Correlation of Irradiation Damaged Microstructure to Localized Mechanical and Thermal Properties: A Proposal

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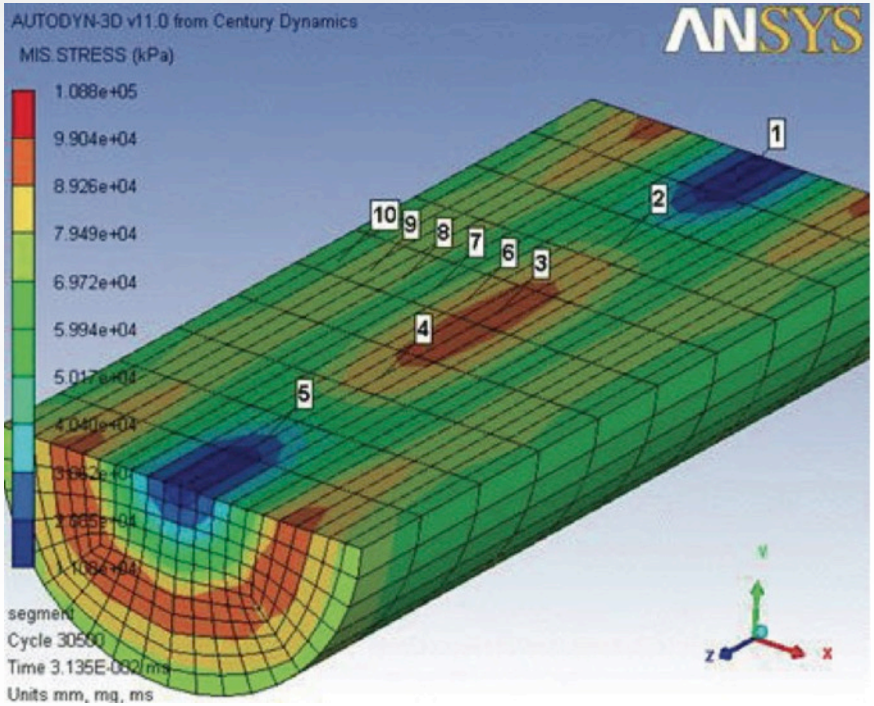
High Power Proton Accelerator Target and Window Design Needs

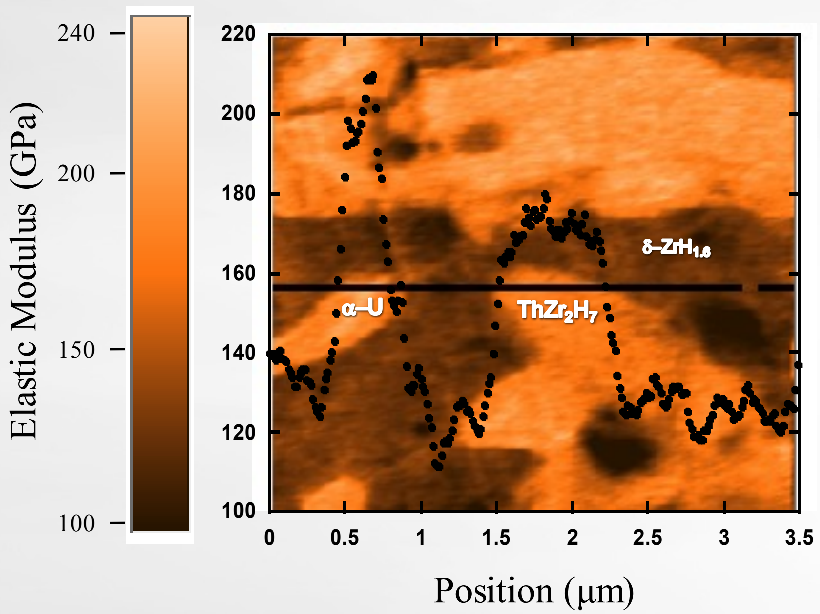
- Microscopic behavior
  - High radiation damage rates rapidly cause unacceptable material degradation
  - Better understanding of high energy proton radiation damage mechanisms needed
  - Enables tailored design of radiation-tolerant target or window materials
- Macroscopic properties
  - Coupled mechanical and thermal transient response to pulsed or rastered beams
  - During proton irradiation experiments, small beam size causes large thermal (and therefore, property) gradients
  - Difficult to measure properties on irradiated samples using traditional techniques
- Need to correlate effects of microstructural radiation damage to mechanical and thermal properties for a given combination of localized dose and irradiation temperature



Hurh, P. 2012. “High Power Target Challenges at Fermilab,” Project X Forum on Spallation Sources for Particle Physics.

Measuring Localized Mechanical Properties

- Atomic Force Microscopy
  - Commonly used for high-resolution topographic imaging
  - Can be used to measure hardness with sub-micron resolution using appropriate tips
  - Capable of measuring elastic modulus with sub-micron resolution using appropriate displacement control



Elastic modulus map of three-phase $(U_4Th_2Zr_9)H_{1.5}$ fuel produced using AFM

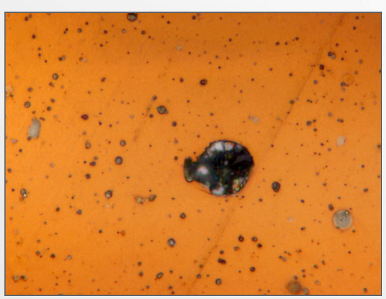
Olander, D, Balooch, M, et. al. 2012. "Investigation of Feasibility of Incorporation of Hydride in Fuels," ATR NSUF User Meeting.

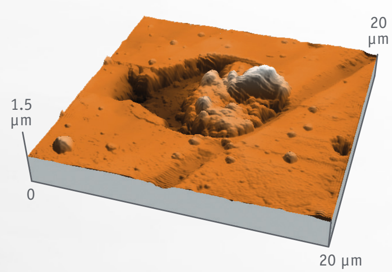
Measuring Localized Mechanical Properties

Nikon E400 POL Microscope

- PNNL has the capability to examine irradiated fuels/materials using an AFM attachment to an optical microscope
- Internal PNNL funding is being sought to develop this capability to produce elastic modulus maps in collaboration with Prof. Balooch of UC-Berkeley

Nanosurf LensAFM





Comparison of 100X Optical Microscope Image (left) to Nanosurf LensAFM Topographic Image (right) – LensAFM Product Literature, 2015.

Measuring Localized Mechanical Properties

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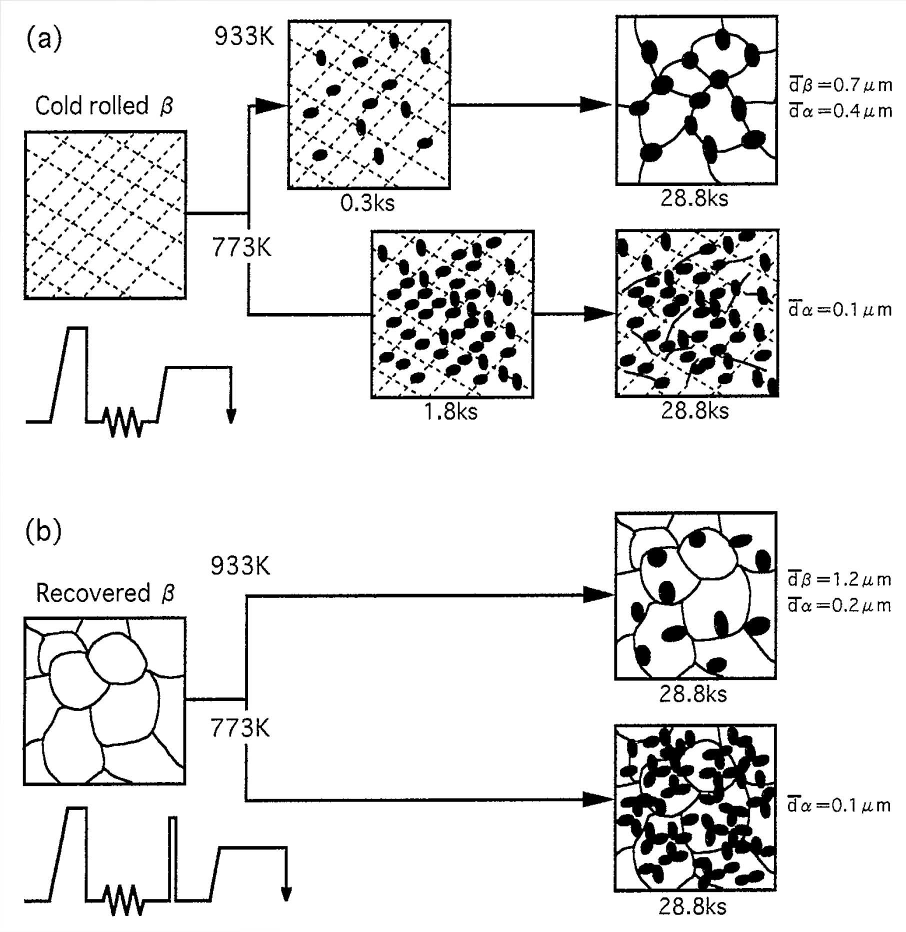
- If successful, the proposed technique could be useful for studying localized mechanical properties of multi-phase accelerator materials
- PNNL will be receiving and performing post-irradiation microscopy on OTR foils irradiated at T2K in Japan
- The Ti-15V-3Cr-3Sn-3Al alloy is a metastable β-Ti alloy
  - While nominally single-phase, aging will produce α-Ti precipitates
  - Irradiation at elevated temperatures is likely to produce a similar microstructure
  - Significant modulus difference between α-Ti and β-Ti phases
  - Irradiation experience with α-Ti and α+β Ti alloys has shown a tendency to precipitate additional Al- and V-rich phases
  - Ti-S and Ti-P phases have also been observed due to S and P impurities



Makino, T, et al. 1996. "Microstructure Development in a Thermomechanically Processed Ti-15V-3Cr-3Sn-3Al Alloy," *Materials Science and Engineering*, A213:51-60.

Measuring Localized Thermal Properties

- The laser flash method is a standard technique for measuring thermal diffusivity (α)
  - Typically requires samples 5-10 mm diameter and 1-3 mm thick (l)
  - Thermal diffusivity is related to the time required for the back surface of the sample to rise to half its peak value ($t_{1/2}$) after an incident laser flash
  - Not useful for evaluating thermal properties of proton beam irradiated samples due to geometry and gradient effects

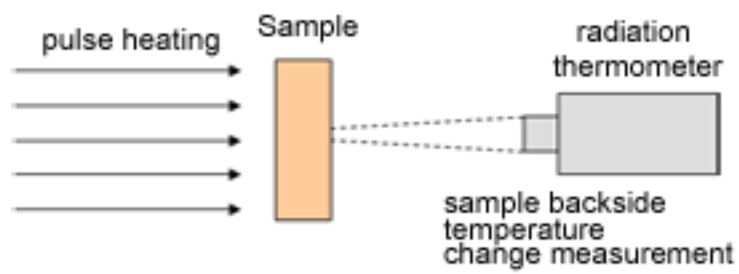


Fig. 1. Laser flash method principles

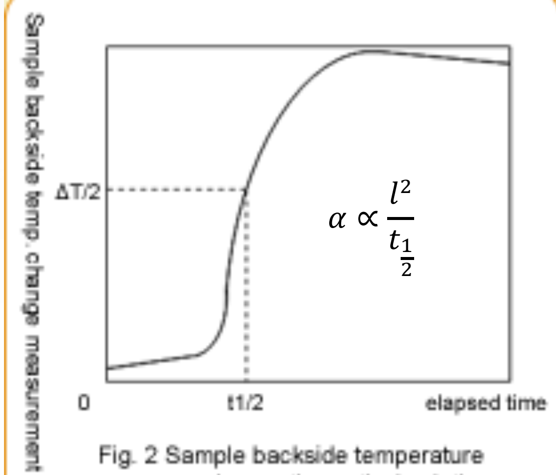
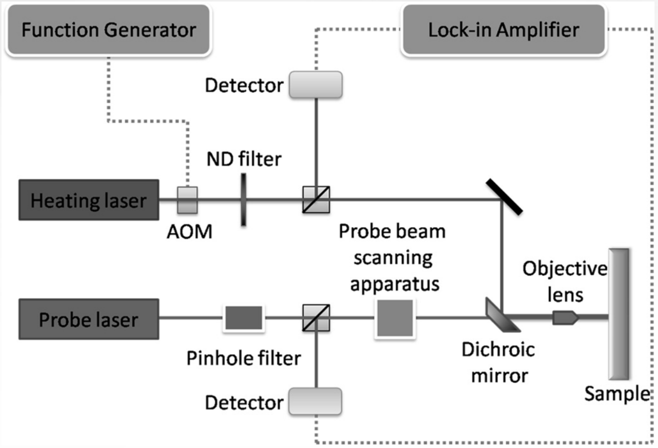


Fig. 2 Sample backside temperature change theoretical solution

Measuring Localized Thermal Properties

- Photothermal radiometry utilizes the same principles of thermal diffusion as the laser flash method but measures thermal diffusivity in a very small volume
  - A laser is used to heat the sample and create thermal waves on the surface
  - The frequency of the laser can be varied to deposit energy at different depths (useful for measuring diffusivity of thin layers)
  - The thermal diffusion length (μ) of the thermal waves is related to the thermal diffusivity (α) and laser frequency (ω)
  - The amplitude and phase components of the complex thermal wave number (σ) relates these measurable values to the thermal diffusion length and thermal diffusivity

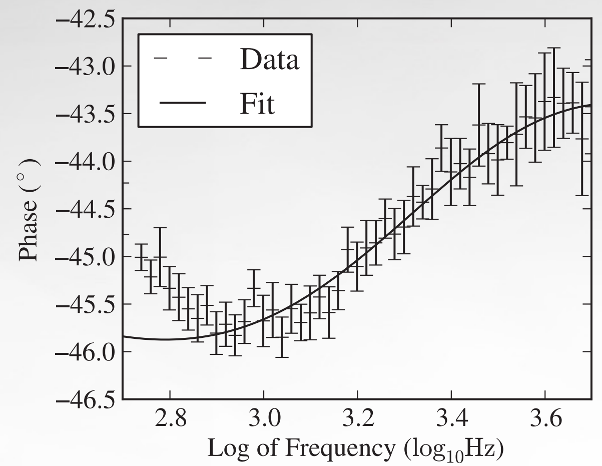


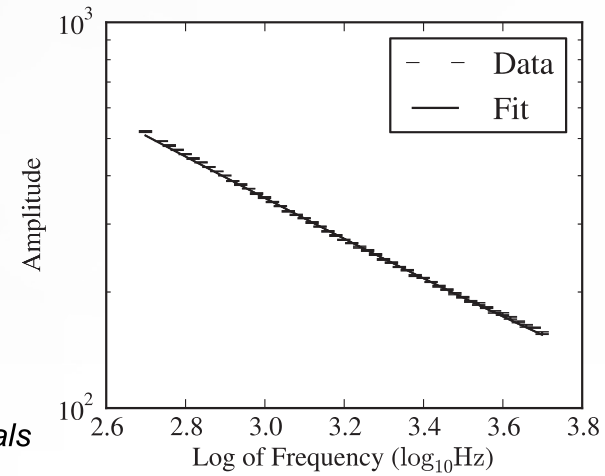
$$\mu_j = \sqrt{\frac{2\alpha_j}{\omega}}$$

$$\sigma_j = \frac{1+i}{\mu_j}$$

Measuring Localized Thermal Properties

- PNNL is collaborating with Prof. Heng Ban of Utah State University has developed photothermal radiometry systems for use with ion irradiated materials
  - Spatial resolution of 10s to 100s of μm
  - Depth sensitivity as low as 5 μm
  - Measured diffusivity in ZrC of ion-damaged, ion-implanted, and bulk regions by varying incident laser frequency
- Seeking internal PNNL funding to develop a system for irradiated materials/fuels





Horne, K, Ban, H, et al. 2012. "Photothermal Radiometry Measurement of Thermophysical Property Change of an Ion-Irradiated Sample," *Materials Science and Engineering*, B177:164-167.

Table 1
Thermal properties of irradiated ZrC at room temperature [15,16].

Property	Layer I	Layer II	Bulk	Units
Layer thickness L	~23	~5	~470	μm
Thermal conductivity k	10.4	10.0	20.5	W/(m K)
Thermal diffusivity α	1.95	6.95	8.88	1×10^{-6} m²/s
Specific heat C_p	813	219	352	J/(kg K)
Thermal effusivity e_f	7430	3800	6880	$W\sqrt{s}/(m^2 K)$