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(54) **ELECTROCHEMICAL REDUCTION OF TITANIUM OXIDE**

ELEKTROCHEMISCHE REDUKTION VON TITANOXID

REDUCTION ELECTROCHIMIQUE D'OXYDE DE TITANE

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Description

[0001] The present application relates to electrochemical reduction of metal oxides.

[0002] The present application relates to electrochemical reduction of metal oxides in the form of powders and/or pellets in an electrolytic cell containing a molten electrolyte to produce reduced material, namely metal having a low oxygen concentration, typically no more than 0.2% by weight.

[0003] The present invention is concerned with controlling electrochemical reduction of titanium oxide.

[0004] The present invention applies to situations in which the process is carried out on a batch basis, a continuous basis, and a semi-continuous basis.

[0005] The present invention was made during the course of a research project on electrochemical reduction of metal oxides carried out by the applicant.

[0006] The research project focussed on the reduction of titania (TiO_2).

[0007] During the course of the research project the applicant carried out a series of experiments, initially on a laboratory scale and more recently on a pilot plant scale, investigating the reduction of metal oxides in the form of titania in electrolytic cells comprising a pool of molten $CaCl_2$-based electrolyte, an anode formed from graphite, and a range of cathodes.

[0008] The $CaCl_2$-based electrolyte used in the experiments was a commercially available source of $CaCl_2$, which decomposed on heating and produced a very small amount of CaO.

[0009] The applicant operated the electrolytic cells at a potential above the decomposition potential of CaO and below the decomposition potential of $CaCl_2$.

[0010] The applicant found in the laboratory work that the cells electrochemically reduced titania to titanium with low concentrations of oxygen, i.e. concentrations less than 0.2 wt.%, at these potentials.

[0011] The applicant operated the laboratory electrolytic cells under a wide range of different operating parameters and conditions.

[0012] The applicant operated the laboratory electrolytic cells on a batch basis with titania in the form of pellets and larger solid blocks in the early part of the laboratory work and titania powder in the later part of the work.

[0013] The applicant also operated the laboratory electrolytic cells on a batch basis with other metal oxides.

[0014] Recent pilot plant work carried out by the applicant was carried out on a pilot plant electrolytic cell that was set up to operate initially on a continuous basis and subsequently on a batch basis.

[0015] During the course of the laboratory scale experimental work the applicant found that the concentration of CaO in the electrolyte had a significant impact on the process for electrochemically reducing metal oxides in the laboratory cells.

[0016] In particular, the applicant found that the concentration of CaO in the electrolyte should be controlled by maintaining the concentration above a minimum CaO concentration and below a maximum CaO concentration in order to optimise operation of the process.

[0017] More particularly, the applicant found that there were lower reduction rates and lower current efficiencies at higher CaO concentrations in the electrolyte.

[0018] In addition, the applicant found that there was a minimum CaO concentration below which there was practically no reduction of titania.

[0019] The applicant believes that, in any situation, the lower limit of CaO is a function of scavenger or other reactions that consume CaO.

[0020] For example, CaO and titania react spontaneously and form perovskite ($CaTiO_3$). Thus, depending on the amount of CaO in the electrolyte and the amount of titania introduced into the electrolyte, the amount of CaO may decrease substantially as a consequence of the perovskite reaction.

[0021] In addition, calcium metal reacts with titania and forms calcium titanates, thus producing another sink for CaO.

[0022] In addition, the applicant found that the minimum and maximum CaO concentrations in any given situation are dependent to a certain extent on the physical characteristics of the titania.

[0023] An important physical characteristic is the surface area of titania in contact with electrolyte, particularly when this characteristic is considered in the context of a given mass of the electrolyte and a given mass of the titania.

[0024] In turn, the titania surface area for a given mass of titania is dependent on physical characteristics of the titania, such as powder/pellet shape and porosity.

[0025] The applicant also believes that the above-mentioned significance of the CaO concentration in a $CaCl_2$-based electrolyte also applies to other alkali earth metal oxides, alkali metal oxides, and yttrium oxides. Specifically, the applicant believes that where one or more than one of the metal oxides is present in a $CaCl_2$-based electrolyte, controlling the concentrations of the metal oxides within ranges is a means of optimising operation of an electrochemical reduction process for titanium oxide in a solid state in the electrolyte.

[0026] The metal oxides may be present as an impurity or impurites in the feed material used to produce the $CaCl_2$-based electrolyte and/or by way of specific addition to the electrolyte.

[0027] According to the present invention there is provided a process as defined in the appended independent claim, to which reference should now be made. The invention may therefore advantageously provide a process for electrochemically reducing a titanium oxide in a solid state in an electrolytic cell that includes an anode, a cathode, a molten $CaCl_2$-based electrolyte containing CaO, and titanium oxide feed material in contact with the molten electrolyte, which electrochemical process includes applying an electrical potential across the anode and the cathode and electrochemically reducing titanium oxide

feed material in contact with the molten electrolyte and producing reduced material, and which process is characterised by controlling the process by controlling the concentration of CaO in the electrolyte.

[0028] Preferably the process includes controlling the concentration of CaO in the electrolyte by maintaining the concentration of CaO in the electrolyte between lower and upper concentration limits.

[0029] Preferably the maximum CaO concentration in the electrolyte is 0.5 wt.%.

[0030] More preferably the maximum CaO concentration in the electrolyte is 0.3 wt.%.

[0031] Preferably the minimum CaO concentration in the electrolyte is 0.005 wt.%.

[0032] More preferably the minimum CaO concentration in the electrolyte is 0.05 wt.%.

[0033] Typically, the minimum CaO concentration in the electrolyte is 0.1 wt.%.

[0034] Typically, the CaO concentration in the electrolyte is in the range of 0.1-0.3 wt.%.

[0035] More typically, the CaO concentration in the electrolyte is in the range of 0.15-0.25 wt.%.

[0036] The process includes controlling the concentration of CaO in the electrolyte by reference to the surface area of contact of the titanium oxide with the electrolyte.

[0037] The process includes controlling the concentration of CaO in the electrolyte by reference to the surface area of contact of the titanium oxide with the electrolyte when considered in the context of the mass of the electrolyte and the mass of the titanium oxide.

[0038] The practical option for controlling the CaO concentration in the electrolyte is to control the mass ratio of the electrolyte and the titanium oxide in the cell.

[0039] In the context of controlling the CaO concentration in the electrolyte above the minimum concentration limit, the process includes controlling the mass ratio of the electrolyte and titanium oxide in the cell to be at least 10:1 in situations where the initial CaO concentration is below 0.3, more preferably below 0.2 wt.% in the electrolyte and the titanium oxide is in a form of pellets and/or powders having a specific surface area of 0.1 to 100 m^2/g of titanium oxide.

[0040] The process may include controlling the CaO concentration in the electrolyte by adding CaO to the electrolyte during the course of the process.

[0041] The process may include controlling the CaO concentration in the electrolyte by selecting an initial CaO concentration that is sufficiently high.

[0042] The electrochemical process may be carried out on any one of a batch basis, a semi-continuous basis, or a continuous basis.

[0043] The titanium oxide feed material is in a powder and/or a pellet form.

[0044] Preferably the titanium oxide feed material is titania.

[0045] Preferably the electrochemical reduction process includes applying a potential across the anode and the cathode that is above the decomposition potential of CaO and below the decomposition of $CaCl_2$.

[0046] The electrochemical reduction process may be carried out as a single stage or a multi-stage process.

[0047] In more general terms, according to the present application there is provided a process for electrochemically reducing a titanium oxide in a solid state in an electrolytic cell that includes (a) an anode, (b) a cathode, (c) a molten $CaCl_2$-based electrolyte containing any one or more than one metal oxide selected from the group including alkali earth metal oxides, alkali metal oxides, and yttrium oxides, and (d) a titanium oxide feed material in contact with the molten electrolyte, which electrochemical process includes applying an electrical potential across the anode and the cathode and electrochemically reducing titanium oxide feed material in contact with the molten electrolyte and producing reduced material, and which process is characterised by controlling the process by controlling the concentration of the selected metal oxide or metal oxides in the electrolyte.

[0048] According to the present invention the selected metal oxide is CaO.

[0049] The above-mentioned laboratory scale experimental work carried out by the applicant was carried out in a high temperature laboratory electrolytic cell.

[0050] The cell comprised a reaction vessel, a furnace, a crucible assembly, an electrode assembly, and a power supply.

[0051] The reaction vessel was manufactured from a high temperature stainless steel and had an internal diameter of 110mm and height of 430mm, and a water-cooled flange.

[0052] The vessel was contained within a resistance-heated furnace capable of reaching 1400°K. A positioning pedestal within the vessel allowed for the crucible containing the molten salt to be properly fixed within the vessel.

[0053] A water-cooled lid was a critical feature of the reaction vessel and had provision for a viewing port which facilitated accurate positioning of the electrodes and the thermocouple within the molten salt bath. The viewing port also allowed the surface of the molten salt to be monitored during the reaction.

[0054] Carbon monoxide and carbon dioxide are the main by-products of the electrochemical reduction process. In order to monitor the progress of reduction and to ensure that the only source of oxygen was the titanium dioxide, special care was taken in controlling the gas atmosphere. A measured flow of high purity argon gas was passed through a furnace containing copper turnings at 873°K, then through a furnace containing magnesium flakes at 673°K before entering the vessel. After passing through the vessel, the gas stream was stripped of any chlorine-containing species and moisture and continuously analysed for CO and CO_2.

[0055] A solid electrolyte based oxygen analyser monitored the partial pressure of O_2 in the off-gas immediately after the furnace.

[0056] Data logging and control was performed using

a LabView software interface.

[0057] Furnace pressure, applied voltage, reference voltage, cell current, electrolyte temperature, oxygen, carbon monoxide and carbon dioxide content of the off-gas were monitored and logged for subsequent analysis as a function of time.

[0058] As is indicated above, the chemistry of the molten electrolyte was found to have a significant impact on the process operation. Consequently, the applicant ensured that the composition of the electrolyte at the start of a run was well controlled.

[0059] The electrolyte was prepared from analytical grade dihydrate, $CaCl_2.H_2O$ obtained from APS Chemicals. Typically, 680g of molten salt were used in the experiments. Prior to melting, a dehydration step was carried out as follows.

[0060] The $CaCl_2.H_2O$ was slowly heated under vacuum to 525K and kept at this temperature for at least 12 hours during which time the weight loss due to water removal was monitored. Once there was no more weight loss, the anhydrous salt was transferred from the vacuum oven into a platinum crucible and placed in a melting furnace. The salt was melted at 1275°K and kept at this temperature for 30 minutes to allow further removal of any residual water. The molten salt was then cast into a preheated steel mould, removed once solid, and transferred while hot to a drying oven held at 400K.

[0061] By using this standardised procedure for preparing the $CaCl_2$ electrolyte, it was possible to obtain reproducible quality material.

[0062] In the majority of experiments the titania pellets were prepared from 99.5% minimum purity rutile (Alfa Aesar) although occasionally other titania sources were used. The titania pellets were prepared so that control of the total porosity and pore size was exercised by sintering at a predetermined temperature.

[0063] In one series of experiments, the concentration of CaO in the electrolyte was systematically increased up to 2.5 wt.%.

[0064] Specifically, experiments were carried out with concentrations of 0.14, 0.20, 0.42, 1.4, and 2.4 wt.% CaO in the electrolyte and with the electrolyte in molten form at 1275K. The experiments were carried out for a total of 4 hours with an applied voltage of 3V. The IR losses of the cell were calculated to be less than 0.2V.

[0065] The results of the experiments are summarised in Figures 1 to 3.

[0066] Figure 1 is a plot of cell current (in Amps) versus time (in seconds) for each of the above-mentioned experiments. The Figure shows that there was a decrease in cell current over time with each of the CaO concentrations in the electrolyte. The Figure also shows that the cell passed more current with increased concentrations of CaO in the electrolyte. This finding, of itself does not indicate that there was increased reduscton of titania at increased CaO concentrations.

[0067] Figure 2 is a plot of the concentration of oxygen in titanium in the reduced pellets versus the CaO concentration in the electrolyte at the end of the experimental runs of 4 hours. The Figure shows that reduction of titania to titanium was inhibited as the concentration of CaO in the electrolyte increased. Specifically, the Figure shows that there were higher concentrations of oxygen in the titanium produced in the cell as the CaO concentrations in the electrolyte increased. In other words, the Figure shows that the rate of reduction decreased as the concentration of CaO in the electrolyte increased.

[0068] In addition, when read together, Figures 1 and 2 indicate that significantly higher current consumption was required to achieve lower levels of reduction of titania at increased CaO concentrations. Significantly, on the basis of Figures 1 and 2, operating at CaO concentrations of 0.5 wt.% or less produced high levels of reduction - to less than about 0.5 wt.% oxygen - with reasonable current consumption

[0069] Figure 3 is a plot of the amount of carbon removed from the anode in grams and the overall current efficiency versus the CaO concentration in the electrolyte at the end of the experimental runs of 4 hours. The Figure shows that the amounts of electricity and carbon consumed in the process were proportionally higher as the concentration of CaO in the electrolyte increased. In particular, the Figure shows that the current efficiency decreased as the concentration of CaO in the electrolyte increased.

[0070] The above results and other results not summarised here and consideration by the applicant of the results indicate to the applicant that CaO is an important parameter for controlling an electrochemical reduction process to produce optimum results in terms of reduction rate and current efficiency, and that the process could be controlled for example by maintaining the CaO concentration within lower and upper concentration limits. Specifically, above results and other results not summarised here indicate to the applicant that the CaO concentration in the electrolyte should be controlled to be in the range of 0.05-0.5 wt.%.

[0071] By way of example, whilst the above-described laboratory work was carried out on a $CaCl_2$-based electrolyte containing CaO, the applicant believes that the findings of the work extend to other other alkali earth metal oxides, alkali metal oxides, and yttrium oxides.

Claims

1. A process for electrochemically reducing a titanium oxide in a solid state in an electrolytic cell that includes an anode, a cathode, a molten $CaCl_2$-based electrolyte containing CaO, and titanium oxide feed material in contact with the molten electrolyte, which electrochemical process includes applying an electrical potential across the anode and the cathode and electrochemically reducing titanium oxide feed material in contact with the molten electrolyte and producing reduced material, and which process is **char-**

acterised by controlling the process by controlling the concentration of CaO in the electrolyte by controlling the mass ratio of the electrolyte and titanium oxide in the cell to be at least 10:1, the initial CaO concentration to be below 0.3 wt.% in the electrolyte and the titanium oxide to be in a form of pellets and/or powders having a specific surface area of 0.1 to 100 m^2/g of titanium oxide.

2. The process defined in claim 1 includes controlling the concentration of CaO in the electrolyte by maintaining the concentration of CaO in the electrolyte between lower and upper concentration limits.

3. The process defined in claim 2 includes maintaining the concentration of CaO in the electrolyte at or below a maximum CaO concentration of 0.5 wt.%.

4. The process defined in claim 2 includes maintaining the concentration of CaO in the electrolyte at or below a maximum CaO concentration of 0.3 wt.%.

5. The process defined in any one of claims 2 to 4 includes maintaining the concentration of CaO in the electrolyte at or above a minimum CaO concentration of 0.005 wt.%.

6. The process defined in any one of claims 2 to 4 includes maintaining the concentration of CaO in the electrolyte at or above a minimum CaO concentration of 0.05 wt.%.

7. The process defined in any of claims 2 to 4 includes maintaining the concentration of CaO in the electrolyte at or above a minimum CaO concentration of 0.1 wt.%.

8. The process defined in claim 2 includes maintaining the concentration of CaO in the electrolyte in the range of 0.1-0.3 wt.%.

9. The process defined in claim 2 includes maintaining the concentration of CaO in the electrolyte in the range of 0.15-0.25 wt.%.

10. The process defined in any one of the preceding claims includes controlling the concentration of CaO in the electrolyte by reference to the surface area of contact of the titanium oxide with the electrolyte.

11. The process defined in any one of the preceding claims includes controlling the concentration of CaO in the electrolyte by reference to the surface area of contact of the titanium oxide with the electrolyte when considered in the context of the mass of the electrolyte and the mass of the titanium oxide.

12. The process defined in any one of the preceding claims includes controlling the CaO concentration in the electrolyte by controlling the mass ratio of the electrolyte and the titanium oxide in the cell.

13. The process defined in any one of the preceding claims in which the initial CaO concentration is below 0.2 wt. % in the electrolyte.

14. The process defined in any one of the preceding claims includes controlling the CaO concentration in the electrolyte by adding CaO to the electrolyte during the course of the process.

15. The process defined in any one of the preceding claims includes controlling the CaO concentration in the electrolyte by selecting an initial CaO concentration that is sufficiently high.

Patentansprüche

1. Prozess zum elektrochemischen Reduzieren eines Titanoxids in einem festen Zustand in einer elektrolytischen Zelle, die eine Anode, eine Kathode, einen geschmolzenen Elektrolyt auf $CaCl_2$-Basis, der CaO enthält, und ein Titanoxid-Ausgangsmaterial, das mit dem geschmolzenen Elektrolyt in Kontakt ist, beinhaltet, wobei der elektrochemische Prozess das Anlegen eines elektrischen Potentials an die Anode und die Kathode und das elektrochemische Reduzieren von mit dem geschmolzenen Elektrolyt in Kontakt befindlichem Titanoxid-Ausgangsmaterial und Erzeugen von reduziertem Material beinhaltet und wobei der Prozess **dadurch gekennzeichnet ist, dass** der Prozess durch Regeln der Konzentration von CaO in dem Elektrolyt durch Regeln des Massenverhältnisses des Elektrolyts und Titanoxids in der Zelle, so dass es wenigstens 10:1 beträgt, die anfängliche CaO-Konzentration unter 0,3 Gew.-% in dem Elektrolyt ist und das Titanoxid in einer Form von Pellets und/oder Pulvern mit einer spezifischen Oberfläche von 0,1 bis 100 m^2/g Titanoxid ist, gesteuert wird.

2. Prozess nach Anspruch 1, der das Regeln der Konzentration von CaO in dem Elektrolyt durch Halten der Konzentration von CaO in dem Elektrolyt zwischen einer unteren und einer oberen Konzentrationsgrenze beinhaltet.

3. Prozess nach Anspruch 2, der das Halten der Konzentration von CaO in dem Elektrolyt auf oder unter einer maximalen CaO-Konzentration von 0,5 Gew.-% beinhaltet.

4. Prozess nach Anspruch 2, der das Halten der Konzentration von CaO in dem Elektrolyt auf oder unter einer maximalen CaO-Konzentration von 0,3 Gew.-

% beinhaltet.

5. Prozess nach Anspruch 2, der das Halten der Konzentration von CaO in dem Elektrolyt auf oder über einer Mindest-CaO-Konzentration von 0,005 Gew.-% beinhaltet.

6. Prozess nach einem der Ansprüche 2 bis 4, der das Halten der Konzentration von CaO in dem Elektrolyt auf oder über einer Mindest-CaO-Konzentration von 0,05 Gew.-% beinhaltet.

7. Prozess nach einem der Ansprüche 2 bis 4, der das Halten der Konzentration von CaO in dem Elektrolyt auf oder über einer Mindest-CaO-Konzentration von 0,1 Gew.-% beinhaltet.

8. Prozess nach Anspruch 2, der das Halten der Konzentration von CaO in dem Elektrolyt in dem Bereich von 0,1 bis 0,3 Gew.-% beinhaltet.

9. Prozess nach Anspruch 2, der das Halten der Konzentration von CaO in dem Elektrolyt in dem Bereich von 0,15 bis 0,25 Gew.-% beinhaltet.

10. Prozess nach einem der vorhergehenden Ansprüche, der das Regeln der Konzentration von CaO in dem Elektrolyt durch Bezug auf den Flächeninhalt der Kontaktfläche des Titanoxids mit dem Elektrolyt beinhaltet.

11. Prozess nach einem der vorhergehenden Ansprüche, der das Regeln der Konzentration von CaO in dem Elektrolyt durch Bezug auf den Flächeninhalt der Kontaktfläche des Titanoxids mit dem Elektrolyt beinhaltet, wenn im Zusammenhang mit der Masse des Elektrolyts und der Masse des Titanoxids betrachtet.

12. Prozess nach einem der vorhergehenden Ansprüche, der das Regeln der CaO-Konzentration in dem Elektrolyt durch Regeln des Massenverhältnisses des Elektrolyts und des Titanoxids in der Zelle beinhaltet.

13. Prozess nach einem der vorhergehenden Ansprüche, bei dem die anfängliche CaO-Konzentration in dem Elektrolyt unter 0,2 Gew.-% ist.

14. Prozess nach einem der vorhergehenden Ansprüche, der das Regeln der CaO-Konzentration in dem Elektrolyt durch Zugeben von CaO zu dem Elektrolyt während des Prozessverlaufs beinhaltet.

15. Prozess nach einem der vorhergehenden Ansprüche, der das Regeln der CaO-Konzentration in dem Elektrolyt durch Auswählen einer anfänglichen CaO-Konzentration, die hoch genug ist, beinhaltet.

Revendications

1. Procédé de réduction électrochimique d'un oxyde de titane sous forme solide en une cellule électrolytique qui inclut une anode, une cathode, un électrolyte fondu basé sur $CaCl_2$ contenant du CaO, et une matière de charge d'oxyde de titane en contact avec l'électrolyte fondu, le procédé électrochimique incluant l'application d'un potentiel électrique sur l'anode et la cathode et la réduction électrochimique de la matière de charge de l'oxyde de titane en contact avec l'électrolyte fondu et la production d'un matériau réduit, le procédé étant **caractérisé par** le contrôle du Procédé par le contrôle de la concentration de CaO dans l'électrolyte par le contrôle du rapport de masse de l'électrolyte et de l'oxyde de titane dans la cellule, devant être d'au moins 10:1, la concentration initiale en CaO étant inférieure à 0,3 % en poids dans l'électrolyte et l'oxyde de titane étant sous la forme de pastilles et/ou de poudres possédant une superficie spécifique de 0,1 à 100 m^2/g d'oxyde de titane.

2. Procédé selon la revendication 1, incluant le contrôle de la concentration de CaO dans l'électrolyte en maintenant la concentration de CaO dans l'électrolyte entre les limites de concentration inférieure et supérieure.

3. Procédé selon la revendication 2, incluant le maintien de la concentration de CaO dans l'électrolyte à ou en dessous d'une concentration maximale en CaO de 0,5 % en poids.

4. Procédé selon la revendication 2, incluant le maintien de la concentration de CaO dans l'électrolyte à ou en dessous d'une concentration maximale en CaO de 0,3 % en poids.

5. Procédé selon l'une quelconque des revendications 2 à 4, incluant le maintien de la concentration de CaO dans l'électrolyte à ou au-dessus d'une concentration minimale en CaO de 0,005 % en poids.

6. Procédé selon l'une quelconque des revendications 2 à 4, incluant le maintien de de la concentration de CaO dans l'électrolyte à ou au-dessus d'une concentration minimale en CaO de 0,05 % en poids.

7. Procédé selon l'une quelconque des revendications 2 à 4, incluant le maintien de de la concentration de CaO dans l'électrolyte à ou au-dessus d'une concentration minimale en CaO de 0,01 % en poids.

8. Procédé selon la revendication 2, incluant le maintien de la concentration de CaO dans l'électrolyte dans la plage de 0,1 à 0,3 % en poids.

9. Procédé selon la revendication 2, incluant le maintien de la concentration de CaO dans l'électrolyte dans la plage de 0,15 à 0,25 % en poids.

10. Procédé selon l'une quelconque des revendications précédentes, incluant le contrôle de la concentration de CaO dans l'électrolyte par référence à la superficie de contact de l'oxyde de titane avec l'électrolyte.

11. Procédé selon l'une quelconque des revendications précédentes, incluant le contrôle de la concentration de CaO dans l'électrolyte par référence à la superficie de contact de l'oxyde de titane avec l'électrolyte lorsque considéré dans le contexte de la masse de l'électrolyte et de la masse de l'oxyde de titane.

12. Procédé selon l'une quelconque des revendications précédentes, incluant le contrôle de la concentration de CaO dans l'électrolyte par le contrôle du rapport de masse de l'électrolyte et de l'oxyde de titane dans la cellule.

13. Procédé selon l'une quelconque des revendications précédentes, dans lequel la concentration initiale en CaO est inférieure à 0,2 % en poids dans l'électrolyte.

14. Procédé selon l'une quelconque des revendications précédentes, incluant le contrôle de la concentration de CaO dans l'électrolyte par l'ajout de CaO à l'électrolyte au cours du procédé.

15. Procédé selon l'une quelconque des revendications précédentes, incluant le contrôle de la concentration de CaO dans l'électrolyte par la sélection d'une concentration initiale en CaO qui est suffisamment élevée.

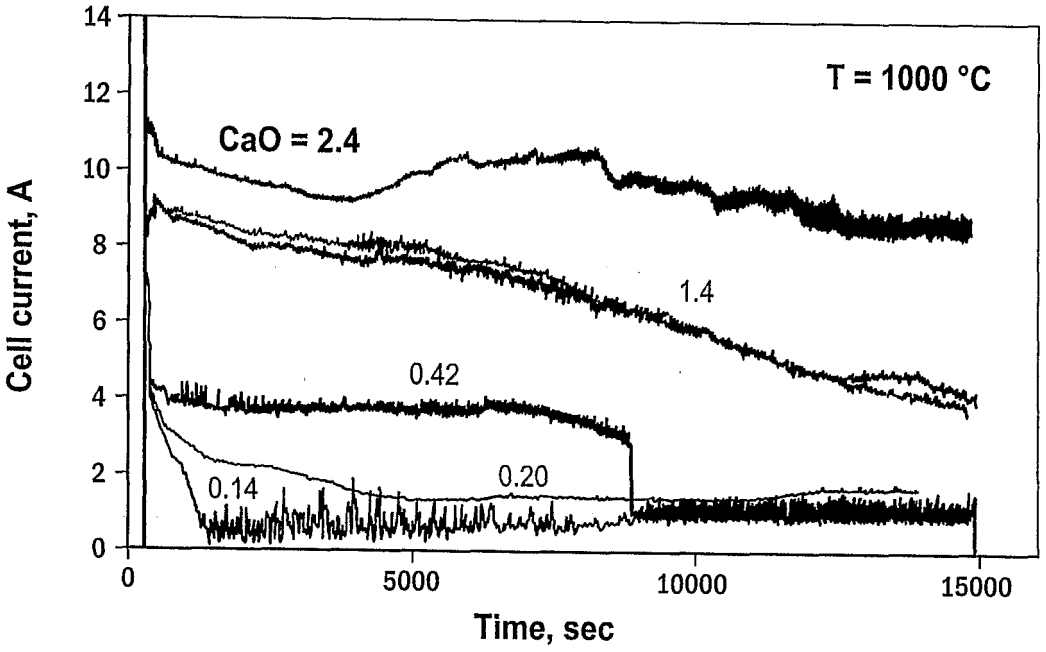

T = 1000 °C
CaO = 2.4
1.4
0.42
0.20
0.14
Cell current, A
Time, sec
0
2
4
6
8
10
12
14
0
5000
10000
15000

FIG. 1

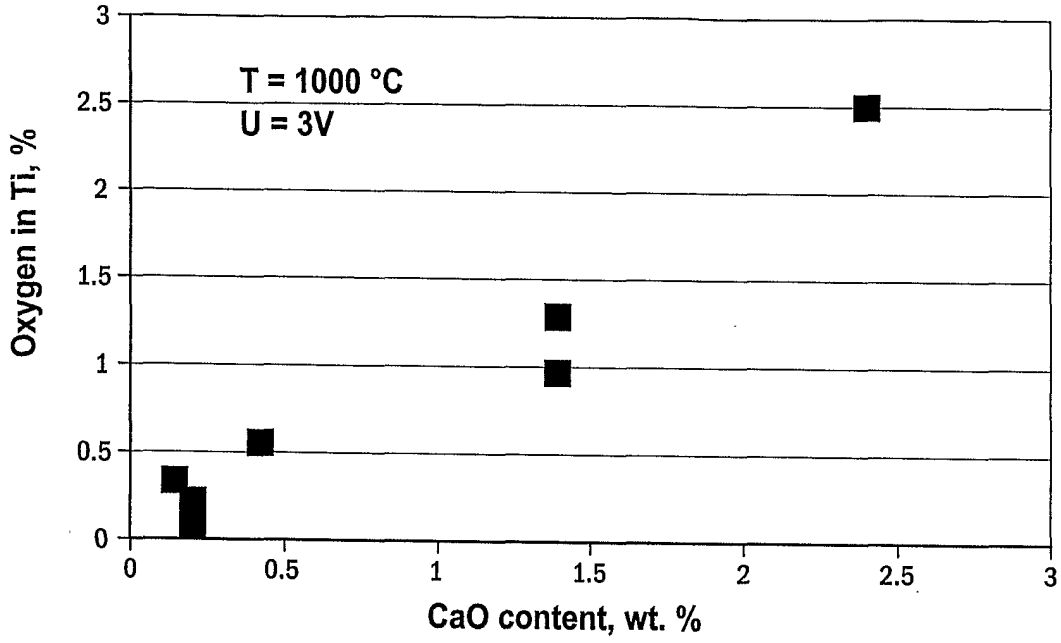

T = 1000 °C
U = 3V
Oxygen in Ti, %
CaO content, wt. %
0
0.5
1
1.5
2
2.5
3

FIG. 2

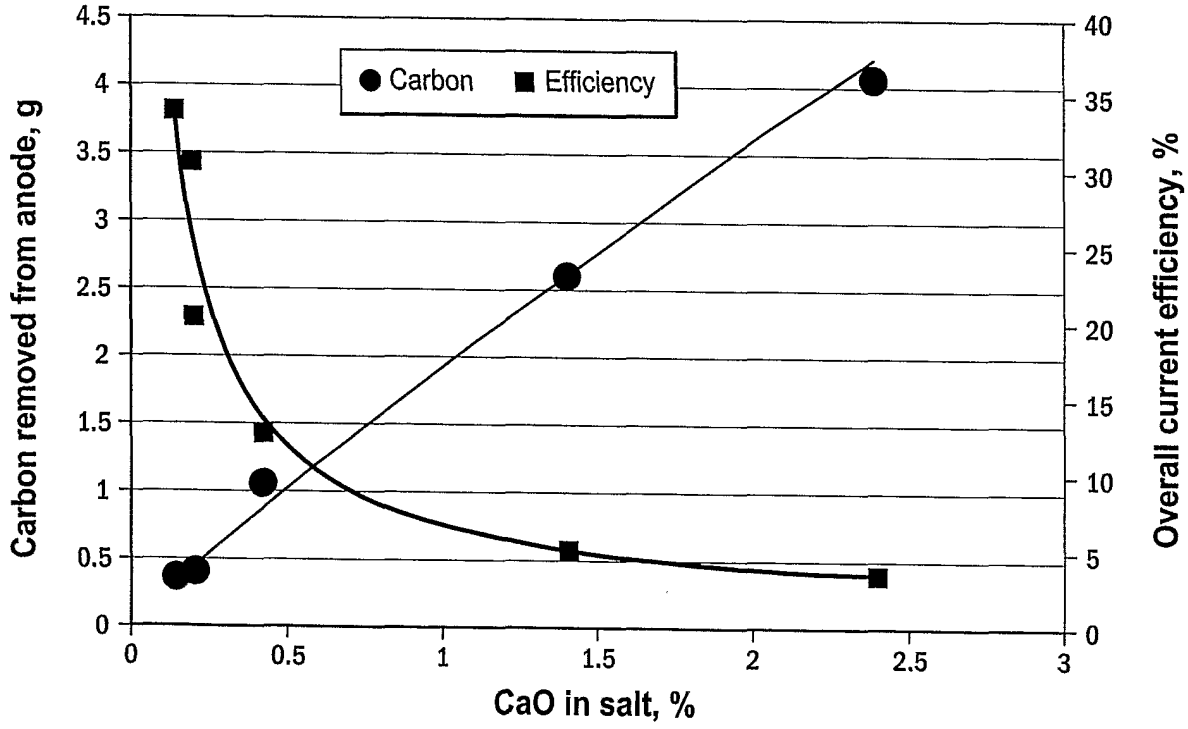

Carbon
Efficiency
Carbon removed from anode, g
Overall current efficiency, %
CaO in salt, %
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
0
5
10
15
20
25
30
35
40

FIG. 3