Assessment of laser peening induced effects on Ti6Al4V by non-destructive measurements

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One of the most appropriate biomaterials for **load-bearing implants**:

- Good mechanical properties
- High corrosion resistance
- Good biocompatibility (Bioinert)

Biological response is regulated by the material / tissue interface
SURFACE MODIFICATIONS BASED ON SEVERE PLASTIC DEFORMATION

GRIT BLASTING (GB)

Orthopedic and dental implant applications

Beneficial effects
- Increases surface roughness
- Increases compressive residual stress
- Increases fatigue resistance

$\text{Al}_2\text{O}_3 / \text{ZrO}_2$

$Ra$

GB-$\text{Al}_2\text{O}_3 \approx 5.1 \mu m$
GB-$\text{ZrO}_2 \approx 1.0 \mu m$
SURFACE MODIFICATIONS BASED ON SEVERE PLASTIC DEFORMATION

GRIT BLASTING (GB)

Beneficial effects
- Increases surface roughness
- Increases compressive residual stress
- Increases fatigue resistance

Detrimental effects
- Surface contamination
- Stress concentrators
- Decreases fatigue resistance

Orthopedic and dental implant applications

GB - \( \text{Al}_2\text{O}_3 \approx 5.1 \text{ µm} \)
GB - \( \text{ZrO}_2 \approx 1.0 \text{ µm} \)

\[
\begin{align*}
R_a & \begin{cases} 
\text{GB-}\text{Al}_2\text{O}_3 & \approx 5.1 \text{ µm} \\
\text{GB-}\text{ZrO}_2 & \approx 1.0 \text{ µm} 
\end{cases}
\end{align*}
\]
Problem solving approach

LASER PEENING WITHOUT COATING

Alternative to achieve **compressive residual stress** at a depth of 1 mm, avoiding stress concentrators:

- **Delaying** the nucleation and propagation of cracks.

- **Improving** the fatigue resistance.

**Q-switched Nd-YAG Laser irradiation:**

- \( E = 2.8 \text{ J/pulse} \)
- \( \tau = 9 \text{ ns} \)
- \( \phi \text{ pulse} = 1.5 \text{ mm} \)
- \( \text{EOD} = 5000 \text{ cm}^2 \)
- **Confining medium:** water
- **No sacrificial coating**
Evaluating LP induced effects

**RELEVANT ITEMS**

- Topography and surface roughness
  - Strong effect on biological response
- Compressive residual stress
  - Cold work (hardening, texture)
  - Strong effect on mechanical behaviour
- Grain refinement / precipitates
Evaluating LP induced effects

RELEVANT ITEMS

Scanning Electron Microscopy (SEM)
X-Ray Diffraction (XRD)
Mechanical profilometry

Topography and surface roughness

compressive residual stress

cold work (hardening, texture)

grain refinement / precipitates

X-Ray Diffraction (XRD)
Syncrotron X-Ray Diffraction
Neutron Diffraction
Hole Drilling (HD)
Optical Microscopy (OM)
Scanning Electron Microscopy (SEM)
Transmission Electron Microscopy (TEM)
Electron Backscatter Diffraction (EBSD)
Vickers Microhardness (HV), ...
Evaluating LP induced effects

RELEVANT ITEMS

- Topography and surface roughness
- Scanning Electron Microscopy (SEM)
- X-Ray Diffraction (XRD)
- Mechanical profilometry

Our proposal:

THERMOELECTRIC POWER MEASUREMENTS
SEEBECK PRINCIPLE: Thermoelectric property that causes the conversion of a temperature difference into electricity.

**METHODS**

- **Hot tip method**
  - Mainly sensitive to:
    - Solute content and lattice defects

- **Magnetic method**
  - Mainly sensitive to:
    - Residual stress

*Insensitive to the sample geometry and the surface roughness*
Thermoelectric power measurements (TEP)

METHODS

Hot tip method

- When a closed loop is made of two metals with a temperature difference at the joints between them, a potential difference ($\Delta V$) is induced (Seebeck effect).

- The thermoelectric power ($\Delta S$) of the sample ($S_M$) relative to the reference metal ($S_{\text{tip}}$) is given by the relation:

$$\Delta S = S_M - S_{\text{tip}} = \Delta V / \Delta T \quad (\text{nV/K})$$

Measuring time 1 s
Accuracy $\pm$ 0.5%
Resolution 1 nV/K
Thermoelectric power measurements (TEP)

**METHODS**

**Hot tip method**

**Magnetic method**

- Reference metal
- Fluxgate gradiometer (lift-off = 2 mm)

\[ \Delta V \]
Combination of LP with two standard heat treatments

1- Partial residual stress relief (595ºC / 1h)
2- Total residual stress relief (710ºC / 2h)

Five different conditions
Surface Evaluation

TOPOGRAPHY

As machined

Ti6Al4V

S4800CENIM 10.0kV 8.0mm x20.0k SE(U) 2.00µm
TOPOGRAPHY

Surface Evaluation

As machined

Ti6Al4V

710ºC / 2h

TiO₂

S4800CENIM 15.0kV 14.9mm x20.0k SE(M) 2.00µm
As machined

Ti6Al4V

710°C / 2h

TiO₂

Laser Peened

TiO

Surface Evaluation

TOPOGRAPHY
TOPOGRAPHY

Surface Evaluation

As machined → 710°C / 2h → Laser Peened

595°C / 1h

Ti6Al4V → TiO2 → TiO
TOPOGRAPHY

Surface Evaluation

As machined

710°C / 2h

Laser Peened

595°C / 1h

710°C / 2h

TiO₂

Ti₆Al₄V

TiO₂

TiO

TiO₂

S4800CENIM 15.0kV 15.0mm x20.0k SE(M) 2.00μm
Surface Evaluation

ROUGHNESS

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ra (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As machined</td>
<td>1.84 ± 0.05</td>
</tr>
<tr>
<td>710°C / 2h</td>
<td>1.70 ± 0.10</td>
</tr>
<tr>
<td>Laser Peened</td>
<td>5.43 ± 0.40</td>
</tr>
<tr>
<td>595°C /1h</td>
<td>5.67 ± 0.29</td>
</tr>
<tr>
<td>710°C /2h</td>
<td>5.33 ± 0.31</td>
</tr>
</tbody>
</table>

595°C /1h

GB-Al$_2$O$_3$ ≈ 5.1 µm
GB-ZrO$_2$ ≈ 1.0 µm

710°C /2h

R$_a$
**TEP MEASUREMENTS: MICROSTRUCTURAL CHANGES**

**Hot tip method**

**Laser Peening**

<table>
<thead>
<tr>
<th>Method</th>
<th>(\Delta S) [(\mu V/K)]</th>
</tr>
</thead>
<tbody>
<tr>
<td>As machined</td>
<td>15</td>
</tr>
<tr>
<td>710(^\circ)C/2h</td>
<td>16</td>
</tr>
<tr>
<td>LP</td>
<td>17</td>
</tr>
<tr>
<td>LP + 595(^\circ)C/1h</td>
<td>16</td>
</tr>
<tr>
<td>LP + 710(^\circ)C/2h</td>
<td>16</td>
</tr>
</tbody>
</table>

**Grit blasting**

<table>
<thead>
<tr>
<th>Condition</th>
<th>(\Delta S) [(\mu V/K)]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference</td>
<td>15</td>
</tr>
<tr>
<td>GB-Alumina + 595(^\circ)C/1h</td>
<td>16</td>
</tr>
<tr>
<td>GB-Alumina + 710(^\circ)C/2h</td>
<td>16</td>
</tr>
</tbody>
</table>

**LP induces less plastic deformation than GB**

LP might induce higher residual stress than GB

CONCLUSIONS

• **Laser Peening without coating** is a good method to generate **biocompatible surfaces** with **roughness** of **clinical interest**.

• **Thermoelectric Power** measurements is a good method to evaluate in a **fast**, **non destructive** and **qualitative** way the **laser peening induced effects**.

• **Laser peening** induced effects on **Ti6Al4V** can be detected by **TEP** and the contribution of the **residual stress** can be **distinguished** from the **microstructural changes**.

• Strong support for these conclusions should be confirmed by **microstructural analyses** (SEM, TEM,...) and **residual stress measurements** (hole drilling, synchrotron).
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