}_3}/M_{\text{Cr}_2\text{O}_3})(\% \text{Cr}_2\text{O}_3) + (M_{\text{Ti}_2\text{O}_3}/M_{\text{Al}_2\text{O}_3})[\% \text{Al}_2\text{O}_3] - (\% \text{SiO}_2)/3\]  

In expression (3b), the vanadium content of slag is expressed as V₂O₅ since this is the convention for the analyses, although the vanadium is expected to be present in the trivalent form.¹³ As the expression shows, part of the Al₂O₃ is not taken into account when the equivalent Ti₂O₃ content is calculated, because some Al₂O₃ (a mass taken to be one-third of that of the silica) reports to the separate glass phase.

The sum of \((\% \text{FeO})_{eq}\), \((\% \text{Ti}_2\text{O}_3)_{eq}\) and \((\% \text{TiO}_2)\) is then normalised to 100%, where \((\% \text{TiO}_2)\) is the Ti⁴⁺ content of the slag, expressed as a mass of TiO₂.

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⁸ CaO and SiO₂ are not taken into account in this calculation since these oxides are assumed to report fully to the separate glass phase.
The result of this normalisation procedure is given in Figure 6, which demonstrates that – when the effect of the impurity oxides is taken into account – the slag composition closely follows that expected for stoichiometric $M_3O_5$. This is the case for slags produced from both the lower-impurity (South African) and higher-impurity (Canadian) ilmenites. The former slags generally lie slightly below the stoichiometric line, probably reflecting a slight degree of oxidation of these slags (which is not unexpected, since these slags contain a higher amount of $Ti_2O_3$ than those produced from the Canadian ilmenite – see Figure 2).

It is worth noting that simply normalising the FeO, $Ti_2O_3$ and TiO$_2$ content of the slag to a total of 100% (i.e. neglecting any effect of the impurity oxides other than dilution) does not improve agreement between the slag composition and $M_3O_5$ stoichiometry or equilibrium, as shown by Figure 7.

4. Conclusion

The FeO-$Ti_2O_3$ relationship in ilmenite smelter slags appears to follow a single pattern for different smelter furnace sizes and designs, and is apparently only altered by the impurity content of the ilmenite feed. Of the possible origins of this consistent relationship, equilibrium does not hold, and kinetic effects are not expected to play the dominant role. Rather, it appears that the amount of $Ti_2O_3$ for a given amount of FeO is fixed by the tendency of the slag to solidify as essentially a single phase ($M_3O_5$). The deterministic nature of this relationship implies that, given the degree of reduction (determined by the amount of reductant fed to the furnace), and the impurity content of the ilmenite and reductant, it should be possible to predict the tap composition (%FeO, %$Ti_2O_3$ and %TiO$_2^{equiv}$) of the smelter slag.

References


Table 1. Slag compositions from the literature, as used to analyse the FeO-Ti$_2$O$_3$ relationships. Compositions are in mass percentages; "TiO$_2$$_{\text{equiv}}$" gives the total Ti$^{4+}$ and Ti$^{3+}$ of the slag, expressed as an equivalent amount of TiO$_2$.

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Plant: RBM - Richards Bay Minerals; NS - Namakwa Sands; IP - Iscor pilot plant
Figure 1. Conjectural liquidus diagram of ilmenite smelter slags, based on the TiO$_2$-TiO$_{1.5}$ and TiO$_2$-FeO binaries,\textsuperscript{11} 1500°C and 1600°C isotherms,\textsuperscript{8} and quasichemical model parameters for the liquid slag region, to calculate the slag composition in equilibrium with iron.\textsuperscript{5,11} Compositions are plotted as mole fractions, and temperatures are in °C. Corrected form of diagram presented earlier.\textsuperscript{3} Data points show compositions of slags produced with South African ilmenites, with the FeO+TiO$_2$+Ti$_2$O$_3$ content normalised to 100% (see compositions in Table 1). Symbols: ● Iscor pilot plant,\textsuperscript{6,10} Δ Richards Bay Minerals,\textsuperscript{7} □ Namakwa Sands,\textsuperscript{8} ○ QIT.\textsuperscript{7,8,9}
Figure 2. Changes in FeO, Ti$_2$O$_3$ and impurity oxide content with increased equivalent total TiO$_2$ content, for slag produced from Canadian ilmenite (open circles) and South African ilmenite (others) (see Figure 1 for references). Solid lines in a) and b) given calculated relationship for pure FeO-Ti$_2$O$_3$-TiO$_2$ slag in equilibrium with pure liquid iron at 1650°C.
Figure 3. Calculated activities in pure liquid FeO-TiO$_2$-TiO$_{1.5}$ slag at 1650°C. Reference states are pure solid TiO$_2$ and TiO$_{1.5}$, and pure liquid FeO. Compositions plotted as mole fractions of TiO$_2$, TiO$_{1.5}$ and FeO. Calculated using literature values of the quasichemical model parameters.$^5,11$

Figure 4. Calculated relationship between FeO and Ti$_2$O$_3$ (mass percentages) in pure FeO-TiO$_2$-TiO$_{1.5}$ slags in equilibrium with pure liquid iron (lines) and liquid iron containing 2% C under an atmosphere with $p_{CO}=1$ atm (open circles), compared with the real FeO-Ti$_2$O$_3$ trend. Activities calculated as in Figure 3, and symbols as in Figure 1.
Figure 5. Comparison of the actual FeO-Ti$_2$O$_3$ trend in slags produced from Canadian ilmenite (open circles) and South African ilmenite (others) (symbols as in Figure 1) with the expected relationship in a solid solution ("M$_3$O$_5$") between stoichiometric Ti$_3$O$_5$ and FeTi$_2$O$_5$, and for equilibrium with liquid iron.

Figure 6. Comparison of the actual trend of "equivalent FeO" (including substitution of FeO by MgO and MnO) with "equivalent Ti$_2$O$_3$" (including substitution of Ti$_2$O$_3$ by Al$_2$O$_3$, Cr$_2$O$_3$ and V$_2$O$_3$) for slags produced from Canadian ilmenite (open circles) and South African ilmenite (others) (symbols as in Figure 1) with the expected relationship for stoichiometric M$_3$O$_5$ (broken line).
Figure 7. Data of Figure 5, but with the total of FeO, Ti$_2$O$_3$ and TiO$_2$ normalised to 100% (i.e. neglecting the effect of impurity oxides).