How TiAl oversized powder coming from EIGA process can be managed?

Results from a study led by 3DMaterials (ex-Silimelt) and ALD Vacuum Technologies
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Silimelt & ALD Vacuum Technologies

- Expert in plasma processes. Recently bought by a subsidiary of the ACE Aéronautique Group (owner of Prismadd that produces parts by additive manufacturing)

- Equipment supplier in the field of vacuum for metallurgy, coatings and heat treatment
Why using TiAl?

- Excellent oxidation and temperature resistance (up to 850°C)
- High strength-to-density ratio
- High thermal conductivity compared to Ti alloys

Source: Techniques de l’Ingénieur
Powder Metallurgy for TiAl part manufacture

• Low ductility at room temperature,
• Low fracture toughness,
• Difficulty to keep an metallurgical homogeneity,
• Low solidification rate,
• Poor machinability...

➢ Powder Metallurgy (PM) and Additive Manufacturing (AM) are promising ways to develop structural parts made in TiAl

➢ However, high quality powder (< 100 µm) is needed
TiAl powder production

Electrode Induction Gas Atomization (EIGA)

- Fine powder (5/200 µm)
- Economic process, low energy consumption
- Some pores in coarse particles
- High Ar consumption

Source: ALD Vacuum Technologies

Plasma Rotating Electrode Process (PREP)

- No pores,
- Low Ar consumption
- Coarse powder (>100 µm),
- High energy consumption, expensive feedstock,

Source: civilengineeringhandbook.tk

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As-atomized powder coming from EIGA

- **Alloy**: TiAl 4822
- Spherical with some satellites
- Quite large size distribution (10-250 \( \mu m \))
- Porosity \( \sim 0.5\% \)

<table>
<thead>
<tr>
<th>Sieving</th>
<th>&gt; 300 ( \mu m )</th>
<th>&gt;100 ( \mu m )</th>
<th>&gt;50 ( \mu m )</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 %</td>
<td>45%</td>
<td>78%</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Laser granulometry</th>
<th>( d_{10} )</th>
<th>( d_{50} )</th>
<th>( d_{90} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>27 ( \mu m )</td>
<td>96 ( \mu m )</td>
<td>219 ( \mu m )</td>
<td></td>
</tr>
</tbody>
</table>
Aim of the study

• Two-stage process for TiAl powder production:

- EIGA
- TiAl oversized powder
  - Sieved: > 100 µm
  - Sieved: < 100 µm
- Plasma fragmentation
  - Sieved: < 100 µm

TiAl powder suitable for PM and AM
The plasma fragmentation process

- Based on non-arc transferred plasma torch (<50 kW)
- Plasma jet: **up to 2300 m/s, up to 13000 K**

**Fragmentation Yield:**

\[
Y_{f-100} = \frac{\text{Amount of powder sieved below } 100 \mu m}{\text{Amount of injected powder}}
\]

\[
Y_{f-50} = \frac{\text{Amount of powder sieved below } 50 \mu m}{\text{Amount of injected powder}}
\]
Why fragmentation occurs?

Surface tension vs Aerodynamic force

\[ \text{We} = \frac{\rho \cdot v^2 \cdot L_c}{\sigma} \]

- **Streamlined approach**: Reynolds and Ohnesorge numbers have also to be accounted for.
- **The particles need to be melted (probably fully)**

\( \rho \): gas density (kg/m³)
\( v \): relative velocity between the particle and the gas flow (m/s)
\( L_c \): droplet diameter (m)
\( \sigma \): surface tension of molten droplets (N/m)

In the plasma jet, for Ti droplet:
- Weber number up to 20-25

Source: Reitz, Lee (2001)
Why fragmentation occurs?

\[ We = \frac{\rho \cdot v^2 \cdot L_c}{\sigma} \]
Influence of the size distribution

- Biggest difference between before and after for particle’s size ranged from 80 to 170 µm
- In theory, the bigger the particle is, the more easy it is to break-up it (surface tension effect)
  - However, particles bigger than 170 µm are quite difficult to melt in the plasma jet (residence time < 5 ms)
Influence of the size distribution

Input Material: sieved below 80 µm

- Fine particles quite difficult to break-up (surface tension effect)
  - Low decrease of size distribution which could be due to vaporization

ArH₂ plasma

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Influence of the gas composition

Input: +50 µm (TiAl)

Same content of secondary/ternary gas

- $ArH_2$ plasma: 25% vol of $H_2$
- $ArHe$ plasma: 25% vol of $He$
- $ArHeH_2$: 22.5% vol of $He$, 2.5% of $H_2$

- The use of $H_2$ increases the amount of powders below 50 µm, after plasma treatment
- Enhancement of droplets break-up
Influence of the gas composition

- Heat transfer to the particles is enhanced due to a higher thermal conductivity of H₂: faster melting in the plasma zones where the gas stream velocity is the highest.

- Higher relative velocity because of a limited momentum transfer to the particles (lower viscosity of H₂) and a higher velocity of the gas stream (lower density of H₂).

**How H₂ has an effect on in-flight plasma treatment?**

**TiAl : +50 µm**

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What about chemistry?

Plasma ArH₂ (up to 25%vol.)

<table>
<thead>
<tr>
<th></th>
<th>Before PF</th>
<th>After PF</th>
</tr>
</thead>
<tbody>
<tr>
<td>O</td>
<td>-</td>
<td>900-1300 ppm</td>
</tr>
<tr>
<td>N</td>
<td>-</td>
<td>~100 ppm</td>
</tr>
<tr>
<td>H</td>
<td>&lt; 10 ppm</td>
<td>300-900 ppm</td>
</tr>
</tbody>
</table>

- When H₂ is used as secondary gas, H content is too high
  - Post-treatment of dehydrating under vacuum?

- Slight decrease of Al content: ~1-2%wt less after plasma treatment
  - High vapor pressure of Al → vaporization

- O and N content perfectible (leaks due to the experimental setup)
What about other materials?

<table>
<thead>
<tr>
<th>Y_{f-50} (&lt; 50 \mu m)</th>
<th>Ti64</th>
<th>TiAl</th>
<th>TiAl</th>
<th>Inconel 718</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feedstock</td>
<td>+50 \mu m</td>
<td>+ 50 \mu m</td>
<td>+100 \mu m</td>
<td>+100 \mu m</td>
</tr>
<tr>
<td>ArH_2 plasma</td>
<td>35%</td>
<td>28%</td>
<td>26%</td>
<td>55%</td>
</tr>
<tr>
<td>ArHe plasma</td>
<td>26%</td>
<td>6%</td>
<td>-</td>
<td>20%</td>
</tr>
</tbody>
</table>

- Ti64 (\rho \sim 4.5 \text{ kg/dm}^3) and Inconel (\rho \sim 8.5 \text{ kg/dm}^3) particles easier to break-up than TiAl (\rho \sim 4 \text{ kg/dm}^3)
- Lower residence time and relative particle velocity for TiAl
Concluding remarks

- A double-stage process was proposed to recover the TiAl oversized particles coming from EIGA process
- The plasma fragmentation is quite efficient for producing fine TiAl powder (<50 µm):
  - for an input powder with particles size ranged from 80 up to 170 µm
  - when H₂ is used, but H content after plasma is too high!
- Experimental development under progress:
  - supersonic plasma jet, dehydriding as post-treatment, more data about chemical composition in particular for Al content...
Influence of the size distribution

<table>
<thead>
<tr>
<th>Influence of the PSD of the input material</th>
<th>After plasma treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Input</td>
</tr>
<tr>
<td>As-atomized</td>
<td>↗</td>
</tr>
<tr>
<td>+50 µm</td>
<td>↗</td>
</tr>
<tr>
<td>+100 µm</td>
<td>↗</td>
</tr>
<tr>
<td>+50-100 µm</td>
<td>↗</td>
</tr>
</tbody>
</table>

ArH₂ plasma

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Influence of the helium content

- Low impact of He content on the amount of particles sized below 50 µm after plasma treatment

![Graph showing the influence of helium content on particle size](image)

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