A Novel Chemical Pathway for Ti Production

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Lead Institution: University of Utah
Partners: Boeing and Alcoa Ti

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A breakthrough technology for making low cost Ti powders
Agenda

- Team Intro
- Technical Concept
- Technical Progress to date
- TEA Highlights
- Demo Requirements
- Future Goals/Closing Thoughts
- Q&A
The flagship research University in the state of Utah.

Dr. Ali Yousefian, Technical Fellow, Boeing Research & Technology
Dr. Don Li, Senior Manager, R&D, Alcoa Ti

**Project Summary**

- Ti sponge (primary metal) is expensive (~$10/Kg Ti sponge).
  Ti powder is more expensive ($30-80/Kg CP-Ti powder).
  Spherical Ti alloy powder for 3D printing is extremely expensive ($200-500/Kg).

- A new chemical pathway was found and demonstrated in a lab scale that can produce normal non-spherical Ti powder at 1/3 or less of its current cost. The process is termed hydrogen assisted magnesiothermic reduction of TiO₂ (HAMR)

- A novel process (GSD) is also developed to make spherical Ti alloy powder for additive manufacturing at a fraction of the cost of the current technologies.

- Both HAMR and GSD processes are now at TRL 4. They are ready for scale up.

**Current goals**

- Produce 10Kg of powder for industrial partners to assess the product quality and market potentials
- Develop a continuation plan to secure funding for pilot production research
Current Processes for Production of Titanium

- **Purified TiO\textsubscript{2}**
  - Ti-slag, Syn. rutile, UGS, ...
  - TiCl\textsubscript{4}
  - Kroll
    - Other R&D processes
      - Armstrong process / ITP
        - Electrochemical FFC process / Metalysis
  - HDH
    - Ti sponge
    - Ti powder
  - Other R&D processes
Utah Processes from UGS to TiO2 and from TiO2 to Ti

- Ti-slag, Syn. rutile, UGS, ...
- Ti powder

Alkaline roasting
Solution hydrolysis

Hydrogen assisted Mg reduction of TiO₂ (HAMR)

Purified TiO₂

Mg reduction & deoxygen
Challenges – Solutions

**Challenges**

- TiO₂ is extremely stable
- H₂ cannot reduce TiO₂
- Mg cannot reduce TiO₂ to lower than 2 wt%O

**Science**

- Scientific discovery:
  
  *Hydrogen destabilizes Ti-O, making the reaction of Mg with Ti-O from being thermodynamically unfavorable to being favorable.*

**Technology**

- Mg reduction in H₂ atmosphere
- Reduction in molten salt – kinetics
- Two step process: reduction and deoxygenation

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Graph showing oxygen potential vs. temperature for TiO₂, TiO, MgO, and CaO. The graph indicates the stability and reducibility of TiO₂ under varying conditions.
The HAMR Process and Product

Impurity contents in final Ti powder

<table>
<thead>
<tr>
<th>Weight percent (%)</th>
<th>Mg</th>
<th>Al</th>
<th>Fe</th>
<th>Si</th>
<th>Cl</th>
<th>O</th>
<th>N</th>
<th>C</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final Ti powder</td>
<td>&lt;0.1</td>
<td>&lt;0.03</td>
<td>&lt;0.10</td>
<td>&lt;0.04</td>
<td>&lt;0.1</td>
<td>&lt;0.12</td>
<td>&lt;0.02</td>
<td>&lt;0.03</td>
<td>&lt;0.03</td>
</tr>
<tr>
<td>ASTM-B299-13 (GP Ti sponge)</td>
<td>0.5</td>
<td>0.05</td>
<td>0.15</td>
<td>0.04</td>
<td>0.2</td>
<td>0.15</td>
<td>0.02</td>
<td>0.03</td>
<td>0.03</td>
</tr>
</tbody>
</table>

The ASTM standard for general purpose Ti sponge is met!

Purified TiO₂

As-reduced TiH₂

Deoxygenated Ti
Estimated energy consumption and CO$_2$ emission of HAMR process compared with Kroll process
Spherical Ti64 powder for 3D Printing

Current processes and challenges

- Ti is a popular metal for 3D printing.
- Spherical Ti powder needed for 3D printing is prohibitively expensive ($250-500/Kg)
- Current plasma atomization processes suffer from low yield and expensive feed stock material for atomization.

The new Utah GSD (granulation-sintering-deoxygenation process)

- GSD process is inherently high yield. Particle size distribution can be customized.
- GSD process uses low cost feed stock (e.g. scrap metal).
- Low oxygen – owing to a breakthrough deoxygenation technology developed as a part of this program.

Using GSD process, cost of powder is estimated at $20-50/Kg depending on feed stock material, as opposed to $250-500/Kg.
TEA Highlights

Market Needs

3D printing

Powder

CP-Ti powder market

Ti billets
## TEA Highlights

Projected prices based on new technology vs. current market prices based on Kroll process

<table>
<thead>
<tr>
<th>Product</th>
<th>Current market price (approx.)</th>
<th>Projected price based on the new technology</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>HAMR CP-Ti</td>
</tr>
<tr>
<td>Ti sponge (Kroll process)</td>
<td>$8-12 /Kg</td>
<td>N/A</td>
</tr>
<tr>
<td>Ti powder</td>
<td>$30-60/Kg (HDH CP-Ti powder)</td>
<td>$7-15/Kg (depend on scale)</td>
</tr>
<tr>
<td>Ti billet (preform for mill products)</td>
<td>$30-40/Kg (sponge-ingot-billet)</td>
<td>$15-25/Kg (powder-compaction-sintering)</td>
</tr>
<tr>
<td>Spherical Ti powder</td>
<td>$250-500/Kg</td>
<td>N/A</td>
</tr>
</tbody>
</table>
Demo Requirements

Commercialization strategy -

- Start-up company to focus on pilot scale and commercialization
- Short term to start with GSD spherical Ti alloy powder for additive manufacturing
- Long term goal to commercialize HAMR CP-Ti powder for current powder markets and eventually Ti billet market

Pilot scale development

- Current at TRL 4. Aiming to reach TRL 7 through a development pilot scale project.
- Pilot scale plant aims to produce powder at 10Kg/day.
- Develop sufficient quantity of materials to be qualified by customers and the industry
- Demonstrate the engineering feasibility of the process and be ready for large scale production
Future Goals / Closing Thoughts

- Two novel processes are developed for making
  - Conventional Ti powder for a wide range of applications
  - Spherical Ti alloy powder for additive manufacturing

- The new processes reduce costs of production by greater than 50%

- Current at TRL 4. Aiming to reach TRL 7 through a pilot scale development project.

- Seeking funding for scale up development project

- A start-up will be the vehicle of commercialization, in collaboration with the U of Utah.

- Focus on commercializing spherical Ti powder for AM/3D printing in the plus-up project phase

- In long term, commercialize HAMR pure Ti powder for Ti billet markets
QUESTIONS?
## TEA Highlights

### Comparison of the Utah-HAMR process to Kroll and FFC Processes

<table>
<thead>
<tr>
<th></th>
<th>Principle</th>
<th>Feed material</th>
<th>Reducing agent</th>
<th>Product quality</th>
<th>Morphology</th>
<th>Challenge</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kroll</td>
<td>Thermal chemical 2Mg+TiCl4=2MgCl2+Ti</td>
<td>TiCl4</td>
<td>Mg</td>
<td>Low to extremely low impurity</td>
<td>Sponge and sponge fine</td>
<td>Distillation is energy-intensive</td>
<td>Commercial</td>
</tr>
<tr>
<td>FFC</td>
<td>Electrochemical TiO2+4e-=Ti+2O²⁻</td>
<td>TiO₂</td>
<td>e⁻</td>
<td>O: 0.29%* C: 0.07% Ca: 0.13%</td>
<td>Partially sintered porous powder</td>
<td>Scaling-up**</td>
<td>Pilot plant</td>
</tr>
<tr>
<td>HAMR</td>
<td>Thermal chemical Mg+TiO2+H2=Ti-H-O +MgO Ti-H-O+Mg+H2=Ti-H +MgO</td>
<td>TiO₂</td>
<td>Mg</td>
<td>O:&lt;0.12% C:&lt;0.02% Mg: &lt;0.10%</td>
<td>Discrete HDH-like powder</td>
<td>Consistency needs to be demonstrated.</td>
<td>Kg scale lab-tested</td>
</tr>
</tbody>
</table>

* According to limited information in public literature. Company internal reality could be different.
Demo Requirements

- What does the demo phase of this project look like?
  - Scale
  - Cost Range
  - Projected Outcomes

- What partnership needs, if any, does your research require?
  - Note: this can be omitted if you don’t feel comfortable telling the group exactly where you are at the moment.
Future Goals / Closing Thoughts

‣ What is the ultimate end goal for this project?
‣ Where do you see yourselves in 5 years? 10?
‣ What is the prospective impact of this project from an industry perspective? Energy perspective? CO2?
‣ What would you like the audience to take away from this presentation?
TEA Highlights

![Diagram showing price and energy consumption for different types of titanium products.](image-url)