PHASE CHARACTERISATION IN SPARK PLASMA SINTERED TiPt ALLOY

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Shape Memory Alloys (SMAs)

- SMAs are capable of “remembering” their original shape after deformation and subsequent input of thermal energy.

- Shape memory depends on a displacive phase transformation.

- Used for couplings, seals, actuators, fire safety valves.

(Calkins, 2010)
TiPt High Temperature SMAs

- Equiatomic TiPt exists as:
  - high temperature BCC austenite $\beta$-phase
  - low temperature orthorhombic martensite $\alpha$-phase

- $T_M \approx 1050 \, ^\circ\text{C}$ at equiatomic composition

- Possible use in actuation for jet engines and other high temperature process control applications

Biggs et al., 2004
Spark Plasma Sintering

- Low voltage, pulsed high DC current to activate consolidation

- High uniaxial pressure for compaction

- Spark plasma between particles and Joule heating (up to 10 000°C)

- Fast heating rates and minimum grain growth, better than PLS, HP or HIP.

- SPS is easy to operate, requires no binders, high capital cost is offset by low running cost

Shen at al., 2002
Spark Plasma Sintering

- Spark plasma and Joule heating results in vaporisation of particle surface and surface melting of particle just behind the vaporised layer.
- Liquid surfaces are drawn together to form “necks”.
- Radiant Joule heat and pressure drives “neck” growth and material transfer.
Objective

- Produce TiPt alloy compacts by Spark plasma sintering (SPS) of equiatomic blended elemental (BE) Ti and Pt powders

- Produce homogenous TiPt alloy phase
Starting Powder

- Ti$_{50}$Pt$_{50}$ (atomic %)
- $\geq$ 99.5 % purity powders

- Ti$_{50}$Pt$_{50}$ (atomic %) as pressed
Sintering Conditions

- Place powder in graphite die setting

- Program temperature-pressure ramp up and hold settings according to the values below:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Temperature (°C)</th>
<th>Time (minutes)</th>
<th>Pressure (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>1200</td>
<td>25</td>
<td>60 MPa</td>
</tr>
<tr>
<td>Sample 2</td>
<td>1300</td>
<td>15</td>
<td>60 MPa</td>
</tr>
<tr>
<td>Sample 3</td>
<td>1300</td>
<td>25</td>
<td>60 MPa</td>
</tr>
<tr>
<td>Sample 4</td>
<td>1300</td>
<td>30</td>
<td>60 MPa</td>
</tr>
<tr>
<td>Sample 5</td>
<td>1400</td>
<td>10</td>
<td>60 MPa</td>
</tr>
</tbody>
</table>

- Full density was achieved in all samples
Solid State Sintering

- During solid state sintering of Ti and Pt, sintering is controlled by interdiffusion of the components.
- All equilibrium and non-equilibrium phases at the sintering temperature are expected to form.
- With time, the alloy will homogenise to the bulk starting composition.
SPS: 1200 °C_25mins_60MPa

- Fully sintered
- Solid state reaction controlled
SPS: 1300 °C_25mins_60MPa

- Fully sintered
- Solid state reaction controlled
SPS: 1400 °C_10mins_60MPa

- Fully sintered
Homogenising: 1300° C_10hrs_FC

Sintering condition: 1200 ° C_25mins_60MPa
Homogenising: 1300° C_10hrs_FC

Sintering condition: 1400 °C_10mins_60MPa
Conclusions

- SPS of BE TiPt powder produces fully sintered specimens, with incomplete homogenisation

- TiPt phase is formed from the BE powder

- Post sintering heat treatment is required to homogenise the microstructure

- There is a need for improved furnace atmosphere control so that contamination can be eliminated as a possible reason for deviation of the final microstructure from the expected equiatomic TiPt composition.