

**University of Campinas, Brazil**

***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



# Outline

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- **Introduction**
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  - **Total Hip Replacement Requiriments**
  - **Bone Elastic Deformation**
  - **Ti Alloys Phase Transformations**
- **Objectives**
- **Experiments**
- **Results**
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  - **High Temperature X-Ray Diffraction**
  - **Aging Heat Treatment and Mechanical Behavior**
  - **Cold Forged Femoral Stem**
- **Conclusions**

# 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



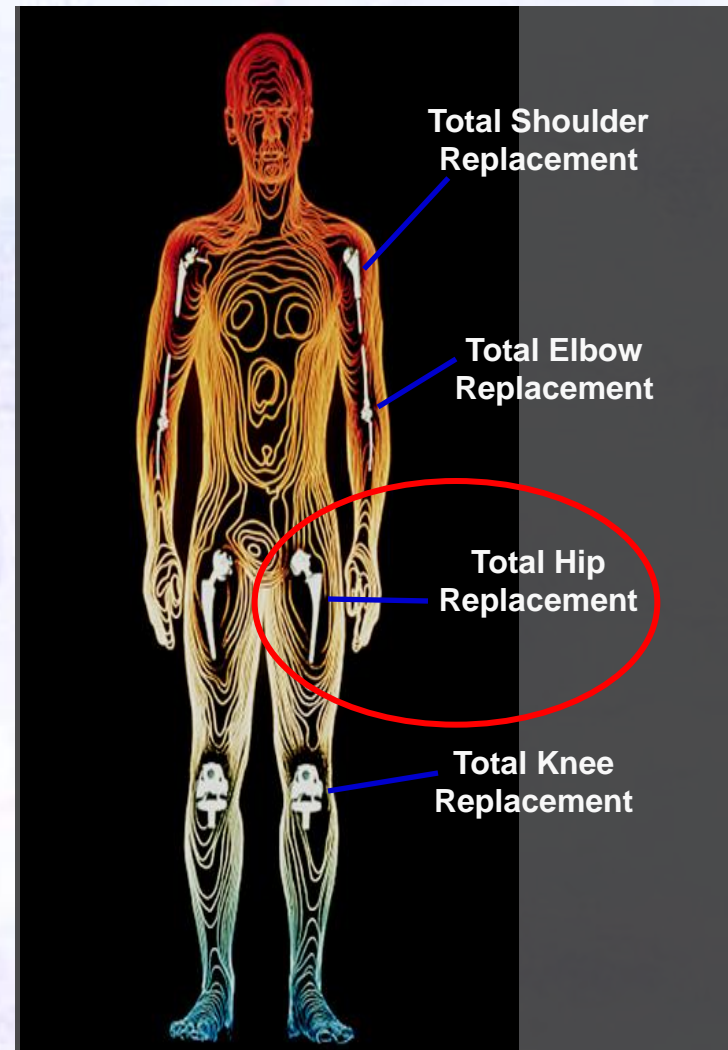
- **Ancient mediterranean civilization**

- dental bridge

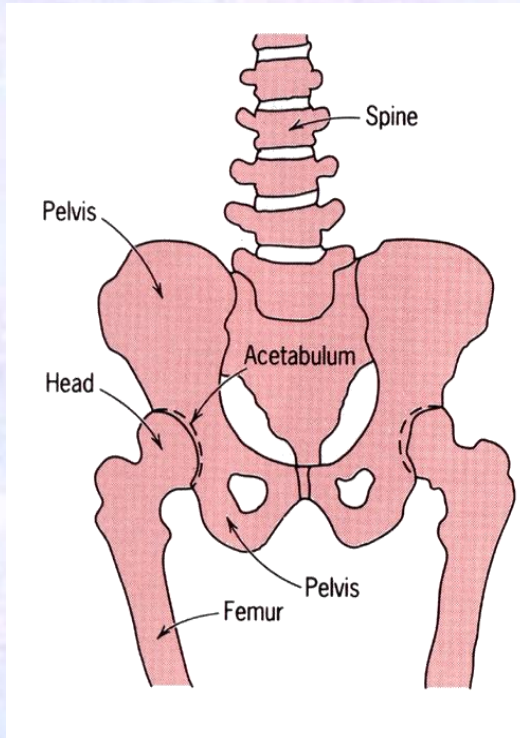


# Total Joint Replacement

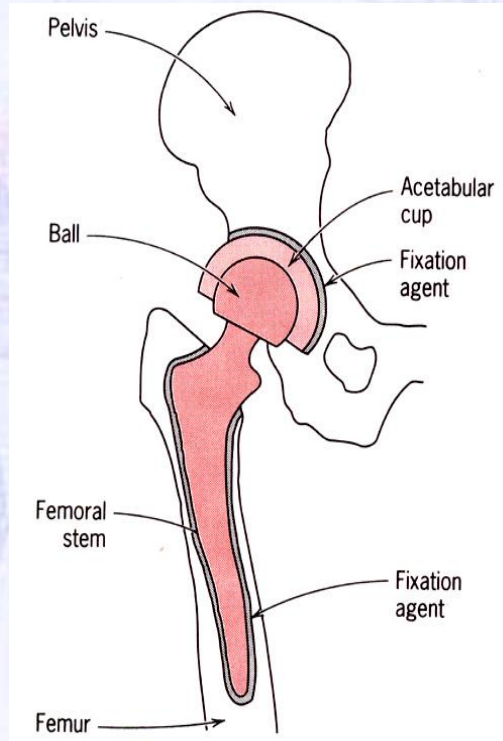
- **TJR** is a surgical procedure in which certain parts of a damaged joint, are removed and replaced with a plastic or metal device called a prosthesis
- Prosthesis is designed to enable the artificial joint to move just like a healthy joint



# Total Hip Replacement



**Hip joints and adjacent skeletal components**



**Total hip replacement**



**Implant after surgery**

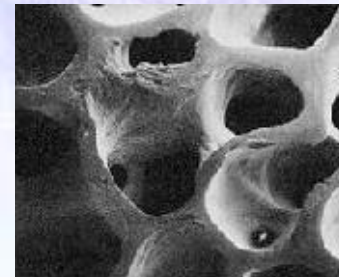
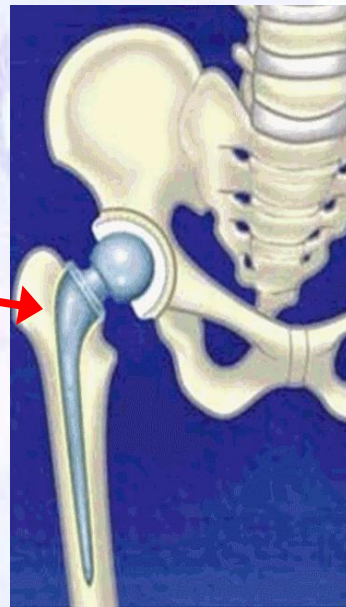
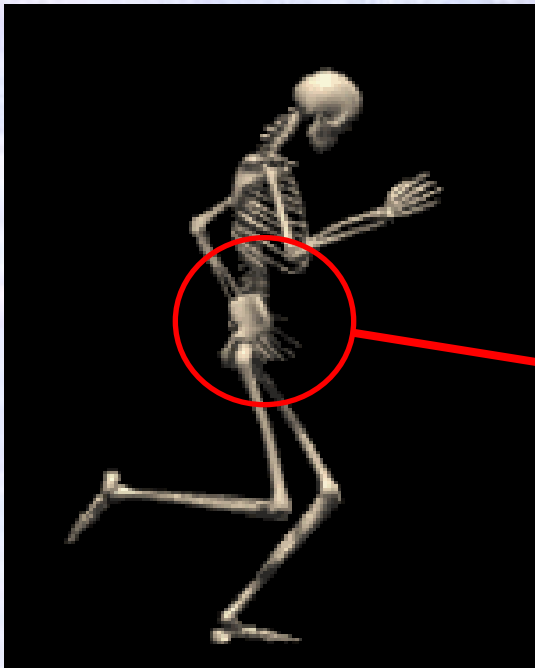
# Total Hip Replacement



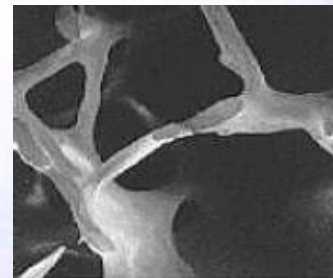
Adapted from Orlin & Cohen Orthopedic Group

# 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



Healthy bone



Bone with osteoporosis

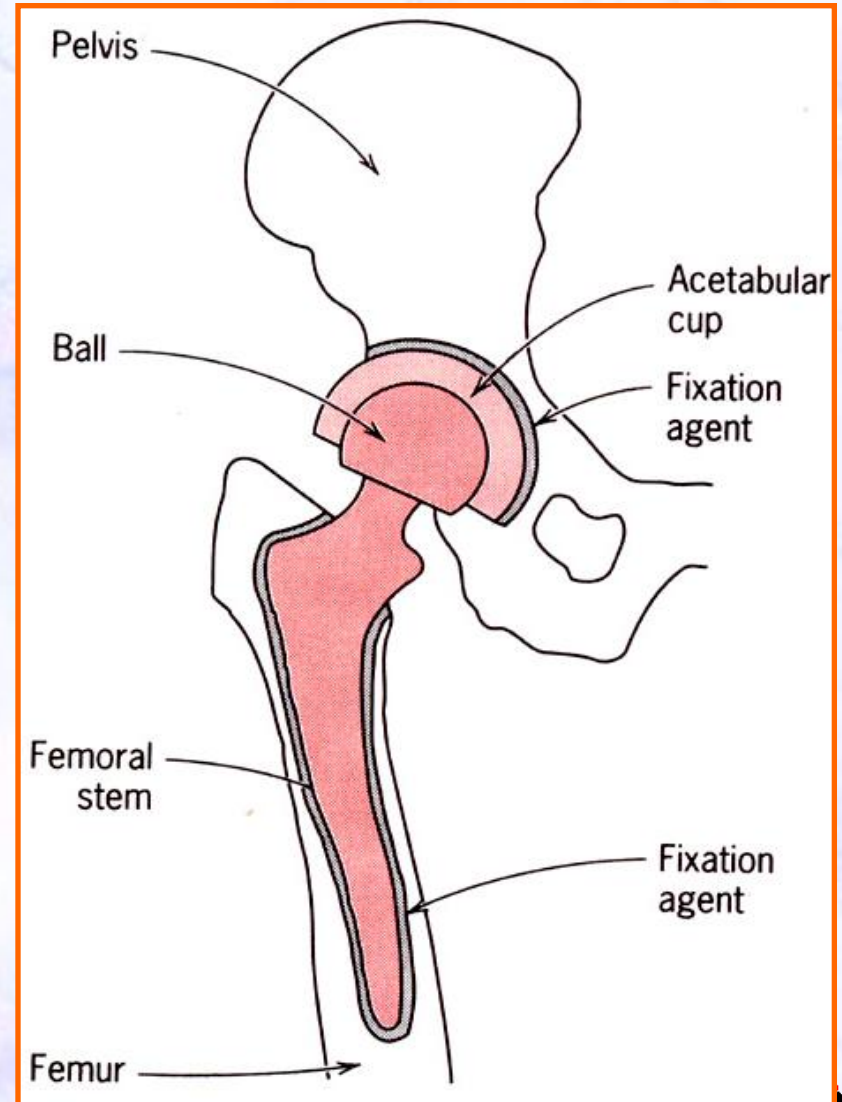
Bone fracture





# 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”

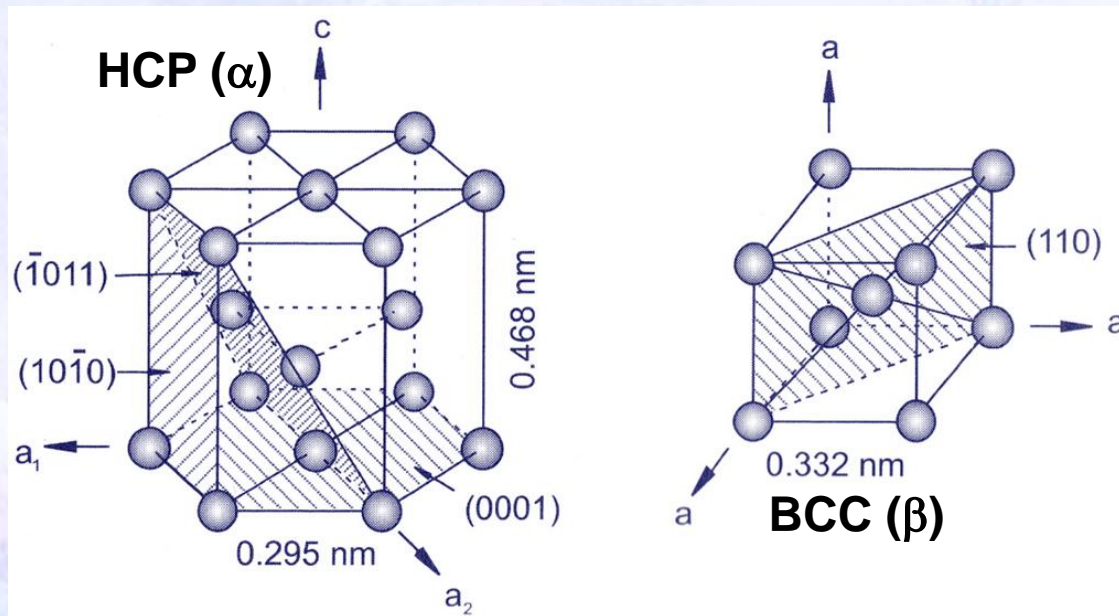


# Objectives

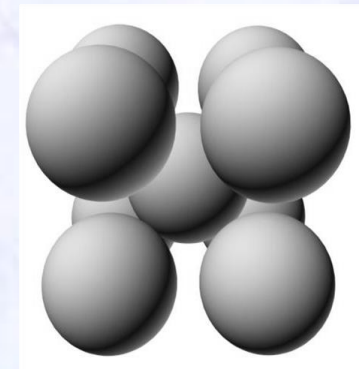
- To discuss phase transformations in  $\beta$  Ti-Nb-Sn alloys:
  - $\alpha$  phase precipitation during aging heat treatment of metastable microstructures
  - Correlation between microstructure and mechanical behavior
  - Application of phase transformations knowledge in Ti-based femoral stem manufacturing

# Titanium Metallurgy

- Titanium shows two allotropic forms:  
HCP and BCC

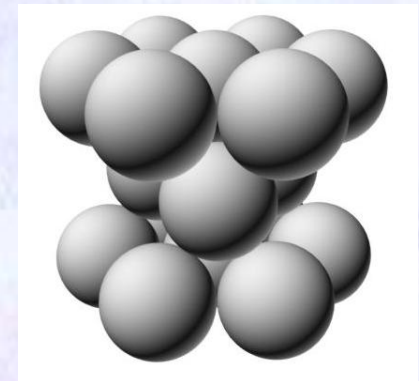


- Addition of alloying elements may change the phase stability and hence, the microstructure and mechanical behavior



BCC ( $\beta$ )

883 °C



HCP ( $\alpha$ )

# $\beta$ Titanium Alloy

## $\beta$ Ti alloys

$\beta$  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**

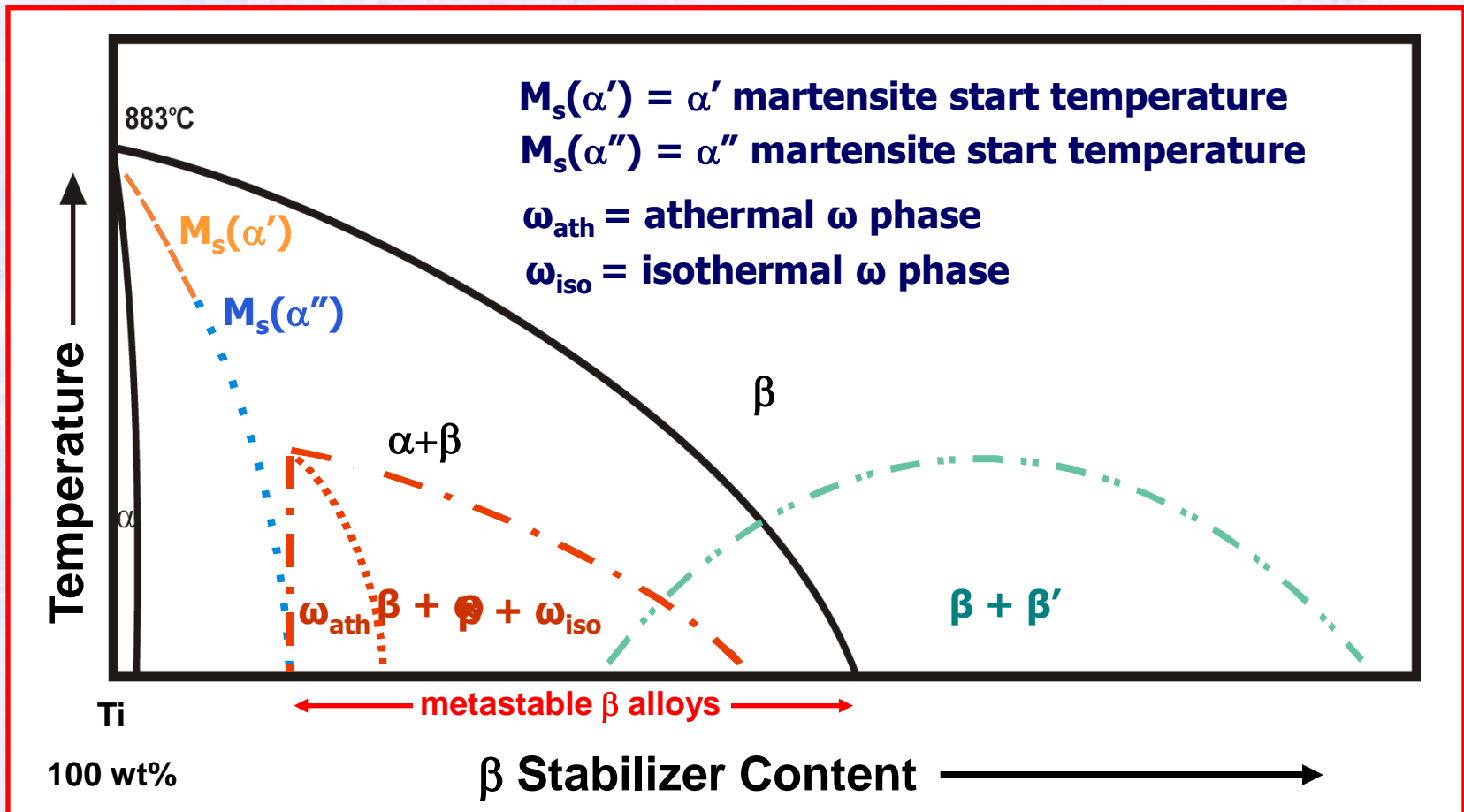
**LOW FORGING TEMPERATURE**

# Ti Alloys Phase Transformations

CHEMICAL  
COMPOSITION AND  
HEAT TREATMENTS

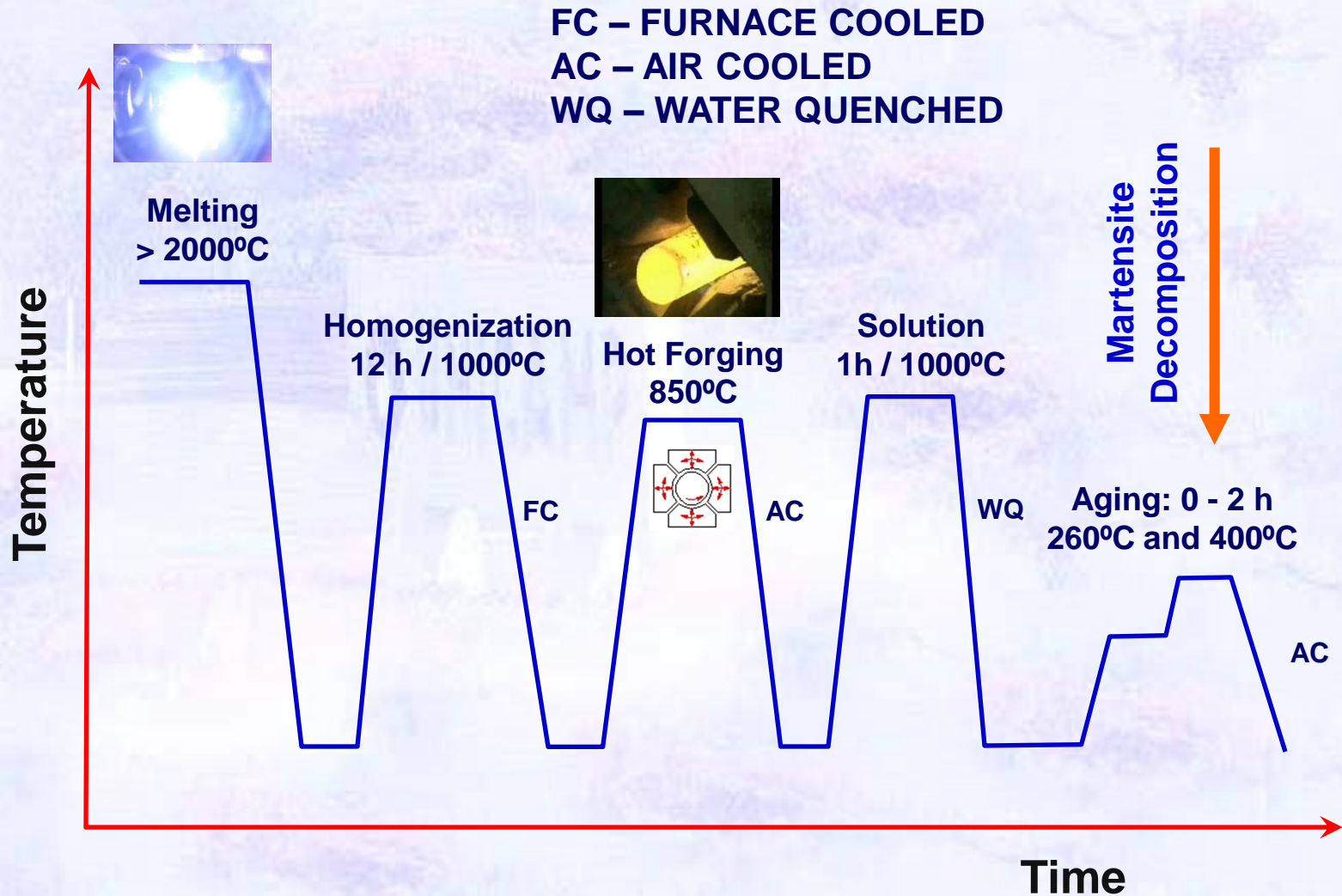


MECHANICAL  
PROPERTIES OF Ti  
ALLOYS



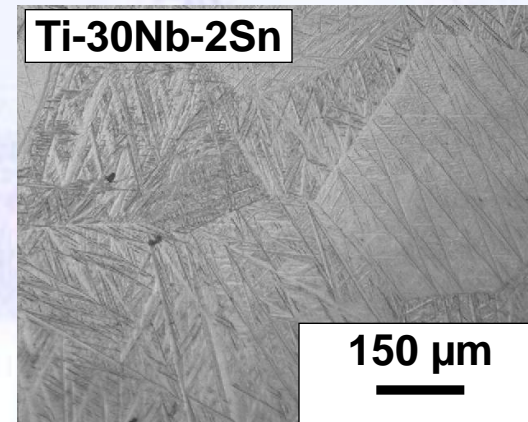
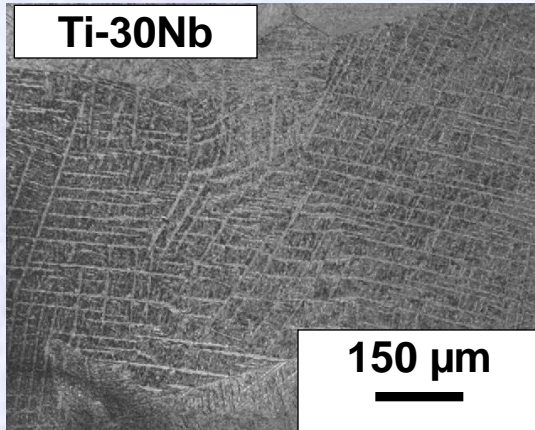
# Processing Route

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

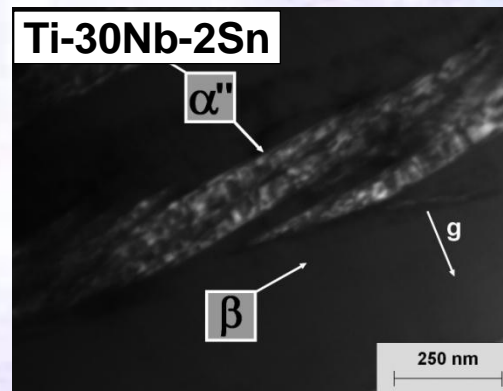
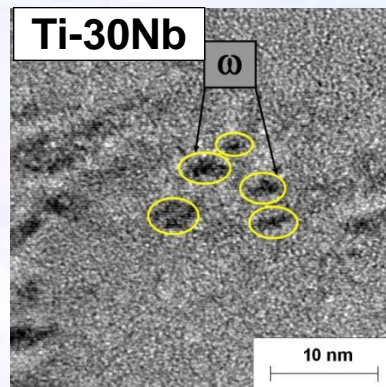


# Effect of Sn on $\alpha''$ Amount

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



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

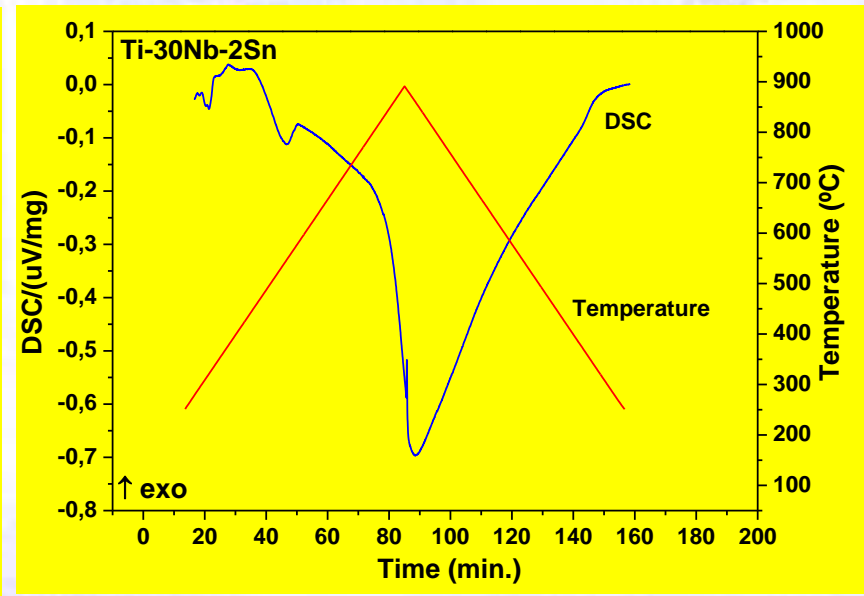
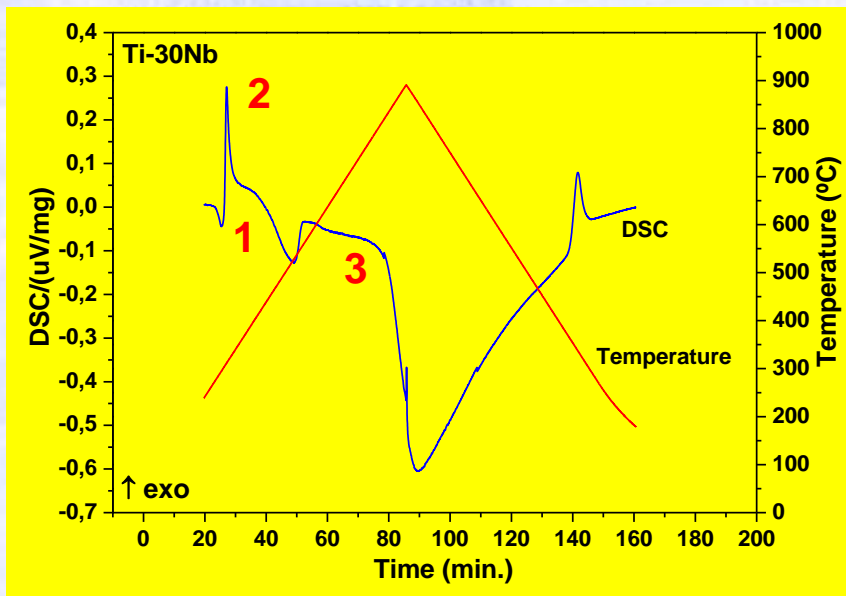


Small amount of nanometric precipitates of  $\omega$  in Ti-30Nb.

# 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

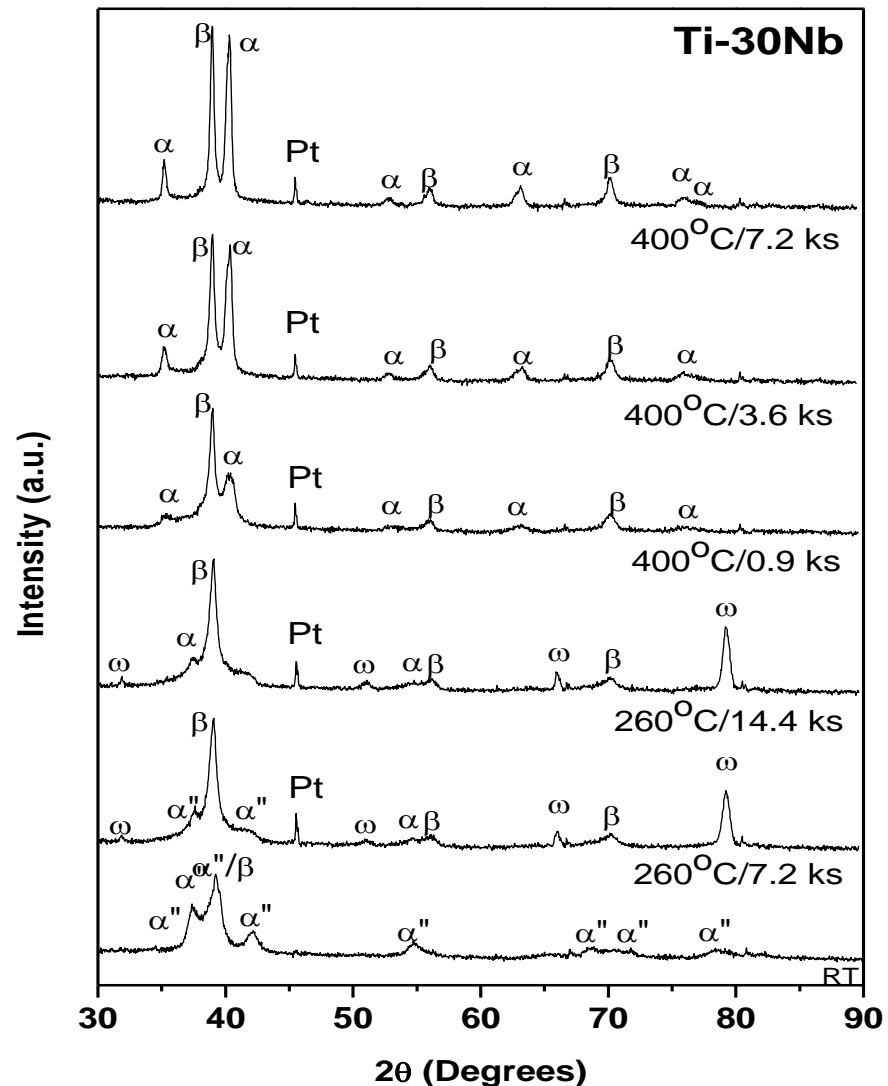
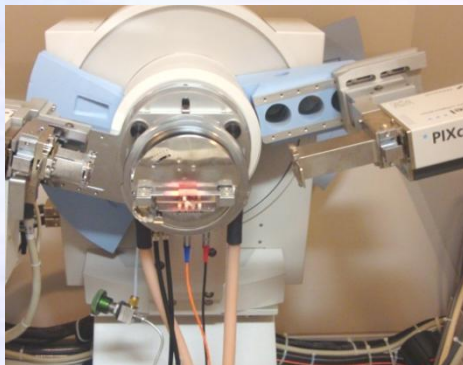




# Martensite Decomposition: Ti-30Nb

## High Temperature X-Ray Diffraction

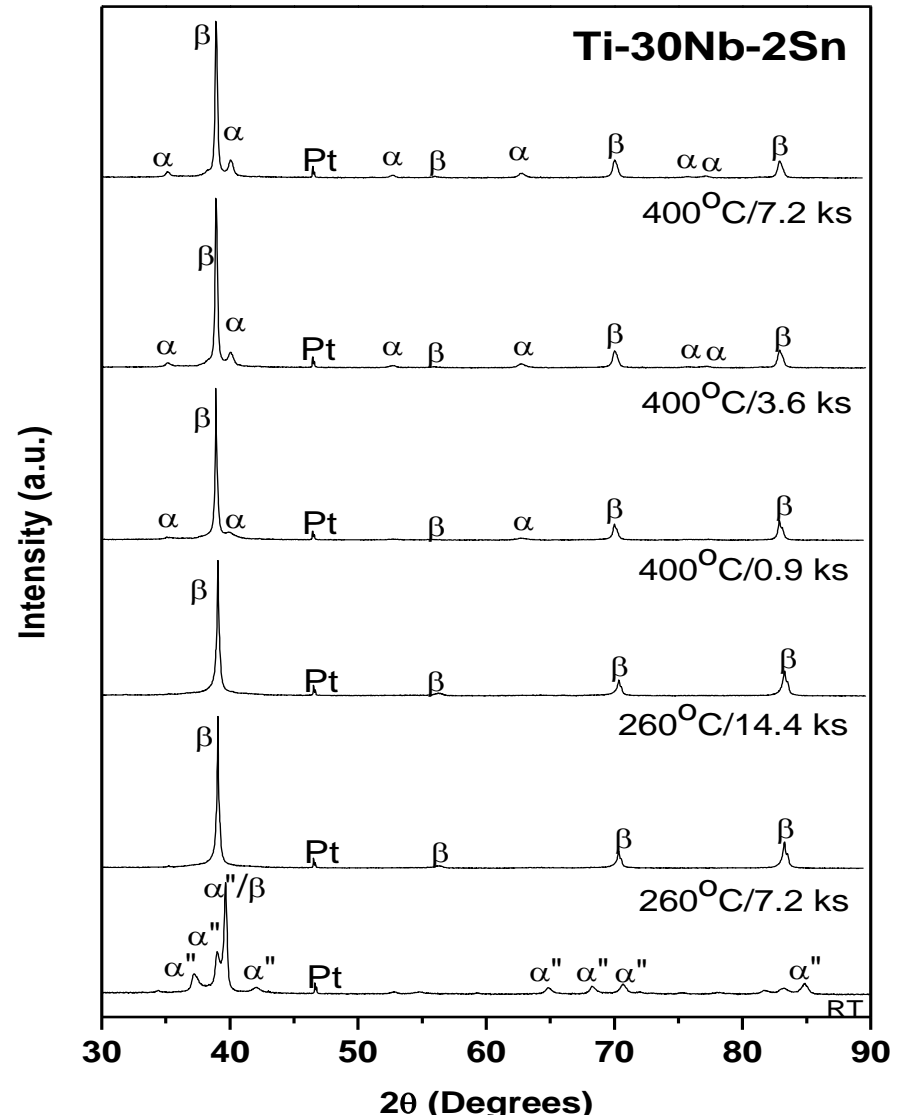
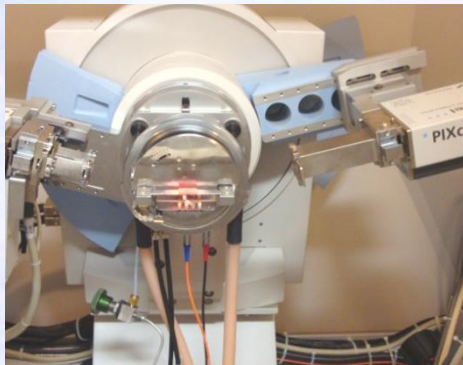
- Martensite decomposition occurred with the aging time
- Reverse transformation of  $\alpha''$  into  $\beta$  phase also took place
- Precipitation of  $\alpha$  and  $\omega$  phases is visible by high temperature XRD



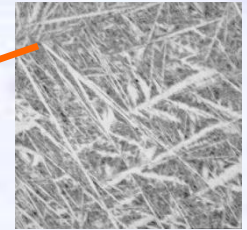
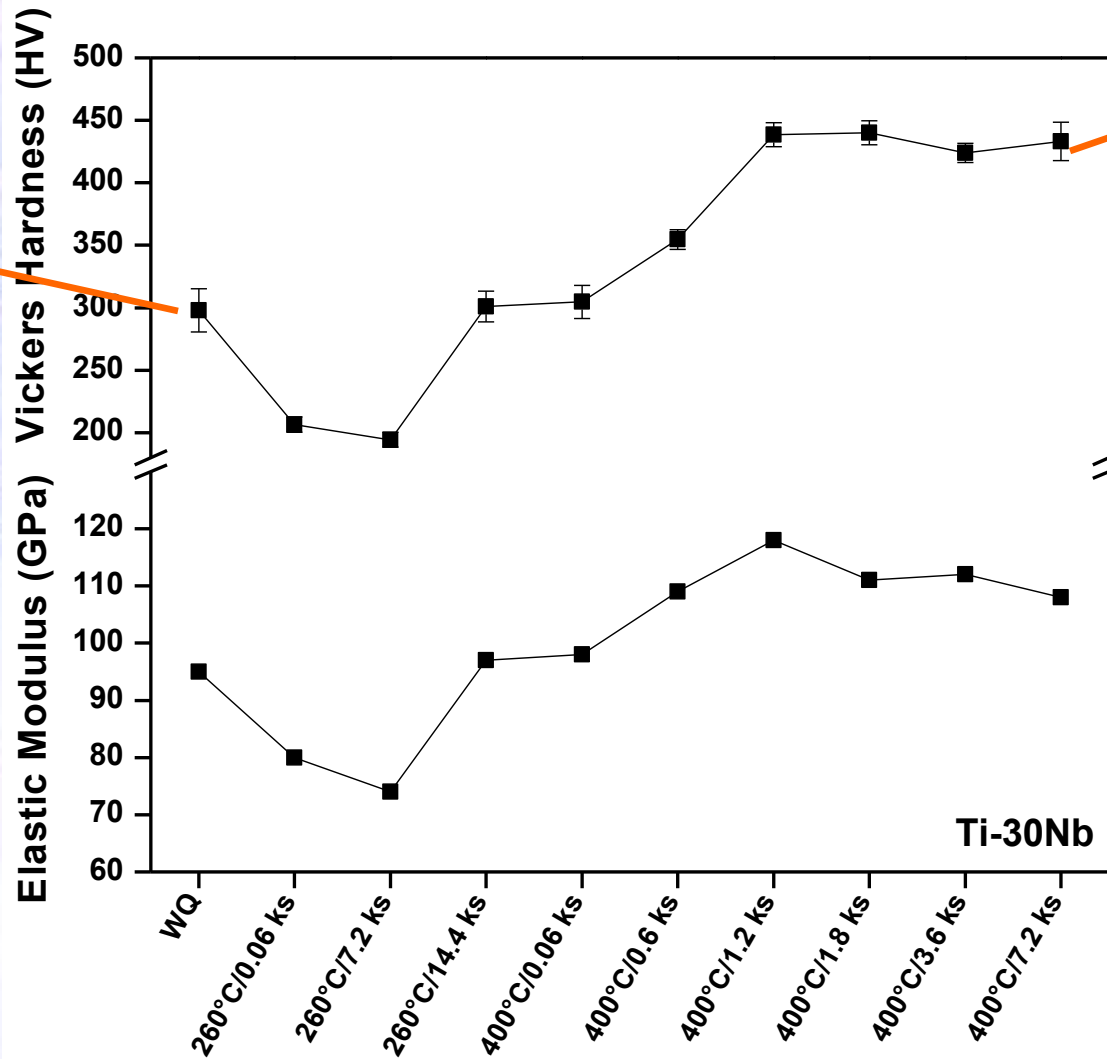
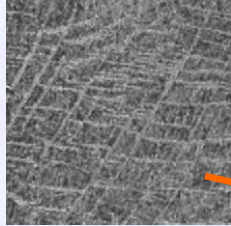
# Martensite Decomposition: Ti-30Nb-2Sn

## High Temperature X-Ray Diffraction

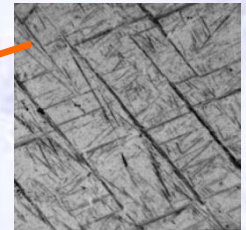
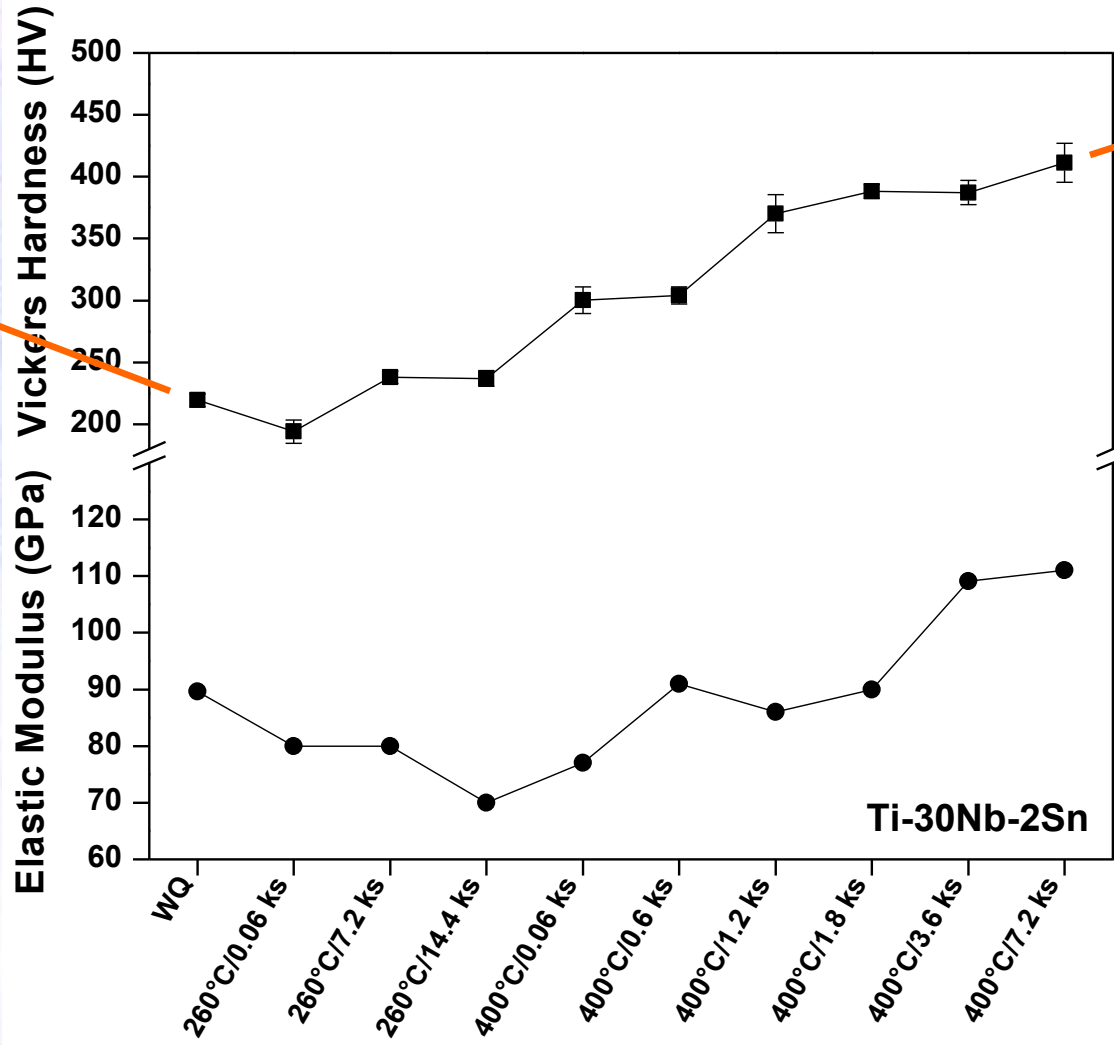
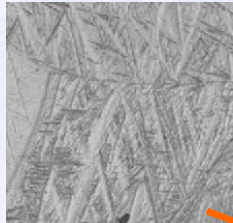
- Martensite decomposition occurred with the aging time
- Reverse transformation of  $\alpha''$  into  $\beta$  phase also took place
- No precipitation of  $\omega$  phase is observed by high temperature XRD



# Mechanical Behavior: Ti-30Nb



# Mechanical Behavior: Ti-30Nb-2Sn



# Tensile Test: Mechanical Properties

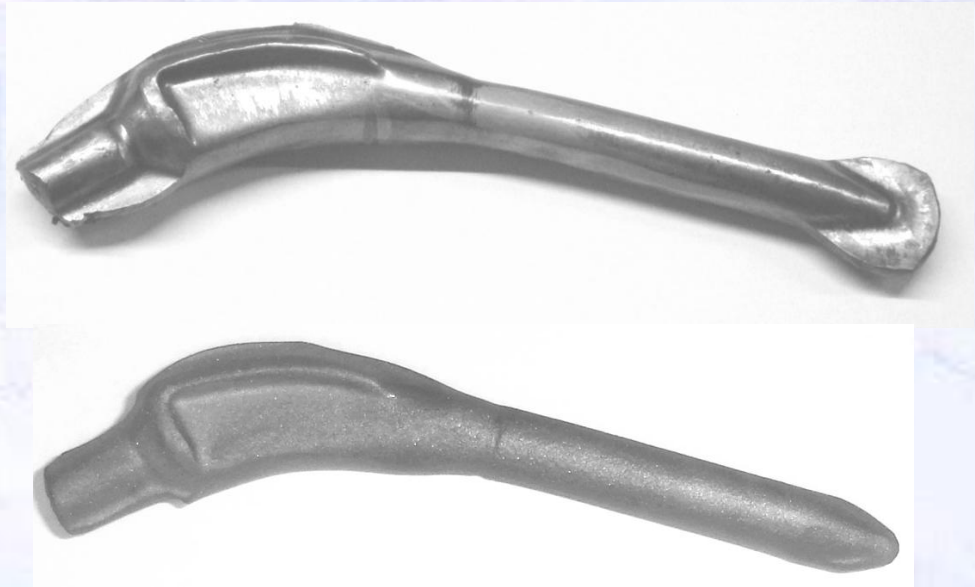
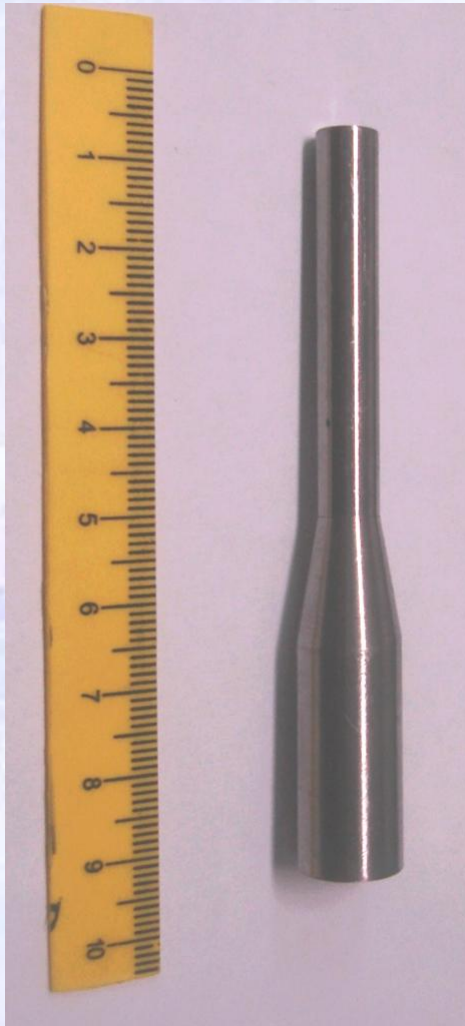
## Effect of aging on mechanical behavior

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

$\omega^{**}$  - 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??**