Effects of Aging Heat Treatment on the Microstructure of Ti-Nb and Ti-Nb-Sn Alloys Employed as Biomaterials

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Mediterranean Conference on Innovative Materials and Applications
15 – 17 March 2011
Beirut - Lebanon
University of Campinas, Brazil
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Introduction

- Concept of implanting materials in the human body is not new
  - Ancient Egypt
    - Mummified foot with an artificial wooden toe
  - Ancient Egypt
    - Dental implant in mummies
  - Ancient Mediterranean civilization
    - Dental bridge
Total Joint Replacement

- **TJR** is a surgical procedure in which certain parts of a damaged joint, are removed and replaced with a plastic or metal device called a prosthesis.

- Prosthesis is designed to enable the artificial joint to move just like a healthy joint.
Total Hip Replacement

Hip joints and adjacent skeletal components

Total hip replacement

Implant after surgery
Total Hip Replacement

Adapted from Orlin & Cohen Orthopedic Group
Bone Elastic Deformation

- Implant material must simulate bone elastic behavior
- Wolff’s Law: Bone modifies its internal architecture and external shape as a result of mechanical stress
- Insufficient load transfer from the implant to the bone causes bone mass loss and osteoporosis

Stainless steel $E=200$ GPa

Healthy bone

Bone with osteoporosis

Bone fracture
Total Hip Replacement Requirements

- Femoral Stem Requirements
- High mechanical strength
- Low prices
- High biocompatibility
- High corrosion resistance
- Low elastic modulus
  - $E_{\text{bone}}$: 10 - 30 GPa
  - $E_{\text{stainless steel}}$: 200 GPa
  - $E_{\text{Ti-6Al-4V}}$: 106 GPa
- "Necessary development of low elastic modulus Ti Alloys"
Objectives

- To discuss phase transformations in $\beta$ Ti-Nb-Sn alloys:
  - $\alpha$ phase precipitation during aging heat treatment of metastable microstructures
  - Correlation between microstructure and mechanical behavior
  - Application of phase transformations knowledge in Ti-based femoral stem manufacturing
Titanium Metallurgy

- Titanium shows two allotropic forms: HCP and BCC

- Addition of alloying elements may change the phase stability and hence, the microstructure and mechanical behavior

HCP (α)

BCC (β)

883 °C
β Titanium Alloy

β Ti alloys

β Stabilizer elements: Cr, Nb, V, Ta, Mo

- HIGH STRENGTH-TO-DENSITY RATIO
- LOW ELASTIC MODULUS
- HIGH STRENGTH
- HIGH TOUGHNESS
- BIOCOMPATIBILITY
- EASY TO HEAT TREAT
- EXCELLENT CORROSION RESISTANCE
- LOW FORGING TEMPERATURE
Ti Alloys Phase Transformations

CHEMICAL COMPOSITION AND HEAT TREATMENTS

M_s(α') = α' martensite start temperature
M_s(α'') = α'' martensite start temperature

ω_{ath} = athermal ω phase
ω_{iso} = isothermal ω phase

β + ω

β + β'

100 wt%

β Stabilizer Content
Processing Route

Alloy Compositions: Ti-30Nb and Ti-30Nb-2Sn (wt. %)

- Melting > 2000ºC
- Homogenization 12 h / 1000ºC
- Hot Forging 850ºC
- Solution 1h / 1000ºC
- Aging: 0 - 2 h 260ºC and 400ºC

FC – FURNACE COOLED
AC – AIR COOLED
WQ – WATER QUENCHED

Time

Temperature
Effect of Sn on $\alpha''$ Amount

Effect of Sn addition on the amount of martensite
Water Quenched Samples

Microstructure = orthorhombic martensite ($\alpha''$) and $\beta$ phase.

Small amount of nanometric precipitates of $\omega$ in Ti-30Nb.
Martensite Decomposition

Thermal Analysis – DSC

- WQ Ti-30Nb and Ti-30Nb-2Sn samples with $\alpha''$ and $\beta$ phases
- Peak 1: reverse transformation $\alpha'' \rightarrow \beta$
- Precipitation of $\omega$ in $\beta$ matrix (end of peak 1)
- Peak 2: nucleation of $\alpha$ - “$\omega$ act as substrates”
- Peak 3: $\beta$ transus
Martensite decomposition occurred with the aging time. Reverse transformation of $\alpha''$ into $\beta$ phase also took place. Precipitation of $\alpha$ and $\omega$ phases is visible by high temperature XRD.
Martensite Decomposition: Ti-30Nb-2Sn

High Temperature X-Ray Diffraction

- Martensite decomposition occurred with the aging time
- Reverse transformation of $\alpha''$ into $\beta$ phase also took place
- No precipitation of $\omega$ phase is observed by high temperature XRD
Mechanical Behavior: Ti-30Nb
Mechanical Behavior: Ti-30Nb-2Sn

![Graph showing the relationship between Elastic Modulus (GPa) and Vickers Hardness (HV) for Ti-30Nb-2Sn at different temperatures and times.]

- Elastic Modulus (GPa)
- Vickers Hardness (HV)

Temperatures and Times:
- 260°C/0.06 ks
- 260°C/0.6 ks
- 260°C/1.2 ks
- 260°C/1.8 ks
- 260°C/3.6 ks
- 260°C/7.2 ks
- 400°C/0.06 ks
- 400°C/0.6 ks
- 400°C/1.2 ks
- 400°C/1.8 ks
- 400°C/3.6 ks
- 400°C/7.2 ks
## Effect of aging on mechanical behavior

<table>
<thead>
<tr>
<th>Alloy Condition</th>
<th>Phases (XRD)</th>
<th>$\sigma_{UTS}$ (MPa)</th>
<th>Elong (%)</th>
<th>E (GPa)</th>
<th>Hardness (VH)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-30Nb</td>
<td>$\alpha''+\beta+\omega$</td>
<td>332 ± 21</td>
<td>30 ± 7</td>
<td>74</td>
<td>299 ± 6</td>
</tr>
<tr>
<td>Ti-30Nb Aged</td>
<td>$\beta+\alpha+\omega$</td>
<td>826 ± 24</td>
<td>0.8 ± 0.1</td>
<td>105</td>
<td>430 ± 10</td>
</tr>
<tr>
<td>Ti-30Nb-2Sn</td>
<td>$\alpha''+\beta$</td>
<td>300 ± 32</td>
<td>36 ± 4.0</td>
<td>67</td>
<td>219 ± 5</td>
</tr>
<tr>
<td>Ti-30Nb-2Sn Aged</td>
<td>$\beta+\alpha+\omega^{**}$</td>
<td>800 ± 22</td>
<td>3.0 ± 0.2</td>
<td>85</td>
<td>390 ± 15</td>
</tr>
</tbody>
</table>

$\omega^{**}$ - very small amount
Cold Forging

Cold Forged Femoral Stem using Ti-30Nb-2Sn alloy
Conclusions

- Microstructure of WQ Ti-30Nb and Ti-30Nb-2Sn was formed by $\beta$ and $\alpha''$ phase and the amount of $\alpha''$ decreases with increase of Sn;
- Aging caused $\alpha''$ decomposition and precipitation of $\beta$, $\omega$ and $\alpha$ phases;
- Results suggest that Sn may act as a suppressor of $\omega$ precipitation, which allows the control of microstructure features and hence, mechanical properties;
- WQ Ti-Nb-Sn sample showed yield strength near 300 MPa, which makes easier cold forging process, whose aged sample value increased up to 800 MPa;
- Aged Ti-Nb-Sn alloy showed elastic modulus of 85 GPa;
- These final values are very suitable in terms of orthopedic biomaterial applications.
Questions??