Consolidation of CP and Prealloyed Ti6Al4V Powder

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Process Schematic

Feed Systems

Armstrong Reactor

Na

NaCl Removal

Sodium Removal

NaCl Storage/Transfer

Primary Loop

TiCl₄

ACl₃

Sodium Circulator

Liquid/Solid Separation System

Na, Ti-6Al-4V, NaCl

NaCl Removal

NaCl

Powder Handling

Ti-6Al-4V

NaCl

Sodium

Na

Powder Processing

International Titanium Powder
Cristal US, Inc.

CRISTAL Global
Morphology of ITP Powder

- Oxygen: 1500 – 3500 ppm
- Nitrogen: 100 – 300 ppm
- Hydrogen: 30 – 100 ppm
- Carbon: 50 – 200 ppm
- Aluminum: 5 – 8 wt%
- Vanadium: 3.5 – 4.5 wt%
- Iron: 10 – 100 ppm
- Sodium: 10 – 300 ppm
- Chlorine: < 10 ppm
Oxygen Variability: *Process & Sample*

![Graph showing Oxygen Variability for different samples](image-url)
Sodium Distribution in Ti-6-4 Alloy

Process Capability of Na_Alloy (ppm)

<table>
<thead>
<tr>
<th>Process Data</th>
<th>LB</th>
<th>Target</th>
<th>USL</th>
</tr>
</thead>
<tbody>
<tr>
<td>LB</td>
<td>0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Target</td>
<td>30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>USL</td>
<td>100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample Mean</td>
<td>37.8409</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample N</td>
<td>44</td>
<td></td>
<td></td>
</tr>
<tr>
<td>StDev (Within)</td>
<td>32.4922</td>
<td></td>
<td></td>
</tr>
<tr>
<td>StDev (Overall)</td>
<td>45.7195</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- **Potential (Within) Capability**
  - Cp *
  - CPL *
  - CPU 0.64
  - Cpk 0.64

- **Overall Capability**
  - Pp *
  - PPL *
  - PPU 0.45
  - Ppk 0.45
  - Cpm 0.50

**Observed Performance**
- PPM < LB 0.00
- PPM > USL 90909.09
- PPM Total 90909.09

**Exp. Within Performance**
- PPM < LB *
- PPM > USL 27870.92
- PPM Total 27870.92

**Exp. Overall Performance**
- PPM < LB *
- PPM > USL 86982.41
- PPM Total 86982.41
Where is the Opportunity?

- Fabrication: 47%
- Chlorination: 9%
- Mg Reduction: 25%

MILL PRODUCT

2nd Melt: 3%

HDH Powder
PREP Powder
Gas Atomized Powder
Conventional Alloy Powders

TiO₂: 4%

VAR Melting Process
TITANIUM SPONGE
TITANIUM ALLOY INGOT (i.e. Ti₆Al₄V)

Sponge Fines
HDH Powder
PREP Powder
Gas Atomized Powder
Conventional Alloy Powders
Product Development Framework

**Powder Processing**
- Raw Materials
- Reaction
- Filtration
- Sodium Recovery
- Heat Treatment
- Milling
- Milled Powder

**Conversion**
- Chemical

**Microstructure**
- Microporosity
- \( \alpha/\beta \) Formation

**Mechanical Properties**
- Tensile Strength
- Ductility
- Fatigue
- Toughness

**Fabrication & Application**
Powder Characteristics

Average Particle size of 119µm

Passing (wt-%) vs. Size (microns)

Relative Tap Density and Relative Apparent Density vs. Sieve Cut (mesh)

Apparent Density vs. Tap Density
Milling – Impact on Chemistry

Oxygen Pickup: 200 to 400 ppm

Element Pick-up (wt-%)

N (wt-%)
H (wt-%)
Compaction of ITP Powders

**Graph 1:**
- Apparent density (g/cc)
- Compaction Pressure - TSI
- Flexural Green Strength - MPa

**Graph 2:**
- Green density (%)
- Compaction Pressure - TSI

**Graph 3:**
- Flexural Green Strength - MPa
- Compaction Pressure - TSI
- Materials: ITP CP-Ti, HDH - 1, HDH - 2

*Compacted at 50 TSI*
Compaction of ITP Powders

- **Green density** -
- **Sintered density** - 1250°C/90min

**Graphs:**
- **Density (%)**
  - ITP Ti6/4
  - HDH Ti6/4 (BE)
  - HDH Ti6/4 (PA)
- **Flexural strength (Mpa)**
  - ITP Ti6/4
  - HDH Ti6/4 (BE)
  - HDH Ti6/4 (PA)
- **Fracture strain (%)**
  - ITP Ti6/4
  - HDH Ti6/4 (BE)
  - HDH Ti6/4 (PA)
## Compaction of ITP Powders - CIP

<table>
<thead>
<tr>
<th>Powder</th>
<th>Pre-CIP Size</th>
<th>CIP Bag</th>
<th>CIP Parameters</th>
<th>Relative Green Density (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CP-Ti</td>
<td>1.125” ID x 4.75” int.</td>
<td>50 dur.</td>
<td>58 ksi x 1 min</td>
<td>86</td>
</tr>
<tr>
<td>CP-Ti</td>
<td>1.125” ID x 4.75” int.</td>
<td>65 dur.</td>
<td>58 ksi x 1 min</td>
<td>87</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>1.125” ID x 4.75” int.</td>
<td>50 dur.</td>
<td>58 ksi x 1 min</td>
<td>75</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>1.125” ID x 4.75” int.</td>
<td>65 dur.</td>
<td>58 ksi x 1 min</td>
<td>77</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>3” ID x 8” int.</td>
<td>50 dur.</td>
<td>58 ksi x 1 min</td>
<td>75</td>
</tr>
<tr>
<td>Ti-6Al-4V</td>
<td>5” ID x 12” int.</td>
<td>50 dur.</td>
<td>58 ksi x 1 min</td>
<td>70</td>
</tr>
</tbody>
</table>

**Compaction**

- **CP-Ti**: 1.125” ID x 4.75” int. / 65 duro.
- **Ti-6Al-4V**: 3” ID x 8” int. / 50 duro.
- **Ti-6Al-4V**: 5” ID x 12” int. / 50 duro.
Sintering of ITP Ti-6Al-4V Powders

Compacts were sintered at 1250°C

Compacted at 30 TSI, sintered at 1250°C for 30 min

Compacted at 30 TSI, sintered at 1250°C for 60 min

Compacted at 30 TSI, sintered at 1250°C for 90 min
Sintering of ITP Ti-6Al-4V Powders

- Graph 1: Shrinkage and Sintered density vs. Sintering temperature
- Graph 2: Flexural strength and Fracture strain vs. Sintering temperature
HIP Bars: After Machining

<table>
<thead>
<tr>
<th></th>
<th>Before machining</th>
<th>After machining</th>
<th>Yield (ratio after/before machining)</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>B01-20010301-2</td>
<td>1.024 lb</td>
<td>0.780 lb</td>
<td>76.2 %</td>
<td>4.47 g/cm³ (T.D.: 98.9 %)</td>
</tr>
<tr>
<td>B01-20100301-4</td>
<td>2.245 lb</td>
<td>1.584 lb</td>
<td>70.6 %</td>
<td>4.40 g/cm³ ( - )</td>
</tr>
</tbody>
</table>
Summary

- Consistent production of quality powder
- Define the economically optimum consolidation routes
- Achieve desired properties
- Collaborate with practitioners of enabling technologies:
  - Roll compaction
  - Joining
  - Additive manufacturing