

Physico-Chemical Properties of Titaniferous Slags

Published in the Journal, June, 1969

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Discussion

H. J. S. Kriek*: During 1956 Iscor became interested in the acid smelting in a blast furnace of siliceous ores containing 15 per cent silica. This interest was as a result of the successful acid smelting in Germany at a CaO/SiO₂-ratio of between 0.4 and 0.8. Acid smelting can become an economic proposition due to a reduction in slag volume accompanied by a lowering of the coke rate. Paschke and Hannel¹ found that a free flowing slag (i.e. viscosities of less than two poise) would not be obtained at C/S-ratios lower than 0.82 and that the addition of 3 to 4 per cent alkalis became necessary. In titanium bearing slags it was established that maximum fluidity is obtained with 10 per cent TiO₂. Acid smelting resulted in increased sulphur pickup with decreasing O/S-ratio and desulphurization with soda-ash outside the blast furnace was necessary.

At Iscor Mr B. B. Segal established that, for slags with (CaO+MgO)/SiO₂-ratios of 0.5, 0.6 and 0.7, the addition of alkalis and TiO₂ resulted in a substantial decrease in melting point and also of viscosity. At a (CaO+MgO)/ratio of 0.53 the addition of 10 per cent TiO₂ is as effective as the addition of 3.75 per cent alkalis. This means that a blast furnace can be run without the addition of alkalis and consequently the detrimental effect of alkalis on the lining can be avoided; also that fluid slags with 5 to 15 per cent MgO and 8 to 10 per cent Al₂O₃ can be obtained with 10 per cent TiO present. (CaO+MgO)/SiO₂-ratios smaller than 0.5 the addition of titania resulted in a decrease in melting point and also in an increase in viscosity. In ceramic crucibles the slag did not become more viscous with time but thickened appreciably when held in graphite crucibles. No titanium carbonitride could be detected and only Ti₂O₃ was found. Prof Howat also did not obtain the TiC-TiN solid solution and therefore it appears that the thickening of the slag is not solely due to the formation of the titanium carbonitrides. This is in agreement with the work of Michailov and Belyakova² who found that TiO₂ decreased the viscosity of acid slags and increased the viscosity of basic slags; that titanium sesquioxide (Ti₂O₃) and titanium monoxide (TiO) increased the viscosity of both acid and basic slags and that titanium carbide, which is found in larger amounts in basic slags than in acid slags, considerably increases the viscosity of both acid and basic slags. Patzak *et al*³ describes the following oxygen deficient titanias, viz Ti₂O, TiO, Ti₂O₃, Ti₃O₅, Ti₄O₇, Ti₅O₉, Ti₆O₁₁—Ti₁₀O₉, Ti₇O₁₃, Ti₈O₁₅ and Ti₉O₁₇.

Until about 1959 a fair amount of data was available on the viscosity of normal slags to which titania was added^{2, 4, 5, 6, 7, 8, 9}. At Iscor an electromagnetic viscosimeter based on the viscometer developed by Bockris¹⁰ was built. Progress was very slow as our main attention was centred around problems associated with the operation

of the Rotor process. During 1962 and 1963 two papers containing fundamental work on the viscosities of slags in the systems CaO-SiO₂-TiO₂¹¹ and CaO-SiO₂-TiO₂ at 0 per cent Al₂O₃, 10 per cent Al₂O₃ and 20 per cent Al₂O₃¹² appeared. The work of Ross and Ohno obviated the necessity of any further work by Iscor on the viscosity of blast furnace slags containing titania. Ross gave the following optimum compositions for blast furnace slags containing titania.

Titania %	Lime %	Silica %	Lime/Silica-ratio
20	30—42	38—50	0.6—1.10
30	27—36	34—43	0.63—1.06
40	24—32	28—36	0.66—1.14

The optimum temperature is 1,500°C because at this temperature the largest workable composition range exists. Higher temperatures result in narrower optimum composition ranges and in increased rates of thickening. The factors affecting the rate of thickening were also investigated by Ross and Ohno, as the main problem with slags containing titania is not finding compositions which are fluid at operating temperatures but the thickening due to a lack of oxygen. With these slags when there is insufficient oxygen to co-ordinate each silicon atom with its own individual shell of four oxygen ions, some sharing of oxygen ions must occur, resulting in the formation of large silicate ions and a rise in viscosity.

The formation of oxygen deficient titania can be prevented as long as iron oxide or manganese oxide remain in the liquid slag. Iwase¹³ and also Viens, Campbell and Rogers¹⁴ obtained in the experimental smelting of ilmenite ores fluid slags with more than 3 to 5 per cent manganese oxide and ferrous oxide in the slags.

It appears that titaniferous ores can be smelted in blast furnaces under the following conditions:

- (1) Acid slag operation, i.e. at C/S-ratios from 0.6-1.0.
- (2) Dolomite can be used as a flux as MgO is a network modifier.
- (3) The Al₂O₃-content must be less than 15 per cent as alumina promotes thickening of the slag and decreases the optimum composition range.
- (4) The hearth temperature must not exceed 1,500°C. According to Ross a definite amount of energy is required for each ton of iron produced and in order to increase production an increase in heat

output must be achieved without increasing the hearth temperature. The hearth temperature can be reduced, without reducing the production rate, by injecting steam, oil or natural gas into the blast furnace at the tuyeres.

- (5) Nitrogen does not increase the thickening rate of acid slags, and only slightly increases the thickening rate of basic slags.
- (6) The TiO_2 -content in the slag must be between 20 per cent and 40 per cent.
- (7) By increasing the ferrous oxide and manganese oxide in the slag it is expected that the optimum range of titanium dioxide content can be further increased,
- (8) As thickening of the slag depends on time in contact with coke shorter tap-to-tap times are advocated.
- (9) Due to the acid nature of the titania slags desulphurization will be outside the blast furnace.

Ross¹⁵ in a further paper showed that high titanium ores are amenable to modern beneficiation and blast furnace techniques. From the literature^{15, 9, 2, 16} it appears that titaniferous ores have been successfully smelted in Russian blast furnaces for the past 30 years.

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H. J. Bovey: I think it may be of interest that at the Anglo American Research Laboratory we have attempted to apply some of the findings of the authors of the paper presented this afternoon.

Our aim was the recovery of rutile from an actual titania slag. This slag was quite similar in composition to the synthetic slag used by the authors in their investigation. The proposed rutile recovery method was based

on the remark in the paper that by increasing the silica content of the slag to between 43 and 48 per cent, it might be possible to cause considerable changes in the phase relationships. Thus, by controlled cooling of the silica modified slag, titanium dioxide could be made to crystallize from the molten mass.

The scouting tests we carried out to confirm this proposition were conducted on a scale of 750 grams. The required addition of silica was made to the ground slag to give a total SiO_2 content of 45 to 50 per cent. The mixture was placed in a fireclay pot, melted at $1,400^\circ C$ in a Globar furnace and then allowed to cool slowly in the furnace by turning off the power.

We were able to obtain the needle-shaped rutile crystals only when the melt was cooled extremely slowly, i.e. at about $1^\circ C-5^\circ C$ per minute. The crystals were less than 0.5 mm long and it was not found possible to achieve any concentration of rutile on a superpanner, even after grinding the cooled melt to minus 200 mesh.

These results were disappointing, despite the realisation that our tests were of a very preliminary nature. Because of this the control of cooling rates was probably not as precise as it might have been.

In addition to the factor of cooling rate control, it appears possible that minor components of the actual slag, such as iron oxide for example, might have caused an appreciable difference in crystallization behaviour from the synthetic slag used in the author's investigations.

I feel that the next logical step would be to examine an actual slag sample using the high temperature microscopy technique described in this afternoon's paper. This should be helpful in deciding under what cooling rate conditions the rutile crystallization technique could be made to work on an actual slag. Then, further large scale tests could be conducted using the appropriate cooling rate conditions and attempts made to recover a rutile concentrate.

Other methods were also tried in an effort to separate a titanium-rich compound from an actual slag. The approach involved attempting to selectively remove titanium, leaving the gangue constituents in the slag, or the reverse, of removing the gangue constituents and leaving a titanium concentrate.

In the former category, that is of selective attack on the titanium, were high temperature chlorination and concentrated acid leaching. Neither method was attractive because of the substantial attack on gangue materials such as calcium and magnesium. This resulted in high reagent consumptions.

Attempts at selectively dissolving the gangue by dilute acid leaching were unsuccessful because the titanium values were found to be fairly soluble even in the dilute acids. A substantial quantity of silica was also observed to dissolve.

S. Selmer Olsen: The authors say that—'Until very recently, it was feared that the high titania content of these titaniferous magnetites would militate against their value as useful iron ores'. Doubts existed that these vast deposits of this special type of ore could be smelted in conventional blast furnaces.

If we ask ourselves the reason for this fear, the answer is—lack of knowledge!

We must therefore appreciate that the work presented today has been carried out because it throws a bit more light into the darkness of smelting metallurgy.

Every metallurgist who battles with drainage of difficult slags whether too dry, too sticky or too cold, or whatever we call it, would be very appreciative of diagrams of the particular type given in the paper. In most cases, however, these are not available, or, if available are not sufficiently detailed to be of full value.

The reason for this is very understandable. If we have a look at Figs. 2 and 3, we find that there are all together 63 points. Each point was determined four times totalling 252 determinations. If we assume two determinations a day there is at least half a year's work in just plotting those curves. In addition to this there was the determination of the different slag phases. Let us assume all together about one year's work. These diagrams cover only variations in CaO and MgO, the ratio $\text{SiO}_2:\text{Al}_2\text{O}_3:\text{TiO}_2$ is being kept constant. By varying the quartz, dolomite and limestone fluxes different $\text{SiO}_2:\text{Al}_2\text{O}_3:\text{TiO}_2$ ratios will come into the picture. One can imagine what a long time it would take to get a complete set of such diagrams.

For most slag diagrams published, atmospheric conditions are taken for granted. To me, the change in liquidus temperature as a function of time under reducing conditions is surprising. In Fig. 6 curve No. 2 appears to flatten out after 400 to 500 minutes but there is no indication of flattening in curve No. 1. If reducing conditions were applied to Figs. 2 and 3 one can only imagine how many years it would take to get this complete set of curves.

Where the slag viscosities are concerned, measurement by a commercial viscometer was not considered sufficiently accurate and special equipment had to be built to give results of sufficient accuracy.

All this work takes time and costs money.

A natural question in this connection would be—'But why was work of this type not started earlier particularly in relation to submerged arc smelting generally?' One explanation might be the atmosphere of secrecy which still obtains in the different smelting processes. In most countries the scientists have not even been wanted inside the smelting works because of fear that some or other little secret should reach competitors. Under such conditions it is understandable that an interest for research into the smelting processes could not be created at the scientific centres.

I would therefore like to compliment Highveld Steel and Vanadium Corporation for sponsoring this research project, and also for being broadminded enough to allow publication of the results. Most interesting to read is the 'Discussion of the phase equilibria and the viscosity values' which I take it has been written more for creating useful thinking about what is actually happening in a furnace, than from a few diagrams to try to explain the smelting process.

The authors state that—'Due to turbulence both in the furnace and on tapping, small droplets of metal will occur in the slag'. The loss of metal as droplets in the slag is naturally irritating but there are reasons to believe that they can be formed for reasons other than those due to turbulence.

Some time ago we had to dig out a silico-manganese furnace where the slag just underneath the electrodes had solidified. I found this slag full of metal droplets, or rather what immediately appeared to be metal droplets. By removing these from the slag and having a look at them under a microscope I found to my surprise that what looked like metal droplets, collected in the upper part of the slag, actually consisted of slag, pieces of ore, quartz

and coke all held together by a thin film which I assumed was SiC. The droplets from further down in the slag consisted of slag and metal and small remainders of the coke. The ore and quartz, however, were not there. I would assume that metal was formed inside the droplets and as they became heavier they sank down to the bottom, and when reaching the metal bath separated into metal, final slag and most probably graphitized carbon. It is not impossible that, in any case, part of the reactions in an electric furnace take place inside droplets formed from mixture falling into a sort of primary slag underneath the electrodes. It is also possible that when tapping the furnace some of these droplets are still in the slag. Due to the slag cooling and becoming more viscous these droplets never get a chance to separate. Where graphite dust coming out of the taphole is concerned, I suppose many of us have many a time been surprised by the quantities.

If, in any case, parts of the reactions take place inside droplets in some sort of a primary slag, droplets in the slag tapped could be a natural result of the process itself and might not have anything to do with turbulence.

The authors further say—'The viscosity of the slag will affect the electric arc furnace smelting process in a number of ways. From a chemical viewpoint the most important effect is that, although the distribution of solute in the system is governed by Nernst's law, the kinetics of the overall reaction are governed by the rate of volume diffusion which, in turn, may be related to fluidity'. Allow me to add to this—'May be not!'—(related to fluidity).

The reason why I have mentioned this is to indicate the darkness which still exists, where the understanding of the processes is concerned, and also to make clear, that more than the diagrams presented is required, in order to get a full understanding of many of our smelting processes.

I am very pleased to know that work of this nature is being carried on in the Metallurgy Department at Witwatersrand University by the Pyrometallurgical Research Group sponsored by the National Institute for Metallurgy and I can assure you that we are looking forward to presentation of more papers of the same high standard as today.

Dr Raper: Mr Chairman and gentlemen, I must first of all congratulate Professor Howat on his very fine paper and his very fine presentation. I am very sorry that Mr Hall, our Metallurgical Manager was not with us this afternoon. I do not know what happened to him. He is probably lost between Witbank and Johannesburg, but I am sure he would have been most interested to hear your paper, Professor Howat.

Your Secretary thought it would be interesting if I showed you a few slides illustrating the operation at Witbank. However before I show you the slides which incidentally cover the iron and steelmaking operation and exclude the mill, I thought it would be of interest to give you a brief description of the plant.

I also know that many of you will be visiting Witbank on Friday to tour the works on the visit arranged by the Mining and Metallurgical Society and those people might also be interested in a brief description beforehand.

THE IRON PLANT

The Iron Plant consists essentially of three operations, these are:

1. Receipt, storage and blending of raw materials.

2. Four rotary pre-reduction kilns.
3. Four 30 Mva electric smelting furnaces.

The raw materials used are a titaniferous magnetite ore which is mined at Mapochs and contains an average of 55.5 per cent iron, 13 per cent titanium oxide and 1.6 per cent V_2O_5 . The ore is crushed, washed and screened at Mapochs to -2 in. $+\frac{1}{8}$ in. and transported by rail to Witbank. The ore is received from the bottom dump trucks in a bunker from where it is transferred by belt conveyor to a covered storage yard of 20,000 tons capacity. Coal is used as the reductant in the iron-making process. No. 5 seam coal from Apex mines and Greenside Colliery is used in a size range of $-1\frac{1}{2}$ in. $+\frac{1}{8}$ in. The coal is transported to site either by rail, in which case it is then handled by tippler and placed in storage or alternatively road transport is used.

Part of the fluxing material is provided by dolomite which originates from Littleton Colliery. Additionally, small quantities of silica mined locally are used for slag control.

All these raw materials are transferred to day bunkers and from these bunkers a carefully proportioned feed is achieved by the use of belt weigh feeders, this is fed to each kiln. The burden comprises ore, 2,000 lb, coal 880 lb, dolomite 440 lb and silica 65 lb. This burden composition is designed to produce a slag metal ratio of 0.78 to 1, with a slag basicity of 1.7 and a titania content of 31 per cent in the slag.

KILNS

The four rotary kilns are co-current fired and are 200 ft long and 13 ft ih diameter. In the reaction zone temperatures are approximately $1,050^\circ\text{C}$. The kilns are fired with pulverized coal and a suitable temperature profile is maintained along the length of the kiln by introducing air through air pipes, spaced at regular intervals along the shell of the kiln. This air is used to partially burn the volatiles which are evolved in the first stages of the kiln operation. During the passage of the charge through the kiln, coal carbonization is more or less completed as is calcination of the dolomite. In addition, some 35 per cent of the oxygen associated with the iron in the iron ore is removed.

ELKEM SMELTING FURNACES

Four 30 Mva Elkem submerged arc furnaces having a rated capacity of approximately 40,000 tons pig of iron per month are installed. The pre-reduced hot charge from the kilns is transferred by skip to one of six hot charge hoppers feeding each furnace, from where the furnace is gravity fed.

Molten pig iron is tapped into hot metal transfer ladles and transported by rail to the Steel Plant. Slag is tapped into 30 ton pots and disposed by rail to the slag dump.

The hot metal analysis is 3.9 per cent C, 1.2 per cent V, .25 per cent Si, .25 per cent Ti, .08 per cent P, .06 per cent S and 0.2 per cent Mn.

STEELMAKING

After arrival in the Steel Plant it was intended that the hot metal be transferred to one of two 800 ton mixers which would serve as buffer storage between iron and steel plant production. Shortly after these units were commissioned however it was discovered that the iron tended to be unstable and high melting point titanium complexes were precipitated during the time the metal was in the mixer. This precipitation eventually resulted

in the mixer being taken off line and presently hot metal received from the iron plant is charged directly into one of four nominal 60 ton shaking ladles.

SHAKING LADLES

After charging, the shaking ladle is transported to one of two shaking ladle emplacements to allow the removal of vanadium from the metal as a vanadium pentoxide slag. A water-cooled gas off take hood is swung over the ladle. A water-cooled lance is lowered to within 30 in. of the bath and oxygen is blown at a rate of 1,000 c.f.m. The quantity of oxygen blown is determined by charge weight computation which evaluates the oxygen required to remove the vanadium from the bath. After the blowing period during which time various additives are added from overhead bins via a chute to the ladle, the metal contains approximately 3.2 per cent C and 0.09 per cent V. Floating on top of the metal is the vanadium slag which contains approximately 25 per cent V_2O_5 . The shaking ladle containing the hot metal and slag in total weighing 200 tons is then transported to the B.O.F. where the metal is under poured by means of a teapot spout from the ladle to the B.O.F. The vanadium slag is retained in the shaking ladle and subsequently dumped in a cooling compound.

BASIC OXYGEN FURNACE STEELMAKING

Two 50/60 ton B.O.F.'s are installed, one of which is operational whilst the second is being relined. Tar bonded dolomite bricks or magnesite blocks are used as lining material. Refining is carried out by means of oxygen blowing through a triple holed water-cooled lance. Once again oxygen requirements are computed from the carbon removal required and again fluxes are added to the furnace during the blow this time to provide the correct slag condition for sulphur and phosphorus removal. At Highveld, steels are normally produced for structural purposes and this necessitates a turn down carbon of 14 points with a turn down temperature of $1,660^\circ\text{C}$. The high turn down temperature is due to the subsequent C.C. process which demands a higher degree of metal super heat than conventional casting processes.

Alloying elements are added to the ladle in the form of FeMn and FeSi, all steel being fully silicon killed. The ladles are then transported by electric transfer car to the C.C. plant.

CONTINUOUS CASTING PLANT

Three C.C. machines are provided, two twin strand heavy slab machines and one 4 strand billet machine.

In all cases the machines are of the bow type however, straight not curved moulds are used on the billet machines. The steel is poured to the moulds via a tundish which is equipped with nozzles and stopper roads.

In the mould low melting point powder is used as lubricant together with small quantities of rape seed oil. After primary solidification in the mould the block is withdrawn through the secondary solidification zone, which consists of guide rollers and water cooling sprays.

After solidification, the blocks are straightened in a combined withdrawal and straightening roll system which comprises four pairs of rolls. After straightening, the blocks are automatically cut to pre-determined lengths by means of oxy-propane flames before transfer to cooling banks.

When cooled the blocks are loaded by means of a magnet crane from the banks to road vehicles for transfer to the mill.

Author's Reply

The authors would like to thank the contributors for their very valuable and informative discussions to the paper. It is quite apparent that ISCOR has done considerable work in relation to the question of titaniferous slags in the iron-smelting process. The interesting point is that several of Dr Kriek's comments so closely agree with those of the authors and this is most gratifying. With regard to specific points mentioned by Dr Kriek, the authors would like to add the following:

It has been suggested by several authors^{1, 2, 3} that TiO_2 although its normal co-ordination number with oxygen is 6, can become a network former because the Ti^{4+} cation may be in four-fold co-ordination at elevated temperatures. If a part of the TiO_2 content of the slag was in four-fold co-ordination then the viscosity can be expected to increase because of the relatively larger proportion of network formation in the slag.

Although these slags containing 35-40 per cent TiO_2 exhibited no titanium carbide, nitride or carbo-nitride after subjection to reducing conditions there was evidence that the TiO_2 had become reduced to a series of lower oxides. If these oxides result in an increase of the liquidus temperature (as is shown in the paper) then the viscosity as measured under constant temperature conditions would increase with degree of reduction. This is due to the fact that the viscosity will finally be measured below the rising liquidus temperature, i.e., increased viscosity due to the presence of primary crystals.

Mr Bovey of the Anglo American Research Laboratories mentioned in his discussion that, in attempts to extract the rutile by the proposed method of adding SiO_2 to the slag to permit crystallization of TiO_2 in a glassy matrix, it was found that crystallization was slow and that the individual crystals were very small. This may be attributed to the fact that the slags used in the experiments were actual plant slags and therefore were in a highly reduced condition, i.e., the Ti was present as

a series of lower oxides. Presumably these lower oxides would first have to be re-oxidized to TiO_2 before suitable crystallization can occur. Also, because of the inherently high crystallization potential of TiO_2 , there would be a large number of nuclei, hence a large number of small crystals.

Mr Selmer Olsen of AMCOR raised a number of very interesting points for discussion. The authors believe that there must exist a primary slag in the arc zone, and that the slag and alloy which are tapped are integrated averages, with respect to time, of a series of initial and/or partial processes. This aspect is to be examined in relation to the smelting of ferro-chromium alloys.

It is generally accepted that a low viscosity will improve the kinetics of chemical reactions due to an increase in the rate of volume diffusion. However, in relation to the theory of metal droplet reactions occurring in a slag matrix, perhaps the optimum viscosity of the slag would have to be sufficiently high to permit the reaction to go to completion.

Certainly, an important fourth parameter to be considered in these physico-chemical properties of the titaniferous slags is the electrical conductivity and capacitance. The latter two would appear to be important in any submerged arc smelting process and are being measured for slags associated with the production of ferro-chromium alloys.

In conclusion, the authors would like to express their appreciation for the very kind remarks by Dr Raper and the very enlightening slide show on the Highveld plant.

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