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[Continued on next page]

(54) **Title:** METHOD FOR INCREASING OF TITANIUM OXIDE CONTENT IN SLAG PRODUCED IN CONNECTION WITH ELECTRIC FURNACE SMELTING OF TITANOMAGNETITE

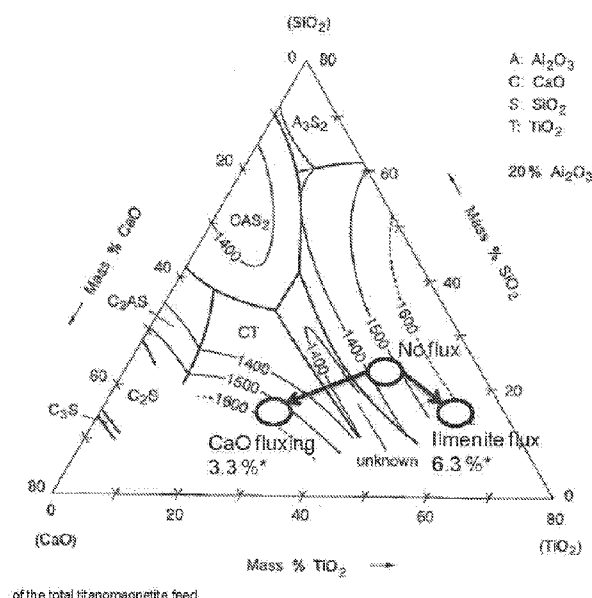


FIG 1

(57) **Abstract:** The invention relates to a method for increasing titanium oxide content in slag produced in connection with electric furnace smelting of titanomagnetite, wherein the method comprises feeding titanomagnetite, reducing agent and fluxing agent into an electric furnace, melting titanomagnetite, reducing agent and fluxing agent in the electric furnace to form a layer containing liquid metal and layer containing slag above the layer containing liquid metal, and withdrawing liquid metal and slag separately from the electric furnace. The fluxing agent comprises in percentages of weight between 3 and 100 % titanium oxide bearing material such as ilmenite.



SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ,
GW, KM, ML, MR, NE, SN, TD, TG).

— *of inventorship (Rule 4.17(iv))*

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Published:

— *as to applicant's entitlement to apply for and be granted
a patent (Rule 4.17(ii))*

— *with international search report (Art. 21(3))*

METHOD FOR INCREASING OF TITANIUM OXIDE CONTENT IN SLAG PRODUCED IN CONNECTION WITH ELECTRIC FURNACE SMELTING OF TITANOMAGNETITE

5 Field of the invention

The invention relates to a method for increasing titanium oxide content in slag produced in connection of electric furnace smelting of titanomagnetite as defined in the preamble of independent claim 1.

10 In this document "titanium oxide" refers to all titanium oxide compounds, such as to TiO (non stoichiometric), to TiO₂, and to Ti₂O₃.

There are many potential titanomagnetite deposits globally. A typical processing method for titanomagnetite is electric furnace smelting. In most cases the main products in titanomagnetite smelting are pig iron (rich in valuable metals, for example vanadium) and slag. Slag is not regarded as a commercial product and it is usually landfilled.

15 Depending on the titanomagnetite quality, slag may need fluxing in order to achieve good smelting properties. Traditionally slag fluxing is done with quartz or calcium oxide. Traditional fluxing leaves slag low in content of titanium oxide (TiO₂) and the slag cannot be upgraded into a commercial product.

20 In the smelting process, raw materials are stored in their separate day bins. The raw materials are mixed together to a conveyor from the day bins, forming the furnace feed mix. The main feed material to the furnace is titanomagnetite concentrate, titanomagnetite prereduced concentrate, titanomagnetite pellets or prereduced titanomagnetite pellets. The used reductant is usually carbon bearing material, such as coke or anthracite. In addition to concentrate and reductant, fluxing agents can be used to modify the slag composition so that the slag has desired
25 liquidus (melting) temperature and low enough viscosity for smooth tappings. Traditionally slag fluxing is conducted by feeding calcium bearing material into the furnace, most commonly burnt lime (CaO). The slag, consisting mainly of TiO₂, SiO₂, Al₂O₃, MgO and CaO has been of no commercial value and is typically landfilled.

30 The furnace feed mix is normally directed into feed bins, located above the electric furnace. The electric furnace can be of two types, AC or DC furnace. The feed mix is fed with vibrating feeders into feed tubes that lead the materials through the furnace roof into the molten bath. The molten slag temperature is kept between 1500 and 1700 °C, depending on the slag liquidus temperature. The metal temperature is typically lower at 1450-1550 °C. The main process target in titanomagnetite smelting is to reduce the iron oxides present in the
35 titanomagnetite concentrate into pig iron and the valuable metal oxides (such as vanadiumoxide) present in the concentrate into the pig iron metal phase. The two basic reactions during the smelting process are the following:

Reduction of FeO:



Reduction of valuable metal oxides (example V_2O_5):



Small amount of other oxides such as SiO_2 and MnO are also reduced to the metal phase. Other oxide components are left in the slag phase including most of the TiO_2 present in the concentrate. Metal phase forms the bottom liquid layer and the slag with lower density remains on top of the metal phase. Metal and slag are tapped to product ladles alternately. The slag product may also be wet granulated to fine particle size directly from the furnace. Traditional fluxing leaves slag low in content of TiO_2 and the slag cannot be upgraded into a commercial product.

In publication AU 656476 a process is provided for the production of a titanium rich slag and pig iron from titanomagnetite. The titanomagnetite is fed continuously, together with carbonaceous reductant, and in the absence of fluxes, to the molten bath of a circular d.c. arc furnace, preferably a plasma arc furnace, wherein the molten bath forms the anode and one or more electrodes in the furnace roof form the cathode. The feed materials are preferably preheated or prereduced using heat contained in the off-gases from the process. Titanium rich slag which can be used as the feed to a sulphate based titanium dioxide production process is recovered continuously, or intermittently, and pig iron is tapped off as a by-product.

Publication WO 2011/143703 presents a direct smelting process that comprises supplying (a) a metalliferous feed material that contains iron oxides and at least 3 wt.% titanium oxides (b) a solid carbonaceous feed material, and (c) an oxygen-containing gas into a direct smelting vessel containing a molten bath of iron and slag and direct smelting the metalliferous feed material in the vessel and producing process outputs of molten iron, molten slag containing titanium oxides, and an off-gas. The process is characterised by controlling the process conditions, as described herein, so that the molten slag has a viscosity in a range of 0.5-5 poise when the slag temperature is in a range of 1400-1550 °C in the molten bath in the direct smelting vessel. Publication WO 2011/143703 presents also a direct smelting vessel when used to smelt a metalliferous feed material that contains iron oxides and at least 3 wt.% titanium oxides via a molten bath-based direct smelting process, with the vessel containing a molten bath of metal and slag, and with the molten slag having a temperature range of 1400-1550 °C and a viscosity in a range of 0.5-5 poise.

Objective of the invention

The object of the invention is to provide an effective method for the increasing of titanium oxide content in produced slag in connection with electric furnace smelting of titanomagnetite, making the slag a valuable by product.

Short description of the invention

The method of the invention is characterized by the definitions of independent claim 1.

Preferred embodiments of the method are defined in the dependent claims 2 to 9.

The invention relates also to the use of slag containing titanium oxide obtained from a method according to any of the claims 1 to 10 as a feedstock in the production of TiO_2 pigment.

In the presented innovation traditional fluxing materials are replaced with titanium oxide bearing materials such as with ilmenite such as ilmenite concentrate in order to produce upgradeable TiO_2 slag. Ilmenite concentrate is a commercial mineral product (rich in TiO_2) which is widely available in the market. Also other titanium oxide bearing materials can be used. Ilmenite concentrate is incorporated into the furnace feed mix with titanomagnetite and reducing agent such as anthracite. The feed amount is calculated based on the target slag TiO_2 content and the target slag liquidus temperature. Slag viscosity and the target slag liquidus temperature are factors that limit the maximum TiO_2 content. In some embodiments of the method, the target slag TiO_2 content in weight percentages is at least 55 %, in other embodiments at least 70 % or at least 85 %.

In the electric furnace, if ilmenite is used as titanium oxide bearing material, the iron oxides in the ilmenite are reduced to metallic form therefore raising the furnace pig iron output. The TiO_2 in the ilmenite is smelted and it dissolves into the slag originating from the titanomagnetite and anthracite ash components, therefore raising the slag TiO_2 content.

The presented slag modification transforms the otherwise dischargeable slag into a commercial product. As a result the smelting operation becomes economically more viable and profitable. The presented innovation makes many potential titanomagnetite projects economically more viable.

List of figures

In the following the invention will be described in more detail by referring to the figures, of which

Figure 1 is a ternary phase diagram showing ilmenite fluxing and CaO fluxing of reference material 1,

Figure 2 is a ternary phase diagram showing ilmenite fluxing and CaO fluxing of reference material 2,

Figure 3 is a ternary phase diagram showing ilmenite fluxing and CaO fluxing of reference material 3, and

Figure 4 shows the relationship between slag capacity and slag vanadium oxide bearing capacity.

Detailed description of the invention

The invention relates to a method for increasing of titanium oxide content i.e. the relative

weight percentage content of titanium oxide with respect to the other components of the slag, in slag produced in connection with electric furnace smelting of titanomagnetite.

The method comprises feeding titanomagnetite, reducing agent and fluxing agent into a smelting furnace such as into an electric furnace.

5 The method comprises melting titanomagnetite, reducing agent and fluxing agent in the electric furnace to form a layer containing liquid metal and layer containing slag above the layer containing liquid metal.

The electric furnace is preferably, but not necessarily, an electric arc furnace, and consequently thermal energy for performing said melting is preferably, but not necessarily
10 produced by electrical energy.

The method comprises withdrawing liquid metal and slag separately from the electric furnace.

In the method, the fluxing agent comprises in percentages of weight between 3 and 100 % titanium oxide bearing material such as ilmenite.

15 The method comprises preferably, but not necessarily, feeding an amount of titanium oxide bearing material such as ilmenite into the electric furnace from about 3 % to about 25 %, preferably from about 10 to about 15 %, more preferably about 15 % on titanomagnetite weight basis.

The method comprises preferably, but not necessarily, using carbonaceous material such
20 as coke or anthracite as reducing agent.

The method comprises preferably, but not necessarily, feeding titanomagnetite containing in weight percentages between 75 and 95 %, preferably between 80 and 90 % magnetite (Fe_3O_4), between 0.25 and 1.5, preferably between 0.45 and 1.5 % vanadium oxide (V_2O_5), and between 2.5 and 15 %, preferably between 3 and 10 % titanium oxide including for example TiO_2 and
25 Ti_2O_3 .

The method comprises preferably, but not necessarily, using titanium oxide bearing material such as ilmenite containing in weight percentages between about 40 and about 60 %, preferably between about 50 and about 55 % titanium oxide including for example TiO_2 and Ti_2O_3 .

30 The method comprises preferably, but not necessarily, calculating the titanium oxide equivalent content in the titanomagnetite, calculating the titanium oxide equivalent content in the titanium oxide bearing material such as in the ilmenite, and adjusting the feed amount of titanomagnetite to the feed amount of titanium oxide bearing material such as of ilmenite.

The method comprises preferably, but not necessarily, feeding an amount of titanium
35 oxide bearing material such as of ilmenite into the electric furnace so that from about 1 % to about 20 % titanium oxide, including for example TiO_2 and Ti_2O_3 , will be fed into the electric furnace with the titanium oxide bearing material such as with the ilmenite on titanomagnetite weight basis.

Theoretical examples

In the following, three theoretical examples of ilmenite fluxing and the effect of the slag chemistry will be presented. In the theoretical calculations, the reference titanomagnetites 1 to 3 (see table 1) was used as feed materials and reference ilmenite 2 (see table 2) was used fluxing agent. On the theoretical calculations, for comparison purposes, CaO was also used fluxing agent. Additionally, the theoretical result if no fluxing agent was used, was calculated in the theoretical calculations. The target slag TiO₂ content in terms of percentages of weight in the calculations was 55 %. This can controlled directly by ilmenite feed amount in relation to the titanomagnetite feed amount.

Based on the theoretical approach in all of the three different titanomagnetite cases, ilmenite fluxing enables good slag liquidus temperature, and produces a slag that is a marketable product. Figures 1 to 3 also illustrate that with CaO fluxing the slag TiO₂ content will become low, rendering the slag a disposable, non-commercial by product.

In the ternary phase diagrams in figures 1 to 3, the MgO component amount was added to CaO component in order to effectively illustrate the slag chemistry in the diagrams.

Table 1: composition of reference materials 1, 2 and 3 in percentages of weight

Reference material		1	2	3
FeO	%		-	
Fe ₃ O ₄	%	91	86	79
Fe ₂ O ₃	%		-	
TiO ₂	%	4	7.6	7.9
SiO ₂	%	0.7	2.9	2.5
Al ₂ O ₃	%	1.4	01.0	3.6
CaO	%	0.4	1.2	0.9
V ₂ O ₅	%	0.7	1.5	0.5
Cr ₂ O ₃	%	0.1	0.01	-
MnO	%	0.2	0.3	0.7
MgO	%	0.5	0.57	3.0
P ₂ O ₅	%	-	-	0.1
C	%	0.02	-	-
S	%	-	-	-

Volatiles	%	-	0.4	-
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Table 2 composition of ilmenite material 3 in percentages of weight

Reference ilmenite		1	2	3	4	5
TiO₂ekv	%	45	57	40	51	59
FeO	%	1.5	18	19	17	2.2
Fe₂O₃	%	46	20	27	27	28
TiO₂	%	45	57	41	47	55
Ti₂O₃	%	-	-	7.8	3.4	3.6
MgO	%	0.3	0.2	0.7	0.3	1.1
Al₂O₃	%	0.5	0.4	0.5	0.6	1.1
P₂O₅	%	-	-	0.1	<0.02	0.2
CaO	%	0.4	0.2	0.02	0.1	0.1
V₂O₅	%	0.2	0.3	0.2	0.1	0.2
Cr₂O₃	%	0.1	0.1	0.1	0.1	1.3
MnO	%	0.3	0.9	0.6	1.6	0.9
ZrO	%	0.1	0.04	0.01	0.01	0.1
SiO₂	%	1.9	1.5	0.6	0.3	0.2
Volatiles	%	-	-	0.4	0.9	3.1

5 The slag composition projections were conducted by normal mass balance calculations, where the titanomagnetite amount was fixed and ilmenite addition was calculated (% of titanomagnetite feed) based on the slag TiO₂ content target (55 %). Recovery rates (distribution of elements into slag and metal) were estimated based on experimental tests. For the ternary phase diagrams in figures 1 to 3, the main slag component contents (SiO₂, CaO, Al₂O₃ and TiO₂) were projected to 100 %, therefore excluding minor components from the ternary phase diagrams. Also due to the multiphase slag system, MgO was summed up with CaO in order to simplify the system and to enable phase diagram study.

The used recovery rates are illustrated in table 3.

Table 3: Used recovery rates.

Element	Recovery % to metal
Fe	100
Cr	20
Ti	10
Mn	40
V	95
Si	15
S	50
P	90
C	10

5 Reference Material 1

In the ternary phase diagram in figure 1, ilmenite and CaO fluxing of titanomagnetite reference 1 is represented. In the ternary phase diagram the slag compositions of the three cases (no flux, ilmenite flux and CaO flux) and the corresponding liquidus temperatures are shown.

From figure 1 it can be seen that in order to achieve slag TiO₂ content of 55 %, the
 10 ilmenite flux feed amount should be 6.3 % of the titanomagnetite feed. At the same time a liquidus temperature of about 1550°C is achieved. This temperature enables good vanadium recoveries and a high TiO₂ content of the slag which makes the slag a marketable product.

When CaO fluxing is used the CaO flux amount should be 3.3 % of titanomagnetite feed resulting in slag CaO content of 35 % and slag liquidus temperature of 1550 C. This fluxing
 15 leaves the slag TiO₂ content at 27 %, rendering the slag a non-profitable side product.

Reference Material 2

In the ternary phase diagram in figure 2, ilmenite and CaO fluxing of titanomagnetite reference 2 is represented. In the ternary phase diagram the slag compositions of the three cases (no flux, ilmenite flux and CaO flux) and the corresponding liquidus temperatures are shown.

From figure 2 it can be seen that in order to achieve slag TiO₂ content of 55 %, the
 20 ilmenite flux feed amount should be 3.6 % of the titanomagnetite feed. At the same time a liquidus temperature of above 1600°C is achieved. This temperature enables good vanadium recoveries and a high TiO₂ content of the slag which makes the slag a marketable product.

With CaO fluxing the fluxing amount should be 5.3 % of titanomagnetite feed resulting
 25 in slag CaO content of 35 % and liquidus temperature of 1550 °C. This fluxing leaves the slag TiO₂ content at 35 %, rendering the slag a non-profitable side product.

Reference Material 3

In the ternary phase diagram in figure 3, ilmenite and CaO fluxing of titanomagnetite
 30 reference 3 is represented. In the ternary phase diagram the slag compositions of the three cases

(no flux, ilmenite flux and CaO flux) and the corresponding liquidus temperatures are shown.

From figure 3 it can be seen that in order to achieve slag TiO_2 content of 55 %, the ilmenite flux feed amount should be 15 % of the titanomagnetite feed. At the same time a liquidus temperature of above 1500°C is achieved.

With CaO fluxing the fluxing amount should be 3.8 % of titanomagnetite feed resulting in slag CaO content of 35 %. This fluxing leaves the slag TiO_2 content at 35 %, rendering the slag a non-profitable side product.

Higher recovery rates of valuable metals

High slag basicity lowers valuable metal recoveries, for example the recovery of vanadium into the pig iron. In figure 4, the relationship between slag optical basicity and slag vanadium oxide bearing capacity is seen.

CaO is a slag component which raises slag basicity. On the other hand TiO_2 acts as an acid component, which lowers slag basicity. As CaO content increases in traditional fluxing, the vanadium capacity of the slag increases, therefore lowering the amount of vanadium that is reduced into metallic form. When ilmenite flux is used, slag CaO content is very low, and TiO_2 content high. As a result slag vanadium oxide capacity remains low therefore raising the vanadium recovery to metal.

Ilmenite fluxing enables higher recovery of valuable metals making the process economically more viable, as less vanadium is lost into the slag.

Broader operating area with high TiO_2 versus high CaO

As seen in figures 1 to 3, slag with high TiO_2 resides in a temperature area where the liquidus temperature lines are further apart when compared to the area where CaO is used as flux. This means that deviations in normal smelter operation, such as raw material chemical composition changes, feed system inaccuracies, power and temperature changes, have less effect in slag characteristics (viscosity, liquidus temperature) compared to operation with CaO flux, making the smelter operation more smooth and predictable.

Possibility for productification of the TiO_2 rich slag

The slag TiO_2 needs to be above 55 % for TiO_2 pigment production. Therefore if ilmenite flux addition is adequate this limit can be reached.

It is apparent to a person skilled in the art that as technology advanced, the basic idea of the invention can be implemented in various ways. The invention and its embodiments are therefore not restricted to the above examples, but they may vary within the scope of the claims.

Claims

1. A method for increasing titanium oxide content in slag produced in connection with electric furnace smelting of titanomagnetite, wherein the method comprises:

feeding titanomagnetite, reducing agent and fluxing agent into an electric furnace,

melting titanomagnetite, reducing agent and fluxing agent in the electric furnace to form a layer containing liquid metal and layer containing slag above the layer containing liquid metal, and

withdrawing liquid metal and slag separately from the electric furnace

characterized

by the fluxing agent comprises in percentages of weight between 3 and 100 % titanium oxide bearing material.

2. The method according to claim 1, **characterized** by feeding an amount of titanium oxide bearing material into the electric furnace from about 3 % to about 25 %, preferably from about 10 to about 15 %, more preferably about 15 % on titanomagnetite weight basis.

3. The method according to claim 1 or 2, **characterized** by using carbonaceous material such as coke or anthracite as reducing agent.

4. The method according to any of the claims 1 to 3, **characterized** by feeding titanomagnetite containing in weight percentages between 75 and 95 % magnetite, between 0.25 and 1.5 % vanadium oxide, and between 2.5 and 15 % titanium oxide.

5. The method according to any of the claims 1 to 4, **characterized** by using titanium oxide bearing material containing in weight percentages between about 40 and about 60 %, preferably between about 50 and about 55 % titanium oxide.

6. The method according to any of the claims 1 to 5, **characterized** by calculating the titanium oxide equivalent content in the titanomagnetite, by calculating the titanium oxide equivalent content in the titanium oxide bearing material, and by adjusting the feed amount of titanomagnetite to the feed amount of titanium oxide bearing material.

7. The method according to any of the claims 1 to 6, **characterized** by feeding an amount of titanium oxide bearing material into the electric furnace so that from about 1 % to about 20 % titanium oxide will be fed into the electric furnace with the titanium oxide bearing material on titanomagnetite weight basis.

8. The method according to any of the claims 1 to 7, **characterized**
by using ilmenite as titanium oxide bearing material.

5 9. The method according to any of the claims 1 to 8, **characterized**
by the electric furnace being an electric arc furnace, and
by thermal energy for performing said melting being produced by electrical energy

10. Use of slag containing titanium oxide obtained from a method according to any of the
claims 1 to 9 as a feedstock in the production of TiO₂ pigment.

1 / 4

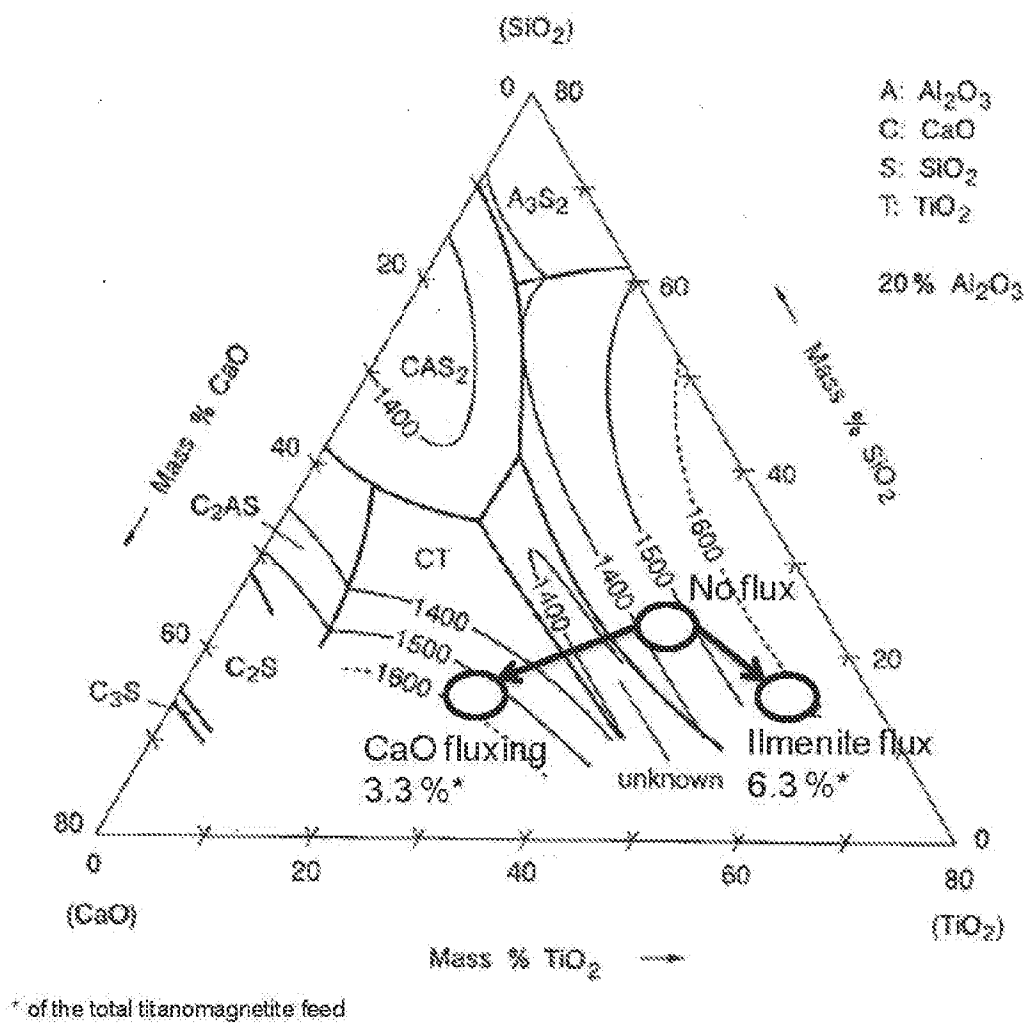
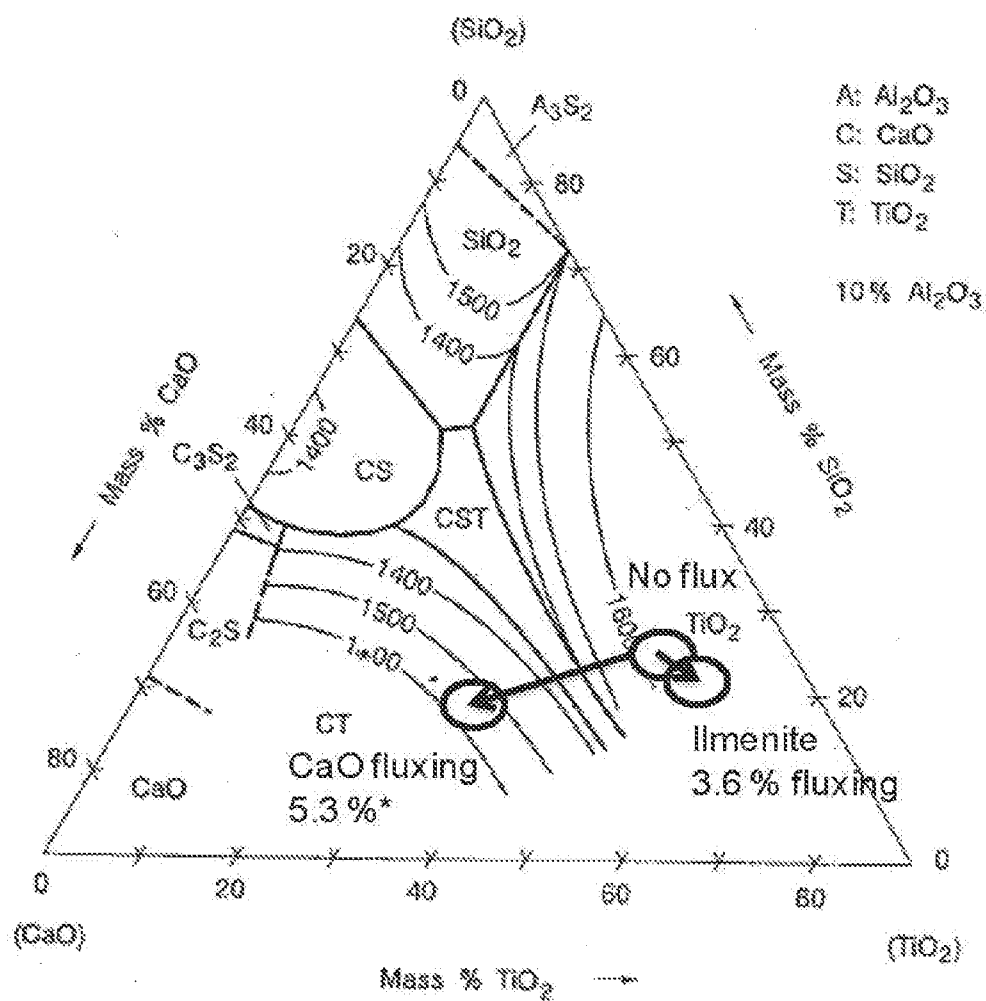


FIG 1

2 / 4



* of the total titanomagnetite feed

FIG 2

3 / 4

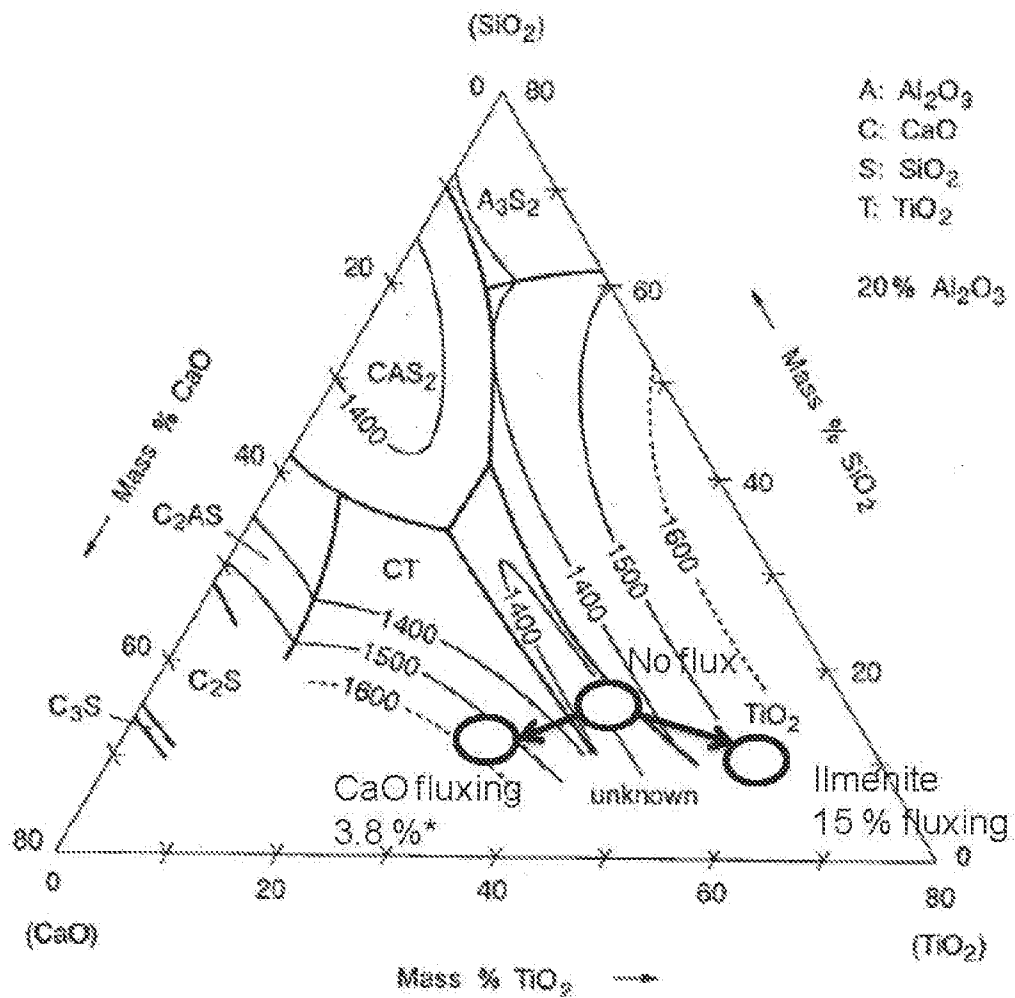


FIG 3

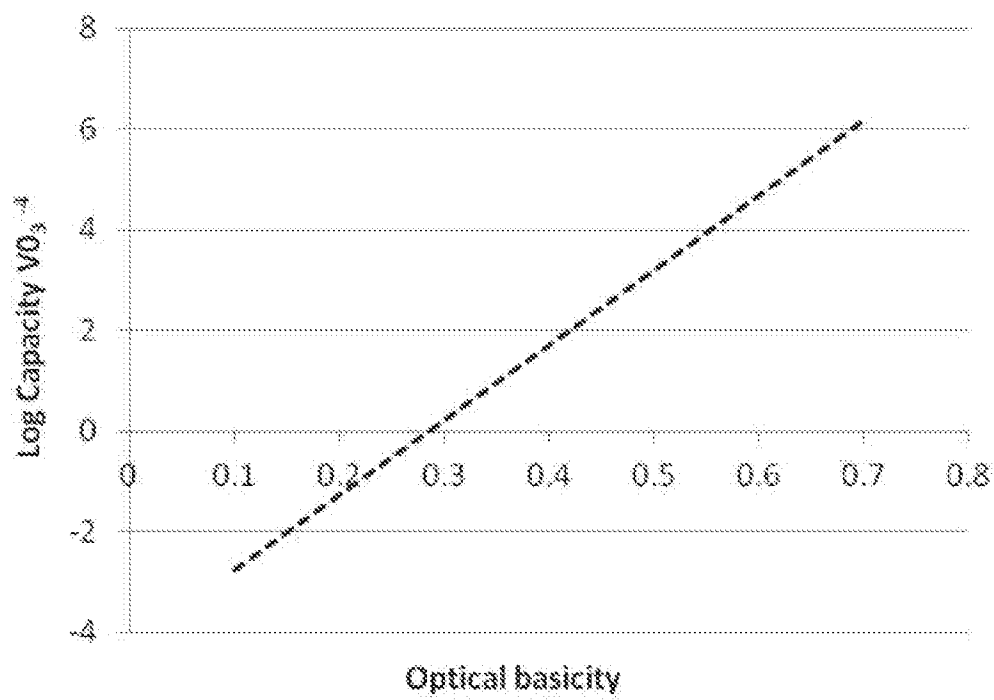


FIG 4

INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2016/050087

A. CLASSIFICATION OF SUBJECT MATTER
INV. C22B34/12 C21B11/10
ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C22B C21B

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 practicable, search terms used)

EP0-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	WO 2011/143703 A1 (TECH RESOURCES PTY LTD [AU]; DRY RODNEY JAMES [AU]; PILOTE JACQUES [AU] 24 November 2011 (2011-11-24) cited in the application the whole document -----	10
A	-----	1-9
X	US 2001/052272 A1 (SCHOUKENS ALBERT F S [ZA] ET AL) 20 December 2001 (2001-12-20) the whole document -----	10
A	-----	1-9
	-/--	

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search

20 April 2016

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29/04/2016

Name and mailing address of the ISA/

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Swiatek, Ryszard

INTERNATIONAL SEARCH REPORT

International application No
PCT/FI2016/050087

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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