Phase Evolution in Proton-irradiated (α+β) Ti6Al4V

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Brookhaven Linac Isotope Producer

NSLS-II

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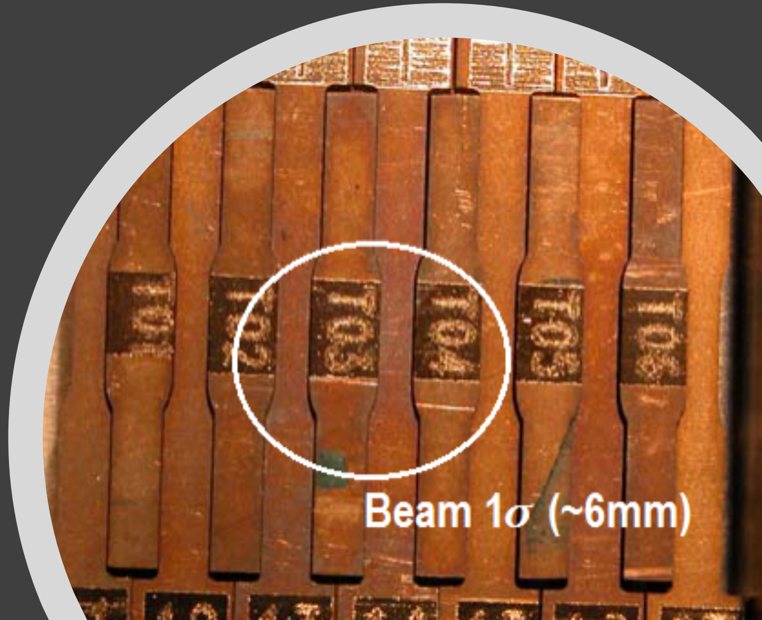
Overview

(α+β) Ti6Al4V considered in Neutrino Factory-Muon Collider (mid-Z material)

- Irradiation:
  - With protons at BNL-BLIP in two (2) phases to ~5×10^{20} p/cm^2 (total)
  - Post-irradiation annealing between phases
- Post-irradiation thermal analysis (precision dilatometry) and mechanical testing:
  - Thermal analysis to ~810 ^{o}C revealed phase transitions and the effects of irradiation in shifting the temperature ranges for the transitions
  - Mechanical testing revealed
    - Loss of ductility
    - More significantly ⇨ ***almost complete loss of UNIFORM ELONGATION***
- Energy Dispersive X-ray Diffraction with in-situ pure bending stress at NSLS synchrotron revealed:
  - The appearance of a ***faint new phase that looks like the ω phase***
  - Symmetries between tension and compression
- XRD experiments at NSLS-II with in-situ pure bending stress state combined with Refined Rietveld analysis showed:
  - Fluctuations between α and β phases as a result of the level of straining
  - ***Further evidence that the ω phase in <10nm size has formed as a result of irradiation***

Ti6Al4V Irradiation Experiments

- **Phase-I**: 140 MeV protons at BLIP
  - Peak fluence of ~ **1.1×10^{20}** p/cm^2
- **Phase-II**: Selected samples from the array were re-irradiated following thermal analysis between irradiations.
  - CTE Samples: Peak fluence of **~2.6×10^{20} p/cm^2** following 2nd irradiation (Samples: T02 and T04)
  - TENSILE samples: Peak fluence of **~5.14×10^{20} p/cm^2** after 2nd irradiation
- **Post-irradiation Analyses:**
  - Thermal analysis (dilatometry) on CTE type specimens
  - Mechanical testing (tensile fracture)
  - EDXRD analysis (NSLS) with 200 keV polychromatic X-rays
    - In-situ 4-point bending (pure bending stress)
  - XRD analysis with 67 keV monochromatic X-rays (NSLS-II XPD beamline)
    - Also with in-situ 4-point bending stress
  - Refined Rietveld analysis for phase ID

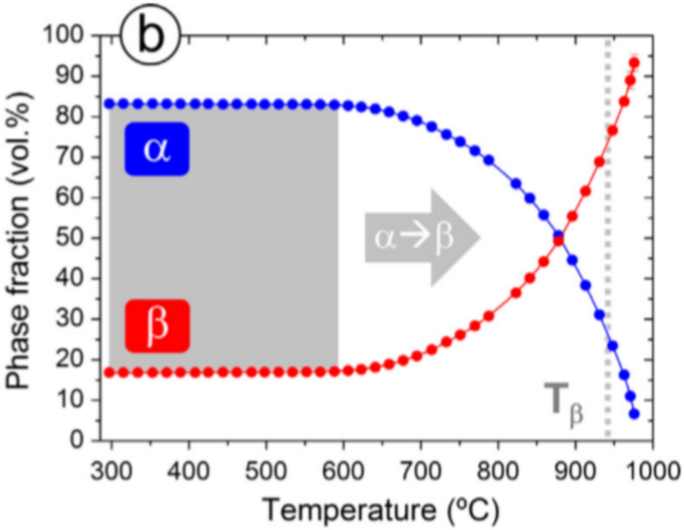


Phase Transformation Kinetics

Linear Heating of (α+β) Ti6Al4V
Dilatometry-based Study

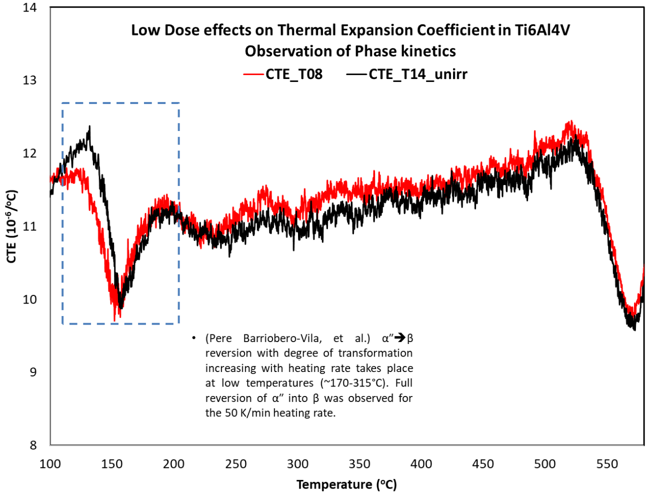
- Several studies have studied the kinetics of ***unirradiated*** Ti6Al4:
  [Dilatometry, Differential Scanning Calorimetry, and HEXRD (Rietveld)]
  - Pere Barriobero-Vila, et al.
    - "Role of element partitioning on the α-β phase transformation kinetics of a bi-modal Ti-6Al-6V-2Sn alloy during continuous heating", J. of Alloys and Compounds, 626 (2015), 330-339
    - "Phase transformation kinetics during continuous heating of a b-quenched Ti–10V–2Fe–3Al alloy," J Mater Sci (2015) 50:1412–1426
  - P. Homporova et al.
    - "Dynamic phase evolution in titanium alloy Ti6Al4V

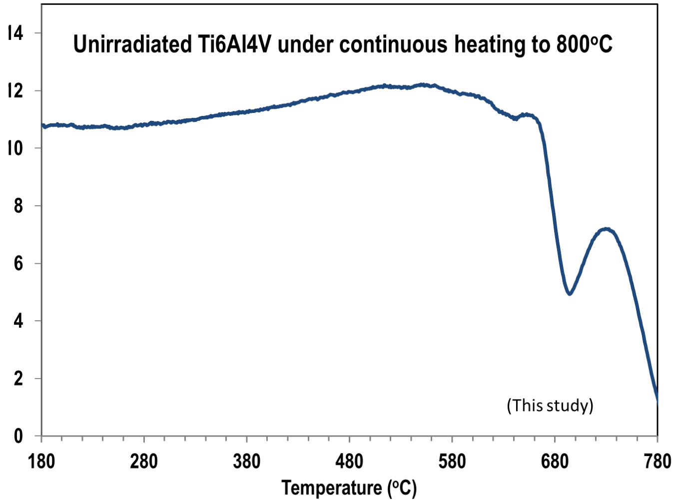
Volume fraction evolution of α and β obtained by Rietveld analysis as a function of temperature during continuous heating (Barriobero, et al)



- ***BNL Study***
  - Observe the effect of proton irradiation on the phase transformations observed through heating
    - **α" ⇨ β at low temperatures**
    - **α ⇨ β at high temperatures**
    - **Possible indication of radiation-induced ω phase**

- ***Additional*** transformation kinetics:
  - A fast athermal α″⇨β reversion
    - Degree of transformation increases with heating rate
    - Takes place at low temperatures ~170-315°C
    - Full reversion of α″ into β observed for 50 K/min heating
  - Ti alloys:
    - ω phase forms from β via:
      - Quenching from β field
      - During isothermal aging of β metastable phase at low temperatures (< 500 ºC)



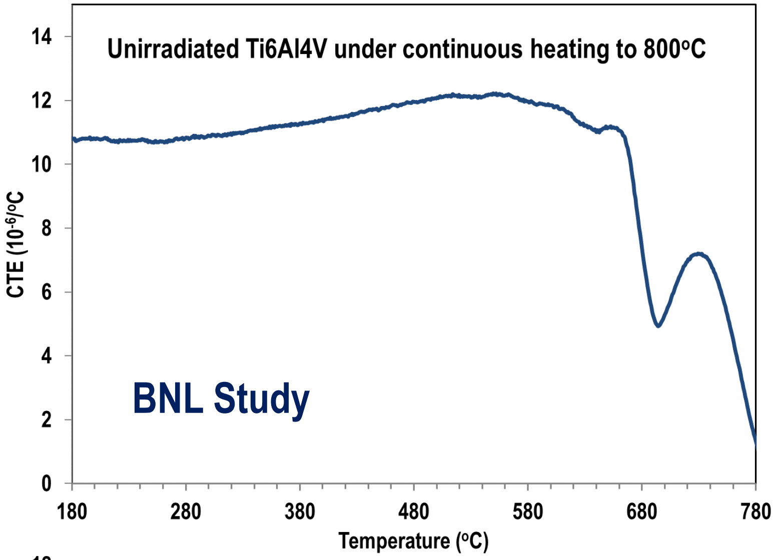


The of α" ➔ β at low temperatures and the α ➔ β at high temperatures are observed in this study using precision dilatometry (see above). Also shown is the low dose irradiation effect on the α" ➔ β.

Phase Transformation Kinetics 2

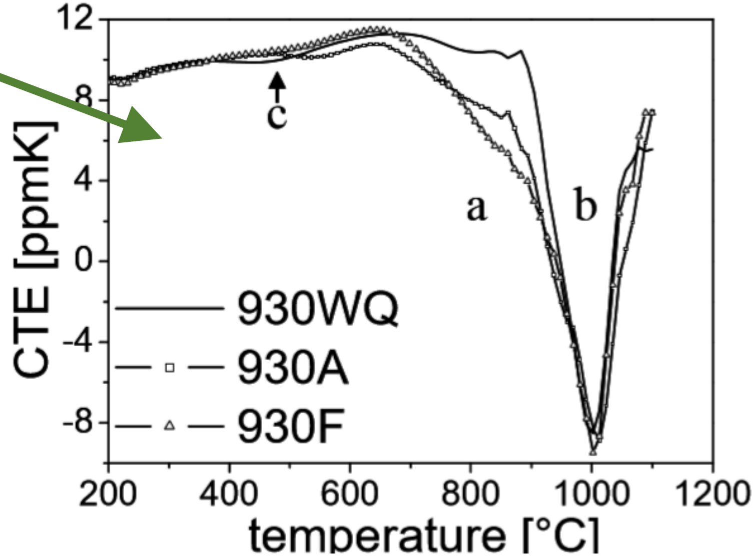
Linear Heating of (α+β) Ti6Al4V
Dilatometry-based Study

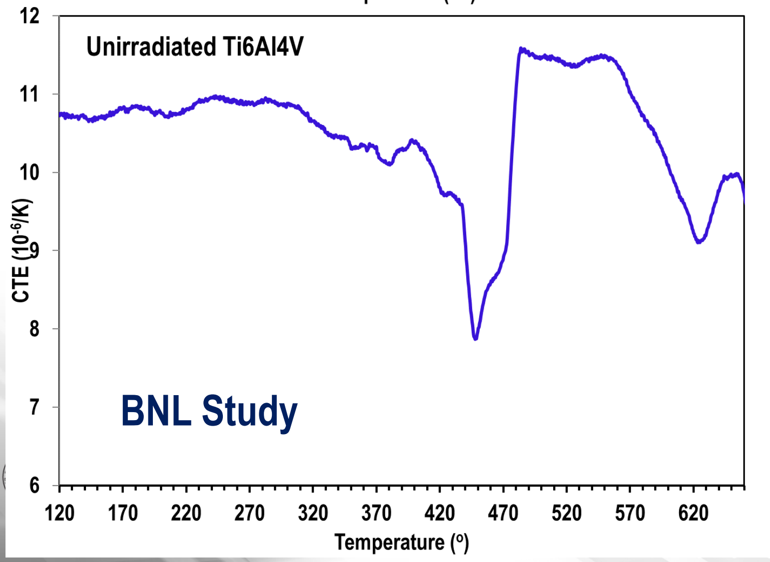
- Phase transformations
  - Detectable retardation of CTE
- Phase transformations
  - α ⇨ β identified by endothermic peaks
  - Martensite ⇨ β+α produces an exothermic peak



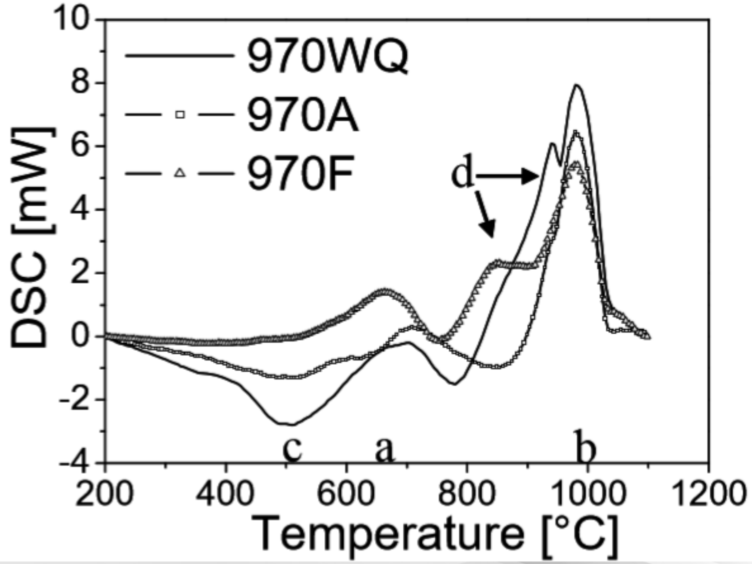
Homporova et al. correlated dilatometry and DSC in ***unirradiated*** *Ti6Al4V*

α ⇨ β



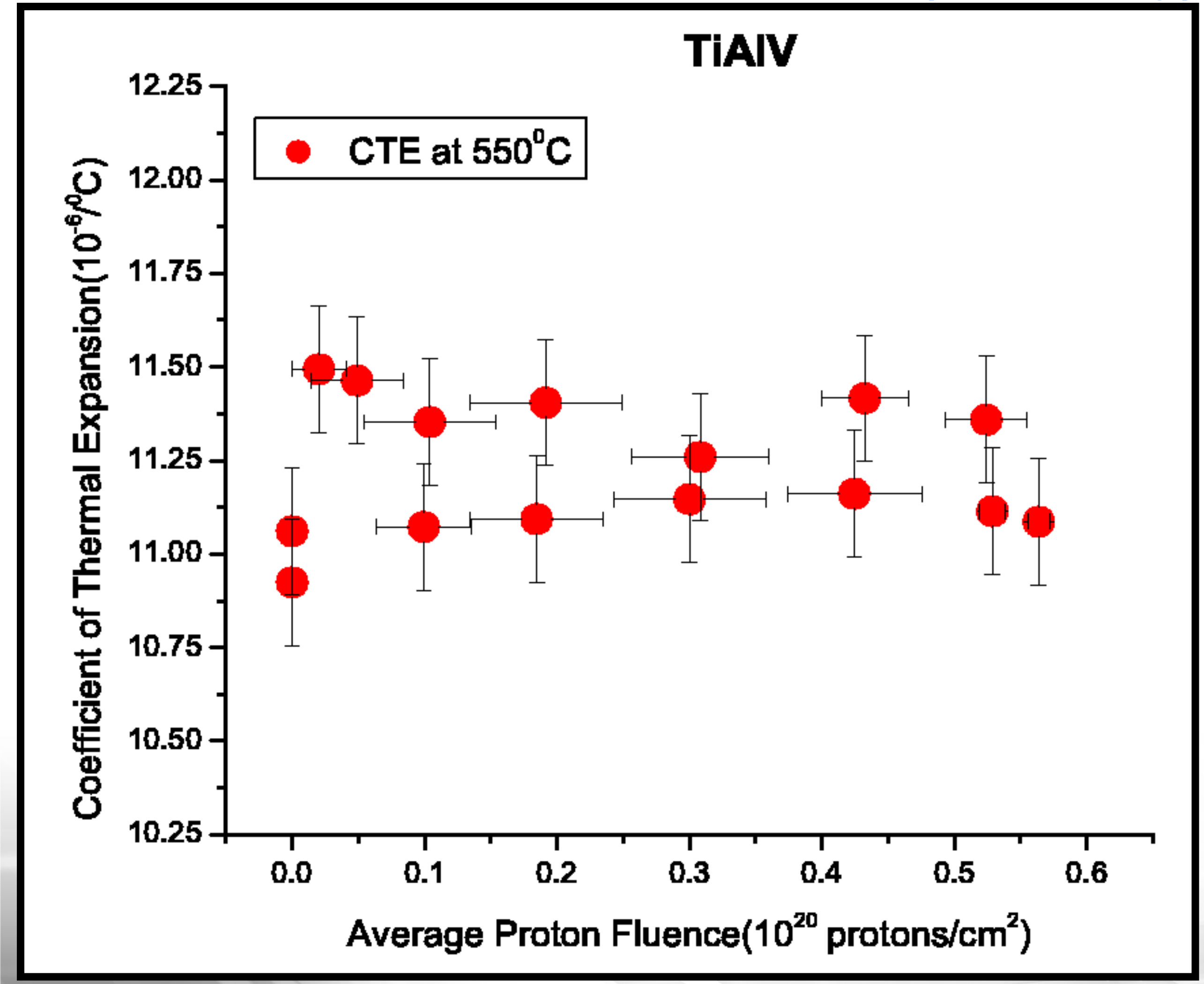


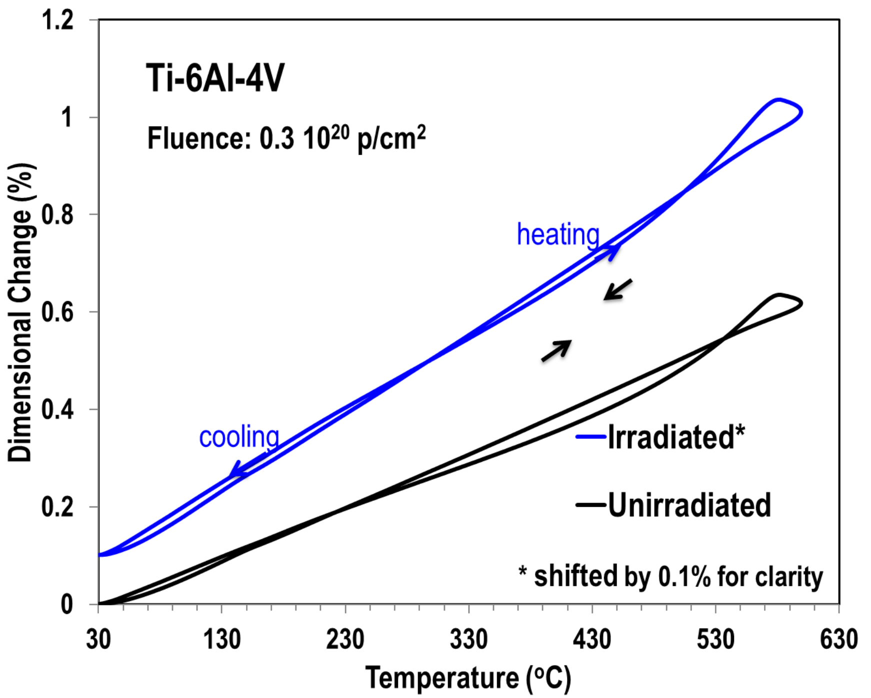
Martensite ⇨ β+α

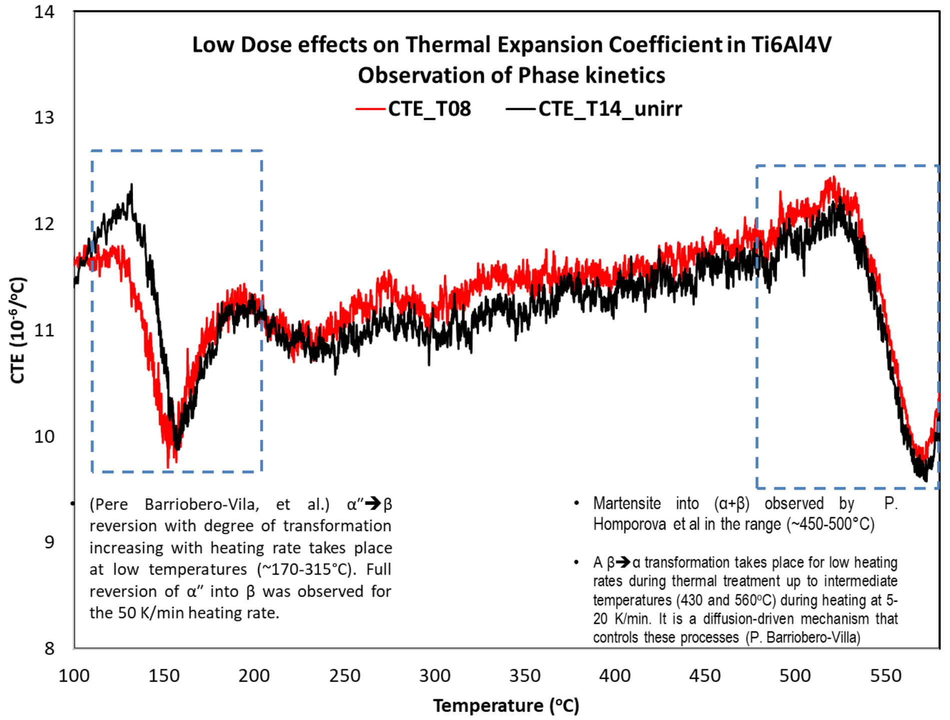


Ti6Al4V: Irradiation Studies

Variation of CTE with fluence at 550°C (BNL study)







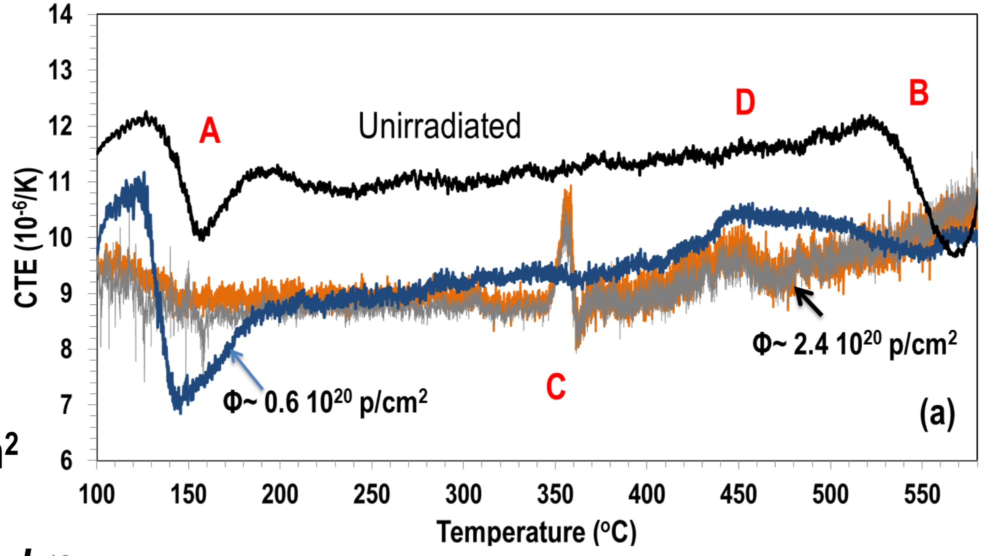
Ti6Al4V: Irradiation Damage Effects

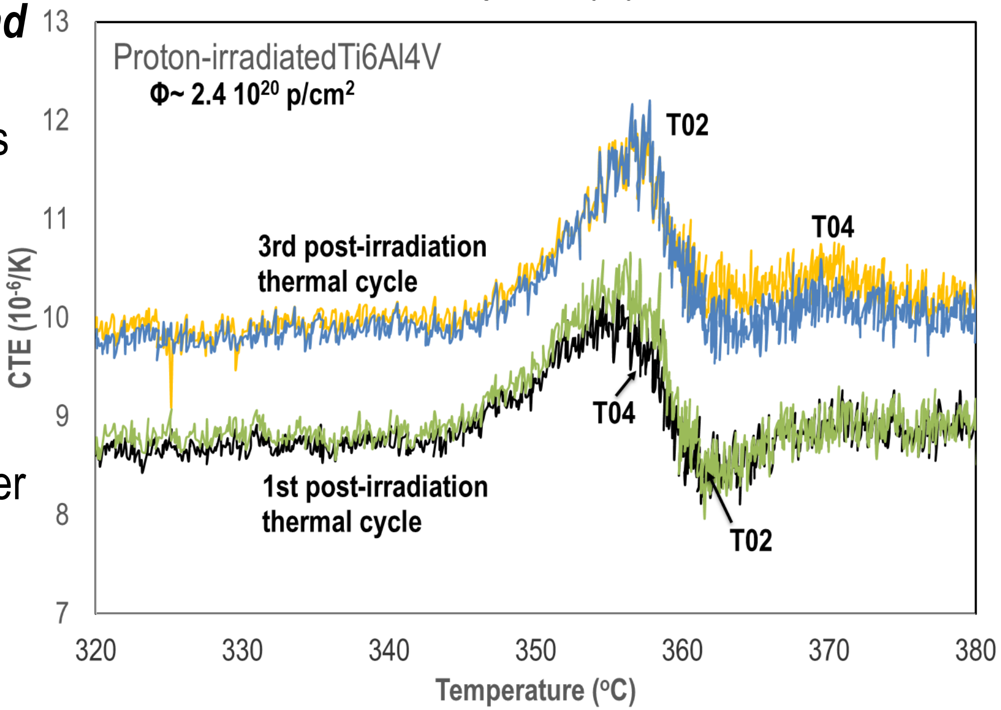
- Higher dose samples were re-irradiated to 2.4×10^{20} p/cm^2
- Sample with 0.6×10^{20} p/cm^2 shown has undergone several thermal cycles of annealing
- Post-irradiation heating to only 600^{o}C

IMPORTANT OBSERVATIONS:

- At **C:** New transformation seen for 2 different samples irradiated to **2.4×10^{20} p/cm^2**
    - Peak is exothermic indicating **martensitic transformation**
    - Transformation seems to appear above a threshold fluence - ***location and occurrence not impacted by thermal cycling!!!***
    - *Recall:* Ti-02 and Ti-04 CTE samples thermally annealed between irradiations
- At **A:** α"⇨β takes place (as discussed previously)
    - Very faint evidence after irradiation to 2.4×10^{20} p/cm^2
- At **D:** Local minima of the α_β observed in 430-530^{o}C range
    - 5^{o}C/min
    - Rietveld analysis
    - α⇨β transition observed at ~550^{o}C (unirradiated) apparently shifts to lower temperature as a result of irradiation (D location)

Phase Transformation Kinetics

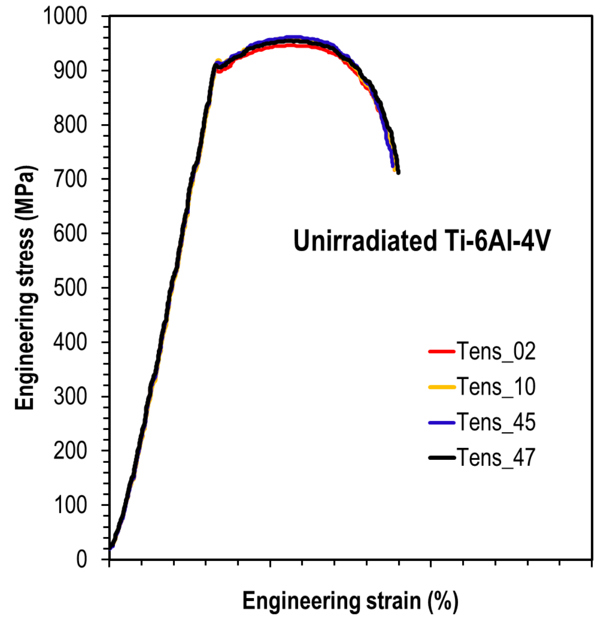


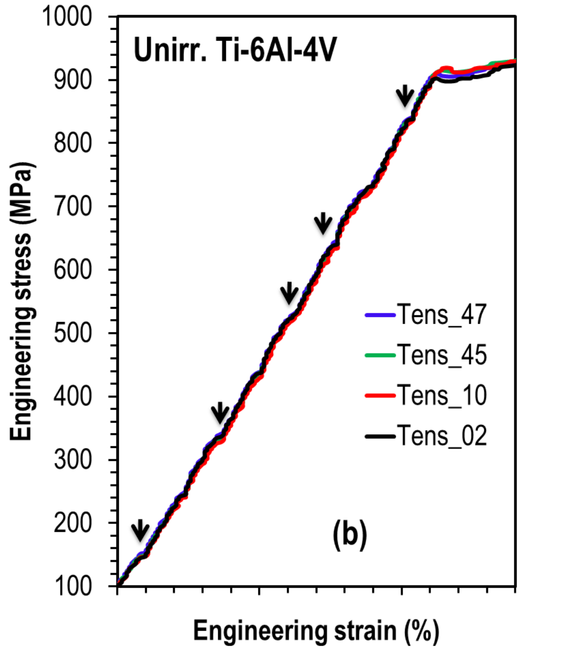


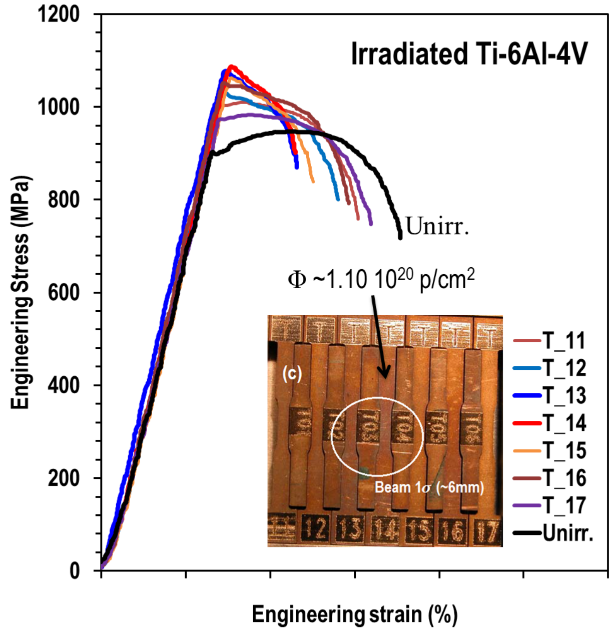
(α+β) Ti6Al4V Irradiation: Stress-Strain

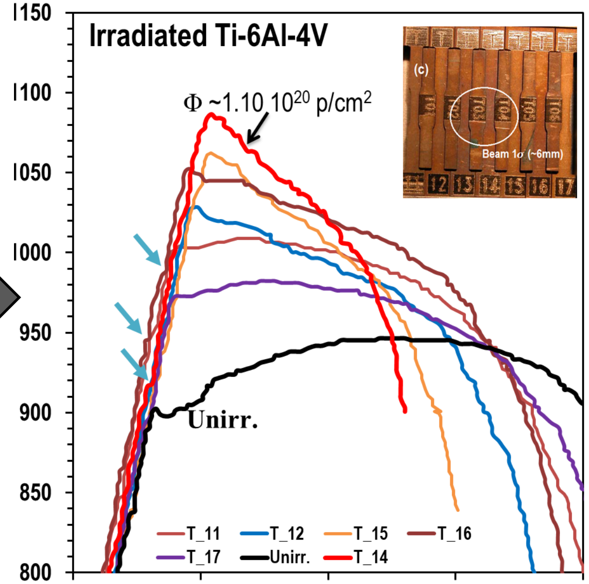
Assessment:

- Unirradiated Ti6Al4V shows very reproducible behavior
  - Kinks within the elastic range at same locations are most likely attributable to the hcp alpha phase
  - Signs of ductile failure and some work hardening
- Irradiation at ~180-240°C leads to:
  - Yield stress increase
  - Almost no uniform elongation at higher fluences
  - Some ductility remains in Ti6Al4V – to be compared with Ti alloy Gum metal, which turned completely brittle after the same irradiation process
- "Kinks" are still observable following irradiation





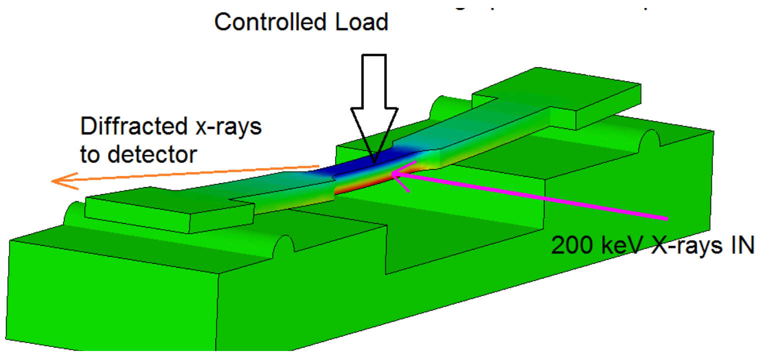




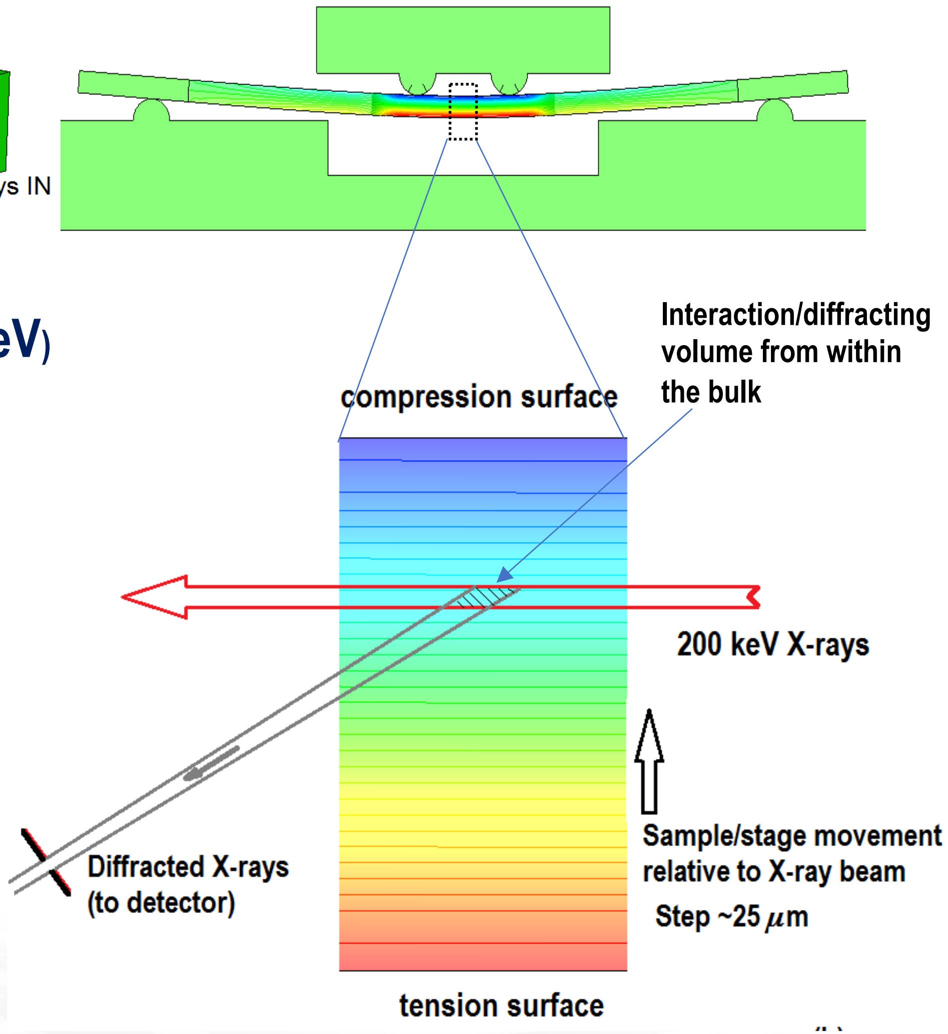
Ti6Al4V Irradiation: X-Ray Diffraction Experiments

Experiments were conducted using:

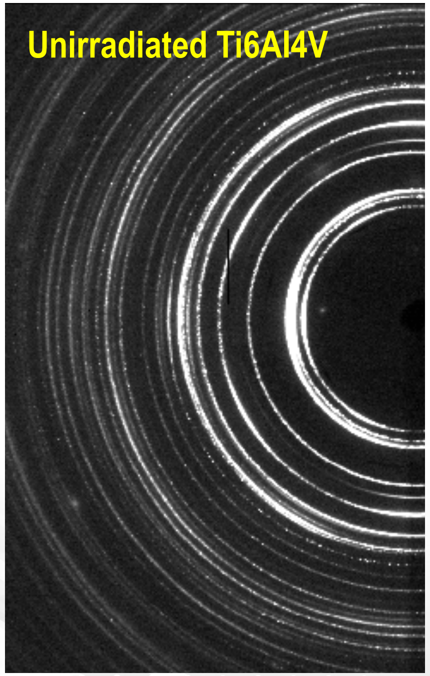
- NSLS X17B1 Beamline
  - 200 keV polychromatic X-rays
  - In-situ four-point bending stress
  - EDXRD techniques
- NSLS-II XPD Beamline
  - 67 keV monochromatic X-rays
  - In-situ four-point bending
  - Refined Rietveld technique

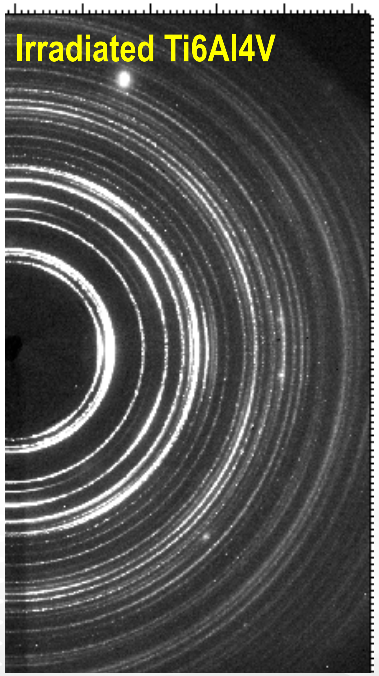


EDXRD (200 keV)



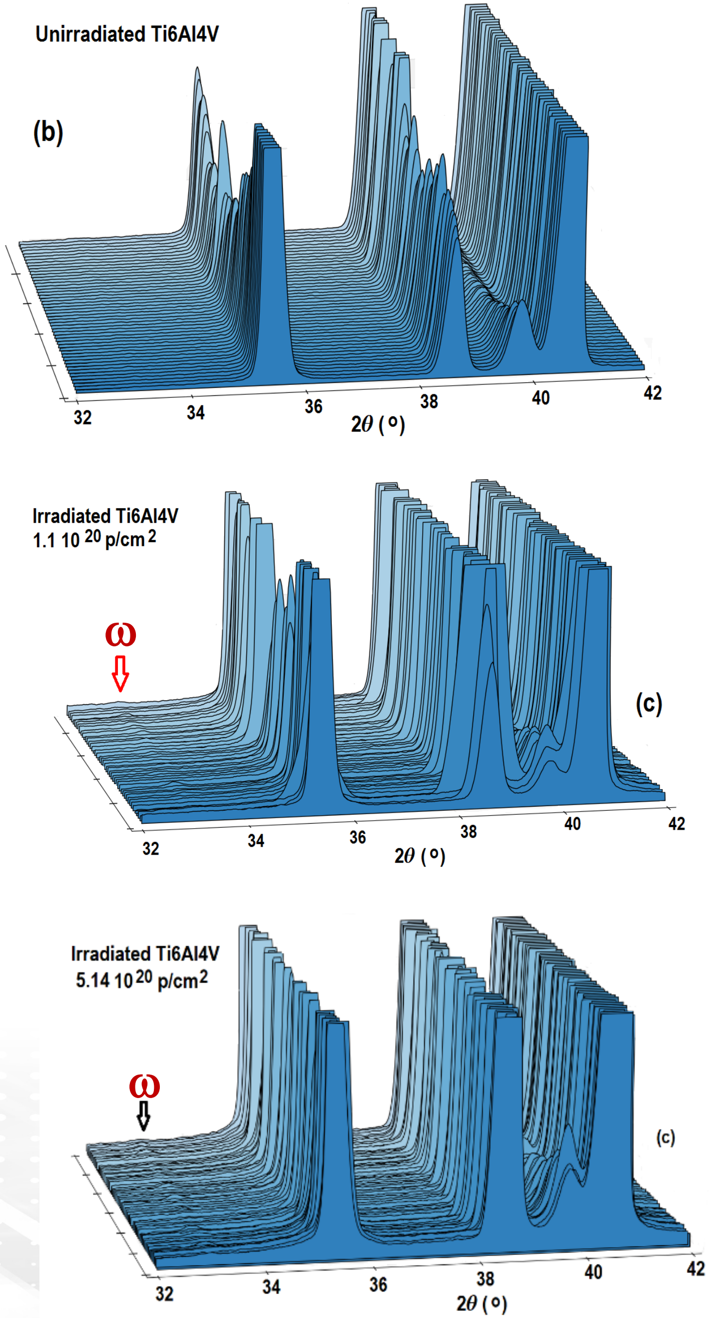
XRD (67 keV)

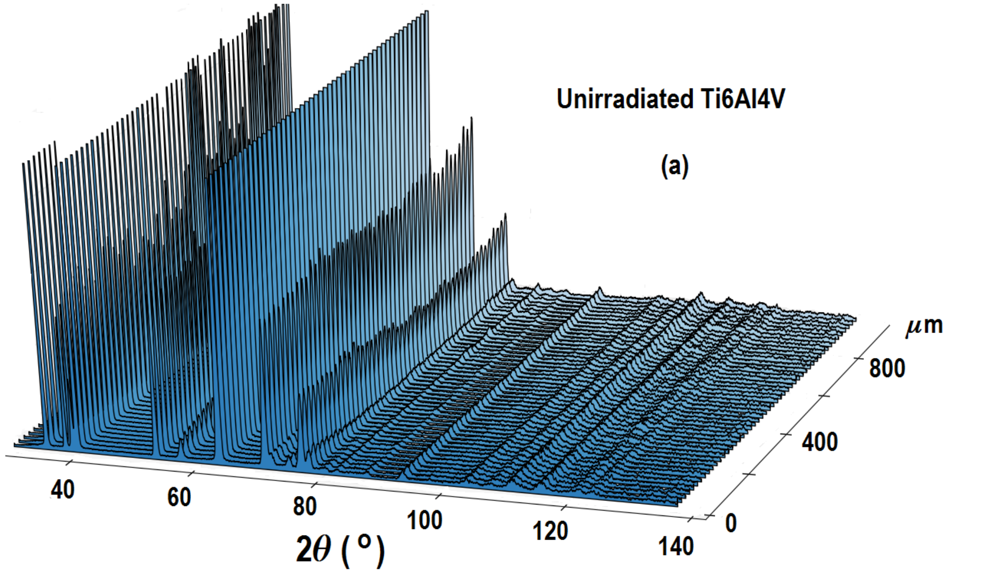




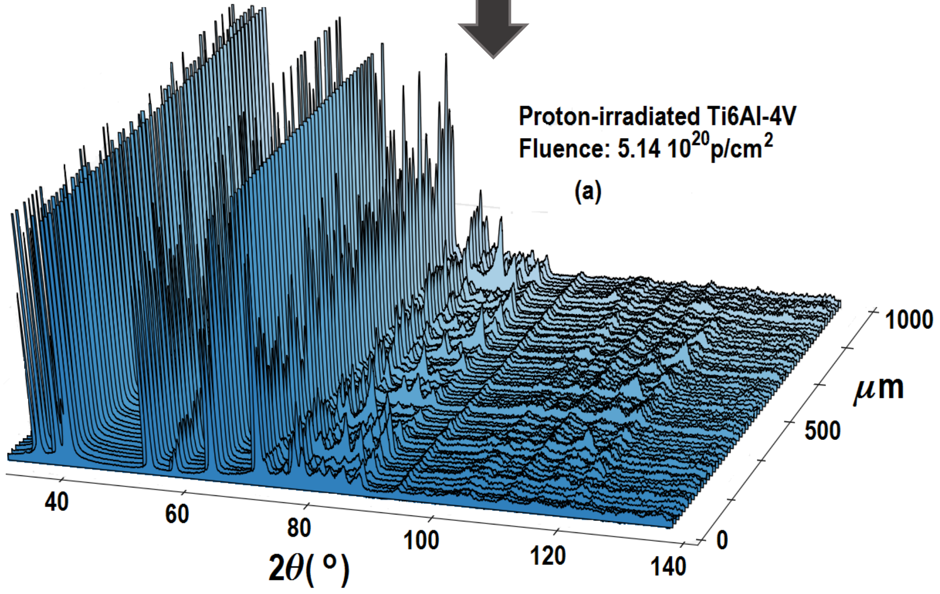
Ti6Al4V EDXRD Results: ω Phase Appearance

Increasing Fluence





Irradiation effects on the microstructure (high 2θ values)



Consistent with appearance of new ω phase throughout bulk

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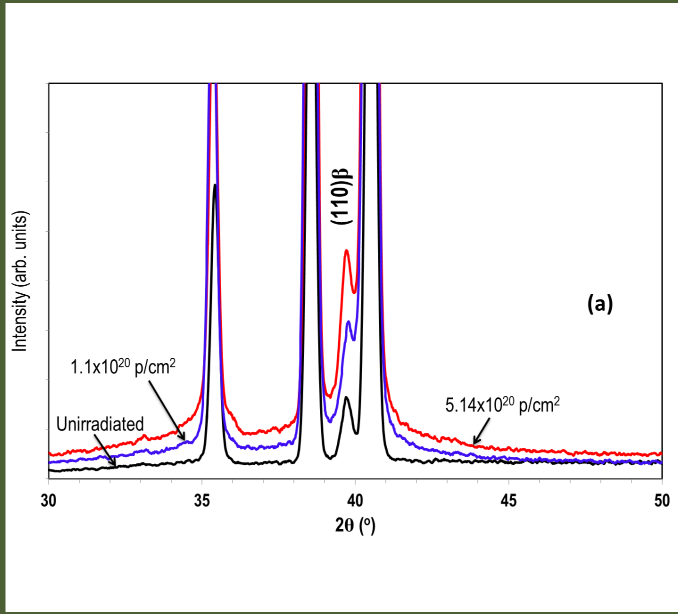
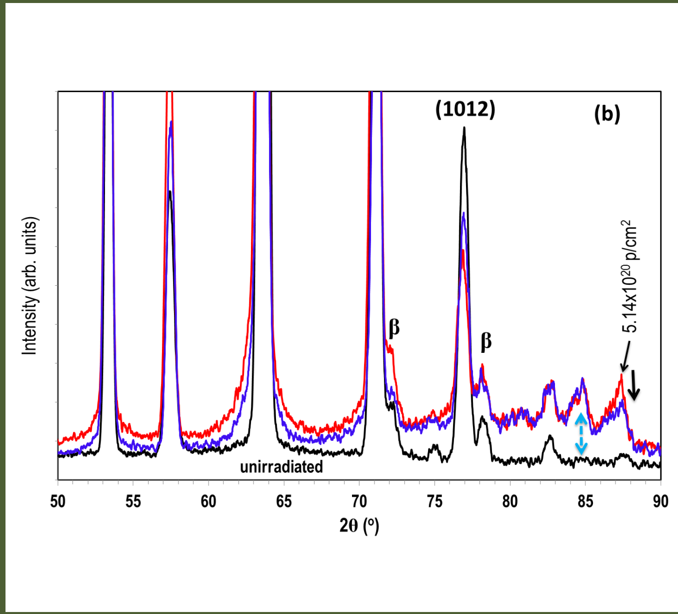
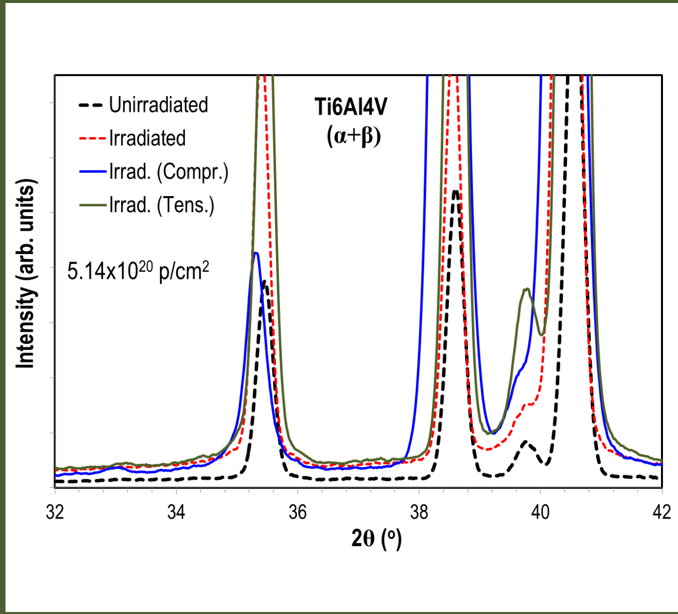
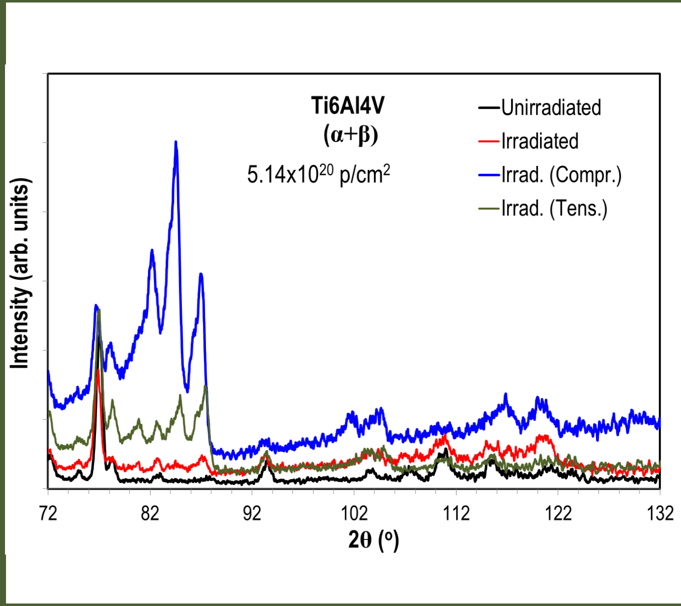




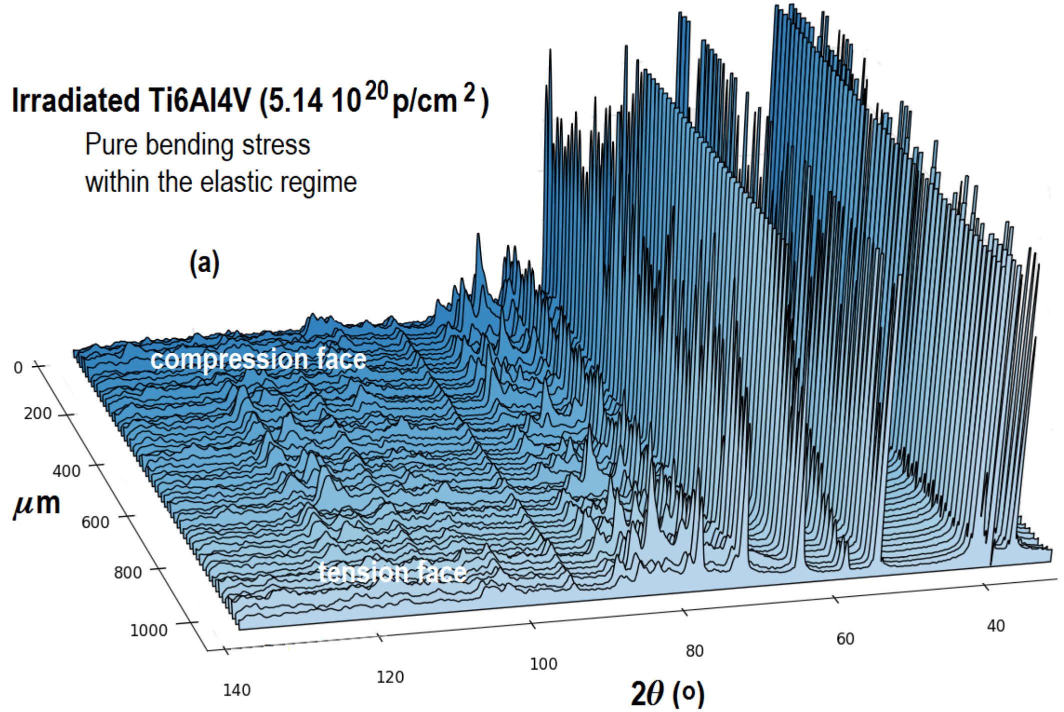




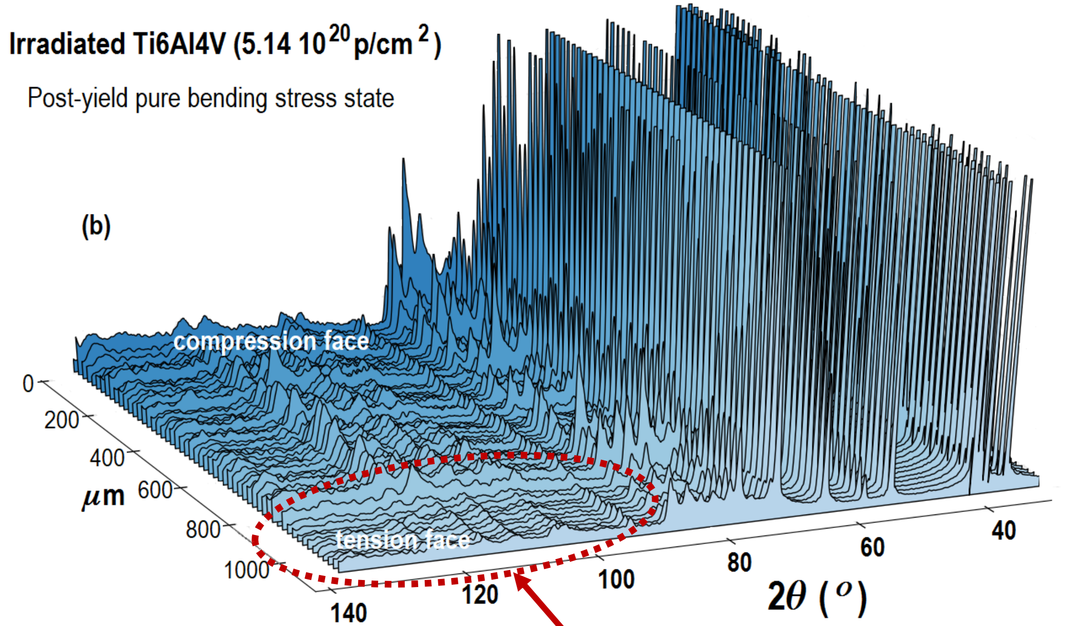
Ti6Al4V EDXRD Results: Irradiation Effects on α, β Phases

Tension-Compression Asymmetry

Ti6Al4V EDXRD Results: Tension-Compression Asymmetry

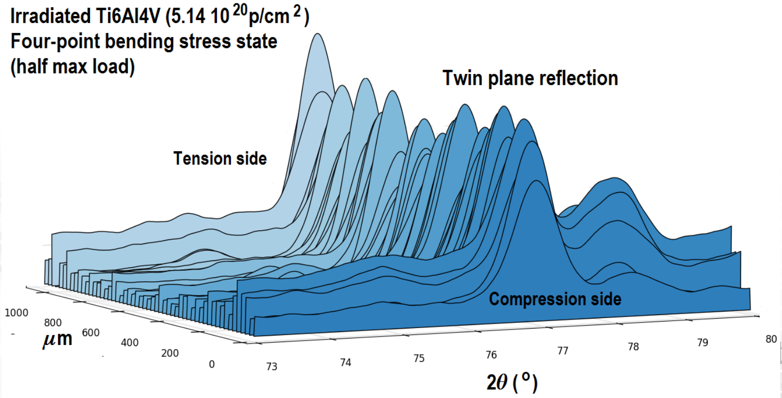


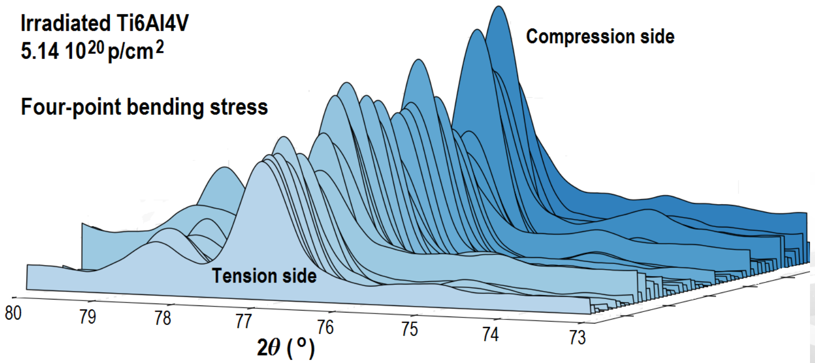
Stress in the elastic range



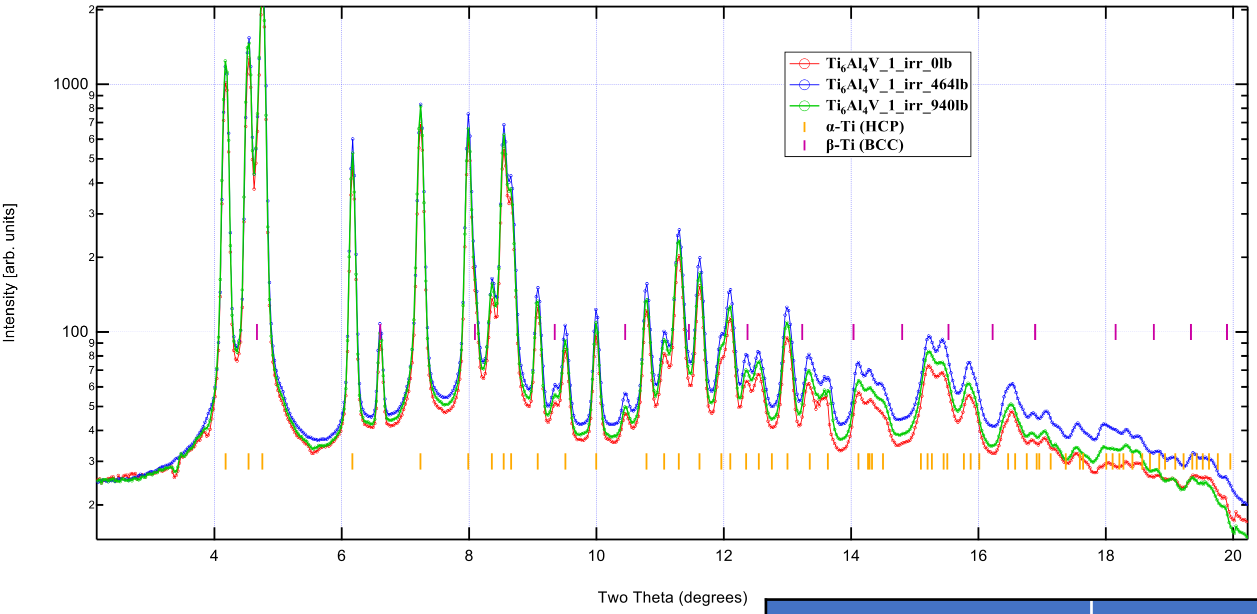
Stress beyond the elastic range
(note reflection map change over the plasticized zone)

Evolution of Twinning with applied stress
Note the change in reflection intensity as material goes plastic on the tension face





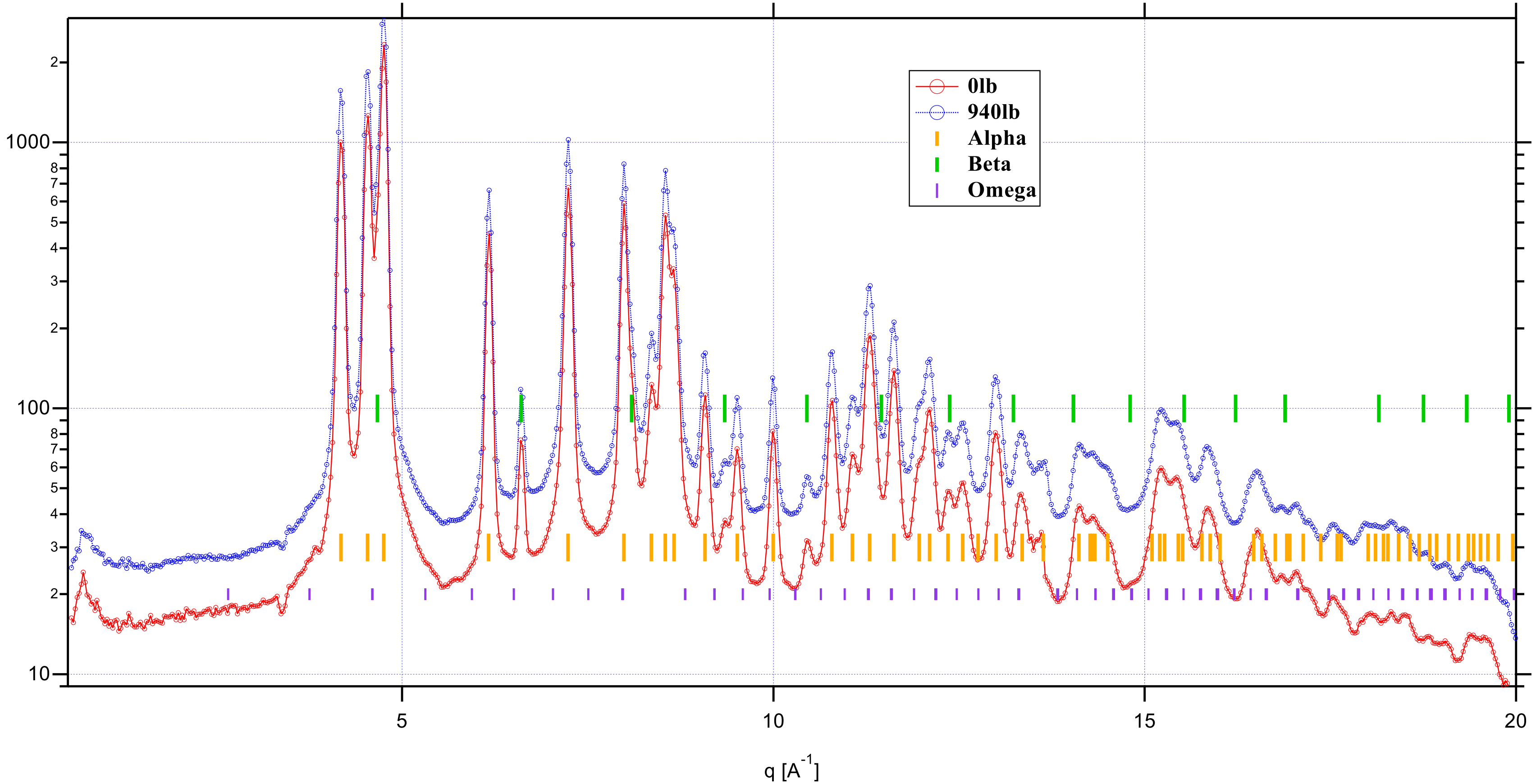
Irradiated Ti6Al4V: Refined Rietveld Analysis



α and β phase fraction as a result of applied in-situ stress

Stress-state		a	±a	c	±c	size	± size	Fraction
	phase	Å	Å	Å	Å	nm	nm	%
0 Load c/a (a)=1.5948	β (bcc)	3.20841	0.00128			46.12	17.81	3
	α (hcp)	2.92541	0.00019	4.66542	0.00045	125.60	4.42	97
Elastic c/a=1.5950	β	3.20561	0.00109			35.76	8.40	2.5
	α	2.92452	0.00018	4.66484	0.00041	128.72	9.86	97.5
Post-yield	β	3.20699	0.00098			35.96	2.39	2
	α	2.92605	0.00018	4.66713	0.00044	121.77	8.10	98

Irradiated Ti6Al4V: Refined Rietveld Analysis

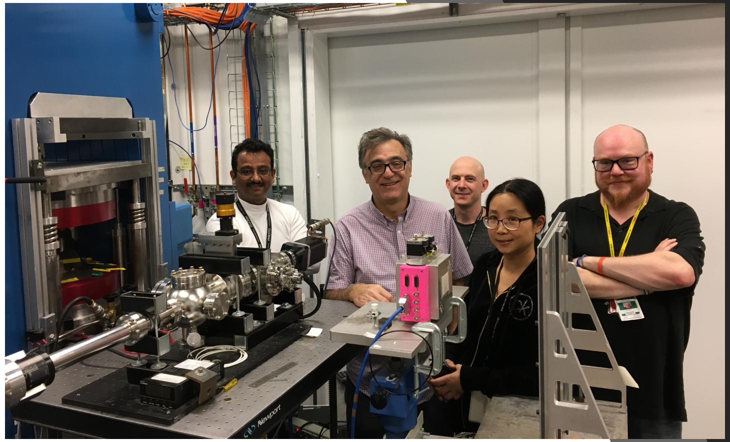


Indications of a radiation-induced ω phase

Comments:

- Stability of α and β phases of Ti-6Al-4V following irradiation (ω phase nano? i.e. <10nm).
- To unequivocally say, one would need better angular resolution.
  - Future experiments: longer sample to detector distance will to resolve the peaks from this phase.
  - These experiments planned for the NSLS-II XPD
- Note: *EDXRD technique at NSLS has also revealed the appearance of the phase that resembles the ω phase*

Ti6Al4V: Summary and Next Steps



Commissioning Run for NSLS-II XPD Spring Campaign Now Complete

Summary:

- The BNL irradiation study followed by dilatometric and X-ray characterization revealed
  - The effects of irradiation on the α, β evolution
  - The appearance of ω-phase as a result of proton exposure
- Observed asymmetries between tension and compression and activation of twinning from the hcp(α) phase
- Conducted X-ray diffraction with Refined Rietveld analysis and in-situ four-point bending stress
  - NSLS: EDXRD with polychromatic 200 keV x-rays
  - NSLS-II: XRD with monochromatic 67 keV x-rays

Next Steps:

- X-ray tomography, small angle scattering with in-situ multidirectional loading (3-point, 4-point bending)
- Tension (to fracture) of irradiated samples combined with compression and/or twisting
- Low cycle fatigue
- Further verification of the **ω** phase appearance and the **α-β** phase volume fraction

Upcoming Experimental Plans:

- X-ray tomography/X-ray diffraction with in-situ low-cycle fatigue of proton-irradiated Ti6Al4V
  Planned for NSLS-II XPD during Spring 2020 Run
- Fracture toughness of pre-notched/irradiated Ti6Al4V
  Utilizing the micro-beam achieved at NSLS-II XPD (~20x30 μm)
- X-ray imaging and X-ray tomography
  Utilizing the NSLS-II HEX beamline (currently under construction)

Thank you for your attention!