A METALLURGICAL PROCESS FOR UPGRADING FERRO-TITANIFEROUS MINERAL CONCENTRATE USING TIME DEPENDENT MAGNETIC FIELDS

Abstract: There is provided a metallurgical process for upgrading ferro-titaniferous mineral concentrate (comprising more than 10% by weight of iron content calculated as FeO) using time dependent magnetic fields. The process comprises reducing a substantial portion of the iron content at temperature below the flow point of the titaniferous phase of the mineral concentrate in the presence of a time dependent magnetic field, wherein the time dependent magnetic field is used to at least contribute to the reduced iron content coalescing into iron spheroids and subsequently physically separating the iron spheroids.
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A metallurgical process for upgrading ferro-titaniferous mineral concentrate using time dependent magnetic fields

Field of the Invention

[1] The present invention relates generally to upgrading of ore or mineral concentrate by elimination of iron impurities and specifically, but not necessarily entirely to, beneficiating a ferro-titaniferous mineral concentrate using induced electromagnetic fields to separate out iron content from the mineral concentrate, thereby upgrading the titanium content of titaniferous mineral concentrate.

[2] The titaniferous mineral concentrate may contain significant quantities of iron content, for example, but not confined to, ilmenite, leucoxene, titano-magnetite and titaniferous iron sands.

[3] Whereas the embodiments described herein have been described primarily with reference to use of a time dependent magnetic field as the influencing energy source for separation of the ferrous and/or ferric oxide iron content of ferro-titaniferous mineral concentrate, it should be noted that the invention need not necessarily be limited to use for ferro-titaniferous mineral concentrate and may be utilised for other separating metallurgical processes.

Background of the Invention

[4] Large quantities of titanium are consumed annually in the production of titanium metal and titanium dioxide pigment. The titanium used by these industries must be extracted to exceptionally high purity.

[5] Titanium occurs in nature as the mineral rutile, which consists predominantly of titanium dioxide with low levels of impurities and as various ferro-titaniferous minerals such as ilmenite, which consist of titanium dioxide in combination with ferrous and ferric iron oxides.

[6] The titanium content of rutile can easily be extracted as either metal or as pigment grade, by what is known as the Chloride Route, which is a relatively simple process involving reaction of the rutile with chlorine gas. As rutile is low in impurities and the chlorine can be recovered and reused, this represents a relatively inexpensive one step process.

[7] The titanium content of ferro-titaniferous minerals cannot be economically recovered using the Chloride Route, mainly due to the high iron content, but can be recovered in a form suitable for pigment manufacture by a process known as the Sulphate Route.

[8] The Sulphate Route process involves 27 separate unit operations and thus has much higher operating costs than the chloride route. It also suffers from the inability to economically recover either the sulphuric acid reagent used in the titanium extraction or the high quantities of impurities,
particularly iron, which are solubilised during processing. As a result, this route also suffers from high reagent and effluent disposal costs.

[9] However, the availability of rutile is far short of the demand for titanium, particularly within the pigment industry, a situation which is now compounded by rapidly diminishing reserves.

[10] This situation has historically meant that rutile prices have been maintained at much higher levels than for ilmenite, which is the predominant ferro-titaniferous feedstock, in terms of titanium content.

[11] The titanium industry has faced a dilemma in terms of obtaining feedstock suitable for titanium metal production, which can only be produced by the Chloride Route and for obtaining feedstock suitable for high grade titanium dioxide pigment production.

[12] The titanium industry has also had to contend with the poor environmental reputation attached to the Sulphate route, due to the much greater environmental awareness attached to issues related to effluent disposal.

[13] Prior art attempts at solving this dilemma comprise reduction of the iron content of suitable relatively cheap ferro-titaniferous concentrates, particularly ilmenite, by more acceptable environmentally friendly means, to produce feedstock which may be suitable to either or both of the Chloride or Sulphate route processes, thus realising higher prices in terms of titanium content.

[14] For example, prior art reduction processes comprise the application of carbothermic iron reduction as a means of upgrading iron-bearing titaniferous mineral concentrates.

[15] Carbothermic iron reduction involves either the use of rotary kilns to achieve reaction below the fusion point of the resultant titanium product (<1600°C), referred to herein as the low temperature process, or by use of electric arc furnaces to achieve reaction at high temperature (>1600 °C), referred to herein as the high temperature process, resulting in the fusion of all reaction products.

[16] Both the high temperature process and the low temperature process suffer from economic difficulty.

[17] Specifically, the low temperature process results in the iron being present in the reaction product as finely dispersed metallic particles. These iron particles must then be oxidised and removed by acid leaching in order to produce a suitably upgraded product, resulting in high operating and residue disposal costs.

[18] The high temperature process allows for relatively simple removal of the resultant molten iron from the fused titanium product, as they form separate layers in the furnace. However, the high temperature process involves high operating costs due to the need for higher temperatures, which must be generated by the use of electric power, and high maintenance costs resulting from the severe corrosive nature of the molten titanium product ("slag") on furnace linings.
Due to the environmental difficulties associated with the low temperature process and the high electric power requirements of the high temperature process, upgrading of titaniferous mineral concentrates is currently achieved using arc furnace technology in locations where very cheap power is available.

US 2721793 A (MAGRI, Jr. et al.) 25 October 1955 (DI) discloses the beneficiation of ferrotitaniferous ores at comparatively low temperatures by reducing a substantial proportion of the iron content in the ore to elementary iron in the presence of sufficient of a boron compound to form from the elementary iron thus obtained an iron-boron composition having a flow point below the flow point of the reduced titaniferous phase of the ore, the charge being maintained during the beneficiation at a temperature between the flow point of the iron-boron composition and the flowpoint of the titaniferous phase.

The Magri process decreases the flow point or the viscosity of the elementary iron formed so that a very large part of this iron flows from the ore and coalesces into spheroids or a pool which are removed from the titaniferous phase by physical means.

US20090324440 AI (Marsh) (D2) discloses an extractive metallurgical process for the chemical reduction of an iron-containing ore or concentrate characterised in that the ore or concentrate is exposed to microwave (MW) energy and radio frequency (RF) energy concurrently with heating of the ore or concentrate, the MW and RF energy levels being selected so that there is little or no additional heating of said ore or concentrate.


CN 1775368 A (WUHAN TECHNOLOGY UNIV, et al.) 24 May 2006 (D4) discloses obtaining iron slag from titanium ore by calcining and subsequent magnetic separation.

US 3552564 A (BURGENER et al.) 05 January 1971 (D5) discloses a ferromagnetic ore concentrator (including ilmenite) in a varying magnetic field but do not disclose reduction of the ore.

CA 2302712 AI (COUNCIL SCIENTIFIC & INDUSTRIAL RESERARCH [IN]) 24 October 2001 (D6) discloses preparation of synthetic rutile from ilmenite comprising smelting metallized ilmenite by arc plasma followed by grinding, oxidizing, leaching, filtering, washing and drying titania separated from pig iron.

RU 2295582 CI 20 March 2007 (D7) discloses a method of processing titanium containing slag by subjecting to a rotating electromagnetic field, melting the slag and reducing metals in the slag.
None of the above prior art documents disclose reducing a substantial portion of iron content in a ferro-titaniferous ore in the presence of a time dependent magnetic field to produce iron spheroids.

Furthermore, none of the above prior art documents either individually or in combination obviously suggests to a person skilled in the art, an apparatus (and a process) for upgrading ferro-titaniferous mineral concentrate comprising more than 10% by weight of iron content calculated as FeO, using a time dependent magnetic field at temperatures below the flow point of the titaniferous phase of the mineral concentrate, the apparatus comprising; a time dependent magnetic field source controller for controlling the time dependent magnetic field source to control the reduction of the iron content wherein the time dependent magnetic field is used to contribute to the reduced iron content coalescing into iron spheroids.

It is to be understood that, if any prior art information is referred to herein, such reference does not constitute an admission that the information forms part of the common general knowledge in the art, in Australia or any other country.

Summary of the Disclosure

The invention specifically relates generally to the use of a time dependent magnetic field as the influencing energy source for separation of the ferrous and/or ferric oxide iron content of ferro-titaniferous mineral concentrate at temperatures below the flow point of the titaniferous phase of the mineral concentrate, such as at temperatures below 1600°C or less thereby addressing, or at least providing an alternative, to the high operating and disposal costs of acid leaching of the finely dispersed metallic particles of the above described low temperature process and the high energy costs of the above described high temperature process.

We found that the application of the time dependent magnetic field surprisingly has a substantial influence in being able to separate the components of an ore at significantly lower temperatures and significantly faster than conventional processes which may then be conveniently physically separated.

For example, when applied to separation of the ferrous and/or ferric oxide iron content of ferro-titaniferous mineral concentrate, the time dependent magnetic field contributed to the iron content coalescing into iron spheroids which could be subsequently removed by physical means.

The exact reasons for such influence of the time dependent magnetic field remains speculative at this stage but we theorise that the effects shown belong to an area of physics referred to as magnetohydrodynamics.
For example, Moffatt describes in "Electromagnetic Stirring"¹ that an alternating magnetic field (either single phase or multiphase) applied to a conductor, whether solid or fluid, will induce electric currents in the conductor, and hence a Lorentz force distribution.

Furthermore, Moffatt states that while application may be considered in the field of metallurgical processing (e.g. the major industrial process of aluminium smelting), understanding of the flows generated by electromagnetic stirring in all but the most idealized circumstances is still at a fairly primitive level.

We discovered that the application of time dependent magnetic field to the field of separating ore components confers surprising results.

We discovered the ability to expedite the usual chemical reactions and associated flow of electrons which occurs during a chemical reaction for which one theory is that the time dependant magnetic field creates forces which influence the particles (electrons/molecules etc) of the ore to react at lower temperatures as to opposed at to the usual higher temperatures.

Specifically, for separation of the ferrous and/or ferric oxide iron content of ferro-titaniferous mineral concentrate, we discovered that the time dependent magnetic field influences the iron content to coalesce into spheroids which may advantageously be subsequently removed by physical means, as opposed to acid leaching and the like.

We further discovered the need to invent a new furnace apparatus for the application of our discovery, wherein, as opposed to a conventional induction furnace where the alternating magnetic field is used for heating the ore, the new furnace apparatus has independent heat and time dependent magnetic field sources such that the heating of the ore may be controlled independently of the strength of the time dependent magnetic field such that the time dependent magnetic field may be controlled to adjust the reaction temperatures (such as that of the iron content reduction reaction) and also the physical properties of the resultant separated components (such as the sizing of the iron spheroids).

As such, with the foregoing in mind, in accordance with one aspect, there is provided a metallurgic process for upgrading ferro-titaniferous mineral concentrate using time dependent magnetic fields, the ferro-titaniferous mineral concentrate comprising more than 10% by weight of iron content calculated as FeO, the process comprising: reducing a substantial portion of the iron content at temperature below the flow point of the titaniferous phase of the mineral concentrate in the presence of a time dependent magnetic field, wherein the time dependent magnetic field is used

¹ H. K. Moffatt, "Electromagnetic Stirring" Department of Applied Mathematics and Theoretical Physics, University of Cambridge,
to at least contribute to the reduced iron content coalescing into iron spheroids; and subsequently physically separating the iron spheroids.

[42] The majority of the iron spheroids may be greater than 106 \( \mu \text{m} \) in diameter.

[43] The metallurgic process may further comprise providing a heat source; and providing a separate time dependent magnetic field source; and controlling the time dependent magnetic field source independent of the heat source.

[44] The heat source may be controlled independently of the time dependent magnetic field source to heat the ferro-titaniferous mineral concentrate.

[45] The time dependent magnetic field contributes to an insubstantial amount of heating as compared to the heat source.

[46] Controlling the time dependent magnetic field source may comprise controlling the time dependent magnetic field to control the size of the iron spheroids.

[47] Controlling the time dependent magnetic field source may comprise controlling the time dependent magnetic field to control the rate of generation of iron spheroids.

[48] The heat source may comprise at least one of an oil, gas and coal fired heat source.

[49] Physically separating the iron spheroids may comprise a crushing or grinding phase followed by at least one of sieving, air classification and magnetic separation.

[50] The temperature may be below 1600°C.

[51] The temperature may be below 1500°C.

[52] The temperature may be below 1400°C.

[53] The temperature may be between 1000°C and 1370°C.

[54] The iron content may be at least one of ferrous and ferric oxide.

[55] Providing a carbon source for reducing the iron content.

[56] The carbon source may comprise at least one of coke, charcoal and coal.

[57] The ferrous iron content of the mineral concentrate may be reduced to iron metal according to the following reaction: \( \text{FeO} + \text{C} \rightarrow \text{Fe} + \text{CO} \)

[58] The ferric iron content of the mineral concentrate may be reduced to iron metal according to the following reaction: \( \text{Fe}_2\text{O}_3 + 3\text{C} \rightarrow 2\text{Fe} + 3\text{CO} \)

[59] The carbon source may comprise carbon monoxide.

[60] The ferrous iron content of the mineral concentrate may be reduced to iron metal according to the following reaction: \( \text{FeO} + \text{CO} \rightarrow \text{Fe} + \text{CO}_2 \)

[61] The ferric iron content of the mineral concentrate may be reduced to iron metal according to the following reaction: \( \text{Fe}_2\text{O}_3 + 3\text{CO} \rightarrow 2\text{Fe} + 3\text{CO}_2 \)

[62] The ferro-titaniferous mineral concentrate may be ilmenite (FeTi03) mineral concentrate.
The temperature may be between 1050°C and 1350°C.

The concentrate may be exposed to the time dependent magnetic field for a retention time of between 2 and 60 minutes.

The metallurgic process may further comprise controlling the temperature and retention time according to a predetermined TiO₂ content.

The metallurgic process may further comprise adding magnetic additives to the ferro-titaniferous mineral concentrate.

The magnetic additives comprise at least one of magnetite and titano-magnetite.

The ferro-titaniferous mineral concentrate may be at least one of leucoxene, titanomagnetite and titaniferous iron sands.

The metallurgic process may further comprise inclusion of a chemical additive which reports to the iron product.

The chemical additive may comprise borates and phosphates.

The metallurgic process may further comprise addition of moisture to the ferro-titaniferous mineral concentrate prior the addition of the carbon source.

The metallurgic process may further comprise addition of approximately 20% moisture to the ferro-titaniferous mineral concentrate.

The metallurgic process may further comprise leaching to remove iron spheroids being smaller than 106 μm in diameter.

The leaching may use ferric sulphate.

The metallurgic process as claimed in claim 34, further comprising mechanical agitation for approximately 10 minutes

The leaching may involve the oxidation/reduction reaction Fe + 2Fe³⁺ → 3Fe²⁺.

The metallurgic process may further comprise regenerating ferric ions required for the reaction by oxidation of the resulting ferrous ions by at least one of air, oxygen, ozone or hydrogen peroxide.

According to another aspect, there is provided apparatus for upgrading ferro-titaniferous mineral concentrate comprising more than 10% by weight of iron content calculated as FeO using a time dependent magnetic field at temperatures below the flow point of the titaniferous phase of the mineral concentrate, the apparatus comprising a time dependent magnetic field source controller for controlling the time dependent magnetic field source to control the reduction of the iron content wherein the time dependent magnetic field is used to contribute to the reduced iron content coalescing into iron spheroids.
The apparatus may further comprise a heat source and wherein the heat source may be controlled independently of the time dependent magnetic field source to heat the ferro-titaniferous mineral concentrate.

Other aspects of the invention are also disclosed.

Brief Description of the Drawings

Notwithstanding any other forms which may fall within the scope of the present invention, preferred embodiments of the disclosure will now be described, by way of example only, with reference to the accompanying drawings in which:

Figure 1 shows an exemplary illustrative process circuit for enhancing the beneficiation of ferro-titaniferous mineral concentrate using time dependent magnetic fields in accordance with an embodiment of the present disclosure;

Figure 2 shows exemplary methods of application for the process wherein an AC electric current is passed through a coil to generate the time dependent magnetic field in accordance with an embodiment of the present disclosure;

Figure 3 shows an exemplary process for enhancing the beneficiation of ferro-titaniferous mineral concentrate using time dependent magnetic fields in accordance with an embodiment of the present disclosure;

Figure 4 shows the basic circuit required for the reduction process in accordance with an embodiment of the present disclosure;

Figure 5 shows the circuit of Figure 4 with the inclusion of ferric sulphate leaching into the reduction circuit in accordance with an embodiment of the present disclosure; and

Figure 5 shows the circuit of Figure 4 with the inclusion of ferric sulphate leaching into the reduction circuit in accordance with an embodiment of the present disclosure.

Description of Embodiments

For the purposes of promoting an understanding of the principles in accordance with the disclosure, reference will now be made to the embodiments illustrated in the drawings and specific language will be used to describe the same. It will nevertheless be understood that no limitation of the scope of the disclosure is thereby intended. Any alterations and further modifications of the inventive features illustrated herein, and any additional applications of the principles of the disclosure as illustrated herein, which would normally occur to one skilled in the relevant art and having possession of this disclosure, are to be considered within the scope of the disclosure.

Before the process and apparatus for beneficiating a ferro-titaniferous mineral concentrate using time dependent magnetic fields are disclosed and described, it is to be understood that this
Disclosure is not limited to the particular configurations, process steps, and materials disclosed herein
and as such may vary somewhat. It is also to be understood that the terminology employed herein is
used for the purpose of describing particular embodiments only and is not intended to be limiting
since the scope of the disclosure will be limited only by the claims and equivalents thereof.

In describing and claiming the subject matter of the disclosure, the following terminology will
be used in accordance with the definitions set out below.

It must be noted that, as used in this specification and the appended claims, the singular forms
"a," "an," and "the" include plural referents unless the context clearly dictates otherwise.

As used herein, the terms "comprising," "including," "containing," "characterised by," and
grammatical equivalents thereof are inclusive or open-ended terms that do not exclude additional,
unrecited elements or method steps.

It should be noted in the following description that like or the same reference numerals in
different embodiments denote the same or similar features.

We have discovered that the exposure of a mixture of a ferro-titaniferous mineral such as
ilmenite with a suitable quantity of carbon such as coke, coal or charcoal, to a time dependent
magnetic field produced by a suitably located external electric coil causes the iron content of the ferro-
titaniferous mineral to be rapidly reduced to iron metal at temperatures below 1400 °C and in
particular at temperatures of between 1000 and 1300 °C and that this metal can be rapidly coalesced
into spherical spheroids by controlling the induced time dependent magnetic field.

Typically, the embodiments described herein relates to the reaction between ilmenite
(FeTiO₃) mineral concentrate and a source of carbon such as coke, charcoal, coal and the like,
whereby the ferrous and ferric iron content of the mineral concentrate is reduced to iron metal according to
the reactions:

\[
\text{Ferrous Iron:} \quad \text{FeO} + \text{C} \rightarrow \text{Fe} + \text{CO} \\
\text{Ferric Iron:} \quad \text{Fe}_2\text{O}_3 + 3\text{C} \rightarrow 2\text{Fe} + 3\text{CO}
\]

but may also relate to the use of other reductives, for example carbon monoxide, in which
case the reaction proceeds according to the reactions:

\[
\text{Ferrous Iron:} \quad \text{FeO} + \text{CO} \rightarrow \text{Fe} + \text{CO}_2 \\
\text{Ferric Iron:} \quad \text{Fe}_2\text{O}_3 + 3\text{CO} \rightarrow 2\text{Fe} + 3\text{CO}_2
\]

In the above reactions, the application of the time dependent magnetic field allows the iron
particles to coalesce into metal spheroids, whilst the upgraded titanium bearing fraction remains as a
solid phase.
The process disclosed herein involves the generation of a time dependent magnetic field by application of an alternating electric current (AC) through a coil in close proximity to the charge being heated.

We have found that the sizing of the resultant metal spheroids can be controlled by application of a suitable combination of electromagnetic field strength, temperature and retention time, such that the metal spheroids can be easily removed from the reaction product by simple physical means, as opposed to chemical leaching.

In particular, using ilmenite concentrate as the feedstock, it has been found that by application of a suitable electromagnetic field, iron metal spheroids of diameter > 1 mm can be formed at temperatures of between 1050-1350°C using retention times of 2-30 minutes, such that subsequent to their removal by grinding and screening, an upgraded titanium fraction can be produced, ranging from 70-85% TiO₂.

We have also found that it is possible to control the temperature and retention time to achieve a predetermined level of iron reduction and thus produce an upgraded ilmenite with a predetermined TiO₂ content.

We have also found that any phosphorous contained in the feedstock is extracted into the iron product, leaving a phosphate-free upgraded product, being an important consideration in the potential application of the upgraded product to titanium dioxide pigment production having low tolerance for phosphorous.

We have also found the operating temperature ranges and retention times can be significantly reduced by the presence of even small amounts of highly magnetic materials such as magnetite and titano-magnetite which may result in reduced processing costs.

We have further found that the operating temperature range and retention time required can both be lowered by inclusion of various chemical additives, including borates and phosphates, both of which report to the iron product.

Whereas we did not find benefit from comminution of the titanium bearing feedstock, benefit was found in maintaining the particle size range of the carbon source above 0.5 mm.

We also found that the addition of water to the ilmenite/carbon mixture followed by thorough mixing, prior to charging to the furnace, may result both in improved consistency and improved reduction efficiency at equivalent operating temperatures.

We also found that the iron metal content of the reduced ilmenite after screening at 106 micron or the highly magnetic fraction extracted by magnetic separation after this screening step can be removed by leaching with ferric sulphate solution for 10 minutes.
We also found that the addition of titano-magnetite to the ilmenite/carbon/water mixture prior to mixing allows the operating temperature required to achieve a given level of iron reduction and UGI (Upgraded Ilmenite) quality can be considerably reduced.

Process circuit

Turning now to figure 1, there is shown an exemplary illustrative process circuit 1 for enhancing the beneficiation of ferro-titaniferous mineral concentrate using time dependent magnetic fields.

As can be seen, the circuit 1 comprises a crucible, furnace, kiln 5 or the like comprising the ferro-titaniferous mineral concentrate 4. A reductive 6, such as coke, coal, carbon monoxide or the like is added for the reduction of the iron content of the mineral concentrate 4.

The output from the crucible 5 comprises the separated product 7 and the upgraded tataniferous product 8.

As can be seen, the circuit 1 comprises a heat source 3 and a time dependent magnetic field source 9.

In embodiments, the heat source 3 and the time dependent magnetic field source 9 may be generated by the same apparatus wherein, for example, the heat and the time dependent magnetic field is provided by an induction heating coil.

However, trial and experiment discovered the need to decouple the time dependent magnetic field source from the heat source for independent control such that, for example, as alluded to above, the time dependent magnetic field may be controlled to influence the coalescence of iron content into iron spheroids.

For example, the strength and/or frequency of the time dependent magnetic field may be controlled to influence the size of the iron spheroids generated from the process.

As such, figure 1 represents a preferred embodiment wherein the circuit 1 comprises an independent heat source 3 and time dependent magnetic field source 9.

In this regard, the heat source 3 may provide heat through nonconductive means such as from conventional gas, oil or coal fired heat sources.

Furthermore, the heat source 3 and the time dependent magnetic field source 9 may comprise independent controls therefor.

As such, as can be seen, the heat source 3 may comprise a heat source controller 2 configured to control the operating temperature of the crucible 5 which is controlled independently of a time dependent magnetic field source controller 10 configured to control at least the strength and frequency of the time dependent magnetic field, the latter, as alluded to above, being controlled at
least for influencing the iron reduction and coalescing of the size of the iron spheroids or the rate of generation thereof.

[120] For example, we have found that operation of the circuit 1 can result in the production of pig iron in the form of spheroids with a minimum ball diameter of approximately 150 microns (0.15 mm) and an upgraded ilmenite product with a maximum particle size of 150 microns (0.15 mm).

Method of Application

[121] Turning now to figure 2 there is shown the exemplary methods of application for the process wherein an AC electric current is passed through a coil 12 to generate the time dependent magnetic field passing through and around the coil 12. The time dependent magnetic field can generate eddy currents within any material placed inside the coil 12.

[122] Specifically, application of an alternating current to the coil 12 induces an electromagnetic field through the coil 12, wherein the direction of the field depends on the direction of current flow.

[123] Furthermore, an alternating current passing through the coil 12 will result in a time dependent magnetic field changing in direction at the same rate as the frequency of the alternating current wherein, for example a 60Hz AC current will cause the electromagnetic field to switch directions 60 times per second.

[124] The forces and energy imparted by the electromagnetic field depend on the electrical conductivity, resistivity and magnetic susceptibility of the target material.

[125] As such, potential applications of the process 1 comprise the batch basis as substantially shown in figure 2A comprising a furnace 15 having the coil 12 therein.

[126] Within the furnace 15 may be located a crucible 13 having a graphite lining 14 accommodating the mineral concentrate 4.

[127] Alternatively, the process may be implemented on a continuous basis as substantially shown in figure 2B wherein a rotary kiln 17 is utilised. As alluded to above, the rotary kiln 17 may be heated by conventional means, such as by being oil, gas or coal fired.

[128] As can be seen, the coil 12 surrounds the kiln 17 wherein the mineral concentrate 4 is fed in at one end such that product 16 is discharged from the other end of the kiln 17. In alternative embodiments, as is also shown in figure 2B, the coil 12 can be located alongside the rotary kiln 17 as opposed to being coaxial with the kiln 17.

[129] Ideally, the crucible 13 and the rotary kiln 17 are made from non-magnetic material, such as stainless steel or aluminium.
Process
[130] Turning now to figure 3, there is shown an exemplary process 20 for enhancing the beneficiation of ferro-titaniferous mineral concentrate using time dependent magnetic fields.

[131] At step 17, ferro-titaniferous mineral concentrate 4 comprising more than 10% by weight of iron content calculated as FeO is combined with a suitable reductive and heated in the presence of the time dependent magnetic field.

[132] A substantial portion of the iron content is reduced at a temperature below the flow point of the titaniferous phase of the mineral concentrate.

[133] The application of the time dependent magnetic field at least contributes to the reduced iron content coalescing into iron spheroids.

[134] At step 18, the charge is allowed to cool wherein, at step 19, the product is crushed or ground to approximately 106 μm.

[135] At step 19, the iron content of the crushed product is screened by physical means wherein iron spheroids having sizes of greater than approximately 106 μm are separated as metal product 7.

[136] At step 20, the remaining TiO₂ solids and fine pig iron having sizing of less than 106 μm is separated by a dry roll magnet having 1,000 to 1,500 gauss field strength for recycling to the furnace such that no or little solid waste is produced by operation of the process.

[137] The remainder product remains as upgraded titaniferous product 8.

Exemplary test results
[138] Exemplary test results are provided hereunder to illustrate the performance of the process described herein.

[139] All test results shown below were conducted using an IH15 Series Laboratory Induction Heater, supplied by Across International. The heater was fitted with a 75 mm ID copper water cooled induction coil, which was designed to contain a suitably sized silica jacket. A 150 mL capacity graphite crucible was located inside the jacket.

[140] While in these examples the induction heater was both the source of the heat and the time dependent magnetic field, as alluded to above, in a preferred embodiment, separate heat and time dependent magnetic field sources are desirable for independent control of the heating of the ore and the effects of the time dependent magnetic field.

[141] However, as can be seen from the below, the coil may be utilised initially for heating the ore and then subsequently controlled to impart the effects of the time dependent magnetic field relating to the reduction of the iron content and the coalescence of such into iron spheroids.

[142] All examples below involved placing approximately 100 grams of a selected mixture of titaniferous mineral concentrate and carbon in the graphite crucible and then, by adjustment of the
current input to the coil, heating the mixture to a predetermined temperature range, as measured by a pyrometer and then holding the mixture within this temperature range for a predetermined period, as measured using a stopwatch.

Exemplary test results - Example 1

[143] In this example, 86 grams of Goondicum Ilmenite Concentrate of known analysis was mixed with 12 grams of Yarrabee semi anthracite coal (0.106-0.25 mm) and charged to the graphite crucible.

[144] A current setting known to produce the maximum heating rate was then applied to the coil until the temperature rose to 1350°C, after which the coil current was adjusted as required to maintain the temperature within the range 1300-1375°C for a further 20 minutes.

[145] The final solid was then tipped into a ceramic bowl, cooled to room temperature and was observed to consist predominantly of iron metal in the form of small spheroids, contained within a matrix of titaniferous solids.

[146] After ball milling this product for 5 minutes it was found that the iron metal spheroids could be easily separated from the titaniferous solid by a simple screening step at 106 micron.

[147] The iron spheroids and titaniferous solid (Upgraded Ilmenite - UGI) obtained from this experiment were weighed and analysed.

[148] The size range of the iron spheroids was then determined by screening at 250 and 425 microns.

[149] The results were as follows:-

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>ANALYSIS (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>TiO₂</td>
</tr>
<tr>
<td>Goondicum Ilmenite</td>
<td>86.0</td>
<td>49.9</td>
</tr>
<tr>
<td>Iron spheroids</td>
<td>20.4</td>
<td>0.0</td>
</tr>
<tr>
<td>UGI</td>
<td>58.5</td>
<td>73.3</td>
</tr>
</tbody>
</table>

[150] The size distribution of the iron spheroids was as follows:-

<table>
<thead>
<tr>
<th>SIZE RANGE</th>
<th>CONTENT (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+425 µ</td>
<td>30.5</td>
</tr>
<tr>
<td>-425+250 µ</td>
<td>28.0</td>
</tr>
<tr>
<td>-250+106 µ</td>
<td>41.5</td>
</tr>
</tbody>
</table>

[151] These results indicated that 62.4% of the iron content of the ilmenite had been reduced to iron metal containing 91.6% iron and that 100% of the titanium content had been extracted as UGI containing 73.3% TiO₂.

[152] It should be noted that 100% of the P₂O₅ content was extracted into the iron metal.
The UGI product was subjected to magnetic separation at 600 gauss using a Readings Pilot Laboratory Roll Magnetic Separator. The non-magnetic and magnetic fractions were then weighed and analysed.

The magnetic fraction was then leached for 60 minutes in 100 ml of 10% Hydrochloric Acid using a magnetic stirrer. The solids were then recovered by filtration, dried, weighed and analysed.

The results were as follows:

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>ANALYSIS (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UGI Head</td>
<td>58.5</td>
<td>73.3 0.000 19.1</td>
</tr>
<tr>
<td>Non-Magnetics</td>
<td>35.4</td>
<td>80.6 0.000 9.0</td>
</tr>
<tr>
<td>Magnetics</td>
<td>23.1</td>
<td>57.6 0.000 37.2</td>
</tr>
<tr>
<td>Magnetics Leached</td>
<td>16.4</td>
<td>81.0 0.000 10.9</td>
</tr>
</tbody>
</table>

This result indicated that the initial UGI, expressed as the <106 micron fraction of the product) could be further upgraded as a non-magnetic fraction to 80.6% TiO₂ and 9.0% Fe, by magnetic separation at 1500 gauss and that the magnetic fraction from this separation could then be upgraded to a similar UGI product by hydrochloric acid leaching.

The initial UGI therefore still contained some fine iron metal physically attached to fine UGI particles, indicating that insufficient retention time had been allowed within the desired temperature zone for complete coagulation of the iron into +106 micron spheroids and that in fact 83.5% of the iron content of the ilmenite had been reduced to metal, with 100% recovery of the titanium content as UGI containing 81% TiO₂.

This UGI product is ideally suited to Titanium Dioxide pigment production by the Sulphate Route as an alternative to Titanium Slag, due to its high TiO₂ sulphate solubility (>97%), low iron content and low levels of deleterious impurities.

Exemplary test results - Example 2

In this example, 86 grams of Goondicum Ilmenite Concentrate of known analysis was mixed with 12 grams of Yarrabee semi anthracite coal (1-2 mm) and charged to the graphite crucible. A current setting known to produce the maximum heating rate was then applied to the coil until the temperature rose to 1350°C, after which the coil current was adjusted as required to maintain the temperature within the range 1300-1375°C for a further 40 minutes.

The final solid was then tipped into a ceramic bowl, cooled to room temperature and was observed to consist predominantly of iron metal in the form of small spheroids, contained within a matrix of titaniferous solids.

After ball milling this product for 5 minutes it was found that the iron metal spheroids could be easily separated from the titaniferous solid by a simple screening step at 106 micron.
The iron spheroids and titaniferous solid (Upgraded Ilmenite - UGI) obtained from this experiment were weighed and analysed.

The size range of the iron spheroids was then determined by screening at 250 and 425 microns.

The results were as follows:

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>ANALYSIS (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>TiO₂</td>
</tr>
<tr>
<td>Goondicum Ilmenite</td>
<td>86.0</td>
<td>49.9</td>
</tr>
<tr>
<td>Iron spheroids</td>
<td>30.3</td>
<td>0.0</td>
</tr>
<tr>
<td>UGI</td>
<td>50.3</td>
<td>85.4</td>
</tr>
</tbody>
</table>

The size distribution of the iron spheroids was as follows:

<table>
<thead>
<tr>
<th>SIZE RANGE</th>
<th>CONTENT (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+425 µm</td>
<td>34.5</td>
</tr>
<tr>
<td>-425+250 µm</td>
<td>38.7</td>
</tr>
<tr>
<td>-250+106 µm</td>
<td>26.8</td>
</tr>
</tbody>
</table>

These results indicated that 93.1% of the iron content of the ilmenite had been reduced to iron metal containing 91.6% iron and that 100% of the titanium content had been extracted as UGI containing 85.4% TiO₂.

It should be noted that 100% of the P₂O₅ content was extracted into the iron metal.

Exemplary test results - Example 3

In this example, 86 grams of lower grade Goondicum Ilmenite Concentrate of known analysis was mixed with 12 grams of Yarrabee semi anthracite coal (2.0-4.0 mm) and charged to the graphite crucible. A current setting known to produce the maximum heating rate was again applied to the coil until the temperature rose to 1350°C, after which the coil current was adjusted as required to maintain the temperature within the range 1300-1375°C for a further 10 minutes.

The final solid was then tipped into a ceramic bowl, cooled to room temperature and was observed to consist predominantly of iron metal in the form of small spheroids, contained within a matrix of titaniferous solids.

After ball milling this product for 5 minutes it was found that the iron metal spheroids could be easily separated from the titaniferous solid by a simple screening step at 106 micron.

The iron spheroids and titaniferous solid (Upgraded Ilmenite - UGI) obtained from this experiment were weighed and analysed.
The size range of the iron spheroids was then determined by screening at 250 and 425 microns.

The results were as follows:

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>TiO₂ (%)</th>
<th>P₂O₅ (%)</th>
<th>Fe (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goondicum Ilmenite</td>
<td>86.0</td>
<td>47.9</td>
<td>0.365</td>
<td>33.6</td>
</tr>
<tr>
<td>Iron spheroids</td>
<td>23.1</td>
<td>0.0</td>
<td>1.360</td>
<td>89.4</td>
</tr>
<tr>
<td>UGI</td>
<td>60.8</td>
<td>67.7</td>
<td>0.000</td>
<td>13.5</td>
</tr>
</tbody>
</table>

The size distribution of the iron spheroids was as follows:

<table>
<thead>
<tr>
<th>SIZE RANGE</th>
<th>CONTENT (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+425 µ</td>
<td>36.5</td>
</tr>
<tr>
<td>-425+250 µ</td>
<td>34.0</td>
</tr>
<tr>
<td>-250+106 µ</td>
<td>29.5</td>
</tr>
</tbody>
</table>

These results indicated that 71.6% of the iron content of the ilmenite had been reduced to iron metal containing 89.4% iron and that 100% of the titanium content had been extracted as UGI containing 67.7% TiO₂.

It should be noted that 100% of the considerably higher level of P₂O₅ in the ilmenite used for this example was again extracted into the iron metal.

This example demonstrates that increased particle size of reductive may result in considerable reduction in reaction times.

Exemplary test results - Example 4

In this example, 86 grams of Goondicum Titan-magnetite of known analysis was mixed with 12 grams of Yarrabee semi anthracite coal (2.0-4.0 mm) and charged to the graphite crucible. A current setting known to produce the maximum heating rate was again applied to the coil until the temperature rose to 1150°C, after which the coil current was adjusted as required to maintain the temperature within the range 1100-1200°C for a further 30 minutes.

The final solid was then tipped into a ceramic bowl, cooled to room temperature and was found to consist predominantly of iron metal in the form of small spheroids, contained within a matrix of titaniferous solids.

After ball milling this product for 5 minutes it was found that the iron metal spheroids could be easily separated from the titaniferous solid by a simple screening step at 106 micron.

The iron spheroids and titaniferous solid (Upgraded Ilmenite - UGI) obtained from this experiment were weighed and analysed.
[182] The size range of the iron spheroids was then determined by screening at 250 and 425 microns.

[183] The results were as follows:

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>TiO₂</th>
<th>P₂O₅</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goondicum Titano-magnetite</td>
<td>86.0</td>
<td>13.6</td>
<td>0.046</td>
<td>52.2</td>
</tr>
<tr>
<td>Iron spheroids</td>
<td>43.9</td>
<td>0.0</td>
<td>0.090</td>
<td>91.6</td>
</tr>
<tr>
<td>UGI</td>
<td>24.0</td>
<td>48.7</td>
<td>0.000</td>
<td>8.6</td>
</tr>
</tbody>
</table>

[184] The size distribution of the iron spheroids was as follows:

<table>
<thead>
<tr>
<th>SIZE RANGE</th>
<th>CONTENT (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+425 µ</td>
<td>42.0</td>
</tr>
<tr>
<td>-425+250 µ</td>
<td>36.5</td>
</tr>
<tr>
<td>-250+106 µ</td>
<td>21.5</td>
</tr>
</tbody>
</table>

[185] These results indicated that 95.0% of the iron content of the ilmenite had been reduced to iron metal containing 91.6% iron and that 100% of the titanium content had been extracted as UGI containing 48.7% TiO₂ and 8.6% iron.

[186] This UGI product represents a unique and highly attractive alternative to ilmenite concentrate as a feed-stock for Titanium Dioxide pigment production by the Sulphate Route as, while exhibiting similar TiO₂ content to available ilmenite feed-stocks, it has been found to be much more reactive to sulphate medium (>99.9% TiO₂ solubility) than ilmenite (92-96% TiO₂ solubility), but with much lower iron content, all in ferrous form.

Water Addition

[187] We found that the addition of water to the ilmenite/carbon mixture followed by thorough mixing, prior to charging to the furnace, may result both in improved consistency and improved reduction efficiency at equivalent operating temperatures.

[188] Specifically, the addition of 20% moisture to the ilmenite concentrate prior to addition of the carbon source in the mixing procedure used before charging the furnace crucible, may result in an improvement in repeatability and a lowering of the required operating temperature required to achieve equivalent UGI yield and quality over a given reduction time.

[189] It is thought that this may be due to more even distribution of the reductant carbon within the charge, however the possibility of other physical or chemical effects is not excluded at this stage.
Ferric Sulphate Leaching

[190] We also found that the iron metal content of the reduced ilmenite after screening at 106 micron or the highly magnetic fraction extracted by magnetic separation after this screening step, can be substantially completely removed by leaching with ferric sulphate solution for 10 minutes.

[191] In the application of UGI in the Sulphate Process, the presence of fine iron particles may be undesirable due, for example, to hydrogen emission. It is therefore important that the presence of fine iron particles in UGI product is minimised.

[192] Fine iron particles may be present in the < 106 micron titaniferous fraction of reduced solid discharge from the furnace after the grinding and screening steps, due to incomplete ball formation in the furnace step, or to inefficiency in the magnetic separation step designed to remove residual iron metal.

[193] Magnetic separation inefficiency may occur due to the particles being outside the particle size range for efficient operation of dry roll magnetic separators. It is also possible that a fraction of the solids may have a similar magnetic susceptibility to that of the iron particles.

[194] It has been found that any residual fine iron metal content of the titaniferous solid product can be removed by a subsequent leaching step, involving mechanical agitation of the solids with stoichiometric quantity of ferric sulphate as a dilute solution for 10 minutes, followed by filtration, washing and drying.

[195] This process involves the rapid oxidation/reduction reaction \( \text{Fe} + 2\text{Fe}^{3+} \rightarrow 3\text{Fe}^{2+} \).

[196] This method is preferable to, for example, acid leaching, as no hydrogen emission is involved and the reaction can be shown to be much more rapid and complete.

[197] It is also known that the ferric ions required in this reaction can easily be regenerated by oxidation of the resulting ferrous ions by air, oxygen, ozone or hydrogen peroxide.

[198] The reactions involving ferric sulphate in reactions are as follows:

\[
4\text{Fe} + 4\text{Fe}_2\text{(SO}_4)_3 \rightarrow 12\text{FeSO}_4
\]

\[
12\text{FeSO}_4 + 6\text{H}_2\text{O} + 30_2 \rightarrow 4\text{Fe}_2(\text{SO}_4)_3 + 4\text{Fe(OH)}_3
\]

or overall

\[
4\text{Fe} + 6\text{H}_2\text{O} + 30_2 \rightarrow 4\text{Fe(OH)}_3
\]

[199] It has been found that by using this method, the leached solids obtained from both the < 106 micron size fraction of the furnace discharge and the magnetic fraction extracted from it, are of similar TiO\(_2\) and Fe content to that of the non-magnetic product extracted from the <106 micron size fraction by magnetic separation at 600 gauss.
Addition of Titano-magnetite.

[200] In a further improvement, it has been found that, by the addition of titano-magnetite to the ilmenite/carbon/water mixture prior to mixing, the operating temperature required to achieve a given level of iron reduction and UGI quality can be considerably reduced.

[201] It has been found that operation of the reduction process is greatly improved by the addition of titano-magnetite to the ilmenite charge. Benefits include more rapid reduction at lower operating temperatures and an improvement in the physical nature of the titaniferous solid discharge, in that the solids have an earthy texture and are more easily and completely dischargeable from the furnace crucible.

[202] These effects are particularly evident if a titanomagnetite/ilmenite ratio similar to that occurring within the ore as processed at the Goondicum operation.

[203] It is known that titanomagnetite has a Curie point of around 500°C and therefore can be expected to act in a similar fashion to the graphite crucible liner up to that temperature. However, experimental results indicate that beneficial effects continue beyond this temperature range.

Examples of Improvements

[204] Three additional exemplary examples 5, 6 and 7 are given below, to demonstrate the abovementioned improvements.

Exemplary test results - Example 5

[205] In this example, 150 grams of Goondicum Ilmenite Concentrate of known analysis was thoroughly mixed with 30 grams of Yarrabee semi anthracite coal (0.106-0.25 mm) and 30 ml of water. The solid mixture was then charged to the graphite crucible.

[206] A current setting known to produce the maximum heating rate was then applied to the coil until the temperature rose to 1150°C, after which the coil current was adjusted as required to maintain the temperature within the range 1200-1250°C for a further 20 minutes.

[207] The final solid was then tipped into a ceramic bowl, cooled to room temperature and was observed to consist predominantly of iron metal in the form of small spheroids, contained within a matrix of titaniferous solids.

[208] After ball milling this product for 5 minutes it was found that the iron metal spheroids could be easily separated from the titaniferous solid by a simple screening step at 106 micron.

[209] The iron spheroids and < 106 micron titaniferous solid (Upgraded Ilmenite - UGI) obtained from this experiment were weighed and analysed.

[210] The size range of the iron spheroids was then determined by screening at 250 and 425 microns.
The results were as follows:

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>TiO₂</th>
<th>P₂O₅</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goondicum Ilmenite</td>
<td>150.0</td>
<td>50.8</td>
<td>0.048</td>
<td>34.6</td>
</tr>
<tr>
<td>Iron spheroids</td>
<td>43.0</td>
<td>0.0</td>
<td>0.459</td>
<td>91.6</td>
</tr>
<tr>
<td>&lt;106 micron solids</td>
<td>94.2</td>
<td>80.8</td>
<td>0.000</td>
<td>13.3</td>
</tr>
</tbody>
</table>

The size distribution of the iron spheroids was as follows:

<table>
<thead>
<tr>
<th>SIZE RANGE</th>
<th>CONTENT (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+425 μ</td>
<td>28.5</td>
</tr>
<tr>
<td>-425+250 μ</td>
<td>26.0</td>
</tr>
<tr>
<td>-250+106 μ</td>
<td>45.5</td>
</tr>
</tbody>
</table>

These results indicated that 75.8% of the iron content of the ilmenite had been reduced to iron metal containing 91.6% iron and that 100% of the titanium content had been extracted as UGI containing 80.8% TiO₂ and 13.3% Fe.

It should be noted that 100% of the P₂O₅ content was extracted into the iron metal.

The <106 micron solids product was then subjected to magnetic separation at 600 gauss using a Readings Pilot Laboratory Roll Magnetic Separator. The non-magnetic and magnetic fractions were then weighed and analysed.

The magnetic fraction was then leached for 10 minutes in 100 ml of 10% ferric sulphate using a mechanical stirrer. The solids were then recovered by filtration, dried, weighed and analysed.

The results were as follows:-

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>TiO₂</th>
<th>P₂O₅</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 &lt;106 micron solids</td>
<td>94.2</td>
<td>80.8</td>
<td>0.000</td>
<td>13.3</td>
</tr>
<tr>
<td>2 Non-Magnetics</td>
<td>76.3</td>
<td>89.6</td>
<td>0.000</td>
<td>4.8</td>
</tr>
<tr>
<td>3 Magnetics</td>
<td>17.9</td>
<td>43.0</td>
<td>0.000</td>
<td>49.5</td>
</tr>
<tr>
<td>4 Magnetics Leached</td>
<td>8.6</td>
<td>89.5</td>
<td>0.000</td>
<td>4.8</td>
</tr>
<tr>
<td>UGI (2 + 4)</td>
<td>84.9</td>
<td>89.5</td>
<td>0.000</td>
<td>4.8</td>
</tr>
</tbody>
</table>

This result indicated that a higher grade solids containing 89.6% TiO₂ and 4.6% Fe could be obtained as a non-magnetic fraction by this procedure, that a similar grade product was derived from ferric sulphate leaching of the magnetic fraction. Combination of the non-magnetic solids with the leached magnetics solids yielded a final UGI product containing 89.6% TiO₂ and 4.6% Fe.

This result indicated that the initial <106 micron solids contained fine iron metal particles and that magnetic separation was not fully effective in separating these fine iron particles from non-
metallic solids. This result demonstrated that indicating either insufficient retention time had been allowed within the desired temperature zone for complete coagulation of the iron into +106 micron spheroids, or that the particle size range of the solids is below that required for efficient operation of the magnetic separation equipment, which is designed to operate in the range of 100-450 micron.

[220] Assuming that all iron metal was removed from the UGI these results indicated that in fact 86.9% of the iron content of the ilmenite had been reduced to metal, with 100% recovery of the titanium content, yielding a final UGI product containing 89.5% TiO₂ and 4.8% Fe.

[221] This test demonstrated that UGI containing 80.8% TiO₂ and 13.3% Fe can be produced from high grade (acid leached) Goondicum Ilmenite without the need for magnetic separation, but containing fine iron particles.

[222] By subsequent magnetic separation, it is possible to extract 90% of the TiO₂ content of this product as a non-magnetic fraction free from iron metal and containing 89.5% TiO₂ and 4.8% Fe.

[223] The magnetic fraction, containing the balance of the TiO₂ content and the fine iron metal particles, can be recycled to the furnace or can more advantageously be leached with ferric sulphate, to produce a solid with a similar composition to the non-magnetic fraction, thereby extracting 100% of the TiO₂ content as UGI free from iron metal and containing 89.5% TiO₂ and 4.8% Fe.

Example test results - Example 6

[224] In this example, 150 grams of Intermediate Grade Goondicum Ilmenite Concentrate of known analysis was thoroughly mixed with 30 grams of Yarrabee semi anthracite coal (0.106-0.25 mm) and 30 ml of water. The solid mixture was then charged to the graphite crucible.

[225] A current setting known to produce the maximum heating rate was then applied to the coil until the temperature rose to 1150°C, after which the coil current was adjusted as required to maintain the temperature within the range 1200-1250°C for a further 20 minutes.

[226] The final solid was then tipped into a ceramic bowl, cooled to room temperature and was observed to consist predominantly of iron metal in the form of small spheroids, contained within a matrix of titaniferous solids.

[227] After ball milling this product for 5 minutes it was found that the iron metal spheroids could be easily separated from the titaniferous solid by a simple screening step at 106 micron.

[228] The iron spheroids and titaniferous solid (Upgraded Ilmenite - UGI) obtained from this experiment were weighed and analysed.

[229] The size range of the iron spheroids was then determined by screening at 250 and 425 microns.
The <106 micron solids fraction was then leached for 10 minutes in 100 ml of 10% ferric sulphate using a mechanical stirrer. The solids were then recovered by filtration, dried, weighed and analysed.

The results were as follows:

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>TiO₂</th>
<th>P₂O₅</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goondicum Ilmenite</td>
<td>150.0</td>
<td>48.0</td>
<td>0.471</td>
<td>33.4</td>
</tr>
<tr>
<td>Iron spheroids</td>
<td>42.1</td>
<td>0.0</td>
<td>0.000</td>
<td>91.6</td>
</tr>
<tr>
<td>&lt;106 micron</td>
<td>94.8</td>
<td>75.9</td>
<td>0.000</td>
<td>12.2</td>
</tr>
<tr>
<td>&lt;106 micron solids( leached)</td>
<td>85.4</td>
<td>84.2</td>
<td>0.000</td>
<td>4.7</td>
</tr>
</tbody>
</table>

The size distribution of the iron spheroids was as follows:

<table>
<thead>
<tr>
<th>SIZE RANGE</th>
<th>CONTENT (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>+425 μ</td>
<td>29.5</td>
</tr>
<tr>
<td>-425+250 μ</td>
<td>25.5</td>
</tr>
<tr>
<td>-250+106 μ</td>
<td>45.0</td>
</tr>
</tbody>
</table>

The analysis of the <106 fraction after screening indicated that 76.9% of the iron content of the ilmenite had been reduced to iron metal containing 91.6% iron and that 100% of the titanium content had been extracted as <106 micron solids containing 75.9% TiO₂ and 12.2% Fe.

Subsequent ferric sulphate leaching of these solids indicated that in fact 93.8% of the iron content had been reduced to iron metal and that it was possible to produce a final UGI with 84.2% TiO₂ and 4.7% Fe.

This test demonstrated that UGI containing 75.9% TiO₂ and 12.2% Fe can be produced from intermediate grade Goondicum Ilmenite without the need for magnetic separation, but containing fine iron particles.

It was demonstrated that the fine iron metal particles can be removed from this product by ferric sulphate leaching to yield an improved UGI product free from iron metal and containing 84.2% TiO₂ and 4.7% Fe.

In this example the lower final UGI TiO₂ content compared to that obtained in Exemplary test 5 has resulted from the higher level of impurities (known to be predominantly titano-magnetite, biotite, pyroxene, apatite and feldspar) contained in the ilmenite used.

It was also demonstrated that a higher degree of iron reduction was achieved, indicating that some or all of these impurities have assisted in the reduction mechanism.

This is of considerable interest in the case of titano-magnetite, known to have been completely removed in preparation of the ilmenite used in Exemplary test 5.
Exemplary test results - Example 7

In this example, 116 grams of Intermediate grade Goondicum Ilmenite Concentrate of known analysis was 34 grams of titano-magnetite of known analysis, 30 grams of Yarrabee semi anthracite coal (106-250 micron) and 30 ml of water. After thorough mixing this was charged to the graphite crucible. A current setting was applied such that the temperature rose to 1000°C over a period of 10 minutes. The current setting was then increased to allow the temperature to rise linearly to 1240°C after 15 minutes.

The final solid was then tipped into a ceramic bowl, cooled to room temperature and was observed to consist predominantly of iron metal in the form of small spheroids, contained within a matrix of titaniferous solids.

It was noted that the solids had an earthy consistency and tipped easily from the crucible with little or no adherence to the crucible surface.

After ball milling this product for 5 minutes it was found that the iron metal spheroids could be easily separated from the titaniferous solid by a simple screening step at 106 micron.

The iron spheroids and titaniferous solid (Upgraded Ilmenite - UGI) obtained from this experiment were weighed and analysed.

The size range of the iron spheroids was then determined by screening at 250 and 425 microns.

The results were as follows:

<table>
<thead>
<tr>
<th>FRACTION</th>
<th>WEIGHT (g)</th>
<th>TiO₂</th>
<th>P₂O₅</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goondicum Ilmenite</td>
<td>116.0</td>
<td>48.0</td>
<td>0.471</td>
<td>33.4</td>
</tr>
<tr>
<td>Goondicum Titano-magnetite</td>
<td>34.0</td>
<td>12.1</td>
<td>0.426</td>
<td>34.2</td>
</tr>
<tr>
<td>Iron spheroids</td>
<td>36.7</td>
<td>0.0</td>
<td>1.705</td>
<td>92.0</td>
</tr>
<tr>
<td>&lt;106 micron fraction</td>
<td>87.9</td>
<td>68.0</td>
<td>0.000</td>
<td>18.9</td>
</tr>
<tr>
<td>&lt;106 micron fraction Leached</td>
<td>73.4</td>
<td>81.4</td>
<td>0.000</td>
<td>6.4</td>
</tr>
</tbody>
</table>

The size distribution of the iron spheroids was as follows:-

<table>
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<th>SIZE RANGE</th>
<th>CONTENT (%)</th>
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<tr>
<td>+425 µ</td>
<td>50.5</td>
</tr>
<tr>
<td>-425+250 µ</td>
<td>24.4</td>
</tr>
<tr>
<td>-250+106 µ</td>
<td>25.1</td>
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</table>

This test demonstrated that UGI containing 68.0% TiO₂ and 18.9% Fe can be produced without the need for magnetic separation. Subsequent ferric sulphate leaching of these solids yielded a final
UGI with 81.4% TiO₂ and 6.4% Fe indicating that in fact 90.7% of the iron content had been reduced to iron metal.

[249] It should be noted that 100% of the P₂O₅ content was extracted into the iron metal.

[250] The results from this test clearly identify beneficial effects arising from the addition of titano-magnetite in the demonstrated ability to achieve high levels of reduction at considerably lower temperatures and retention times.

[251] This offers significant potential economic advantages, particularly when combined with the observed ease of removal of the final solids from the furnace.

[252] An important consideration for the Goondicum operation is that the high TiO₂ contents achieved in the final titaniferous solids, both in the unleached and leached UGI solids, indicate that significant quantities of currently valueless titano-magnetite can be utilised to increase the yield of acceptable quality UGI, particularly as the ilmenite used in this test was of intermediate grade.

[253] Combination of higher grade ilmenite feed, such as current final product, with the currently rejected titano-magnetite tailings from LIMS, could be expected to increase the TiO₂ content of UGI produced by this method by at least 2%, thus giving a product more or less equivalent to that produced from ilmenite alone.

Process Implications of Improvements

[254] Apart from the benefits observed within the furnace process the addition of water to furnace feed is a possible advantage in that the final ilmenite prior to drying contains 5-10% moisture. Thus the need for drying may be eliminated.

[255] The basic circuit required for the reduction process is shown as Figure 4.

[256] A critical marketing consideration for UGI into the sulphate pigment industry is an anticipated tight specification on iron metal content, due to safety considerations.

[257] Ferric sulphate leaching offers a simple and safe method of elimination of iron metal from UGI and provides an insurance, should other means of achieving this result, such as alternative magnetic separation equipment more suited to the particle size range involved or longer furnace retention times and the final choice becomes a matter of economics.

[258] Ferric leaching step can be most economically achieved by use of a ferrous sulphate solution at pH 3.4, in a reaction vessel fitted with a particular design of aeration agitator, in which transient ferric ions are generated by oxidation of ferrous ions with oxygen, either as air, oxygen gas or ozone.

[259] Alternatively, oxidation can be achieved by direct addition of hydrogen peroxide to the leach solution at the desired rate.

[260] The inclusion of ferric sulphate leaching into the reduction circuit is shown in Figure 5.
The inclusion of ferric sulphate leaching in conjunction with magnetic separation is shown in Figure 6.

The inclusion of titano-magnetite is shown as an option in all three circuits of Figures 4-6.

The ferric hydroxide by product generated by ferric leaching can either be discharged to tailings, sold to a ferric sulphate manufacturer or recirculated to the furnace as part of the moisture make-up.

In the latter case the ferric hydroxide will be reduced to iron and will appear as increased iron ball product.
Interpretation

Embodiments:

[265] Reference throughout this specification to "one embodiment" or "an embodiment" means that a particular feature, structure or characteristic described in connection with the embodiment is included in at least one embodiment of the present invention. Thus, appearances of the phrases "in one embodiment" or "in an embodiment" in various places throughout this specification are not necessarily all referring to the same embodiment, but may. Furthermore, the particular features, structures or characteristics may be combined in any suitable manner, as would be apparent to one of ordinary skill in the art from this disclosure, in one or more embodiments.

[266] Similarly it should be appreciated that in the above description of example embodiments of the invention, various features of the invention are sometimes grouped together in a single embodiment, figure, or description thereof for the purpose of streamlining the disclosure and aiding in the understanding of one or more of the various inventive aspects. This method of disclosure, however, is not to be interpreted as reflecting an intention that the claimed invention requires more features than are expressly recited in each claim. Rather, as the following claims reflect, inventive aspects lie in less than all features of a single foregoing disclosed embodiment. Thus, the claims following the Detailed Description of Specific Embodiments are hereby expressly incorporated into this Detailed Description of Specific Embodiments, with each claim standing on its own as a separate embodiment of this invention.

[267] Furthermore, while some embodiments described herein include some but not other features included in other embodiments, combinations of features of different embodiments are meant to be within the scope of the invention, and form different embodiments, as would be understood by those in the art. For example, in the following claims, any of the claimed embodiments can be used in any combination.

Different Instances of Objects

[268] As used herein, unless otherwise specified the use of the ordinal adjectives "first", "second", "third", etc., to describe a common object, merely indicate that different instances of like objects are being referred to, and are not intended to imply that the objects so described must be in a given sequence, either temporally, spatially, in ranking, or in any other manner.

Specific Details

[269] In the description provided herein, numerous specific details are set forth. However, it is understood that embodiments of the invention may be practiced without these specific details. In other instances, well-known methods, structures and techniques have not been shown in detail in order not to obscure an understanding of this description.
Term inology

In describing the preferred embodiment of the invention illustrated in the drawings, specific terminology will be resorted to for the sake of clarity. However, the invention is not intended to be limited to the specific terms so selected, and it is to be understood that each specific term includes all technical equivalents which operate in a similar manner to accomplish a similar technical purpose. Terms such as "forward", "rearward", "radially", "peripherally", "upwardly", "downwardly", and the like are used as words of convenience to provide reference points and are not to be construed as limiting terms.

Comprising and Including

In the claims which follow and in the preceding description of the invention, except where the context requires otherwise due to express language or necessary implication, the word "comprise" or variations such as "comprises" or "comprising" are used in an inclusive sense, i.e. to specify the presence of the stated features but not to preclude the presence or addition of further features in various embodiments of the invention.

Any one of the terms: including or which includes or that includes as used herein is also an open term that also means including at least the elements/features that follow the term, but not excluding others. Thus, including is synonymous with and means comprising.

Scope of Invention

Thus, while there has been described what are believed to be the preferred embodiments of the invention, those skilled in the art will recognize that other and further modifications may be made thereto without departing from the spirit of the invention, and it is intended to claim all such changes and modifications as fall within the scope of the invention. For example, any formulas given above are merely representative of procedures that may be used. Functionality may be added or deleted from the block diagrams and operations may be interchanged among functional blocks. Steps may be added or deleted to methods described within the scope of the present invention.

Although the invention has been described with reference to specific examples, it will be appreciated by those skilled in the art that the invention may be embodied in many other forms.

Industrial Applicability

It is apparent from the above, that the arrangements described are applicable to the metallurgic industries.
Claims

1. A metallurgical process for upgrading ferro-titaniferous mineral concentrate using time dependent magnetic fields, the ferro-titaniferous mineral concentrate comprising more than 10% by weight of iron content calculated as FeO, the process comprising:

   reducing a substantial portion of the iron content at temperature below the flow point of the titaniferous phase of the mineral concentrate in the presence of a time dependent magnetic field, wherein the time dependent magnetic field is used to at least contribute to the reduced iron content coalescing into iron spheroids; and

   subsequently physically separating the iron spheroids.

2. A metallurgical process as claimed in claim 1, wherein the majority of the iron spheroids are greater than 106 μm in diameter.

3. A metallurgical process as claimed in claim 1, further comprising:

   providing a heat source; and

   providing a separate time dependent magnetic field source; and

   controlling the time dependent magnetic field source independent of the heat source.

4. A metallurgical process as claimed in claim 3, wherein the heat source is controlled independently of the time dependent magnetic field source to heat the ferro-titaniferous mineral concentrate.

5. A metallurgical process as claimed in claim 3, wherein the time dependent magnetic field contributes to an insubstantial amount of heating as compared to the heat source.

6. A metallurgical process as claimed in claim 3, wherein controlling the time dependent magnetic field source comprises controlling the time dependent magnetic field to control the size of the iron spheroids.

7. A metallurgical process as claimed in claim 3, wherein controlling the time dependent magnetic field source comprises controlling the time dependent magnetic field to control the rate of generation of iron spheroids.

8. A metallurgical process as claimed in claim 3, wherein the heat source comprises at least one of an oil, gas and coal fired heat source.

9. A metallurgical process as claimed in claim 1, wherein physically separating the iron spheroids comprises a crushing or grinding phase followed by at least one of sieving, air classification and magnetic separation.

10. A metallurgical process as claimed in claim 1, wherein the temperature is below 1600°C.

11. A metallurgical process as claimed in claim 1, wherein the temperature is below 1500°C.

12. A metallurgical process as claimed in claim 1, wherein the temperature is below 1400°C.
13. A metallurgic process as claimed in claim 1, wherein the temperature is between 1000°C and 1370°C.
14. A metallurgic process as claimed in claim 1, wherein the iron content is at least one of ferrous and ferric oxide.
15. A metallurgic process as claimed in claim 14, further comprising providing a carbon source for reducing the iron content.
16. A metallurgic process as claimed in claim 15, wherein the carbon source comprises at least one of coke, charcoal and coal.
17. A metallurgic process as claimed in claim 16, wherein the ferrous iron content of the mineral concentrate is reduced to iron metal according to the following reaction:
   \[ \text{FeO} + \text{C} \rightarrow \text{Fe} + \text{CO} \]
18. A metallurgic process as claimed in claim 16, wherein the ferric iron content of the mineral concentrate is reduced to iron metal according to the following reaction:
   \[ \text{Fe}_2\text{O}_3 + 3\text{C} \rightarrow 2\text{Fe} + 3\text{CO} \]
19. A metallurgic process as claimed in claim 15, wherein the carbon source comprises carbon monoxide.
20. A metallurgic process as claimed in claim 19, wherein the ferrous iron content of the mineral concentrate is reduced to iron metal according to the following reaction:
   \[ \text{FeO} + \text{CO} \rightarrow \text{Fe} + \text{CO}_2 \]
21. A metallurgic process as claimed in claim 19, wherein the ferric iron content of the mineral concentrate is reduced to iron metal according to the following reaction:
   \[ \text{Fe}_2\text{O}_3 + 3\text{CO} \rightarrow 2\text{Fe} + 3\text{CO}_2 \]
22. A metallurgic process as claimed in claim 1, wherein the ferro-titaniferous mineral concentrate is ilmenite (FeTiOa) mineral concentrate.
23. A metallurgic process as claimed in claim 22, wherein the temperature is between 1050°C and 1350°C.
24. A metallurgic process as claimed in claim 23, wherein the concentrate is exposed to the time dependent magnetic field for a retention time of between 2 and 30 minutes.
25. A metallurgic process as claimed in claim 22, further comprising controlling the temperature and retention time according to a predetermined TiO$_2$ content.
26. A metallurgic process as claimed in claim 1, further comprising adding magnetic additives to the ferro-titaniferous mineral concentrate.
27. A metallurgic process as claimed in claim 26, wherein the magnetic additives comprise at least one of magnetite and titano-magnetite.
28. A metallurgic process as claimed in claim 1, wherein the ferro-titaniferous mineral concentrate is at least one of leucoxene, titanomagnetite and titaniferous iron sands.

29. A metallurgic process as claimed in claim 1, further comprising inclusion of a chemical additive which reports to the iron product.

30. A metallurgic process as claimed in claim 29, wherein the chemical additive comprises borates and phosphates.

31. A metallurgic process as claimed in claim 15, further comprising addition of moisture to the ferro-titaniferous mineral concentrate prior the addition of the carbon source.

32. A metallurgic process as claimed in claim 31, further comprising addition of approximately 20% moisture to the ferro-titaniferous mineral concentrate.

33. A metallurgic process as claimed in claim 1, further comprising leaching to remove iron spheroids being smaller than 106 \( \mu \text{m} \) in diameter.

34. A metallurgic process as claimed in claim 33, wherein the leaching uses ferric sulphate.

35. A metallurgic process as claimed in claim 34, further comprising mechanical agitation for approximately 10 minutes.

36. A metallurgic process as claimed in claim 34, wherein the leaching involves the oxidation/reduction reaction \( \text{Fe} + 2\text{Fe}^{+++} \rightarrow 3\text{Fe}^{++} \).

37. A metallurgic process as claimed in claim 36, further comprising regenerating ferric ions required for the reaction by oxidation of the resulting ferrous ions by at least one of air, oxygen, ozone or hydrogen peroxide.

38. Apparatus for upgrading ferro-titaniferous mineral concentrate comprising more than 10% by weight of iron content calculated as FeO using a time dependent magnetic field at temperatures below the flow point of the titaniferous phase of the mineral concentrate, the apparatus comprising a time dependent magnetic field source controller for controlling the time dependent magnetic field source to control the reduction of the iron content wherein the time dependent magnetic field is used to contribute to the reduced iron content coalescing into iron spheroids.

39. Apparatus as claimed in claim 31, further comprising a heat source and wherein the heat source is controlled independently of the time dependent magnetic field source to heat the ferro-titaniferous mineral concentrate.
Ferrotitaniferous mineral concentrate 4

Heat + time dependent magnetic field

Cooling

 Crush/grind 150μ

Screen

Dry roll mangnet 1,000 gauss

Magnetics

 Separated Metal product 7

<150μ

Upgraded taniferous product 8

>150μ

Figure 3
Titanomagnetite → Ilmenite → Carbon → Water →

Mixer →

Induction furnace →

Grind →

Screen → Fe(OH)_3 →

Ferric sulphate leach → FeSO_4 →

Filter →

UGI Product → Iron Metal →

Figure 5
**INTERNATIONAL SEARCH REPORT**

**International application No.**  
PCT/AU2017/050746

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### A. CLASSIFICATION OF SUBJECT MATTER

B03C 1/015 (2006.01)  B03C 1/30 (2006.01)  C22B 34/12 (2006.01)  C22B 1/14 (2006.01)  B03B 9/04 (2006.01)

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According to International Patent Classification (IPC) or both national classification and IPC

### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

Class marks; (ISO 9001 Quality Certified Service)

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### C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
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* Further documents are listed in the continuation of Box C  
X See patent family annex

- " " Special categories of cited documents:
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  - "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "&" document member of the same patent family

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Date of the actual completion of the international search  
10 October 2017

Date of mailing of the international search report  
10 October 2017

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**Name and mailing address of the ISA/AU**

AUSTRALIAN PATENT OFFICE  
PO BOX 200, WODEN  ACT  2606, AUSTRALIA  
Email address: pct@ipaustralia.gov.au

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**Authorised officer**

Kosala Gunatillaka  
AUSTRALIAN PATENT OFFICE  (ISO 9001 Quality Certified Service)  
Telephone No. +61262223652

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<td>US 2721 793 A (R.J. MAGRI, Jr. et al.) 25 October 1955 From column 1, line 15 to column 2, line 61</td>
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<td>A</td>
<td>US 3552564 A (BURGENER et al.) 05 January 1971 Abstract; Fig. 1; claims 1-10</td>
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<td>CA 2302712 A1 (COUNCIL OF SCIENTIFIC &amp; INDUSTRIAL RESEARCH [IN]) 24 September 2001 Abstract, from page 8, line 1 to page 11, line 11</td>
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<td>RU 2295582 C1 (OOO DATA TS OOO DATA TS FA [RU]) 20 March 2007, English language machine translation of abstract and specification retrieved from Espacenet Abstract</td>
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This Annex lists known patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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<tr>
<td>CN 103501889 A</td>
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<td>US 3552564 A</td>
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End of Annex

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

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