TITANIUM METAL PRODUCTION VIA OXYCARBIDE ELECTROREFINING

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Keywords: titanium, electrodeposition, electrorefining, electroreduction, oxycarbide, powder

ABSTRACT

A combined electroreduction and refining process has been employed to produce pure titanium metal from an impure feedstock. The process involves the anodic dissolution of titanium oxycarbide with cathodic deposition of titanium in a eutectic mixture of NaCl:KCl at 973-1173K. Characterisation includes scanning electron microscopy, energy dispersive X-ray analysis, X-ray diffraction, and inductively coupled plasma. Deposits consisted of pure titanium metal crystallites, sponge, or powder with a particle size range of 1-30µm.

INTRODUCTION

Global titanium sponge consumption is currently estimated at 150 kTpa and is predicted to grow by 6% annually until 2015 to >200 kTpa. This growth has resulted from an unprecedented worldwide demand for high-performance materials with titanium offering a unique combination of extremely desirable properties; these include its high strength-to-weight ratio, corrosion resistance, refractory nature, biocompatibility, and cryogenic capabilities\cite{1}.

Although titanium is plentiful (it is the 4\textsuperscript{th} most abundant engineering material in the Earth’s crust\cite{2}) and despite its many advantages, the cost of extraction has so far remained a barrier for all but the most demanding applications (e.g. Aerospace). Consequently, the Kroll Process has dominated as the industrial scale extraction method for over 60 years, despite more than a century of research into the topic. Of the potential successors, electro-extractive processes are considered the most likely both technologically and economically.

In addition to the expensive reduction, a significant contributor to the cost of extraction is the requirement for a highly purified titanium tetrachloride (TiCl\textsubscript{4}) feedstock to ensure complete removal of impurities present in the original ore. Whilst some high purity (>92%) TiO\textsubscript{2} ores do exist (e.g. Rutile), the vast majority of global resources (>90%) are present as FeTiO\textsubscript{3} (Ilmenite) which has a TiO\textsubscript{2} content of just 35-60\%. Figure 1 shows the global production of these titanium ores with the insert providing a breakdown by country.
Since the publication of the FFC-Cambridge Process\textsuperscript{[3]} in 2000, an intensive worldwide research programme was undertaken to develop novel titanium extraction techniques\textsuperscript{[4]}. A decade on, only limited progress been made and many of the proposed processes still necessitate similarly expensive feedstock to the Kroll Process, severely limiting their cost reduction potential. As an example, the electro-extractive FFC-Cambridge Process, the flagship Kroll replacement technology, is unable to accept any significant cationic impurities in the feedstock as these would be retained in the final product. It should be noted, however, that whilst this is a bane for pure metal production, it is a boon for producing alloys, where the FFC-Cambridge process understandably excels.

A promising competitor is the Chinuka Process\textsuperscript{[5]} which is a novel hybrid electroreduction-refining method: the process is illustrated schematically in Figure 2.
Historically, titanium oxycarbide (Ti₅CO) has typically been prepared by solid state carbothermic reduction of pigment grade TiO₂. Several researchers have subsequently electrolysed this material in a molten eutectic bath of NaCl:KCl at temperatures of ≥973K[6-22]. Anodic dissolution of the Ti₅CO produces either Ti²⁺[8] or Ti³⁺[9, 12] ions which are plated out at the cathode to generate a powder, sponge, or dendritic product. The Chinuka Process extends this concept by utilising impure titanium ores as a feedstock and thereby integrating electrorefining into the reduction process. Elements more noble than titanium can be selectively retained at the anode to form an extractable sludge, whilst those less noble can be retained in the electrolyte. A particular advantage is the ability to use lower grade ores that are prohibitively impure for other titanium extraction methods, including the capability to handle both cationic and anionic impurities. In addition the Chinuka Process is able to treat ores that exhibit particular physical or chemical characteristics, such as small particle size or significant CaO concentrations, which would otherwise prevent them from being upgraded to pigment grade TiO₂. The possibility to directly produce titanium powders is also extremely attractive due to their significant premium.

In this paper, results are presented from ongoing research on the Chinuka Process at the University of Cambridge. The production and electrolysis of oxycarbide anodes is discussed together with analysis of the anode and cathodic products by SEM/EDX and XRD. ICP analysis of the salt is also discussed.

**EXPERIMENTAL**

Magnetically separated rutile TiO₂ ores (White Mountain Titanium Corp.) with an average particle size of 70µm were blended with graphite powders (1-2µm, Aldrich) in a 1:2 mole ratio. This mixture was then dry ball milled with ZrO₂ media for 24 hours, to an average particle size of 5-15µm. These powders were loaded into an alumina crucible and reacted at 1773-1873K for 4 hours under a vacuum or flowing argon to generate titanium oxycarbide (Ti₂CO) according to the following reaction:

\[ 2\text{TiO}_2 + 4\text{C} \rightarrow \text{Ti}_2\text{CO} + 3\text{CO}(g) \]

The Ti₂CO products were dark red/orange friable agglomerates which were crushed to a powder before being uniaxial pressed. These green preforms were sintered at >1873K for 4 hours under vacuum or flowing argon to produce the final anodes for electrolysis. Due to pore formation during the carbothermic reduction stage, densification sintering was performed separately to improve the mechanical strength and reduce the porosity of the anode. Final porosities were between 25-40% based on external measurements with an estimated theoretical density of 4.94gcm⁻³[11]. Figure 3 shows a photograph of a Ti₂CO anode (left) and an SEM micrograph of the interior (right).
A eutectic mixture of 44% by mass of NaCl and 56% by mass KCl was loaded into an alumina crucible which was placed in a water cooled Inconel retort and heated under vacuum to 723K. The retort was then flushed with argon at 200ml/min and heated to the process temperature. Pre-electrolysis was conducted at 2.8V between two graphite rods (HK-0, Tokai Carbon Europe) until a low and stable background current was achieved, normally ~20mA.

Oxycarbide electrolysis was typically conducted in a 2-electrode arrangement although a AgCl reference electrode or a graphite, molybdenum or titanium pseudo-reference electrode was often employed to monitor the achieved potentials. The anode was either a pure Ti₂CO rod or a carbon based conductive holder containing Ti₂CO pellets (Figure 4). The cathode was a rod of steel, titanium, nickel, or molybdenum.

Electrolysis was performed at 1.8-3.0 V for 4–24 hours. Following electrolysis the electrodes were raised from the salt under argon and the furnace cooled. The electrodes were then washed in water, filtered, dried and analysed.
RESULTS AND DISCUSSION

Ti$_2$CO formation was validated by XRD analysis of feedstock materials sintered at different temperatures for 4 hours under vacuum (Figure 5). Starting with an admixture of carbon and TiO$_2$, reduction can be seen to go through the formation of Ti$_2$O$_3$ at temperatures below 1673K, followed by the formation of a single phase TiC$_x$O$_{1-x}$ with excess carbon. Further elevation of temperatures above 1773K consumes the free carbon with a detectable peak shift in the single phase to the left. The TiC$_x$O$_{1-x}$ peaks are found to lie between those of TiC and TiO as would be expected with a solid solution TiC:TiO phase. The products have been confirmed by carbon and oxygen analysis (combustion and Leco respectively) and are additionally supported by mass loss measurements. No additional products were detected at temperatures up to 2373K.

![XRD patterns](image)

Figure 5. XRD patterns of 1:2 mole ratio rutile ore/C mixtures, sintered at various temperatures for 4 hours under vacuum.

Ti$_2$CO electrolysis followed the previously outlined methodology. A typical chronoamperogram is given in Figure 6, obtained during electrolysis at applied cell voltage of 3.0V with a molybdenum rod cathode. An initially high electrolysis current is seen to decline to a baseline plateau value indicating the complete consumption of the Ti$_2$CO.
Figure 6. Chronoamperogram showing electrolysis of Ti$_2$CO at 3.0V

Figure 7 (left) shows an SEM micrograph of a titanium deposit obtained through electrolysis which exhibits a somewhat crystalline microstructure. The crystallites are approximately 20-30µm. XRD analysis in Figure 7 (right) confirms a pure titanium metal is obtained.

Figure 7. SEM micrograph of titanium deposit (left) and XRD showing pure titanium (right)

Figure 8 shows the Inductively Coupled Plasma (ICP) analyses of a rutile feedstock and a cathodic product. A significant reduction in the concentration of all major impurities can be seen in the product by contrast to the feedstock material. The titanium content has been upgraded from ~96.4% in the ore to >99.7% in the deposit. The retention of comparatively high iron levels is thought to have arisen from the use of a steel cathode in this case.
Given that the feedstock impurities are not transferred to the cathodic product, ICP was conducted on the salt over the course of 4 consecutive electrolyses (Table 1). The low concentrations of the impurities probably prevents their co-deposition, but their gradual build-up will eventually necessitate salt exchange or cleaning, e.g. by titanium gettering. Concentrations may also be kept low through the formation of volatile chlorides, such as AlCl$_3$ or SiCl$_4$.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Al (ppm)</th>
<th>Ca (%)</th>
<th>Cr (%)</th>
<th>Fe (%)</th>
<th>Si (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<tr>
<td>Electrolysis 1</td>
<td>0.00176</td>
<td>0.33831</td>
<td>0.00558</td>
<td>0.00104</td>
<td>--</td>
</tr>
<tr>
<td>Electrolysis 2</td>
<td>0.00122</td>
<td>0.76268</td>
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<td>Electrolysis 3</td>
<td>0.00166</td>
<td>1.38767</td>
<td>0.03570</td>
<td>--</td>
<td>0.05111</td>
</tr>
<tr>
<td>Electrolysis 4</td>
<td>0.00219</td>
<td>1.62361</td>
<td>0.03753</td>
<td>0.00407</td>
<td>0.05483</td>
</tr>
</tbody>
</table>

Table 1. Composition of impurities in salt following consecutive electrolyses analysed by ICP

**CONCLUSIONS**

Impure ores can be partially carbothermally reduced to form Ti$_2$CO with some residual impurities. Electrolysis of this Ti$_2$CO in a molten eutectic mixture of NaCl:KCl at 973-1183K, a method known as the Chinuka Process, can produce a titanium product with a significantly lower impurity content than that of the feedstock. This patented combined electroextraction-refining process permits the use of lower cost feedstocks than competing titanium extraction methods, whilst offering the potential for direct titanium powder production.

**ACKNOWLEDGEMENTS**

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Titanium Metal Production via Oxycarbide Electrorefining

The Chinuka Process: Electroextraction-refining

Daniel Jewell, Mike Kurtanjek, and Derek Fray
Founded in 2003, White Mountain Titanium Corporation (WMTC), an exploration stage titanium mining company, is the parent company of Chinuka Ltd.

WMTC is developing the Cerro Blanco Project; over 8,225 hectares located west of Vallenar in the Atacama region, northern Chile.

The mine has 32.9 Mt of measured & indicated resources at 2.1% TiO₂; mine life of 6-7 yrs, up to 17.5 kt/day.

Rutile and Ilmenite ores available.
Agenda

1. Titanium overview
2. Extraction techniques
3. Chinuka Preparation
4. Chinuka Process
## Overview of titanium market

### Titanium ores by supply and end usage (2011)

<table>
<thead>
<tr>
<th>Ores</th>
<th>End use</th>
<th>Percent of volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>Natural rutile</td>
<td>6,700 tonnes</td>
<td>80</td>
</tr>
<tr>
<td>Ilmenite</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Rutile pigment</td>
<td>0</td>
<td>20</td>
</tr>
</tbody>
</table>

### Ilmenite – Largest feedstock
- Total world resources more than 1.8bn tons
- 90% of global titanium mineral consumption
- Ores typically contain 35-60% TiO$_2$
- Ilmenites containing >60% TiO$_2$ are known as Leucoxene
- Largest producer is South Africa

### Rutile pigment – Largest product
- Global demand over 5.7 million tonnes p.a.
- Accounts for 95% of global titanium ore end use
- Growing demand; up 8% from 2010 – 2011
- Predominantly used in paints, coatings, plastics, & paper
- Largest producer is the United States
Titanium metal market

US Titanium metal usage and world capacity (2011)

Titanium metal sponge
- 5% of world titanium ore usage
- Fast growing (6% p.a.) market
- 283,000 tpa worldwide capacity
- Largest producer is China

Titanium minimum buy weight in aircraft

787 Dreamliner – 116 tonnes titanium
But the real question is…

How do you get from this…

…to this?

Titanium ore

Titanium metal
Titanium: From ore to pure

1. **Ilmenite**
   - FeTiO$_3$
   - Sorel process

2. **Leucoxene**
   - Fe$_2$O$_3$.TiO$_2$
   - Becher process

3. **Natural rutile**
   - TiO$_2$

4. **Titanium slag**
   - 70-85% TiO$_2$
   - Benelte process

5. **Synthetic rutile**
   - 85-96% TiO$_2$

6. **Sulphate process**
    - TiCl$_4$
    - >99.9%

7. **Chloride process**
    - Rutile pigment
    - >99% TiO$_2$

8. **Kroll process**

9. **UGS process**

10. **FFC-Cambridge Process**

11. **Ti**
Our challenge

XRF analysis of Cerro Blanco ores

<table>
<thead>
<tr>
<th></th>
<th>Rutile (wt. %)</th>
<th>Ilmenite (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Standard</td>
<td>Fine</td>
</tr>
<tr>
<td>TiO₂</td>
<td>96.4</td>
<td>97.2</td>
</tr>
<tr>
<td>FeO/Fe₂O₃</td>
<td>0.66</td>
<td>0.77</td>
</tr>
<tr>
<td>SiO₂</td>
<td>1.13</td>
<td>0.88</td>
</tr>
<tr>
<td>MgO</td>
<td>0.04</td>
<td>0.02</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.30</td>
<td>0.18</td>
</tr>
<tr>
<td>MnO₂</td>
<td>&lt;0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>V₂O₅</td>
<td>0.28</td>
<td>0.30</td>
</tr>
<tr>
<td>Nb₂O₅</td>
<td>0.30</td>
<td>-----</td>
</tr>
<tr>
<td>CaO</td>
<td>0.06</td>
<td>0.12</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>0.34</td>
<td>0.34</td>
</tr>
</tbody>
</table>

Challenge #1:
At <53µm, ultrafine rutile is too fine for the chloride route as it is lost from the fluidised bed.
This is important as it comprises ≈10% of the deposit.

Challenge #2:
Calcium oxide content is too high for conventional processing.

*Chloride route:*
CaCl₂ formation defluidises bed

*Sulphate route:*
CaSO₄ formation is problematic.
Chinuka: Ore to metal

- **Ilmenite**: FeTiO$_3$
  - Sorel process
  - Benelite process

- **Leucoxene**: Fe$_2$O$_3$.TiO$_2$
  - Becher process

- **Natural rutile**: TiO$_2$

- **Titanium slag**: 70-85% TiO$_2$
  - Sulphate process
  - Chloride process

- **Synthetic rutile**: 85-96% TiO$_2$
  - Kroll process

- **Chinuka Process**: 

- **Pigment rutile**: >99% TiO$_2$
  - Chloride process

- **Titanium slag**: 70-85% TiO$_2$
  - Sulphate process
  - Chloride process

- **Benelite process**
- **Becher process**
- **Kroll process**

- **Sulphate process**
- **Chloride process**

- **Titanium slag**

- **Sulphate process**
- **Chloride process**

- **Chinuka Process**
- **Ti**

- **Sulphate process**
- **Chloride process**

- **Sulphate process**
- **Chloride process**

- **Sulphate process**
- **Chloride process**

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- **Sulphate process**
- **Chloride process**
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Titanium extractive history

1791: Titanium discovered by W. Gregor

1910: First extracted at 99.9% purity from TiCl₄ by Hunter using liquid Na

1925: First ultra high purity Ti from thermal decomposition of TiI₄ by van Arkel & de Boer

1940: Kroll Process

1950 – 80’s: Various new processes developed

1990 – 00’s: Chinuka Process patented

2011: One-Step Electrorefining & Electroextraction from Ti₂CO

Major Industrial Process
- Kroll Process
- Mg-based reduction of TiCl₄

Electrolytic
- FFC-Cambridge
  - OS
  - GTT HT
  - MOE
  - Polar Ti
  - QIT

Non-Electrolytic
- Armstrong
  - PRP
  - SRI
  - ITP
  - ITT
  - CSIRO

Electrolytic
- GTT LT
- Dow-Howmett
- NJ Zinc Company
- Fluoride

Non-Electrolytic
- AlTi
- Plasma Reduction
- Aerosol Reduction

Limited success
The Chinuka Process

Carbothermic reduction

Vacuum or inert gas

≥1500°C

Ti₂CO

Green forming

Titanium

Electroextraction

Electrorefining

Anode

Ti₂CO = 2Ti⁺ⁿ + 2nₑ⁻ + CO(g)

Cathode

Ti⁺ⁿ + nₑ⁻ = Ti

Molten NaCl:KCl electrolyte

Titanium deposit

Extract

Wash

Crush

Carbon

Ti Ore

Monday, October 08, 2012

Daniel Jewell, University of Cambridge & WMTC

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A closer look at electrorefining

**Anode**

\[ \text{Ti}_2\text{CO} = 2\text{Ti}^{x+} + 2 ne^- + \text{CO}_{(g)} \]

Co-deposition may occur if potential is too negative.

**Cathode**

\[ \text{Ti}^{x+} + ne^- = \text{Ti} \]

Ca, Sr, Ba

**Order of dissolution**

- More noble impurities will form an anode sludge.
- Less noble impurities will dissolve into the salt.

**Order of deposition**

- Less noble impurities should remain in the salt.
Overview

- Invented in 1996 by Derek Fray, Tom Farthing and George Chen at the University of Cambridge
- Electro-deoxidation of metal oxides in a CaCl₂ electrolyte
- Flagship replacement technology for the Kroll Process

Important notes

- Requires pure oxide feedstocks; particularly sensitive to cationic impurities
- Excels in the production of high value metals and alloys
- Employs CaCl₂ which has a high oxygen solubility and is highly hygroscopic

Cathode: \( \text{TiO}_2 + e^- = \text{Ti} + 2\text{O}^{2-} \)
Anode: \( x\text{O}^{2-} + \text{C} = \text{CO}_x + x2e^- \)
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Stage 1:
Partial carbothermic reduction of rutile ores under a vacuum or inert atmosphere at >1500°C

$$2\text{TiO}_2 + 4\text{C} = \text{TiO}:\text{TiC} + 3\text{CO}_\text{(g)}$$

Stage 2:
Anode green forming from oxycarbide followed by sintering; usually performed above >1600°C
**Key points**

- Ti$_2$CO is a good electrical conductor
- Strongly interconnected nodular network provides good mechanical strength
- Low porosity is key to microstructural stability during electrolysis; currently <25% porosity is achieved at 1700°C
What about the impurities?

### Issue?
In addition to Ti$_2$CO formation, impurity reaction products need to be considered.

### Solution?
Depending on level of impurity these can either be handled in electrolysis, or post-sinter processing can include magnetic separation and acid leaching.

### Exceptions?
Ilmenite contains so much iron that slagging can occur.

<table>
<thead>
<tr>
<th>Reaction at 1500°C</th>
<th>Post-reaction processing</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiO$_2$</td>
<td>2TiO$_2$ + 4C = Ti$<em>2$CO + 3CO$</em>{(g)}$</td>
</tr>
<tr>
<td>FeO</td>
<td>FeO + C = Fe + CO$_{(g)}$</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>Fe$_2$O$<em>3$ + 3C = 2Fe + 3CO$</em>{(g)}$</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>No Reaction</td>
</tr>
<tr>
<td>MgO</td>
<td>No Reaction</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>No Reaction</td>
</tr>
<tr>
<td>MnO</td>
<td>3MnO + 4C = Mn$<em>3$C + CO$</em>{(g)}$</td>
</tr>
<tr>
<td>V$_2$O$_5$</td>
<td>V$_2$O$_5$ + 6C = V$<em>2$C + 5CO$</em>{(g)}$</td>
</tr>
<tr>
<td>Nb$_2$O$_5$</td>
<td>Nb$_2$O$<em>5$ + 7C = 2NbC + 5CO$</em>{(g)}$</td>
</tr>
<tr>
<td>CaO</td>
<td>No Reaction</td>
</tr>
<tr>
<td>Cr$_2$O$_3$</td>
<td>1.5Cr$_2$O$_3$ + 6.5C = Cr$_3$C$<em>2$ + 4.5CO$</em>{(g)}$</td>
</tr>
</tbody>
</table>
A possible solution to the Ilmenite problem: blending

The approach
By blending rutile and ilmenite ores, the average iron concentration can be controlled

Advantages
• Eliminates slagging
• Provides tunable impurity levels
• Dilutes feedstock cost

However...
Significant iron contents are still problematic to electrolysis and should be reduced post-reaction
Another aspect of electrorefining: Electrolyte enrichment

Pre-enrichment of electrolyte is typically employed in electrorefining to:

- Reduce deposition overpotential
- Provide sufficient relevant ions to support current
- Prevent decomposition of electrolyte
- Affect electrodeposited product morphology

— This is not essential for process operation, but is preferred

For titanium, typically achieved by high temperature in-situ reaction: \( \text{TiCl}_4 + \text{Ti} = \text{TiCl}_2 \) – Safety Issues!

— Alternative? – Use reactive chlorides of Sn, Ni, Co, Zn, Cu etc.
Conclusions

- Enrichment successful up to at least 10wt%
- Low copper impurities with good physical separation
- Dissolved titanium stable over very long periods
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Electrolysis of Ti$_2$CO

**Experimental conditions**
- Electrolyte: Eutectic NaCl:KCl
- WE = Mo; CE = Ti$_2$CO; RE = Ti
- Temperature: 700 - 900°C
- Time: 2 - 24 hrs
- Voltage: 1.8 V – 3.0 V
- Current density: 50 – 500 mA/cm$^2$
- Current efficiency: 15 - 50%

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**Experimental setup**

- Stainless steel retort (250mm ID)
- Extractor hood
- Data recorder
- Furnace controller
- Power supply & Data acquisition unit
- Vacuum pump
• Anodic gas bubbling observed throughout electrolysis
• Ti sponge-like product adhered to cathode or retained in crucible
• Widely variable product form and size – for nodules typically 1-5µm
• Following washing (H₂O), ‘sponge’ readily crushed to powder
• Salt enriched with Ti – green (Ti²⁺) or blue (Ti³⁺)
• Oxygen content around 1wt.% for these samples*
Comparison of products - Macroscopic

Cathodic deposit - Ti Anode

Cathodic deposit - Ti$_2$CO Anode
Comparison of products - Microscopic

Cathodic deposit - Ti Anode

- Nodules: 2-3 µm
- Particulates: >100 µm

Cathodic deposit - Ti$_2$CO Anode

- Nodules: 1-3 µm
- Crystallites: 20-30 µm
Electrorefined product

- Significant reduction in all cationic impurities with low final values
- Increase in purity from 96.5% feedstock to >99.5% product
- Potential for further purification by process optimisation

Electrolyte

- Gradual build up of impurities; particularly Ca
- Minimal retention of Al and Si
  - Likely due to formation of volatile chlorides
- Residual impurities may be gettered by Ti sponge addition to crucible
A note on oxidation and pyrophoricity

Analysis of critical particle size

Assumption:
Native oxide thickness = 5nm

OXYGEN WT.%

PARTICLE SIZE (MICRONS)

Target core OD

0.1 1 10 100

0.11 wt.% 1100 ppm
0.011 wt.% 110 ppm

10.3 wt.% 103000 ppm

1.1 wt.% 11000 ppm

Core OD

Particle OD

Pyrophoric size limit

CP-4 = 2.8µm
CP-3 = 3.2µm
CP-2 = 4.5µm
CP-1 = 6.1µm

Core OD

Target core OD
Brief overview
Comparison of principle titanium extraction processes

<table>
<thead>
<tr>
<th></th>
<th>Chinuka Process</th>
<th>Kroll Process</th>
<th>FFC – Cambridge Process</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reaction type</td>
<td>Electrolytic</td>
<td>Chemical</td>
<td>Electrolytic</td>
</tr>
<tr>
<td>Operation</td>
<td>Semi-continuous operation</td>
<td>Batch operation</td>
<td>Semi-continuous operation</td>
</tr>
<tr>
<td>Feedstock</td>
<td>Broad range of TiO₂</td>
<td>TiCl₄</td>
<td>High-purity TiO₂</td>
</tr>
<tr>
<td>Electrolyte</td>
<td>Non-hygroscopic NaCl:KCl eutectic; low oxygen solubility</td>
<td>-</td>
<td>Highly hygroscopic CaCl₂ electrolyte; high oxygen solubility</td>
</tr>
<tr>
<td>Operating condition</td>
<td>Operable at voltages well below electrolyte decomposition</td>
<td>-</td>
<td>Typically high cell voltages close to electrolyte decomposition</td>
</tr>
<tr>
<td>Post production</td>
<td>Water washing</td>
<td>Vacuum distillation</td>
<td>Water washing</td>
</tr>
<tr>
<td>Product type</td>
<td>Ti powder or crystallites</td>
<td>Ti sponge</td>
<td>Ti powder or near-net shape</td>
</tr>
<tr>
<td>Scope for development</td>
<td>Emerging technology</td>
<td>Mature technology</td>
<td>Semi-mature technology</td>
</tr>
<tr>
<td>Challenges</td>
<td>Eventually requires salt replacement or purification</td>
<td>Requires electrolytic regeneration of Cl₂ and Mg</td>
<td>Involves complex suboxide and titanate/titanite chemistry</td>
</tr>
</tbody>
</table>
Final words and the next steps...

Elevator pitch

• The Chinuka Process is a novel electrochemical method which simultaneously combines electroextraction with electorefining to produce high purity titanium
• The process offers a number of specific advantages over existing technologies, the most significant of which is the ability to directly treat impure ores

The future

• Continued scale-up of the process with rutile ore feedstock
• Further investigation into the electrolytic performance of ilmenite & blended ores
• Application of the process to non-titanium ores, including niobium (Nb), tantalum (Ta), and the rare earth metals
Question time
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