High quality powder manufacturing and recycling by Induction Plasma Technology

Contact: Romain Vert
R&D Manager - Europe
Phone: +33 3 85 23 91 36
Mobile: +33 6 07 53 77 13
E-mail: romain.vert@tekna.com

Visit our corporate web site: www.tekna.com
Who is Tekna: Processing powders for 25 years

Induction Plasma technology

Powders production and recycling by Induction Plasma

Induction Plasma powders features

Industrial titanium based powder production at Tekna

Conclusions
Who is Tekna: Processing powders for 25 years

- Induction Plasma technology
- Powders production and recycling by Induction Plasma
- Induction Plasma powders features
- Industrial titanium based powder production at Tekna
- Conclusions
Arendals Fossekompani ASA:
- Exists since 1896 (Norway)
- Main business is to operate 2 hydro power plant and electrical energy
- Company turnover (2015): 740 M$ (USD)
- Acquired 100% of Tekna in July 2013
Global Presence

HQ: Canada

Production Sites: Canada (Systems & Powders), Europe (Powders)

Distribution Stocks & Services: Japan, Canada, USA, Brazil, France, India, China, Korea, Singapore
To develop and promote novel industrial solutions using induction plasma technology for high scale manufacturing of advanced high added-value materials
Who is Tekna: Processing powders for 25 years

Induction Plasma technology

Powders production and recycling by Induction Plasma

Induction Plasma powders features

Industrial titanium based powder production at Tekna

Conclusions
Induction Plasma Principle

Precursors

Central gas

Sheath gas

RF Electrical Supply (MHz)

Magnetic Coupling

Torch

Reactor chamber

T [ x 1000°C]
Standard Plasma torches

TEKNA ceramic wall induction plasma torch
U.S. Patent # 5 200 595 and International PCT/CA92/00156

<table>
<thead>
<tr>
<th>Torch</th>
<th>Power (kW)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL-35</td>
<td>15 or 40</td>
</tr>
<tr>
<td>PS-50</td>
<td>80</td>
</tr>
<tr>
<td>PS-70</td>
<td>80 or 200</td>
</tr>
<tr>
<td>PS-100</td>
<td>200</td>
</tr>
</tbody>
</table>
Unique features of ICP Technology

- **No electrodes** (= no consumable; continuous process allowing 24/24 – 7/7 production; no potential W contamination);

- **High purity** environment (absence of electrode erosion);

- **Axial injection** of feed stock in the highest temperature zone of the plasma (homogeneous precursor treatment or reaction);

- **Large plasma volume** which insure a **long in-flight time** in the hot gas stream (up to ~500 ms depending on reactor design, in comparison to typically 1 ms in DC plasma unit) = **ability to treat or purify industrial quantities of a large spectrum of materials** from low to high melting points.

- Discharge sustainable in **various atmospheres** = ensures high purity environment; **chemical reactions** and **purification** possible.

- Rather **high yields**.
Who is Tekna: Processing powders for 25 years

Induction Plasma technology

Powders production and recycling by Induction Plasma

Induction Plasma powders features

Industrial titanium based powder production at Tekna

Conclusions
Precursors

- Powders
- Wire
- Rod

Processed powder

Recycling of plasma gases

By-products

Dust
Residues
Contaminants

....
Industrial applications of Tekna’s ICP technology:

- **Industrial production of spherical powders**
  - Tekna’s spheroidization process
  - Tekna’s atomization process (Wire & Rod)

- **Powder recycling**
  - Refurbishing of scrapped powders:
    - Morphology improvement
    - Flow ability improvement
    - Purification and Oxygen level reduction

- Spherical shape
- High flowability
- High density
- Controlled chemistry
- Low oxygen level
- PSD tailored to customer applications
Industrial applications of Tekna’s ICP technology:

- Spherical shape
- High flowability
- High density
- Controlled chemistry
- Low oxygen level
- PSD tailored to customer applications
Advantages

Reduce powder porosity

Lower powder friability

Improve powder flowing properties (Ti)

Modify particle surface morphology

Enhance powder purity (Mo)

Increase in powder packing density

**Element** | **As-received powder** | **Processed powder**
---|---|---
Ca | 21 | <1
Cr | 16 | 1
Cu | <1 | <1
Fe | 37 | 2
Ni | 22 | 2
O2 | 674 | 136
W | 88 | 79
Zn | 26 | 2
Zr | 20 | <1

| (904) | (222) |

Ni coating on the surface of spherical cast tungsten carbide particles
Plasma powders for PM applications

Added value performances in comparison to standard powders available

Atomized  Crushed  Sponge  Spray-dried

Spherical, dense, no satellite, high flowability, high packing density
Example of Titanium treatment

Tekna’s proprietary process
US Patent 7572315

As-received

Processed / Sieved

Proc. / Sieved / Classified

No flow
0.80 g/cc

34 s/50g
2.20 g/cc

20 s/50g
2.8 g/cc
Industrial applications of Tekna’s ICP technology:

- Industrial production of spherical powders
- Tekna’s spheroidization process
- Tekna’s atomization process (Wire & Rod)
- Powder recycling
  - Refurbishing of scrapped powders:
    - Morphology improvement
    - Flowability improvement
    - Purification and Oxygen level reduction

- Spherical shape
- High flowability
- High density
- Controlled chemistry
- Low oxygen level
- PSD tailored to customer applications
Standard Ti64 Grade 23 from Tekna

Size cuts available (microns)
- 5-25
- 20-45
- 45-105

Custom size available

1070 ppm oxygen

MATERIAL TEST CERTIFICATE

<table>
<thead>
<tr>
<th>Product Name</th>
<th>Ti64-25/5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Description</td>
<td>Spher</td>
</tr>
<tr>
<td>Size</td>
<td>-25 ± 5 μm</td>
</tr>
<tr>
<td>Composition</td>
<td>Ti-6%Al-4%V</td>
</tr>
<tr>
<td>Product no.</td>
<td>34207</td>
</tr>
<tr>
<td>Lot no., Lab no.</td>
<td>34207-15-001, 150964</td>
</tr>
</tbody>
</table>

Chemical Composition

<table>
<thead>
<tr>
<th>Elements</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>Fe*</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>Others, each*</th>
<th>Others, Total*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Results w(%)</td>
<td>Bal.</td>
<td>6.6</td>
<td>4.2</td>
<td>0.23</td>
<td>0.019</td>
<td>0.107</td>
<td>0.024</td>
<td>0.005</td>
<td>&lt;0.03</td>
<td>&lt;0.10</td>
</tr>
</tbody>
</table>

Physical Properties

- Tap Density (g/cc): 2.9
- Apparent Density (g/cc): 2.3

Particle Size Distribution

- D10: 8.2 μm
- D50: 13.4 μm
- D90: 20.4 μm

MORPHOLOGY
Standard Ti64 Grade 23 from Tekna

Size cuts available (microns)
5-25
20-45
45-105

Custom size available

720 ppm oxygen

Ti64 powders produced by atomization

MATERIAL TEST CERTIFICATE

Product Name: Ti64:45/20
Description: Spherical Ti64 Powder
Size: -45 + 20 μm
Composition: Ti-6%Al-4%V, meets ASTM B348-13 Grade 23
Product no.: 34610
Lot no., Lab no.: 34610-15-001, 152033-151973

Chemical Composition¹,²

<table>
<thead>
<tr>
<th>Elements</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>Others, each</th>
<th>Others, Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Results(%)</td>
<td>Bal.</td>
<td>6.42</td>
<td>3.99</td>
<td>0.186</td>
<td>0.015</td>
<td>0.072</td>
<td>0.013</td>
<td>0.002</td>
<td>&lt;0.06</td>
<td>&lt;0.10</td>
</tr>
</tbody>
</table>

Physical Properties¹

Tap Density (g/cc): 2.8
Apparent Density (g/cc): 2.4

Particle Size Distribution

by Laser Light Diffraction; Size in microns (μm)
- D10: 15.0 μm, 23.8 μm
- D50: 34.7 μm, 51.9 μm
- D90: 51.9 μm

by Sieve Analysis (ASTM B9214); wt% Results:
- 46: 15.0, 10.3
- 45-20: 75, 81.0
- ≤20: 10.0, 7.8

¹ Lab #152033

Romain Vert – TEKNA – 19/04/2015
Titanium Europe 2016
Standard Ti64 Grade 23 from Tekna

Size cuts available
(microns)
5-25
20-45
45-105

Custom size available

640 ppm oxygen

Ti64 powders produced by atomization

MATERIAL TEST CERTIFICATE

<table>
<thead>
<tr>
<th>Product Name:</th>
<th>TEKMAT™ Ti64-105/45-1000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Description</td>
<td>Sphe</td>
</tr>
<tr>
<td>Size:</td>
<td>-105 + 45 μm</td>
</tr>
<tr>
<td>Composition:</td>
<td>Ti-6%Al-4%V, Grade 23, Oxygen &lt; 0.1%</td>
</tr>
<tr>
<td>Product no.:</td>
<td>33306</td>
</tr>
<tr>
<td>Lot No.:</td>
<td>15-002</td>
</tr>
</tbody>
</table>

Chemical Composition

<table>
<thead>
<tr>
<th>Elements</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>Others, each</th>
<th>Others, Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Results</td>
<td>wt (%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bel.</td>
<td>6.1</td>
<td>6.1</td>
<td>4.0</td>
<td>0.051</td>
<td>0.005</td>
<td>0.064</td>
<td>0.007</td>
<td>0.001</td>
<td>&lt; 0.02</td>
<td>&lt; 0.07</td>
</tr>
</tbody>
</table>

Physical Properties

<table>
<thead>
<tr>
<th>Test Method</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hall Flow Test (80/60g) ASTM E12134:77</td>
<td>20</td>
</tr>
<tr>
<td>Tap Density (g/cc) ASTM E1657-79</td>
<td>2.9</td>
</tr>
<tr>
<td>Apparent Density (g/cc) ASTM E1657-79</td>
<td>2.0</td>
</tr>
</tbody>
</table>

Morphology

Particle Size Distribution

- D10: 20.5 μm, D50: 52.1 μm, D90: 105.2 μm

By Sieve Analysis (ASTM B214) - wt%:

- +105: 5.0, +45: 95.4, <45: 10.0

BNO CERTIFICATE

ISO 9001:2008
TEKMAT Ti64-250-HD1

**Chemical Composition**

<table>
<thead>
<tr>
<th>Elements</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>Others, each</th>
<th>Others, Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Results  wt (%)</td>
<td>Bal.</td>
<td>6.24</td>
<td>4.02</td>
<td>0.185</td>
<td>0.008</td>
<td>0.062</td>
<td>0.014</td>
<td>0.001</td>
<td>≤ 0.10</td>
<td>≤ 0.05</td>
</tr>
</tbody>
</table>

- **620 ppm oxygen**
- **Meet grade 23 requirements**
- **Size distribution between 45 and 250 µm**
- **Spherical powder**

**Carney Flow Test (260/50g):**
- 11

**Tap Density (g/cc):**
- 2.8

**Apparent Density (g/cc):**
- 2.5

**Particle Size Distribution**

- **by Laser Light Diffraction:** Size in microns (µm)
  - D10: 4.8
  - D50: 182.3
  - D90: 236.3

- **by Sieve Analysis (ASTM E214) – wt%**
  - Size (µm) Min (%) Max (%) Results (%)
    - +250 — — 93.9
    - -250+150 — — 59.7
    - -150+105 — — 20.8
    - -105+45 — — 10.2
    - -45+20 — — 0.1
    - -20 — — 0.1

Romain Vert – TEKNA – 19/04/2015

Titanium Europe 2016
TEKMAT Ti64-250-HD3

Chemical Composition

<table>
<thead>
<tr>
<th>Elements</th>
<th>Ti</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>C</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>Others, each</th>
<th>Others, Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Results</td>
<td>Bal.</td>
<td>6.23</td>
<td>4.04</td>
<td>0.185</td>
<td>0.009</td>
<td>0.071</td>
<td>0.014</td>
<td>0.002</td>
<td>≤ 0.10</td>
<td>≤ 0.05</td>
</tr>
</tbody>
</table>

710 ppm oxygen

Meet grade 23 requirements

Carney Flow Test (sec/50g): 11
Tap Density (g/cc): 3.2
Apparent Density (g/cc): 2.7

Bi-modal size distribution
Highest pack density
Industrial applications of Tekna’s ICP technology:

- **Powder recycling**
  - Refurbishing of scrapped powders:
    - Morphology improvement
    - Flow ability improvement
    - Purification and Oxygen level reduction

- Industrial production of spherical powders
  - Tekna’s spheroidization process
  - Tekna’s atomization process (Wire & Rod)

- Spherical shape
- High flowability
- High density
- Controlled chemistry
- Low oxygen level
- PSD tailored to customer applications
Powder rejected after multiple uses in a laser-based AM system

Sintered particles

Fragmented particles

Satellites
## Ti6Al4V powder recycling

<table>
<thead>
<tr>
<th></th>
<th>Rejected powder (feedstock)</th>
<th>Plasma processed (medium feed rate)</th>
<th>Plasma processed (low feed rate)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Tap density</strong></td>
<td>2.85 g/cc</td>
<td>2.90 g/cc</td>
<td>2.93 g/cc</td>
</tr>
<tr>
<td><strong>App. Density</strong></td>
<td>2.31 g/cc</td>
<td>2.45 g/cc</td>
<td>2.46 g/cc</td>
</tr>
<tr>
<td><strong>Hall Flow</strong></td>
<td>46 s/50g</td>
<td>30 s/50g</td>
<td>26 s/50g</td>
</tr>
<tr>
<td><strong>d5</strong></td>
<td>16.7 um</td>
<td>21.0 um</td>
<td>18.5 um</td>
</tr>
<tr>
<td><strong>d95</strong></td>
<td>53.1 um</td>
<td>51.4 um</td>
<td>51.0 um</td>
</tr>
</tbody>
</table>
Powder rejected after uses in a laser cladding process
### Haynes® 230 powder recycling

<table>
<thead>
<tr>
<th></th>
<th>Scrap powder (feedstock)</th>
<th>Plasma processed (low feed rate)</th>
<th>Plasma processed (medium feed rate)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Tap density</strong></td>
<td>5.57 g/cc</td>
<td>5.92 g/cc</td>
<td>5.90 g/cc</td>
</tr>
<tr>
<td><strong>App. Density</strong></td>
<td>5.08 g/cc</td>
<td>5.45 g/cc</td>
<td>5.43 g/cc</td>
</tr>
<tr>
<td><strong>Hall Flow</strong></td>
<td>13.1 s/50g</td>
<td>10.7 s/50g</td>
<td>10.9 s/50g</td>
</tr>
<tr>
<td><strong>Oxygen</strong></td>
<td>1067 ppm</td>
<td>160 ppm</td>
<td>246 ppm</td>
</tr>
<tr>
<td><strong>d10</strong></td>
<td>46.6 um</td>
<td>49.0 um</td>
<td>48.0 um</td>
</tr>
<tr>
<td><strong>d90</strong></td>
<td>89.3um</td>
<td>93.4 um</td>
<td>91.7 um</td>
</tr>
</tbody>
</table>
Who is Tekna: Processing powders for 25 years

Induction Plasma technology

Powders production and recycling by Induction Plasma

Induction Plasma powders features

Industrial titanium based powder production at Tekna

Conclusions
Induction Plasma powders features

- Composition close monitoring and control
- Suitable for high purity materials and reactive metals
- Low oxygen content = powder recyclability
- High sphericity level & Clean Particles = flow ability + density
- Various size fractions available from 5 to 250 microns = suitable for the highest demanding applications such as MIM, SLM, EBM, HIP...
- Production and development flexibility
- Quality control – Batch to Batch consistency
- Production capacity
Induction Plasma powders features

$$\text{Hausner ratio} = \frac{\text{Tap density}}{\text{Apparent density}}$$

- Cohesive powders
- High flowability powders
Relative apparent density = \frac{\text{Powder apparent density}}{\text{Bulk density}}
Ti64 45-105 µm – Particle cross sections
- Who is Tekna: Processing powders for 25 years
- Induction Plasma technology
- Powders production and recycling by Induction Plasma
- Induction Plasma powders features
- Industrial titanium based powder production at Tekna
- Conclusions
Tekna’s plasma systems solution

Induction Plasma System Solution:
• Pre Processing - Cleaning, Sieving
• Powder Feeding & Plasma Treatment
• Post Processing – Cleaning, Sieving, Packaging

Tekna’s induction plasma torch

Torch features:
• 24/7 continuous operation
• Proprietary ceramic plasma confinement tube;
• No electrodes (consumable & avoid tungsten contamination);
• High Purity process;
• Operation under a wide range of conditions
Tekna’s Integrated Powder Manufacturing

Fully automated & 24/7 operation
CERTIFICATE

Certificate No.: 49449-1-01

The Bureau de normalisation du Québec (BNQ) certifies that the quality management system implemented by:

TEKNA MATÉRIAUX AVANCÉS INC.

covering the following activities:
Manufacturing commercial powders
Permissible exclusions: design and development

carried out at the address:

2895, boul. Industriel, Sherbrooke (Québec) J1L 2T9

has been assessed and found to comply with the requirements of:

ISO 9001:2008

In testimony whereof, have signed in Québec, on June 18, 2015,

Isabelle Landry, Director of Operations
Bureau de normalisation du Québec

Jocelyn Brousseau, Program Leader
Bureau de normalisation du Québec
Who is Tekna: Processing powders for 25 years

Induction Plasma technology

Powders production and recycling by Induction Plasma

Induction Plasma powders features

Industrial titanium based powder production at Tekna

Conclusions
ICP technology allows to produce high quality powders.
- Both spherical micron-sized powders and nanopowders
- High purity materials
- Controlled properties

Technology allows to control the chemistry of the materials and to purify it depending on plasma gas composition.

Possibility to recycle scrap powders from various industries.

Powders produced by ICP meet the requirements of various PM applications (HIP, Additive Manufacturing, SPS, MIM, ...).

Tekna offers production and development flexibility (several systems available).
Thank you for your attention

Romain Vert
R&D Manager - Europe
Mobile: +33 6 07 53 77 13
E-mail: romain.vert@tekna.com