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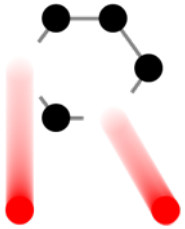

Post-Irradiation Examination of Proton-Irradiated Ti-base Alloys

12 December 2019

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6th RaDIATE Collaboration Meeting
Vancouver, BC

U.S. DEPARTMENT OF ENERGY BATTELLE

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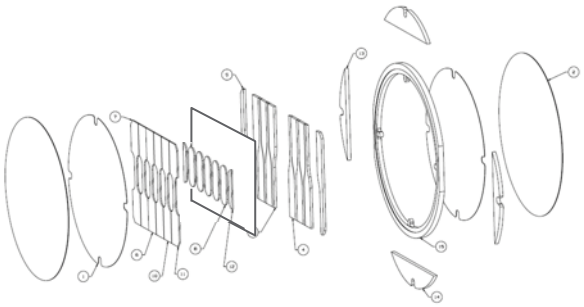
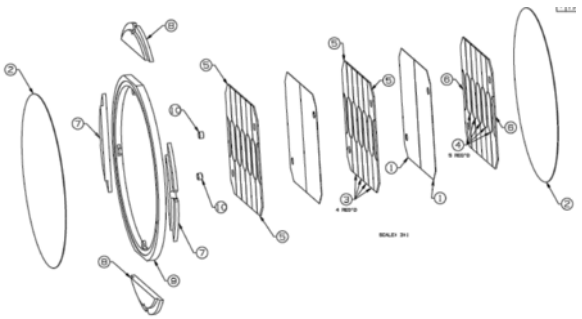
PNNL-SA-149798

Ti-Base Alloy Capsules in the BLIP Experiment

- Capsule DS Ti-1 alloy grades
  - Grade 5 (Ti-6Al-4V)
  - Grade 23 (Ti-6Al-4V ELI)
  - Grade 23F (Ti-6Al-4V forged)
  - Grade 9 (Ti-3Al-2.5V)



- Capsule DS Ti-2 alloy grades
  - Ti-15V-3Cr-3Sn-3Al (β-Ti)
  - Grade 23
  - Grade 23 STA
  - Commercial Purity Ti
  - Grade 5 (Ultra-fine grain size)
  - Grade 6 (Ti-5Al-2.5Sn)



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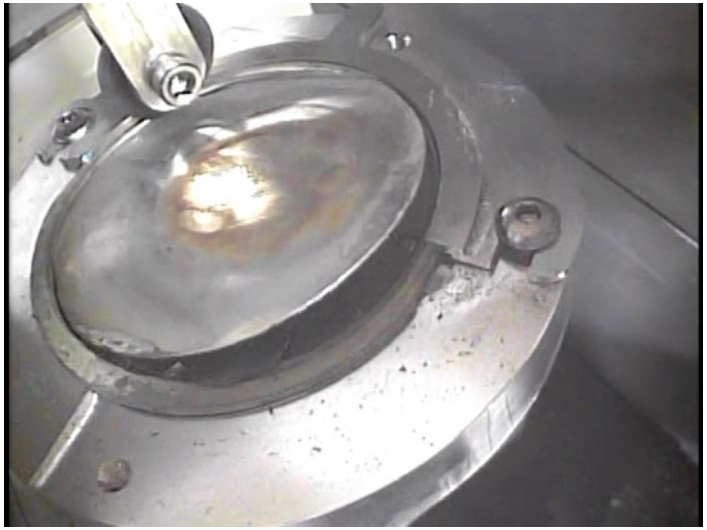
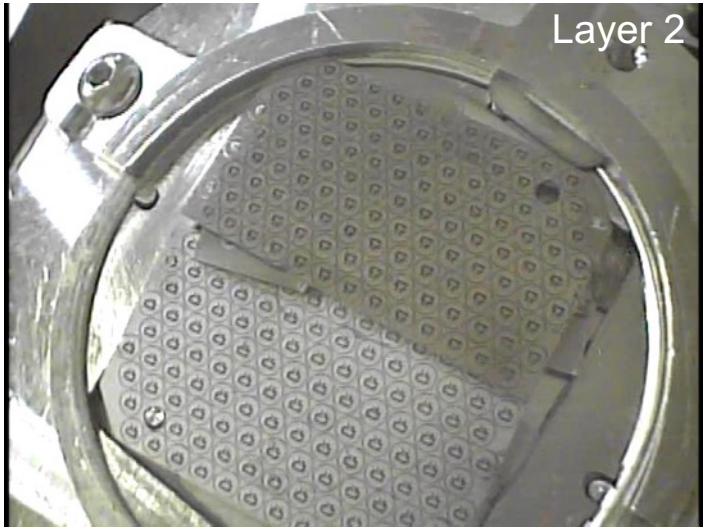
Capsule DS Ti-2 Opening



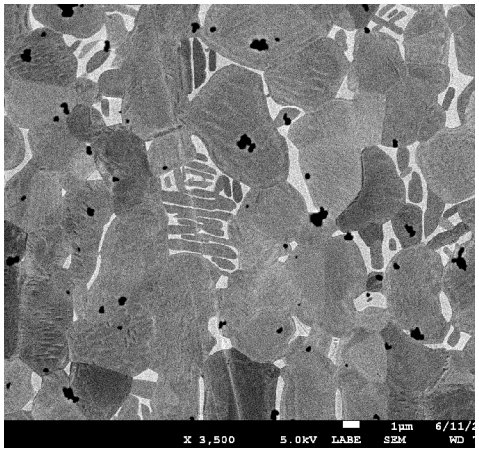


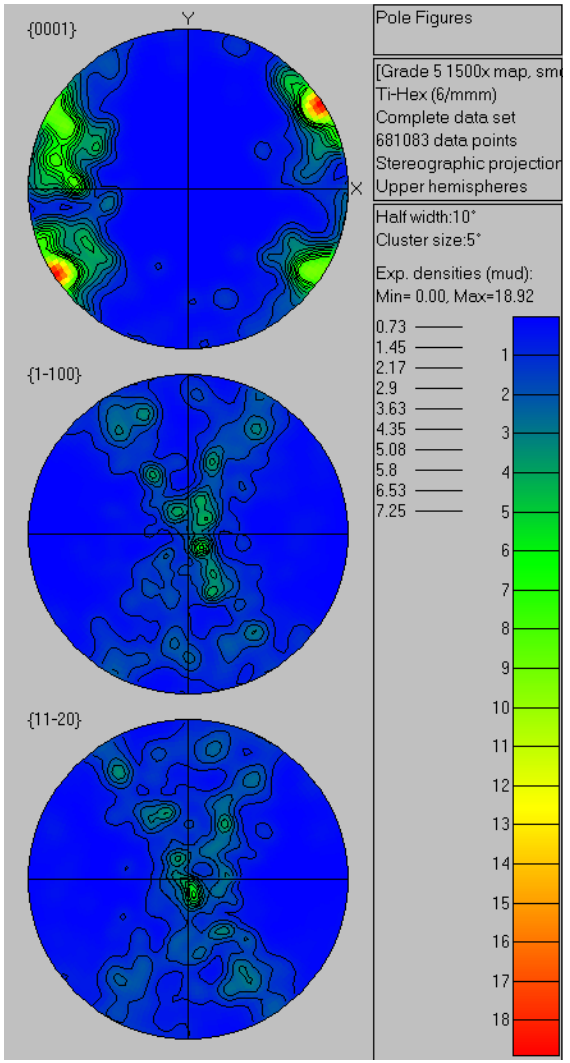
Test Matrix and Current Status

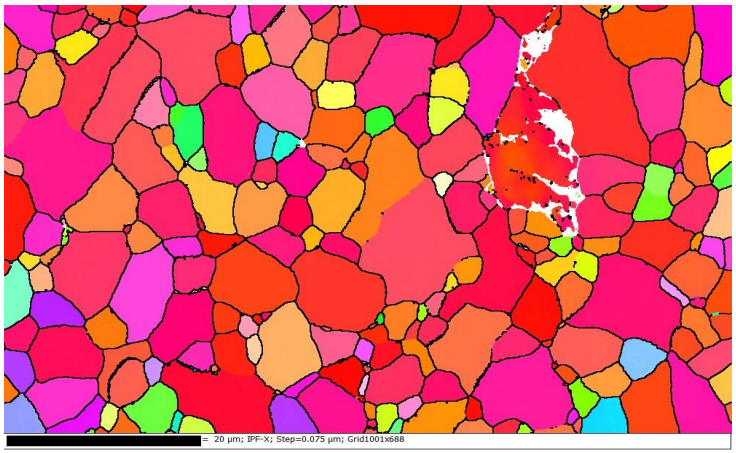
Alloy	EBSD		AFM		TEM		Modulus		Tensile			
	U	I	U	I	U	I	U	I	U		I	
							RT	RT	RT	200	RT	200
DS Ti-1												
Grade 5	Re-do	Done	Re-do	Done	Done	Done	4	1	2	2	1	0
Grade 23	Re-do	Done	Re-do	Done	Done	Done	4	3	2	2	2	1
Grade 23F	Re-do	Done	Re-do	Done	Done	Done	2	1	2	0	1	0
Grade 9	Re-do	Done	Re-do	Done	Done	Done	2	1	2	0	1	0
DS Ti-2												
Ti-15-3-3-3	Done		Started		Started		3	3	2	1	2	1
Grade 23	Done		Started		Started		3	3	2	1	2	1
Grade 23 (STA)	Done		Started		Started		3	3	2	1	2	1
CP Ti	Done		Started		Started		3	3	2	1	2	1
Grade 5 (UFG)	Done		Started		Started		3	3	2	1	2	1
Grade 6	Done		Started		Started		3	3	2	1	2	1

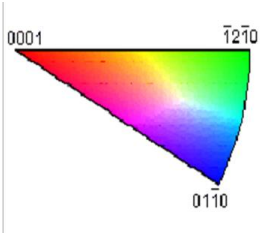
Microstructural Texture

- Large-area EBSD analysis provides overall assessment of texture using unirradiated samples
  - Two phase (α+β) microstructure with small β grains
  - EBSD does not index the β–phase well at this spatial resolution, although β-phase grains are evident in backscatter images
    - ✓ Similar difficulty indexing the Grade 5 ultrafine grain size material in DS Ti-2 capsule
  - Strong α texture in the plane of the sample





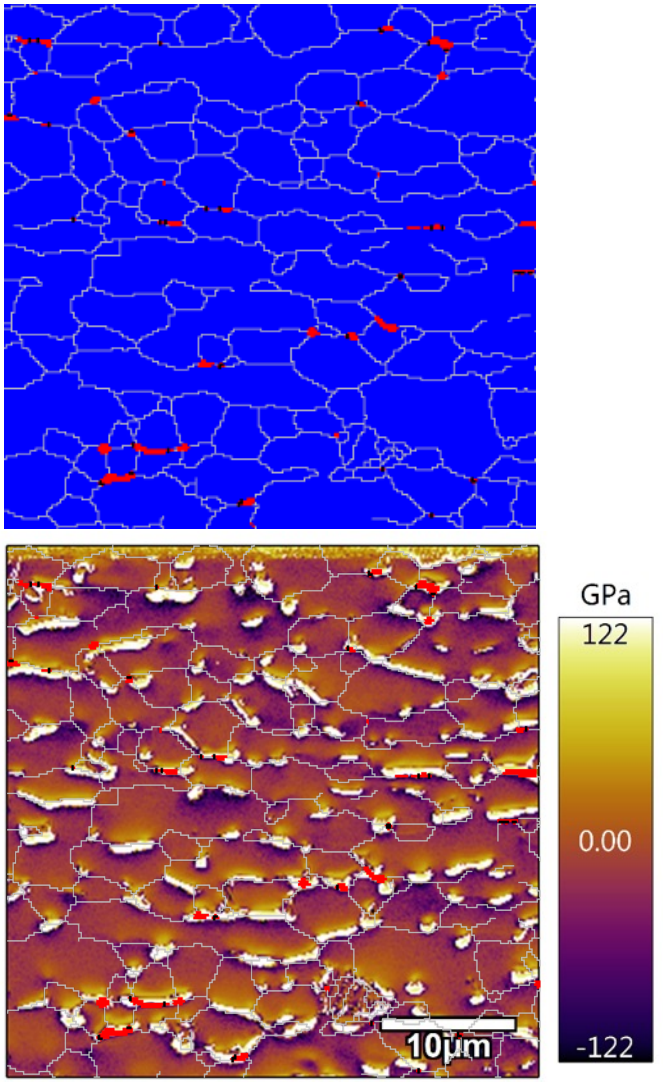




Unirradiated Grade 5 (sample F01)

Correlated EBSD/AFM and Nanohardness

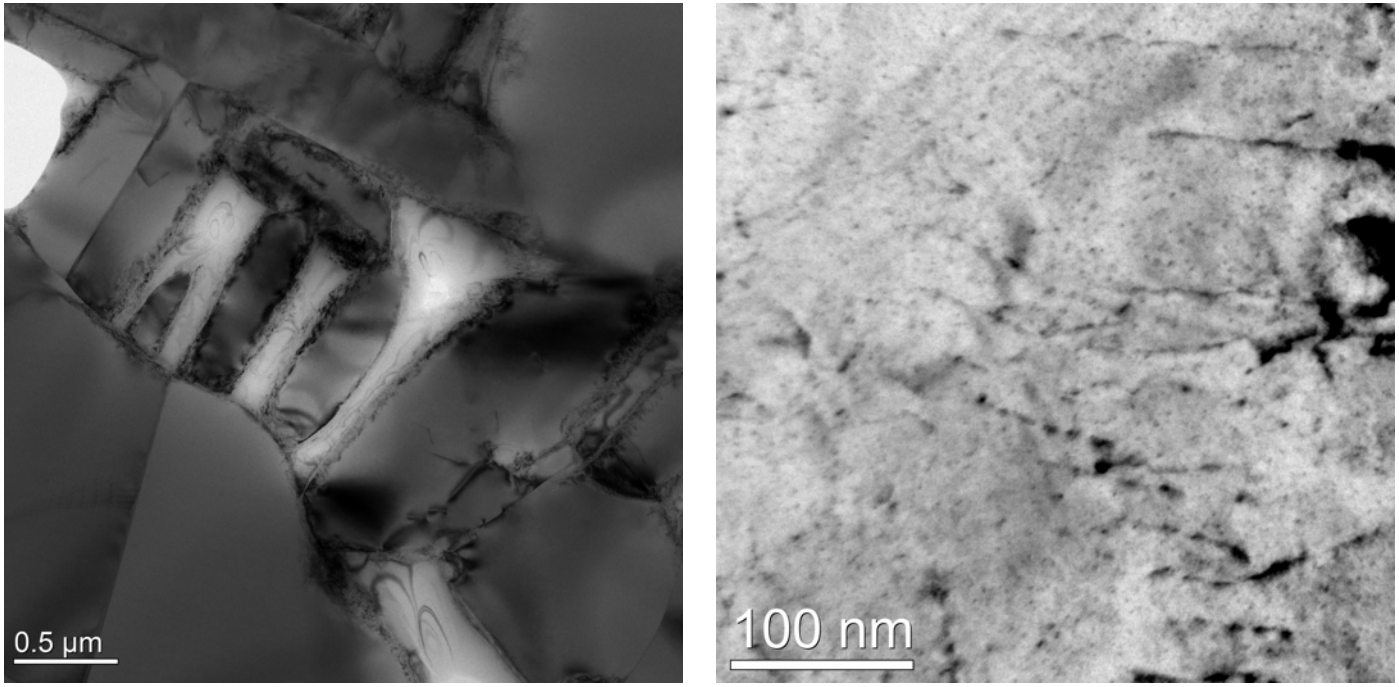
- Correlated EBSD and AFM measurements
  - Allows quantitative correlation of individual grain nanohardness to grain orientation
  - Differences in nanohardness or relationship to grain orientation between unirradiated and irradiated will supplement insight gained from microstructural studies
  - Based on AFM results, it appears not all of the β-Ti grains were resolved and indexed by EBSD
    - ✓ Good correlation between the indexed β-Ti phase regions and the AFM nanohardness results
  - Significant relative hardness contrast between α-Ti and β-Ti phases



Irradiated Grade 5 (sample F05) EBSD (top) and AFM (bottom)

Transmission Electron Microscopy

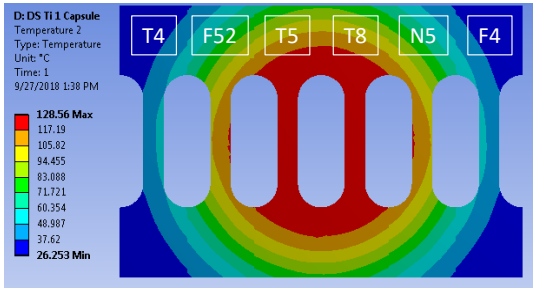
- TEM studies focused on evaluating microstructural evolution with radiation damage
  - Minimal ”classical” radiation damage microstructures evident in irradiated DS Ti-1 samples (~0.1 dpa)
  - There does seem to be some evidence of very fine-scale ω-phase precipitate growth within the β-phase grains with irradiation
  - DS Ti-2 (~1 dpa) and US Ti (~1.5 dpa) samples may display more obvious radiation damage features

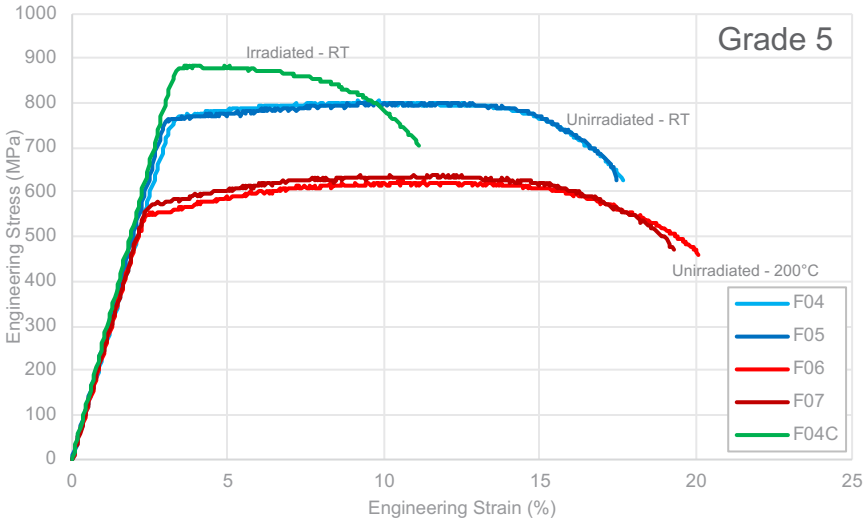


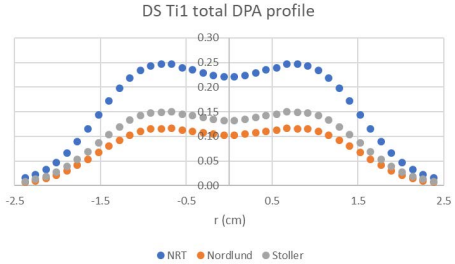
Grade 5 Unirradiated (sample F01, left) and Irradiated (sample F05, right) TEM images

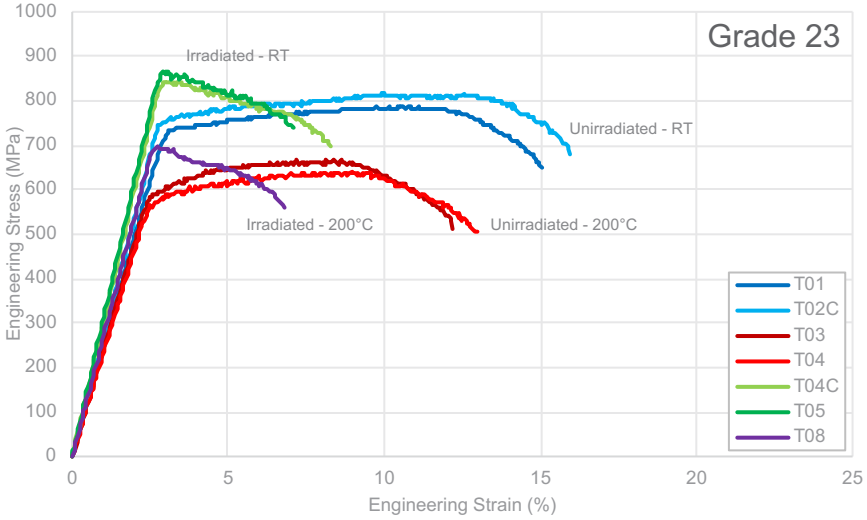
Ti-Base Alloy Tensile Testing and Microscopy

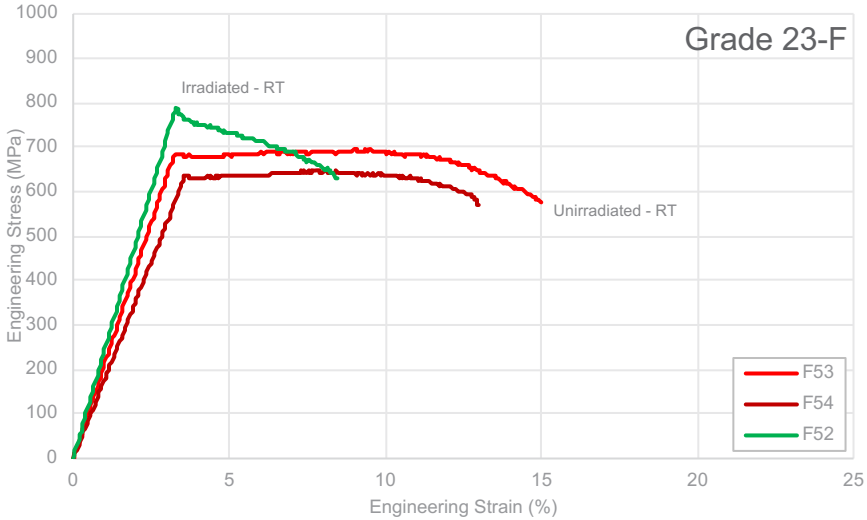
Calcs and plots by K Ammigan





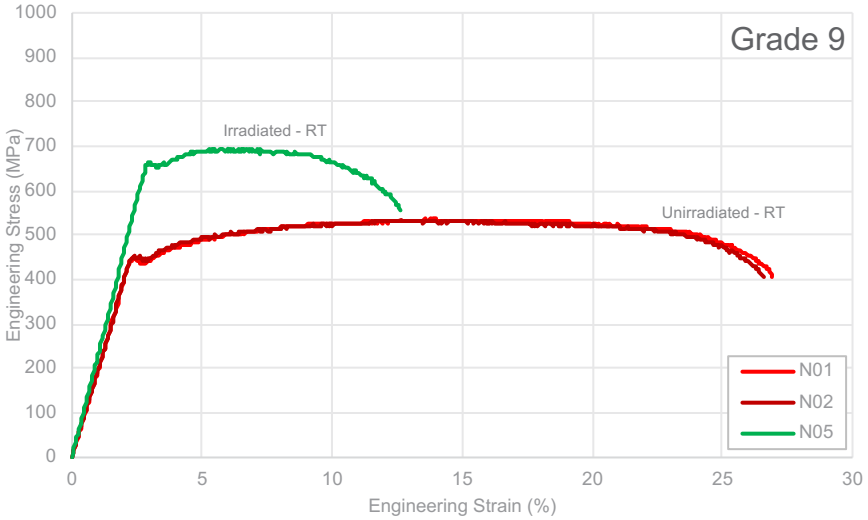


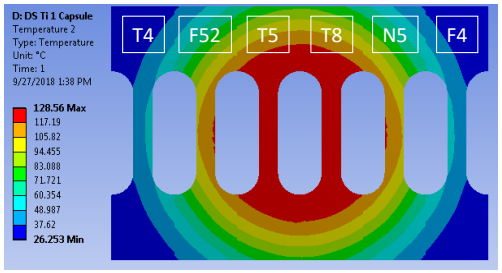


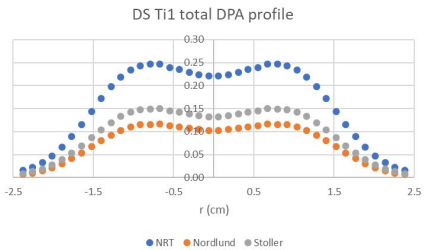


Ti-Base Alloy Tensile Testing and Microscopy

- All four grades have lower unirradiated RT YS and UTS than ASTM Standard minimum values
- Ti-6Al-4V grades stronger than Ti-3Al-2.5V but less ductile
- Distinct radiation hardening observed for each of the four grades
- Ti-3Al-2.5V retains some uniform elongation after irradiation while Ti-6Al-4V grades have essentially none
- Grade 23F tensile data shows more scatter than other grades in unirradiated condition and also shows more scatter in modulus







Calcs and plots by K Ammigan

Ti-Base Alloy Tensile Testing and Microscopy

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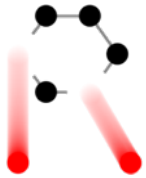


Unirradiated

	Room Temperature				200°C			
	YS (MPa)	UTS (MPa)	Uniform Elongation (%)	Total Elongation (%)	YS (MPa)	UTS (MPa)	Uniform Elongation (%)	Total Elongation (%)
Grade 5	763	803	6.26	15.0	545	625	7.78	18.1
Grade 23	740	801	8.41	12.9	575	654	5.89	10.4
Grade 23-F	654	671	5.11	11.0				
Grade 9	448	535	10.5	24.7				

ASTM B 265/338/348 Minimum Values

	Room Temperature		
	YS (MPa)	UTS (MPa)	Total Elongation (%)
Grade 5	827	896	10
Grade 23	759	827	10
Grade 9	483	621	15

Irradiated - DS Ti-1

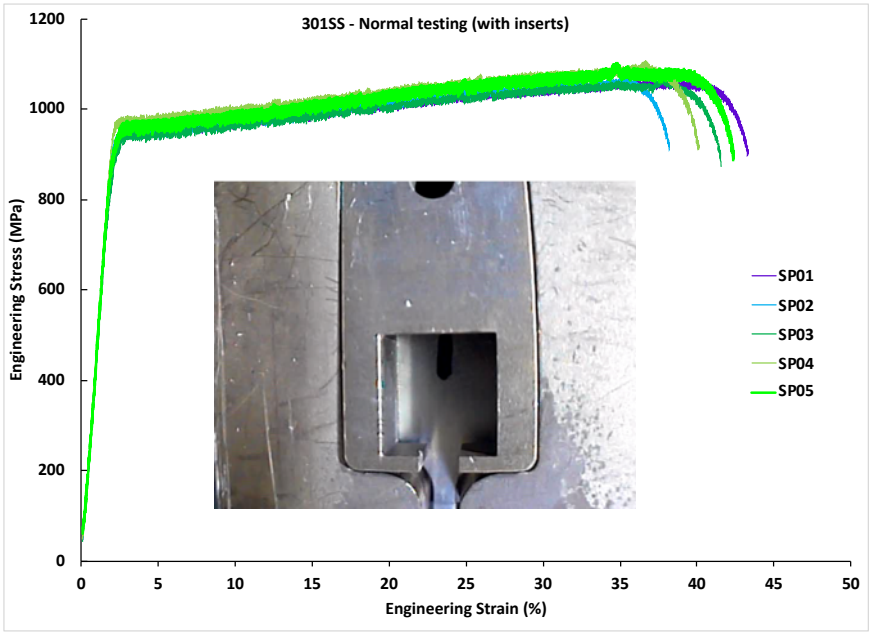
	Room Temperature				200°C			
	YS (MPa)	UTS (MPa)	Uniform Elongation (%)	Total Elongation (%)	YS (MPa)	UTS (MPa)	Uniform Elongation (%)	Total Elongation (%)
Grade 5	875	884	0.717	8.48				
Grade 23	815	854	0.141	5.33	683	696	0.188	4.74
Grade 23-F	789	789	0.0452	5.85				
Grade 9	651	694	3.00	10.2				

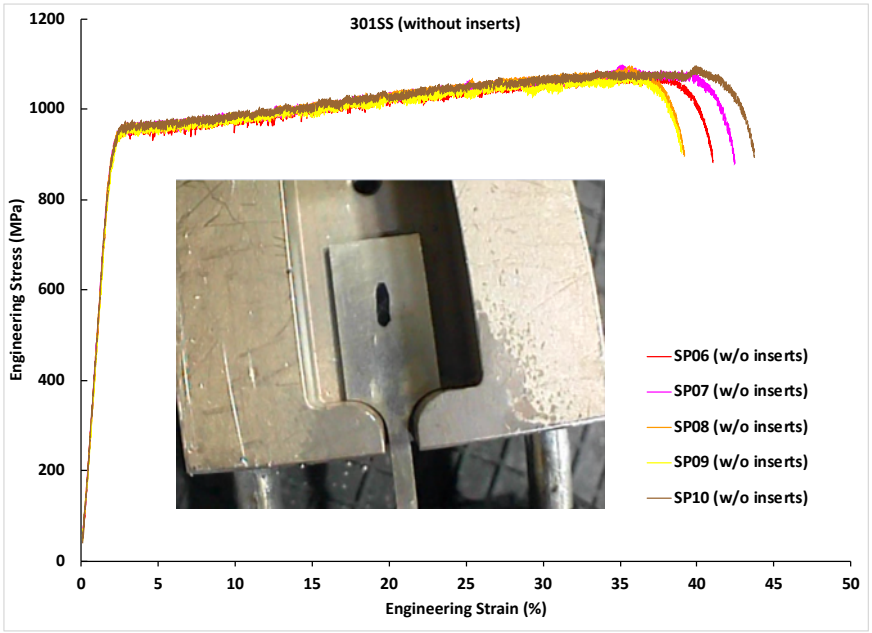
Change Due to Irradiation

	Room Temperature				200°C			
	YS (%)	UTS (%)	Uniform Elongation (%)	Total Elongation (%)	YS (%)	UTS (%)	Uniform Elongation (%)	Total Elongation (%)
Grade 5	15	10	-89	-44				
Grade 23	10	6.6	-98	-59	19	6.5	-97	-55
Grade 23-F	21	17	-99	-47				
Grade 9	45	30	-72	-59				

Stainless Steel Reference Tensile Tests – 301SS

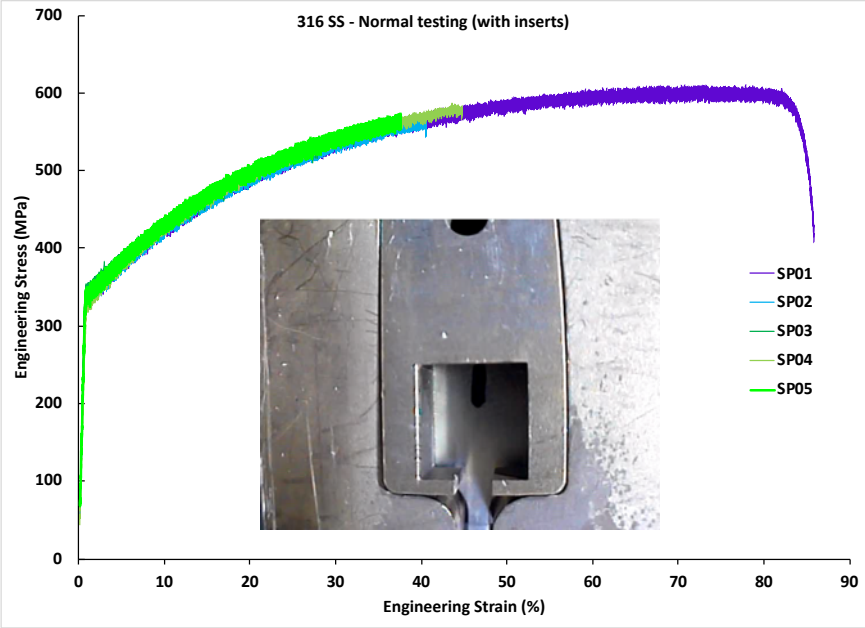
	301SS 1/2-Hard			
	YS (MPa)	UTS (MPa)	Uniform Elongation (%)	Total Elongation (%)
With Inserts	915 ± 27	1085 ± 19	33.7 ± 1.2	39.2 ± 2.1
Without Inserts	906 ± 4	1091 ± 9	33.6 ± 2.4	39.2 ± 2.1
Vendor	907 ± 5	1145 ± 49		33.0 ± 4.2
ASTM A666-10	≥758	≥1034		≥18

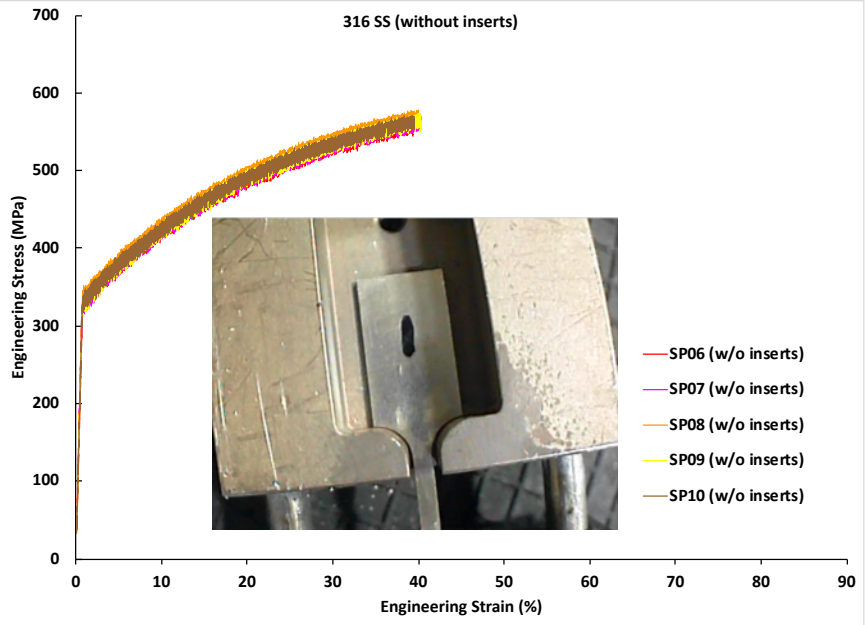




Stainless Steel Reference Tensile Tests – 316SS

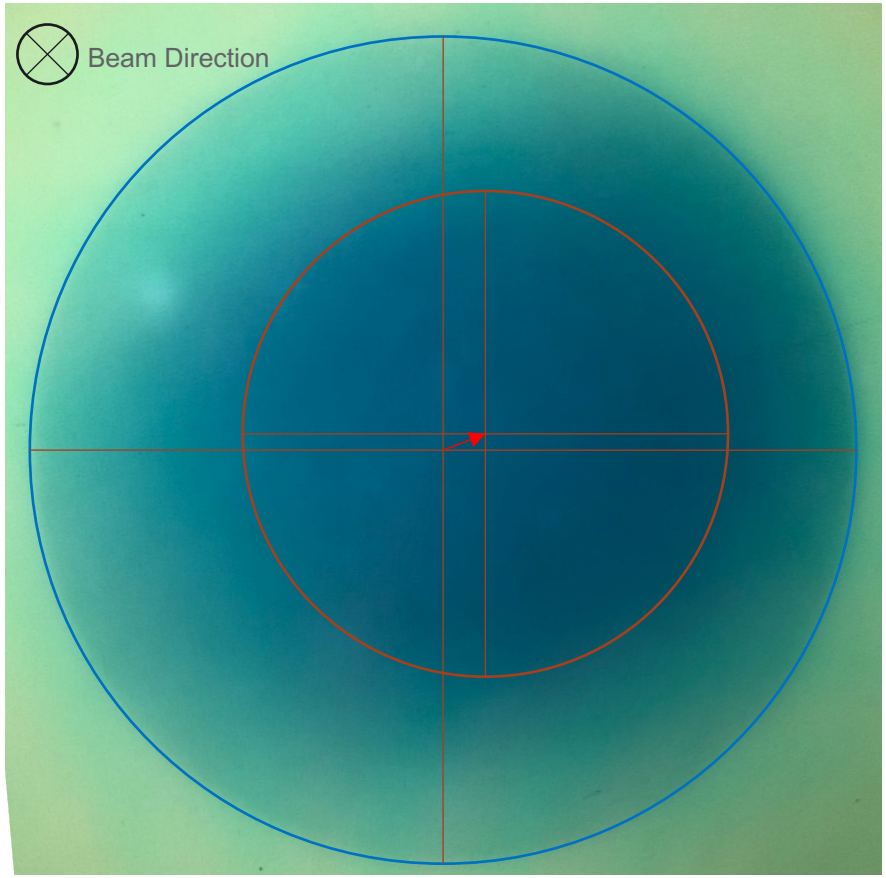
	316SS - Annealed			
	YS (MPa)	UTS (MPa)	Uniform Elongation (%)	Total Elongation (%)
With Inserts	341 ± 9	611	~78	~86
Without Inserts	335 ± 5			
ASTM A240-18	≥275	≥552		≥60





DS Ti-1 Film Dosimetry

- Dosimetry film image reasonably well correlated to capsule orientation via PIE video
- No photos available of DS Ti-1 capsule in BLIP holder
  - Up-down orientation assumed based on Be capsule film dosimetry results
  - Reinforces the need for orientation marks on the capsule face in future BLIP experiments
- DS Ti-1 results suggest offset
  - ~1 mm upward
  - ~3 mm right
- Compare to Be capsule results
  - ~5 mm upward
  - ~1 mm right



Fatigue Sample Shipment to Fermilab

- The 10 cantilever beam fatigue samples from DS Ti-1 were packaged and shipped to Fermilab in June
- Sample dose rates ranged from 70 to 120 mrem/hr at contact
- Samples were wiped with DI water and alcohol until β-γ surface contamination was <MDA on each sample

Mesoscale Fatigue Foil Shipment to CCFE

- Mesoscale fatigue foil dose rate and activity measured and provided to CCFE
- CCFE transport container design reviewed and accepted by PNNL
- Ready to package and ship foils when container is received

Measured Contact Dose Rates (mrem/hr)

	DS Ti-1	CP Ti Foil 1	Ti-15-3-3-3 Foil 2	Grade 23 Foil 4	Grade 23 STA Foil 5
β+γ	800	7000	800	12,000	8000
β only	45	300	400	600	500

Measured Activity (μCi)

Isotope	DS Ti-1	CP Ti Foil 1	Ti-15-3-3-3 Foil 2	Grade 23 Foil 4	Grade 23 STA Foil 5
Na-22	$1.99 \times 10^{2} \pm 5\%$	$1.24 \times 10^{0} \pm 10\%$	$1.00 \times 10^{2} \pm 5\%$	$2.64 \times 10^{2} \pm 5\%$	$2.31 \times 10^{2} \pm 5\%$
Sc-44	$3.00 \times 10^{1} \pm 5\%$	$2.25 \times 10^{1} \pm 5\%$	$2.03 \times 10^{1} \pm 5\%$	$3.09 \times 10^{1} \pm 5\%$	$2.89 \times 10^{2} \pm 5\%$
Ti-44	$3.09 \times 10^{1} \pm 5\%$	$2.69 \times 10^{1} \pm 5\%$	$2.37 \times 10^{1} \pm 6\%$	$3.66 \times 10^{1} \pm 5\%$	$3.25 \times 10^{1} \pm 5\%$
Sc-46	$6.11 \times 10^{1} \pm 5\%$	$4.93 \times 10^{2} \pm 5\%$	$4.86 \times 10^{2} \pm 5\%$	$7.27 \times 10^{2} \pm 5\%$	$6.52 \times 10^{2} \pm 5\%$
Mn-54	$6.55 \times 10^{0} \pm 8\%$	$<3.0 \times 10^{0}$ MDA	$1.59 \times 10^{1} \pm 9\%$	$1.58 \times 10^{1} \pm 12\%$	$1.44 \times 10^{1} \pm 12\%$
Co-57	$<6.0 \times 10^{-1}$ MDA	$1.48 \times 10^{0} \pm 25\%$	$1.30 \times 10^{0} \pm 15\%$	$3.75 \times 10^{0} \pm 12\%$	$2.20 \times 10^{0} \pm 12\%$
Sn-113	$<1.0 \times 10^{0}$ MDA	$<3.0 \times 10^{0}$ MDA	$4.20 \times 10^{1} \pm 5\%$	$<4.0 \times 10^{0}$ MDA	$<3.0 \times 10^{0}$ MDA

Upcoming Ti-base Alloy PIE Activities at PNNL

- Perform DS Ti-2 (unirradiated and irradiated) and US Ti tensile testing
  - Possibly to include ultrasonic measurement of elastic modulus
  - Six surviving US Ti tensile samples (2-Grade 5, 1-Grade 9, 3-Grade 23), all to be tested at RT
- Complete analysis of film dosimetry on DS Ti-2 to characterize position of proton beam and compare to DS Ti-2 and Be capsules
- Complete microscopy of DS Ti-2 and US Ti samples
  - SEM/EBSD, TEM, AFM (nanohardness)
  - Four surviving US Ti microscopy samples (2-Grade 5, 1-Grade 9, 1-Grade23)
- Ship Ti foils from DS Ti-1 and DS Ti-2 to CCFE
- PIE on Ti-base alloy samples from BeGrid2/HRMT43 experiment

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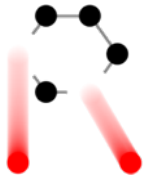
Backup Slides



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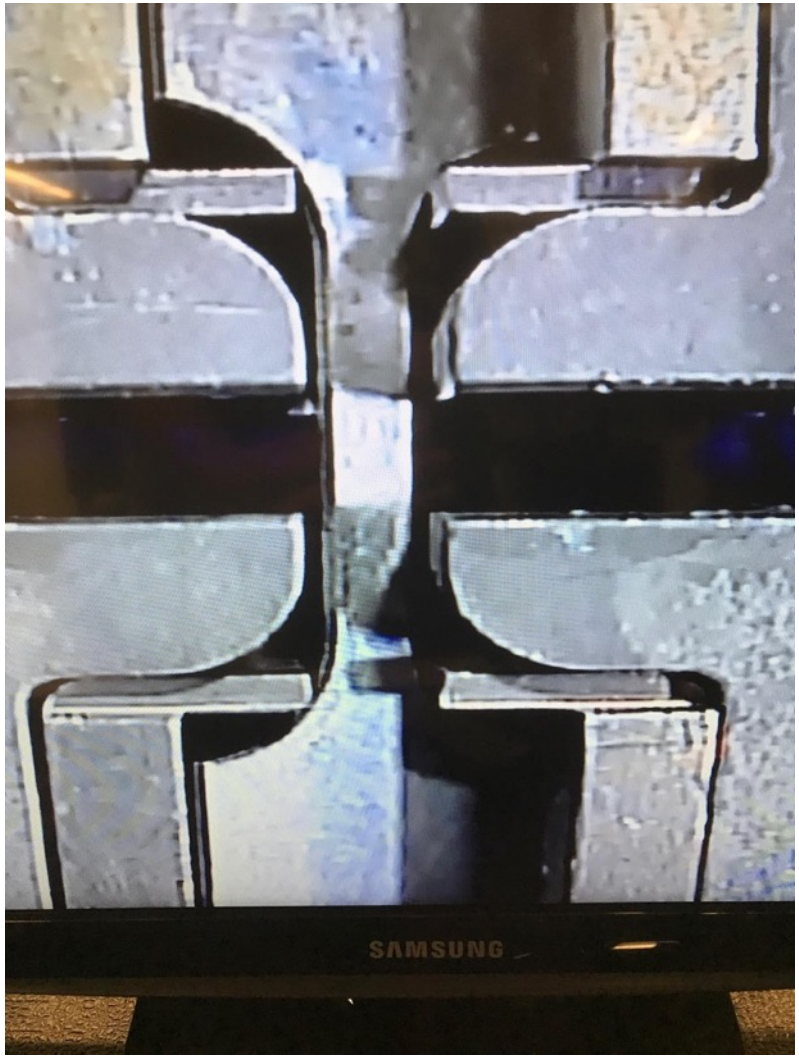
Do Not Try This at Home!



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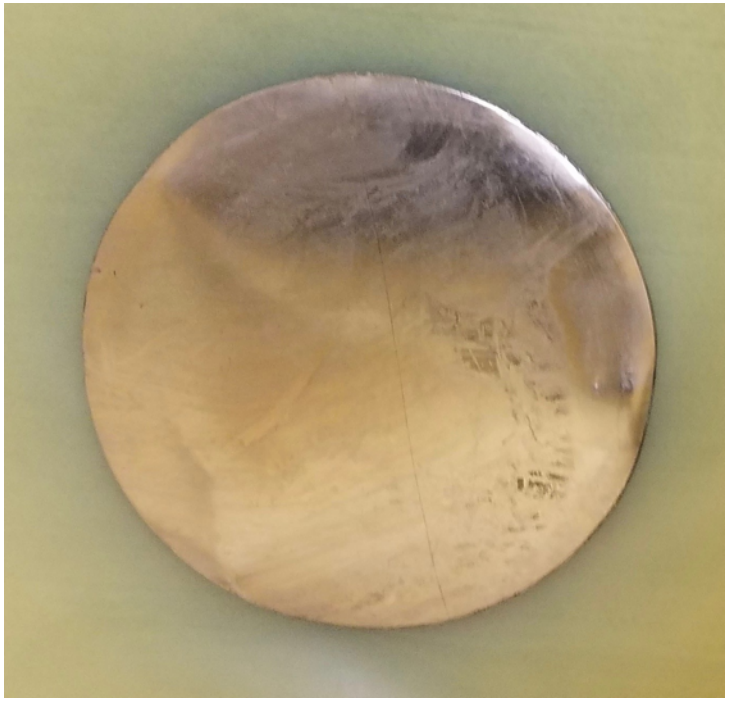
Correlating DS Ti-1 Upstream Face to Capsule Orientation



Be Capsule Film Dosimetry

- GafChromic MD-V3
- Sensitivity 1-100 Gy
- Upstream face of the Be capsule
- 30 hr exposure
- Orientation preserved
  - Direction of bend bar marks on inside of capsule face
  - Registration marks on film
  - Clipped corner of film

- Center of high dose region appears to be offset
  - ~5 mm upward
  - ~1 mm right