In Situ Aging Characterization of Ti Alloys Using High Temperature X-Ray Diffraction

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5th International Conference on Diffusion in Solids and Liquids
24 – 26 June 2009
Rome, Italy

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  - Total Hip Replacement / THR Requiriments
  - Bone Elastic Deformation / Implant Elastic Modulus
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- Results
  - High Temperature X-Ray Diffraction
  - DSC
  - 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

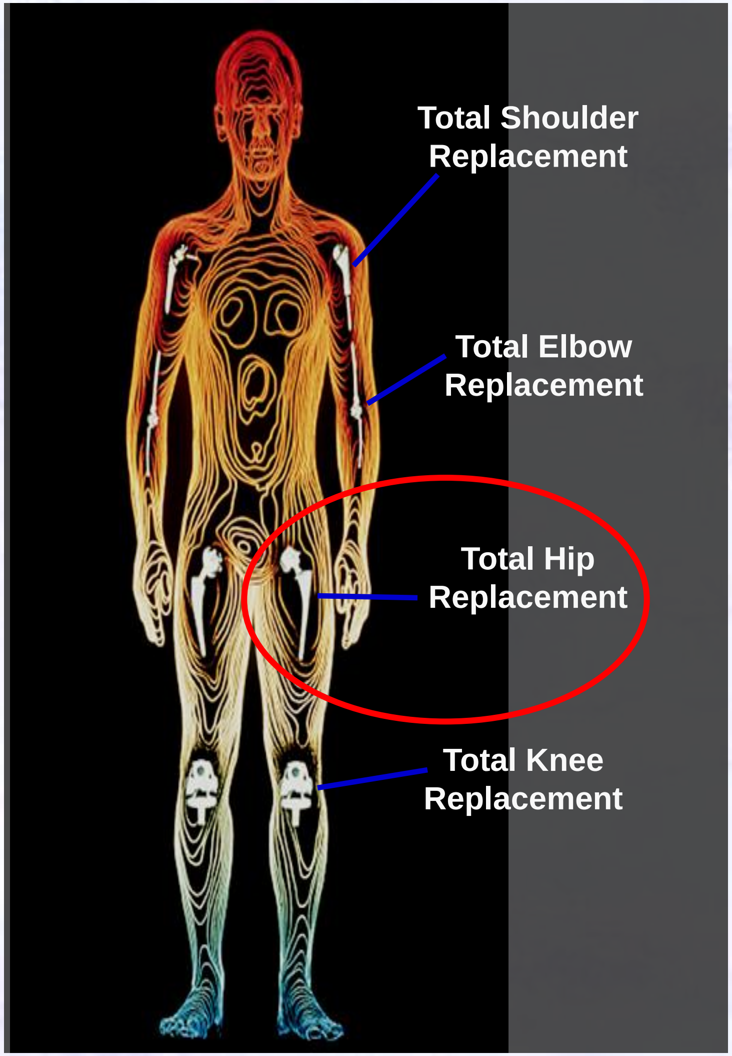
    dental bridge

Orthopedic Implants

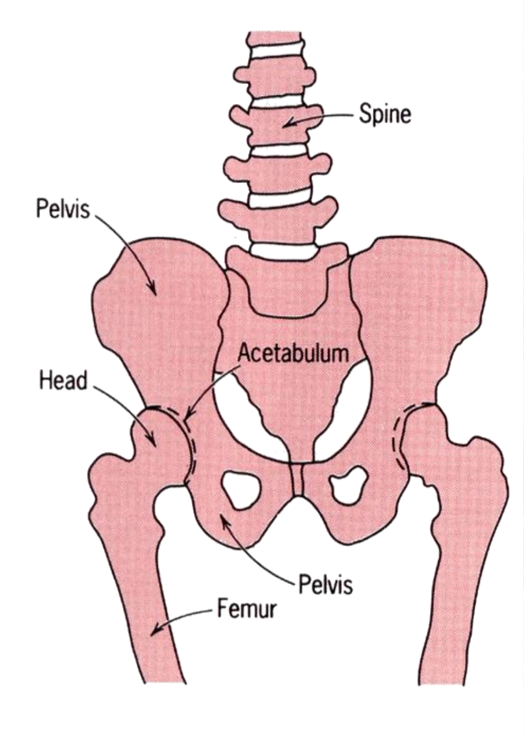
- Orthopedic biomaterials are successful in restoring mobility and quality life to millions of individuals each year
- Success of orthopedic biomaterials may be exemplified by their world market:
  - Annual growth rate of 7% to 9%
  - 2002 Sales of US$ 14 billion
  - Joint replacements: US$ 12 billion
    - Hip implant products: 2.5 billion (700,000 knee replacement surgeries)
    - Knee implant products: US$ 2.5 billion (700,000 knee replacement surgeries)

Total Joint Replacement

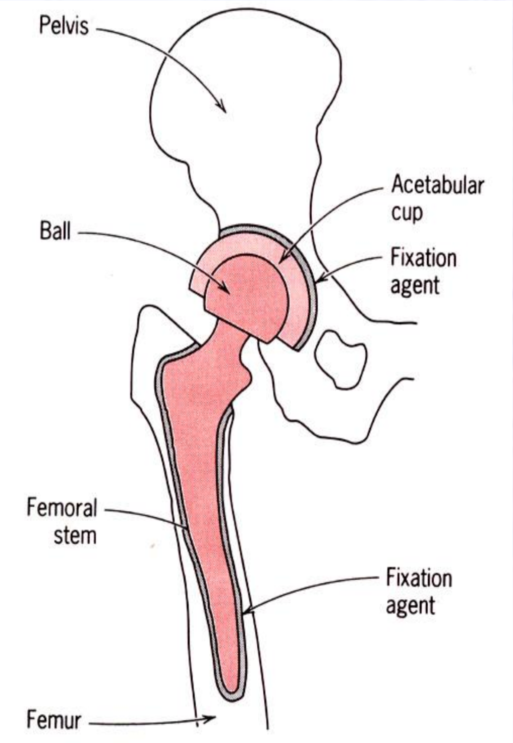
- **TJR is a surgical procedure in which certain parts of an arthritic or damaged joint, are removed and replaced with a plastic or metal device called a prosthesis**
- **Prosthesis is designed to enable the artificial joint to move just like a normal, healthy joint.**



Total Hip Replacement



Hip joints and adjacent skeletal components



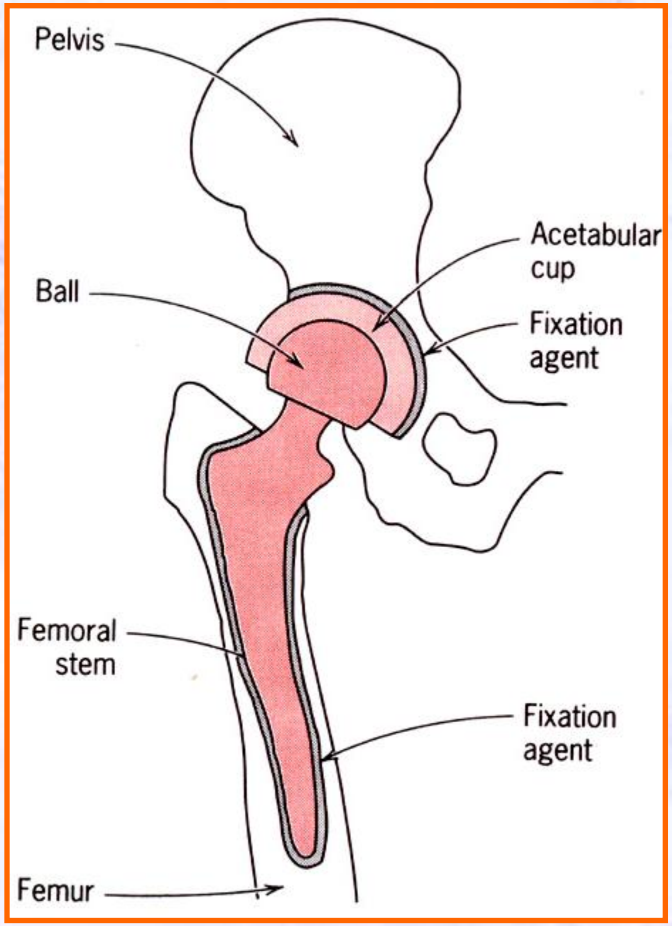
Total hip replacement

Implant after surgery

THR Requiriments

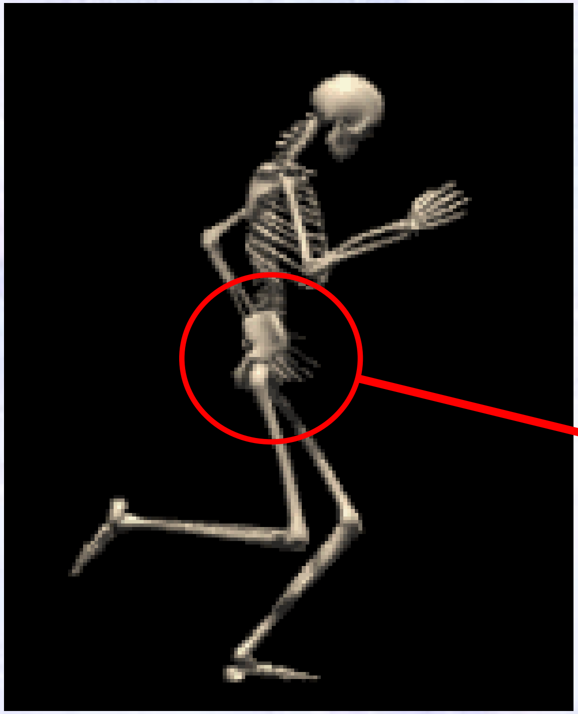
- Biomaterials must show the following properties:
  - High mechanical strength
  - Processability
  - Low prices
  - High biocompatibility
  - High corrosion resistance
  - Must simulate bone elastic behavior

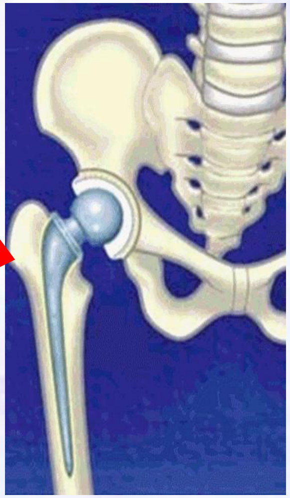
    → low elastic modulus
    - $E_{stainless\ steel}$: 200 GPa
    - $E_{Co\text{-}Cr\text{-}Mo\ Alloys}$: 230 GPa
    - $E_{Ti\text{-}CP}$: 110 GPa
    - $E_{Ti\text{-}6Al\text{-}4V}$: 106 GPa
    - $E_{\beta\text{-}alloys}$: <60 GPa
    - E_{bone}: 10 - 30 GPa



Bone Elastic Deformation

- Implant material must simulate bone elastic behavior
- Insufficient load transfer from the implant to the bone causes bone re-absorption and loosening of the implant device
- Reduction of load applied to the bone causes bone mass loss and osteoporosis





Bone fracture

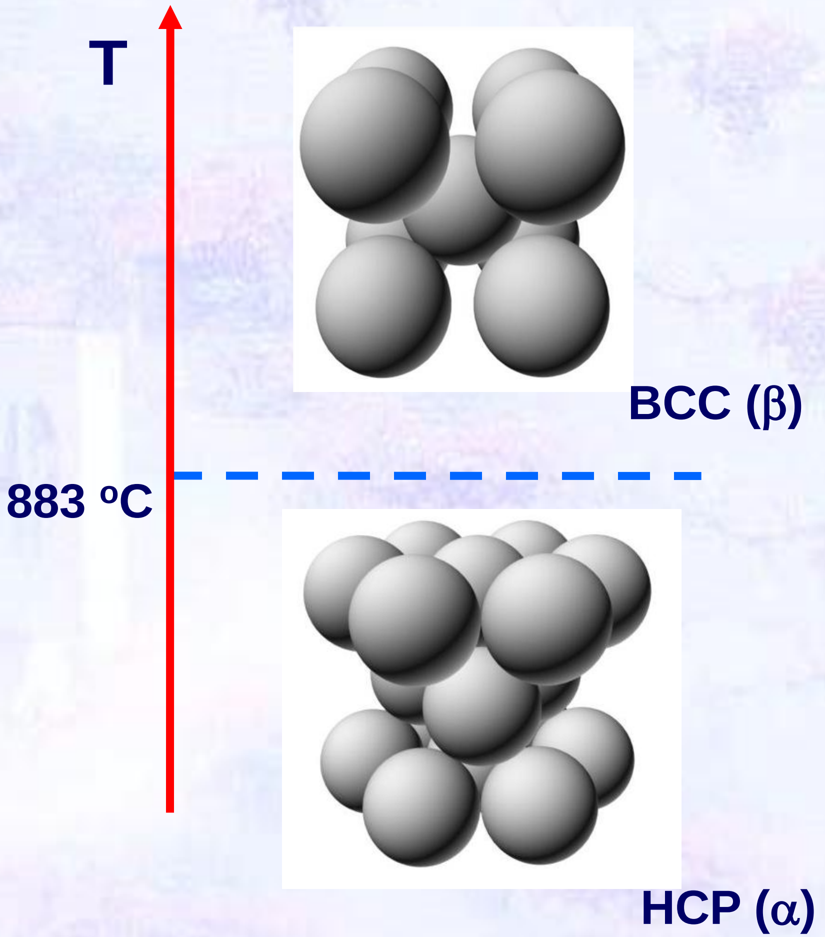
Stainless steel 316L
E = 200 GPa

Objectives

- The main aim of this research is to investigate β titanium alloys to be used as orthopedic biomaterials
- This work attempts to examine phase transformations during aging heat treatment of β Ti-Nb alloys with Sn additions and to correlate microstructure and mechanical behavior using high temperature X-ray diffraction.

Titanium Metallurgy

- Titanium shows two allotropic forms: HCP and BCC
- Addition of alloy elements may change the phase stability and hence, the microstructure and mechanical behavior



β Titanium Alloy

β Ti alloys

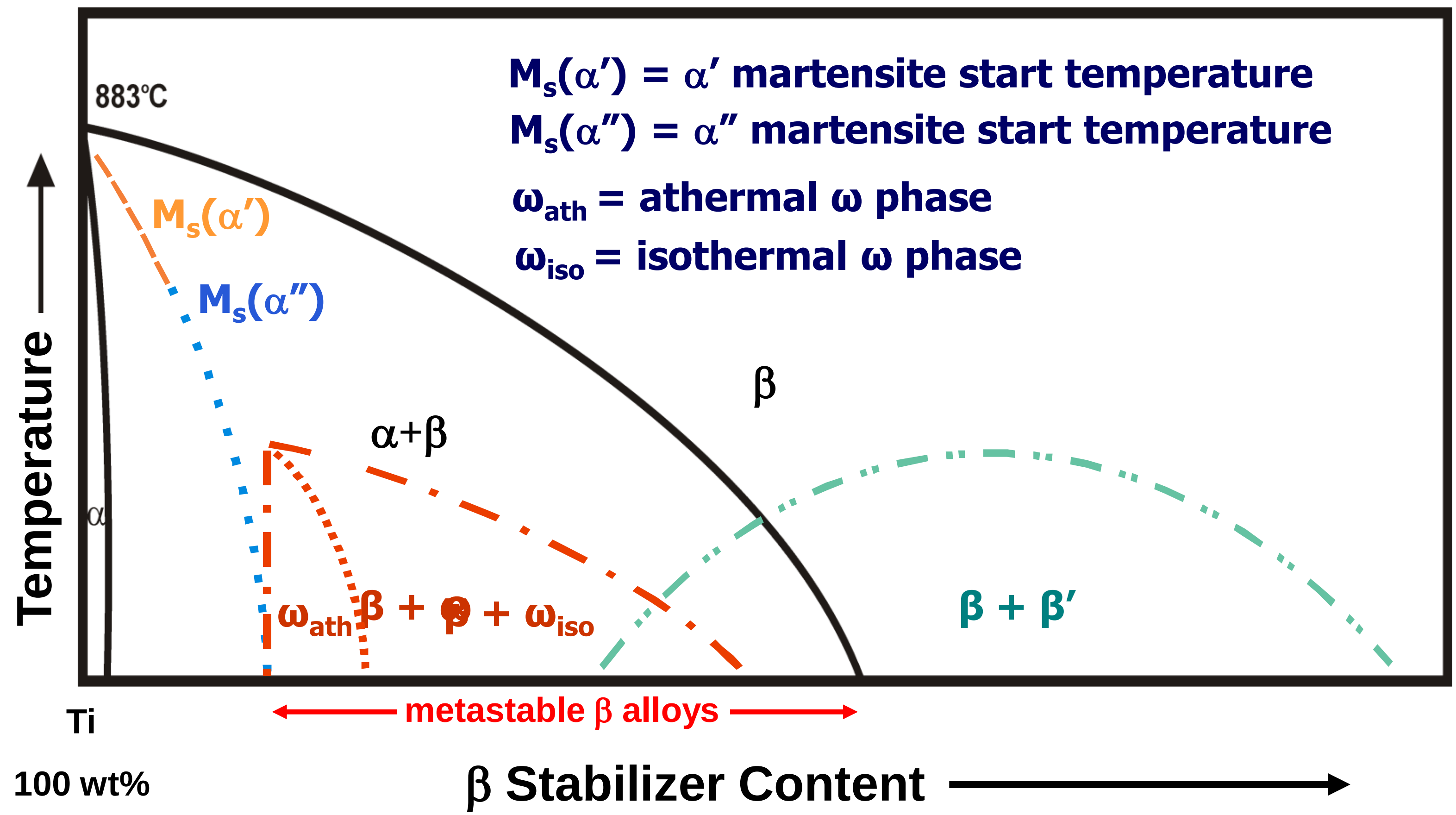
β Stabilizer elements:
Cr, Nb, V, Ta, Mo

- **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

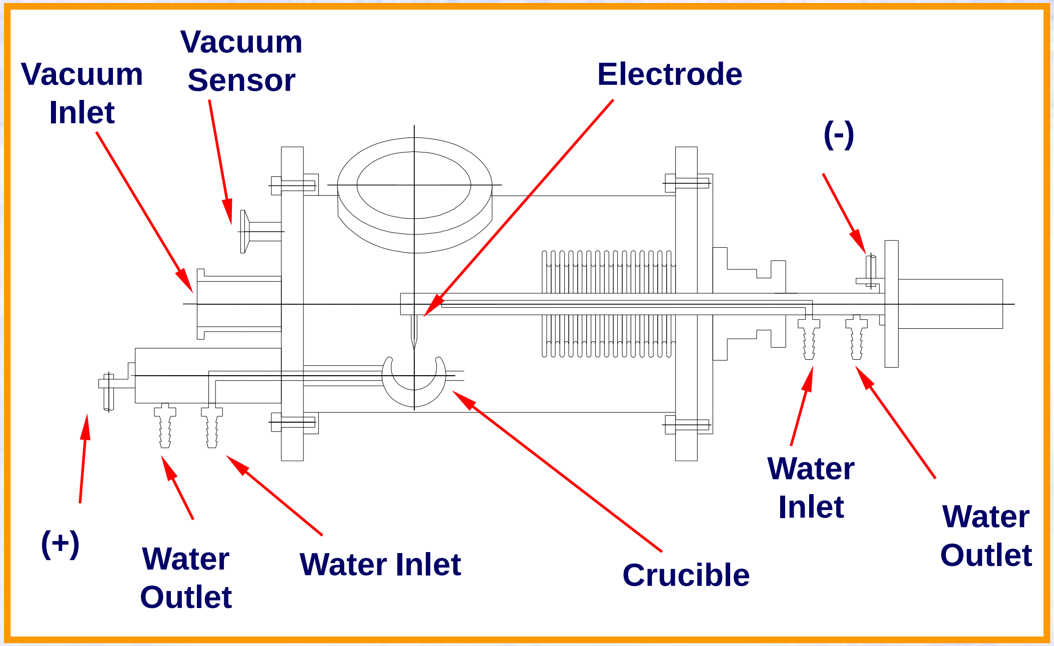
MECHANICAL PROPERTIES OF Ti ALLOYS → CHEMICAL COMPOSITION

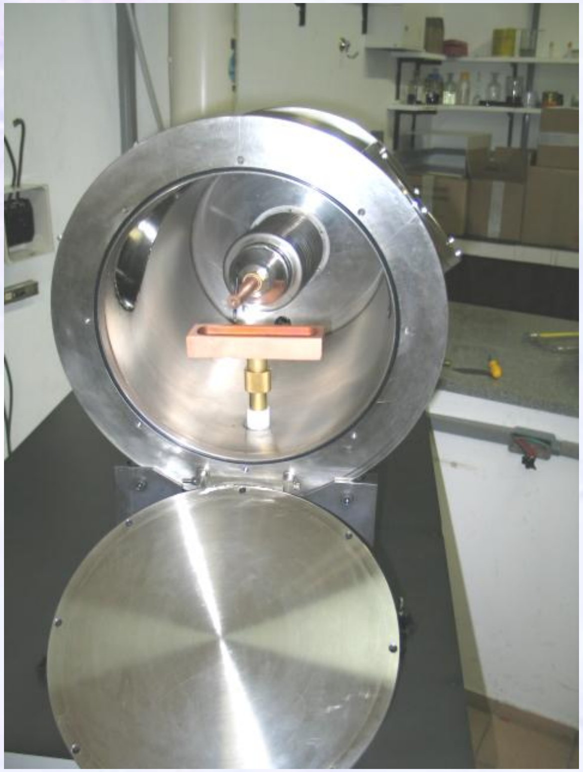
MICROSTRUCTURE



Alloy Preparation

- Alloys were prepared by using high purity Ti, Nb and Sn
- Alloys were melted in arc furnace with non-consumable W electrode and water cooled copper hearth under Ar atmosphere

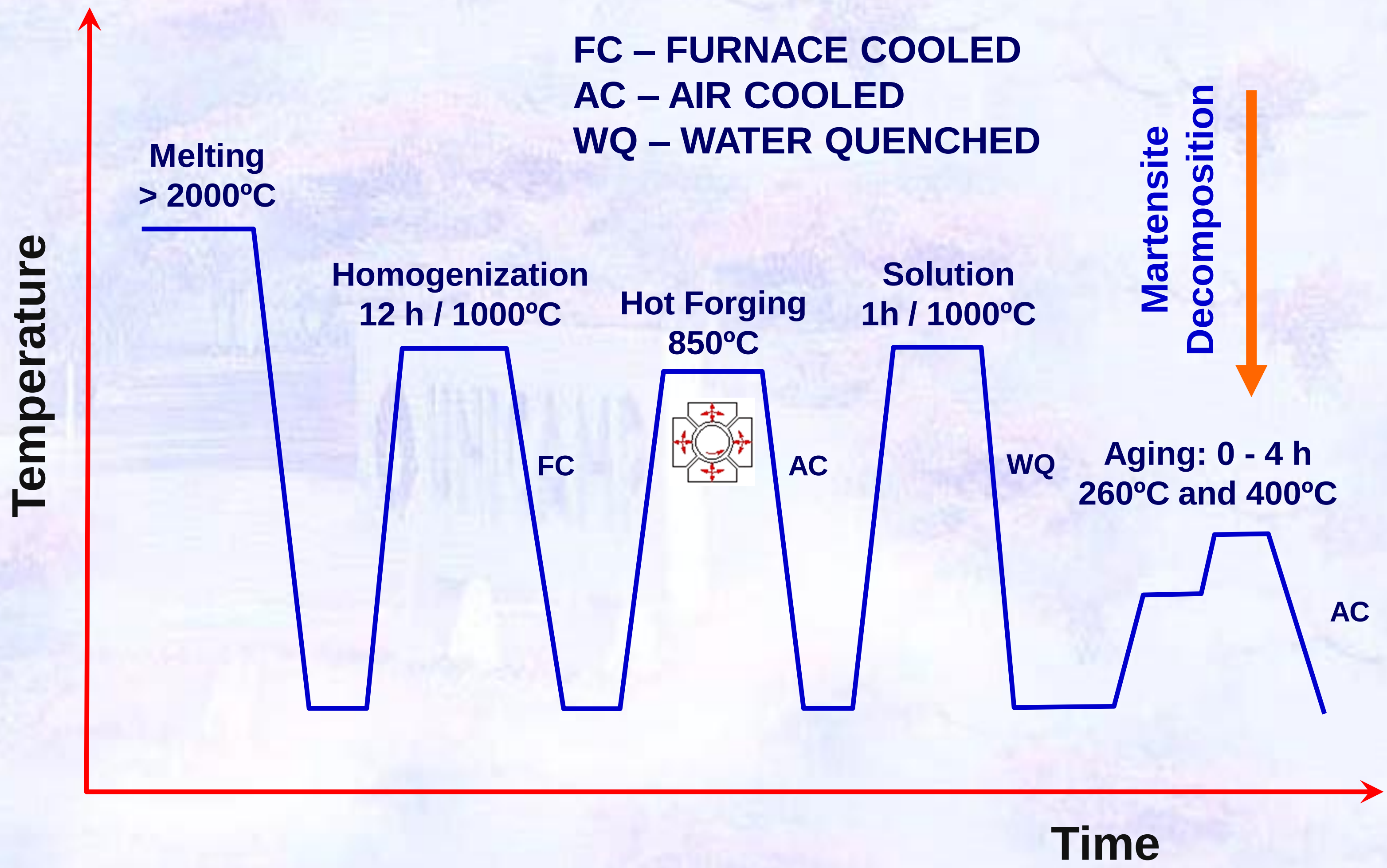




Alloy Composition

Nominal (%wt)	Measured (%wt)
Ti-30.0Nb	Ti-30.4Nb
Ti-30.0Nb-2.0Sn	Ti-30.5Nb-2.1Sn
Ti-30.0Nb-4.0Sn	Ti-30.6Nb-1.9Sn

Processing Route



Sample Characterization

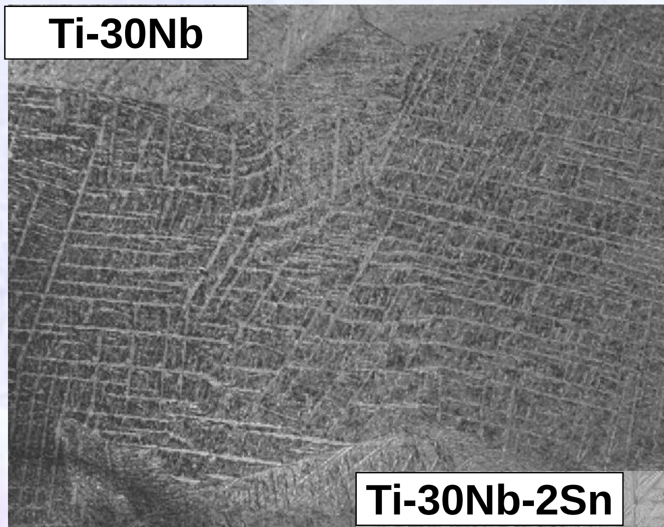
- **Alloys chemical composition: X-ray fluorescence spectrometry**
- **Phase transformations : differential scanning calorimetry**
- **Phase detection: X-ray diffraction**
- **Phase evolution (Aging): high temperature X-ray diffraction**
- **Metallographic preparation: mechanical grinding using SiC sandpaper up to 1200 mesh, polishing with 6 and 1 μm diamond paste**
- **Samples were etched in a Kroll's solution:**

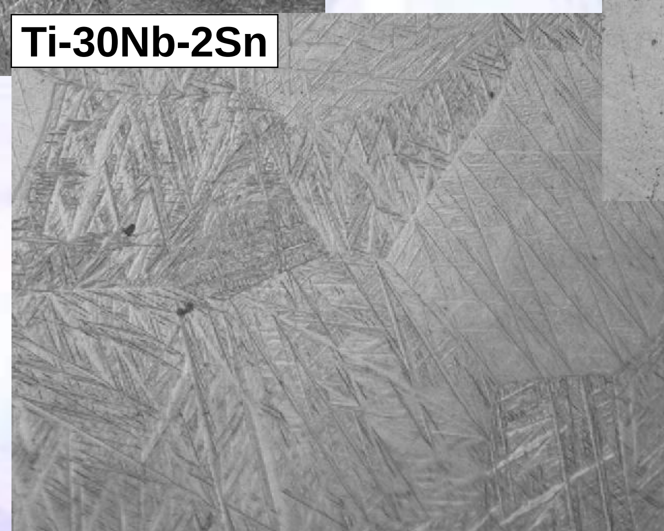
  5 % vol HF, 30 % vol HNO_3 and 65 % vol H_2O

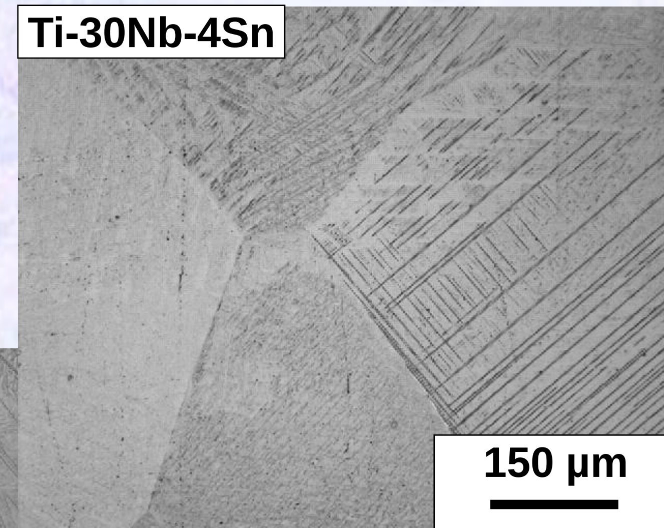
Effect of Sn on α” Amount

Effect of Sn addition on the amount of martensite

Water Quenched Samples

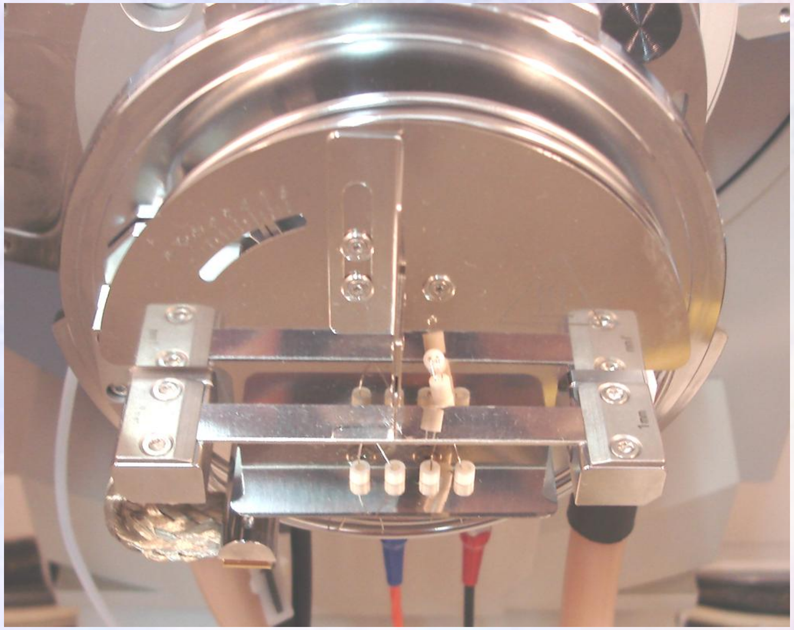


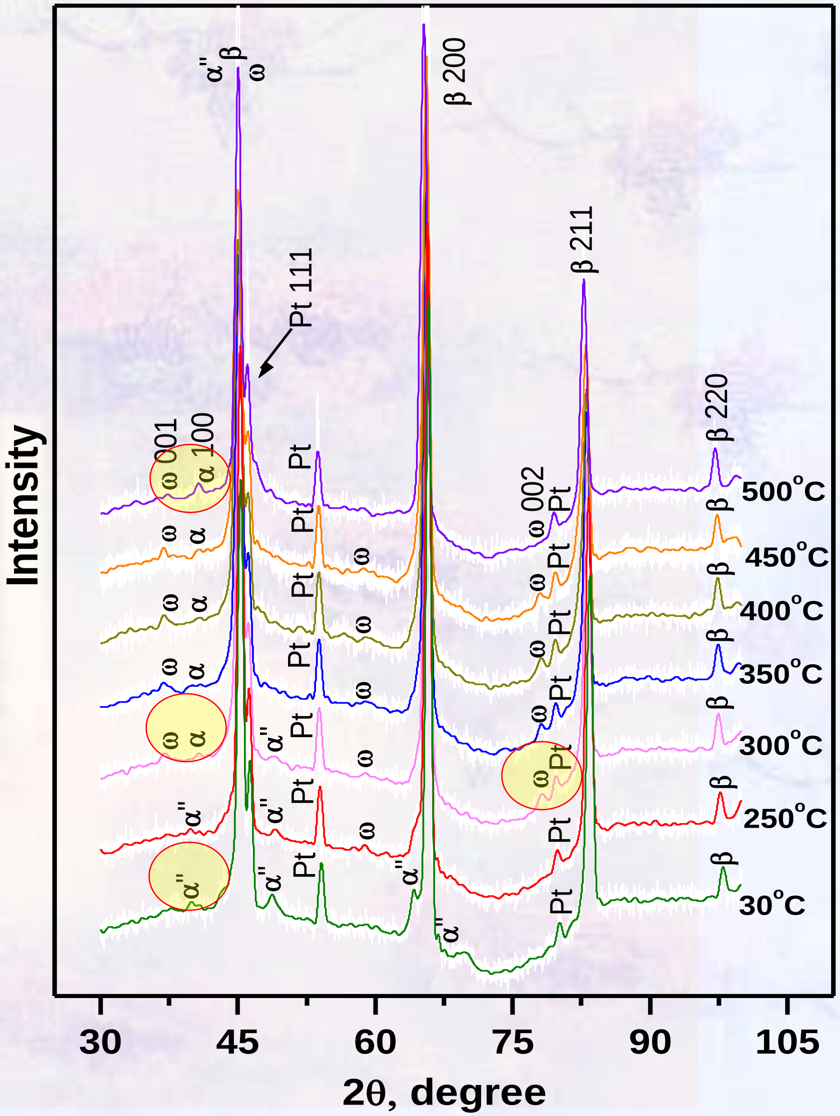




α” Decomposition

In Situ Aging Characterization of Ti Alloys Using High Temperature X-Ray Diffraction Kα Co: λ=0.17890 nm

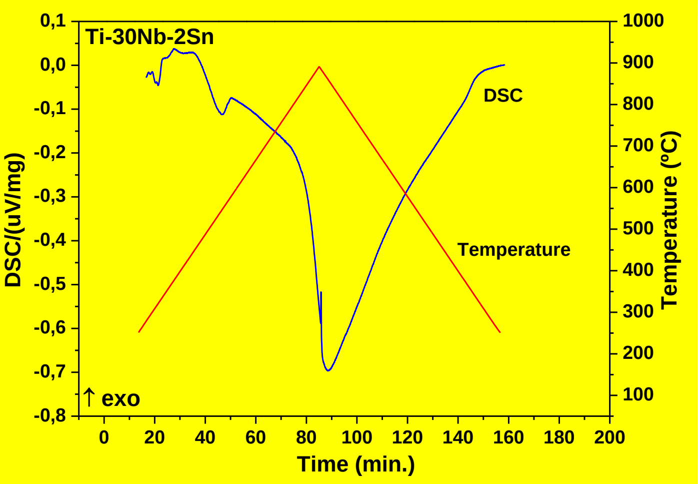


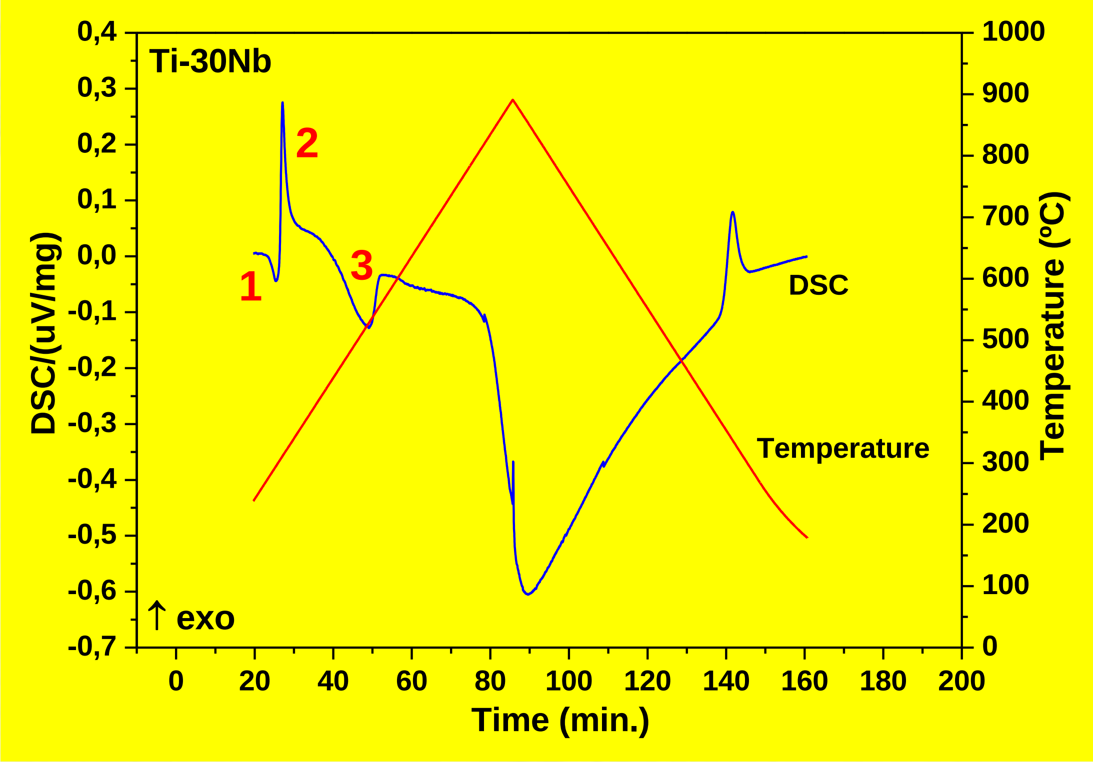


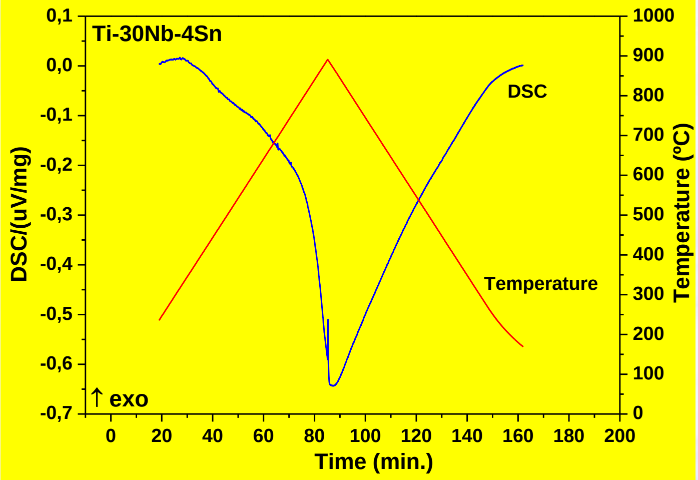
α” Decomposition

Thermal Analysis – DSC

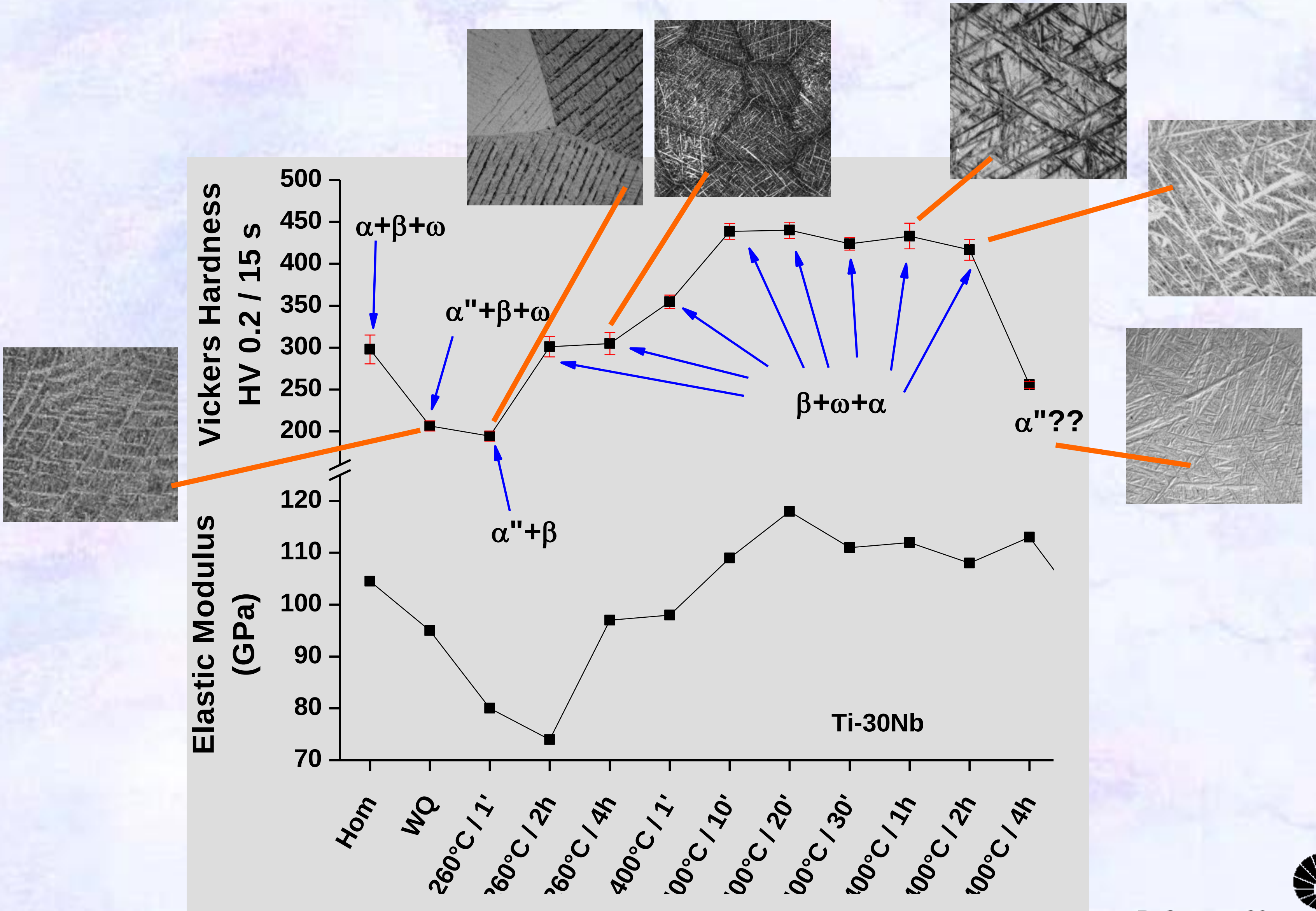
- WQ Ti-30Nb sample with α” and β phases
- Peak 1: reverse transformation α”→ β
- Precipitation of ω in β matrix (end of peak 1)
- Peak 2: nucleation of α - “ω act as substrates”
- Peak 3: β transus



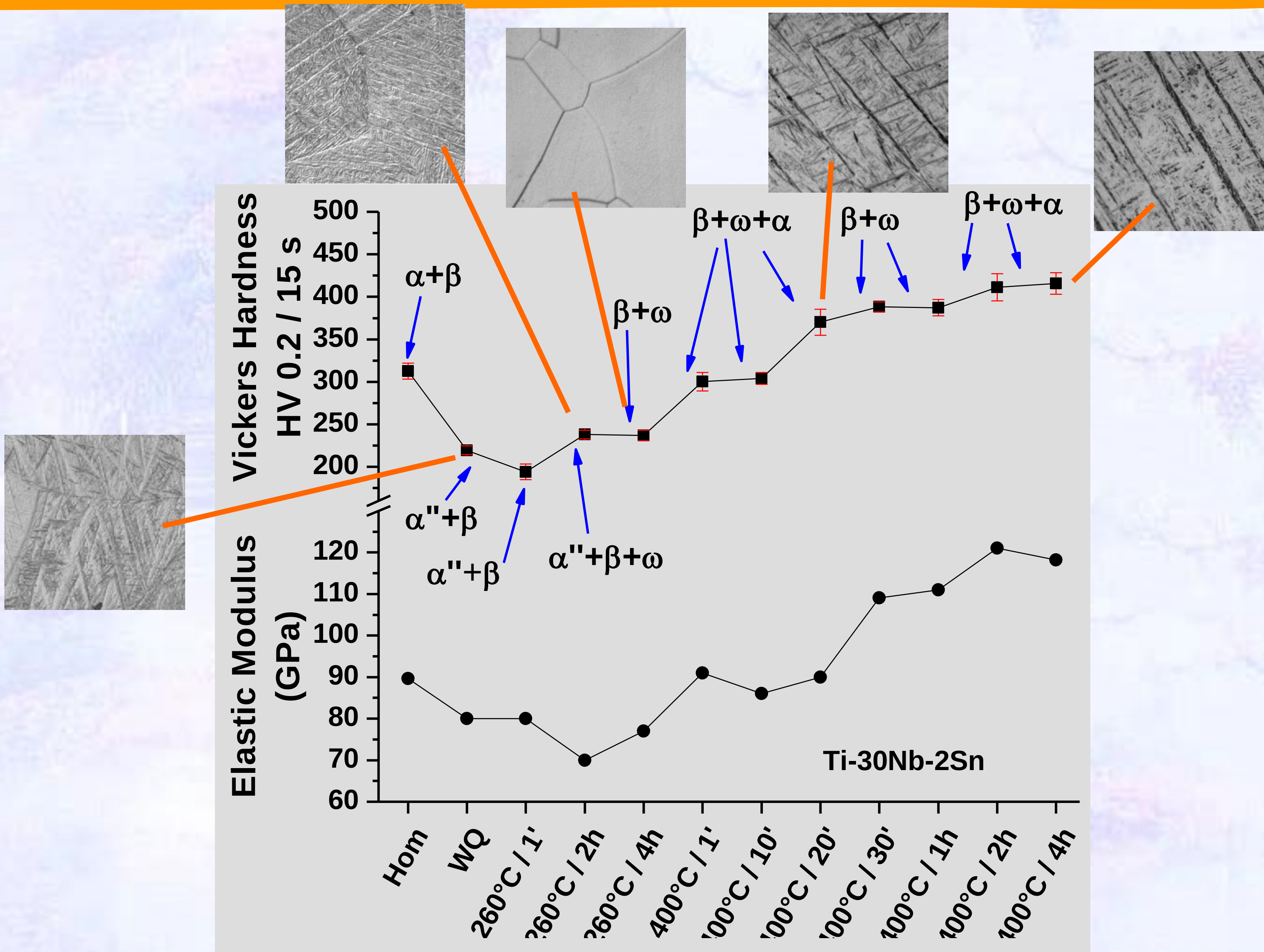




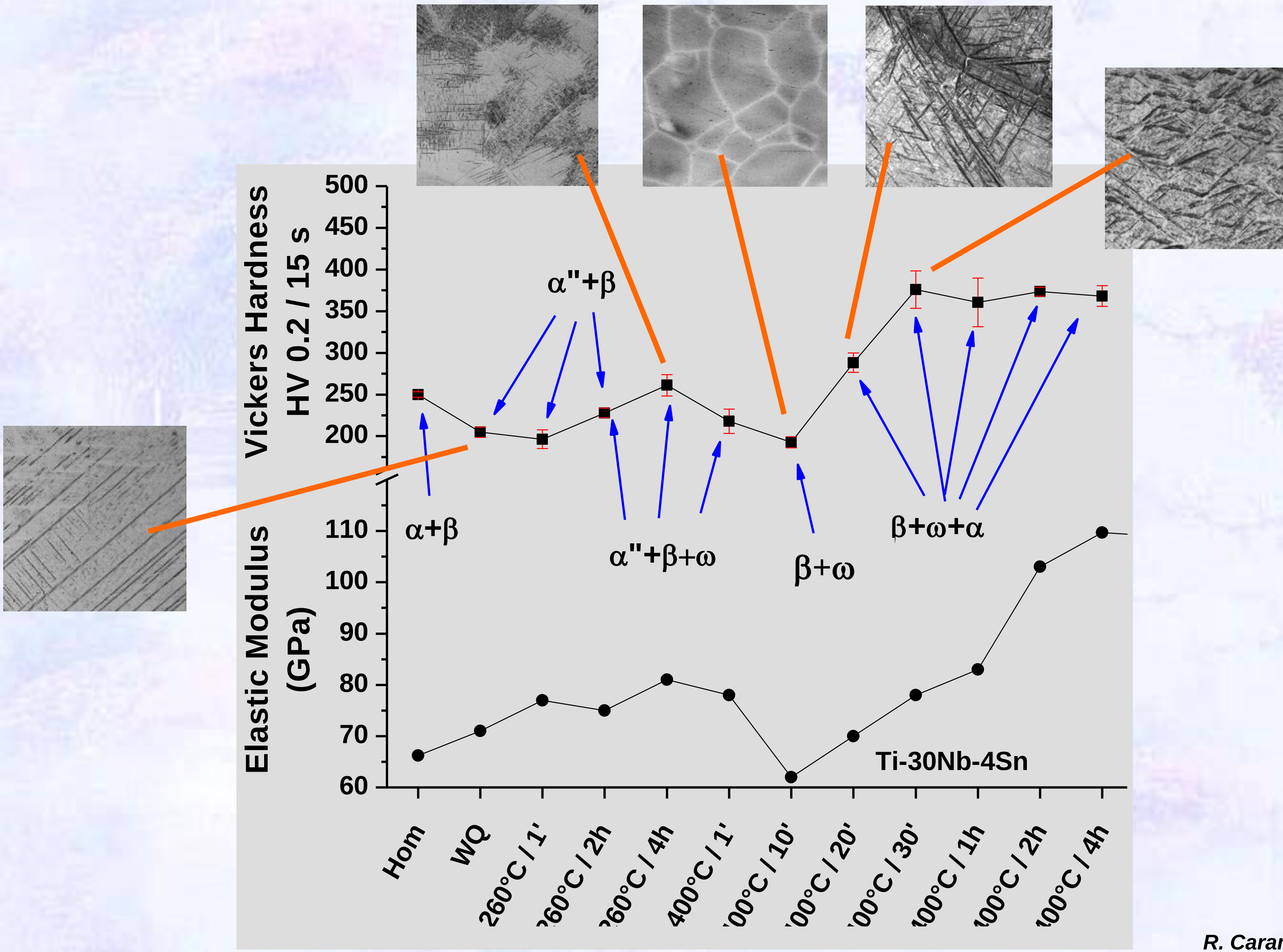
Mechanical Behavior



Mechanical Behavior



Mechanical Behavior



Tensile Test: Mechanical Properties

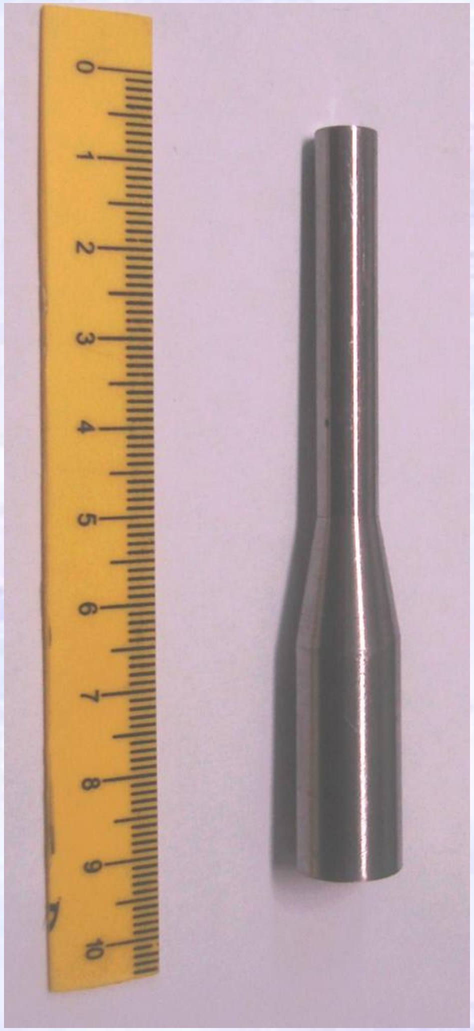
Effect of aging on mechanical behavior

Alloy Condition	Phases (XRD)	σ_{UTS} (MPa)	Elong (%)	E (GPa)	Hardness (HV)
Ti-30Nb Full β	β+ ω	532 ± 21	30 ± 7	74	199 ± 6
Ti-30Nb Aged	β+α+ω	826 ± 24	0.8 ± 0.1	105	424 ± 10
Ti-30Nb-2Sn Full β	β	500 ± 32	36 ± 4.0	70	219 ± 5
Ti-30Nb-2Sn Aged	β+α+ω*	857 ± 22	0.8 ± 0.2	100	432 ± 15
Ti-30Nb-4Sn Full β	β	531 ± 20	21.6 ± 1.2	62	211 ± 7
Ti-30Nb-4Sn Aged	β+α+ω**	937 ± 18	1.2 ± 4.3	101	387 ± 11

ω* - small amount **ω** - very small amount**

Cold Forging

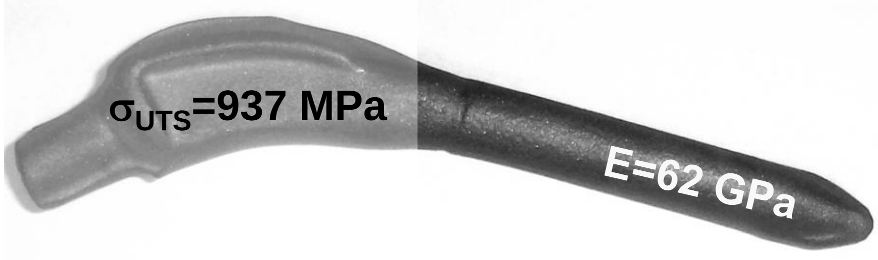
Cold Forged Femoral Stem using Ti-30Nb-4Sn alloy



Optimized Mechanical Behavior

- **Problem: When using aging process, it is virtually impossible to obtain a β Ti alloy with high mechanical strength and low elastic modulus**
- **Solution: Application of different heat treatment procedures according to the region of the prosthesis**

Ti-30Nb-4Sn



Conclusions

- In WQ condition (1h/1000ºC/WQ) the microstructure of Ti-30Nb, Ti-30Nb-2Sn and Ti-30Nb-4Sn alloys was formed by β and α" phase and the amount of α" decreases with increase of Sn;
- Aging procedure allowed to verify that α" decomposition results in precipitation of β, ω and finally, α phases;
- Results suggest that Sn may act as a suppressor of ω phase precipitation, which allows the control of microstructure features and hence, mechanical properties
- While rapid quenched Ti-Nb-Sn samples showed yield strength below 310 MPa, which makes easier cold forging process, whose aged sample value increased up to 900 MPa
- Full β alloy showed elastic modulus below of 62 GPa
- These final values are very suitable in terms of orthopedic biomaterial applications

Questions??