



**GAS-COOLED REACTOR**

ADVANCED REACTOR TECHNOLOGIES PROGRAM

07-16-2024

# Microtensile Specimen Analysis

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**DOE ART GCR Review Meeting**

*Hybrid Meeting at INL*

**July 16–18, 2024**

# Experimental Motivation

- Mechanical and structural properties of TRISO particle layers needed for more detailed modeling of failure probability and licensing.
- Through the work done by Mauseth (2023), a capability that enables micrometer scale tensile strength characterization of the buffer, IPyC, and buffer-IPyC interface of TRISO particles was developed.
- The capabilities developed by Mauseth (2023) can be applied to other TRISO particle layers and compacts.



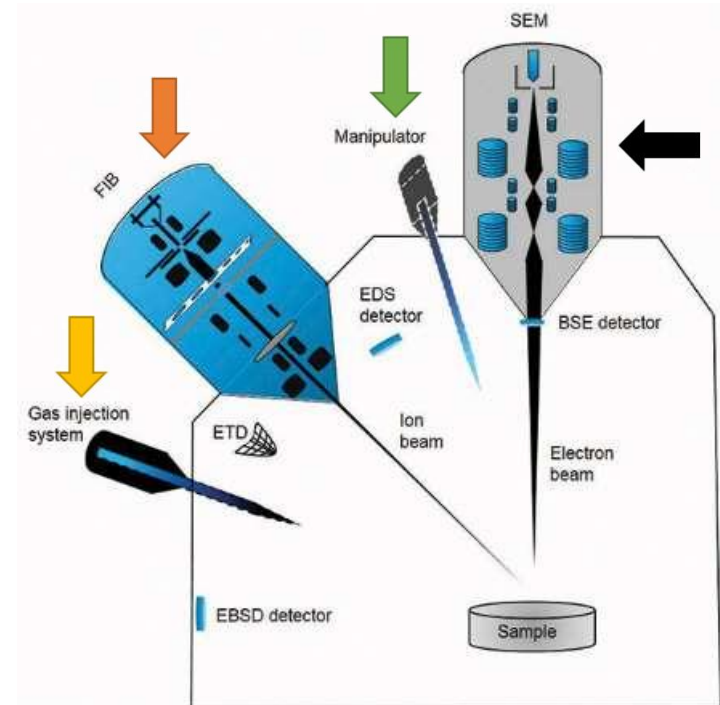
# Methods and Materials

- Instrumentation
- Sample Fabrication
- Testing and Data Acquisition



# FIB SEM Microscopes

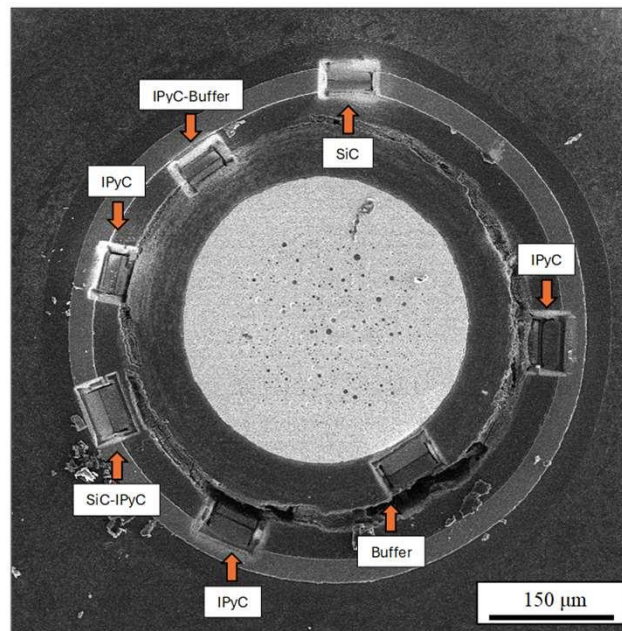
- Focused ion beam (FIB) and scanning electron microscope (SEM) dual beam systems were used to fabricate microtensile samples.
- Two FIB SEM microscopes were used at IMCL: a gallium FEI Quanta 3D Dual Beam and a Thermo G3 Plasma Dual Beam.
- Micro-tensile fabrication technique same as technique developed in study done by Mauseth et al. (2023), with a few modifications due to the use of two different FIBs.



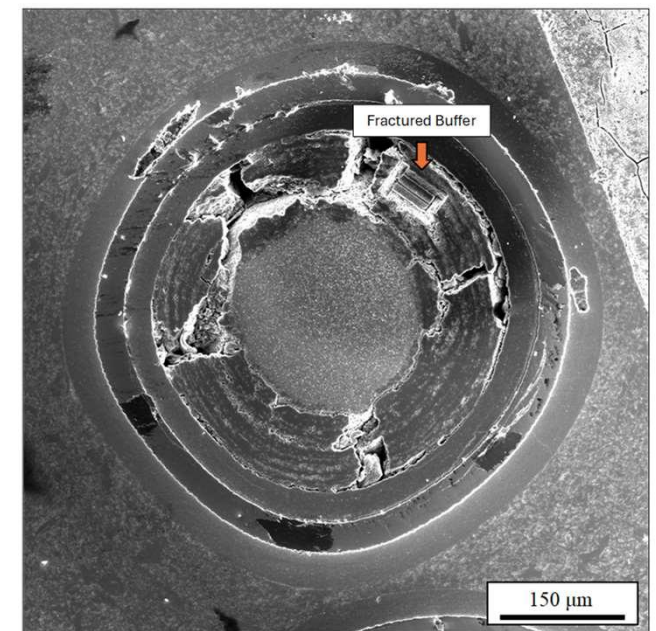
# Microtensile Sample Locations



Compact Overview

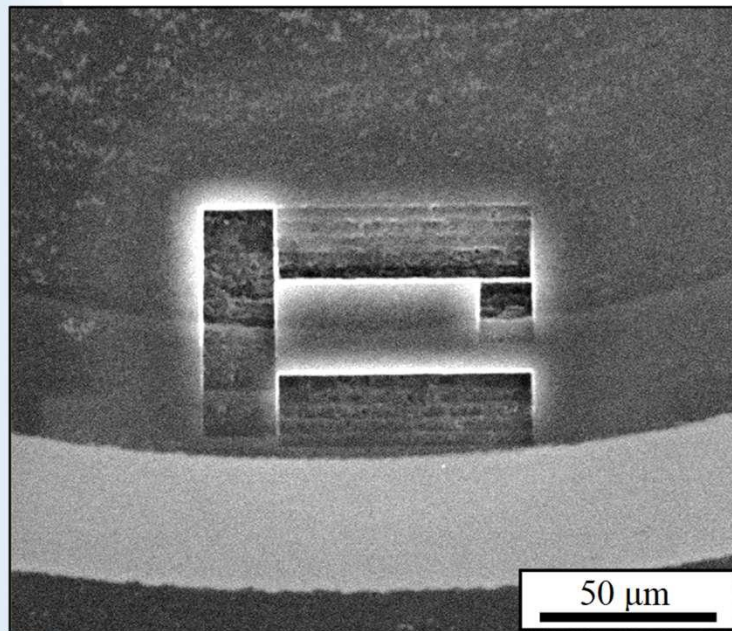


Standard Particle

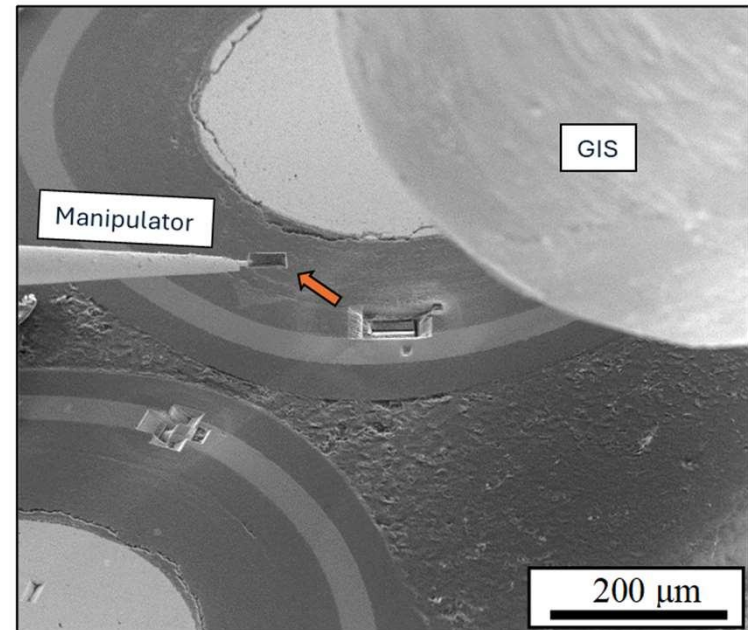


Fractured Particle

# Microtensile Sample Fabrication

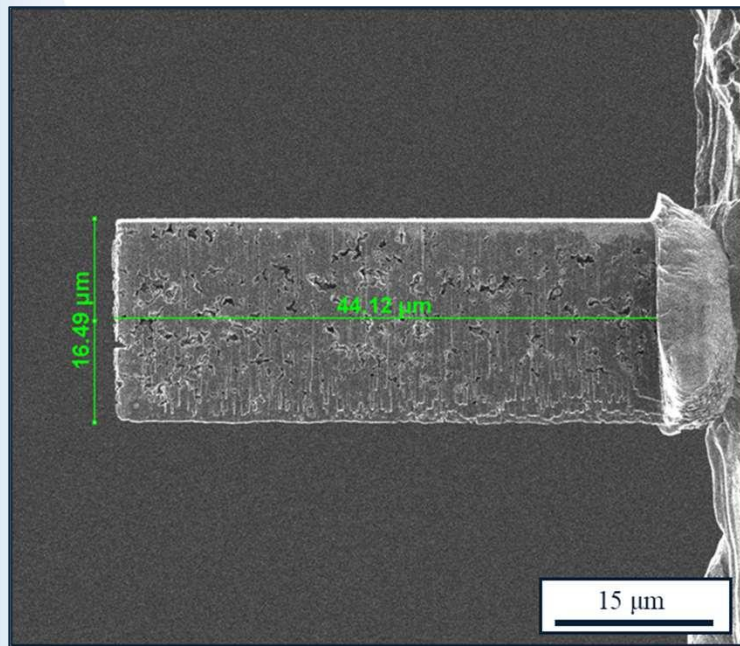


Trenched Block

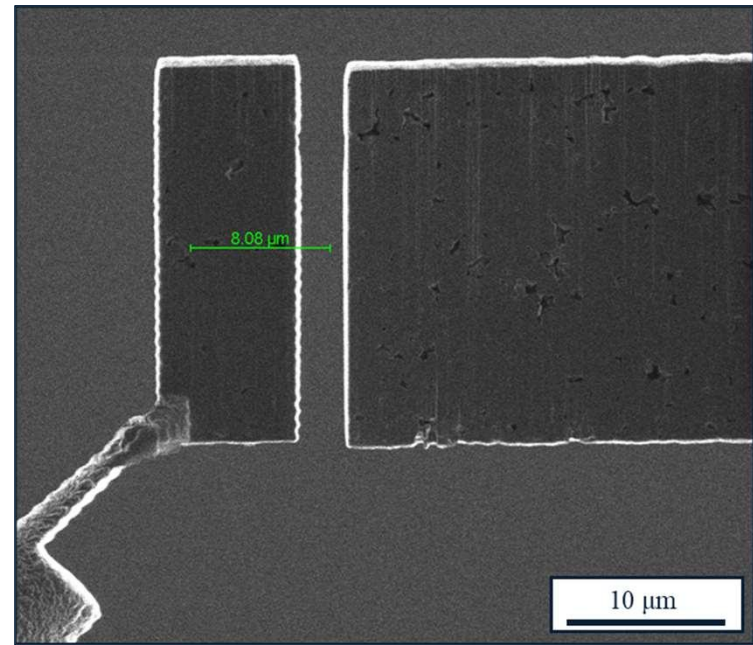


Lifted-Out Block

# Microtensile Sample Fabrication (cont.)

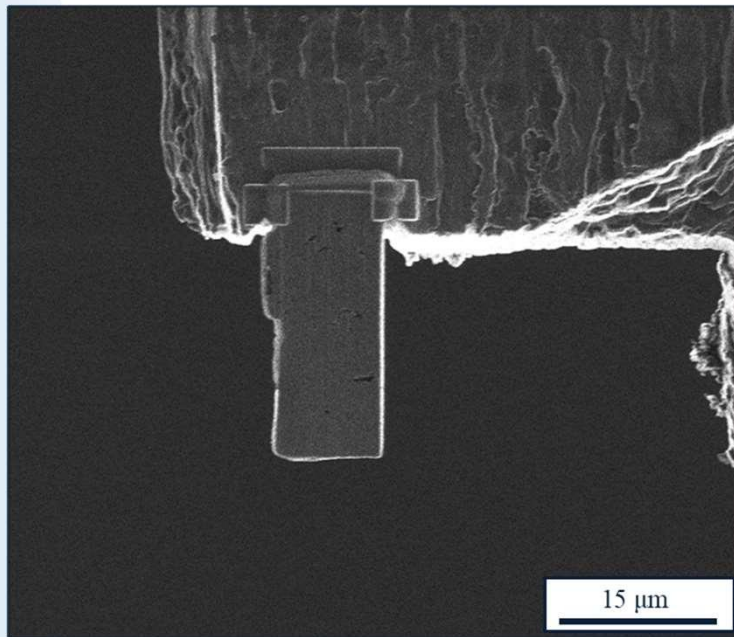


Thinned Block

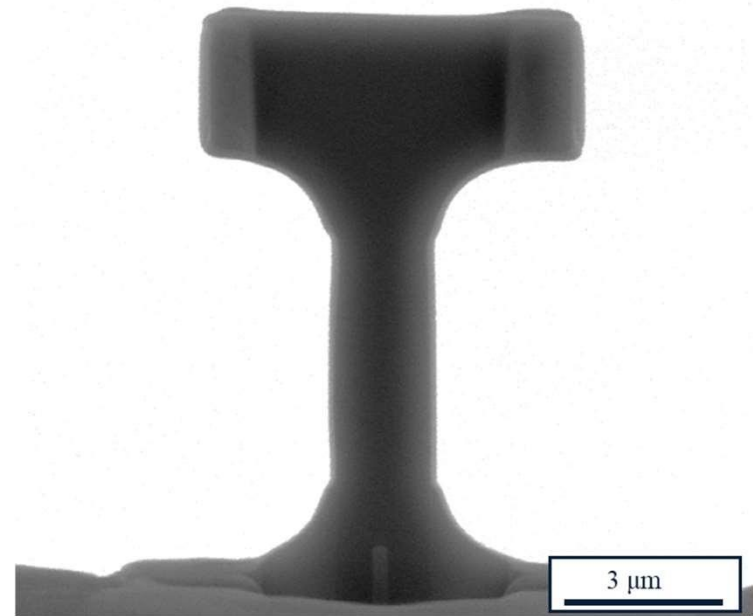


Individual Lamella

# Microtensile Sample Fabrication (cont.)



Mounted Lamella

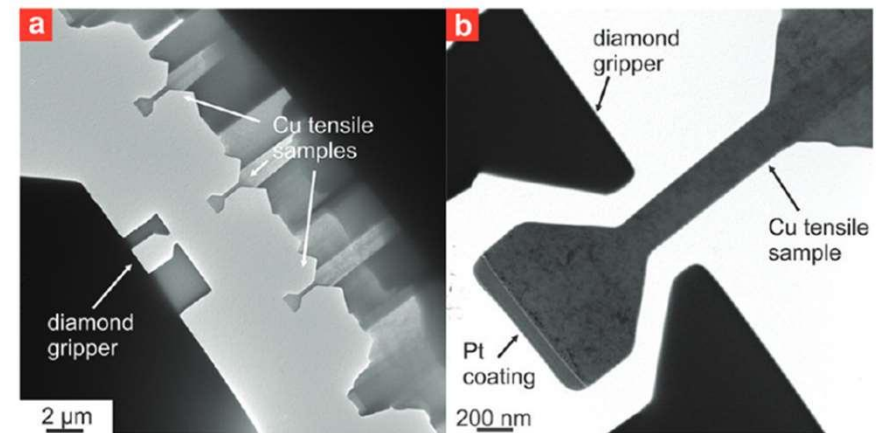


Final Microtensile Sample



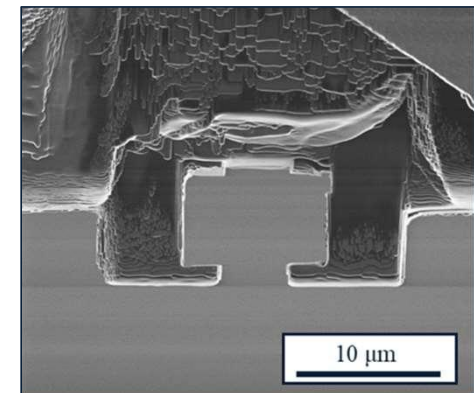
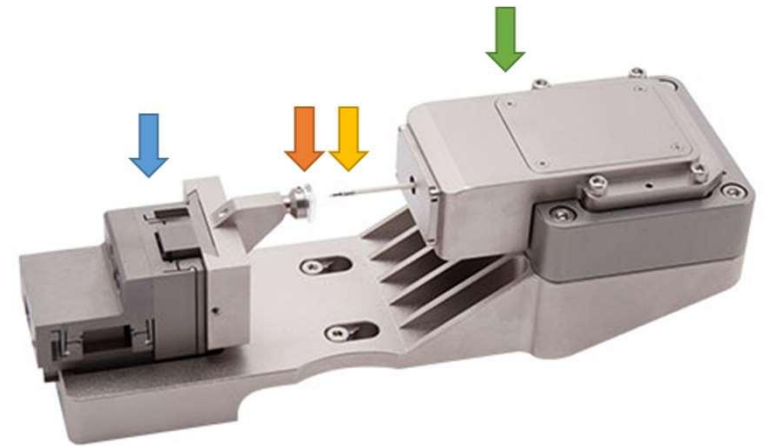
# Tensile Testing

- Tensile testing is an old practice but is new for mechanical testing of TRISO particle layers.
- Kiener and Minor developed a novel quantitative, in situ nano-tensile testing technique that was applied in a TEM (Transmission Electron Microscope) setting.
- The diamond gripper, tensile samples, and testing technique for this research were inspired by Kiener and Minors work.
- Microtensile testing and data acquisition techniques same as techniques developed in study done by Mauseth et al. (2023).

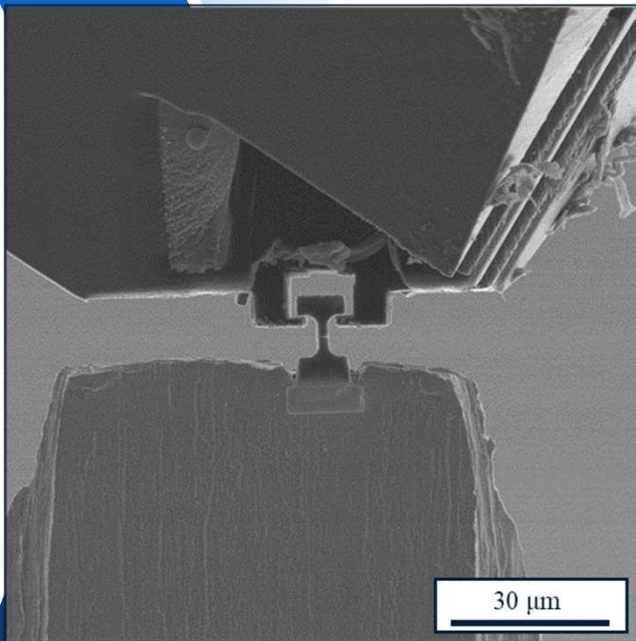


## Micromechanical Load Cell

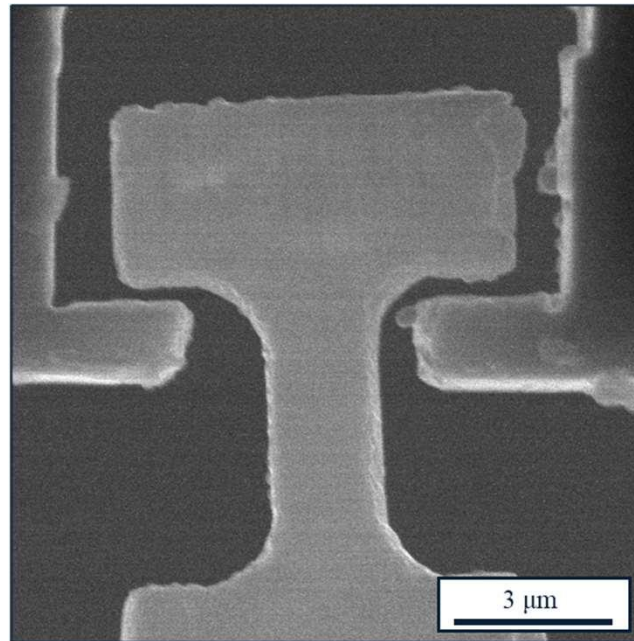
- Bruker Hysitron PI 88 SEM PicoIndenter was used to perform microtensile test in conjunction with SEM.
- The PI 88 is typically used to conduct nanoindentation, was retrofitted with a diamond gripper to enable microtensile testing.



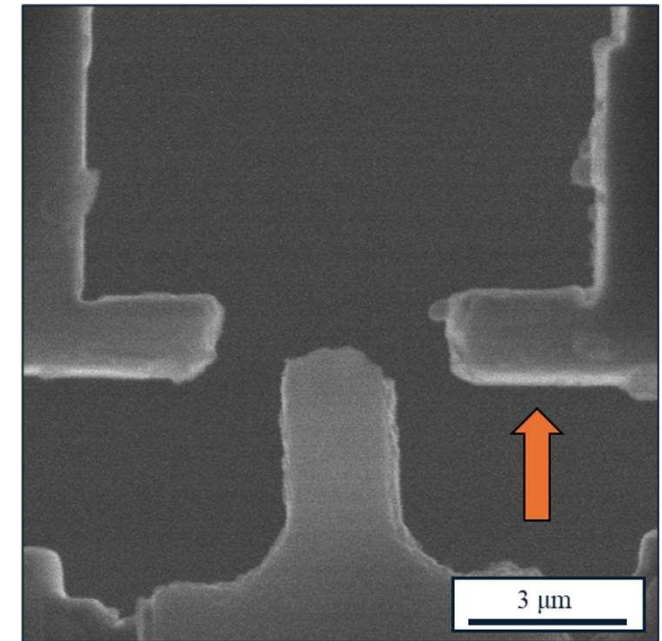
# PI 88 Microtensile Testing



Testing Overview

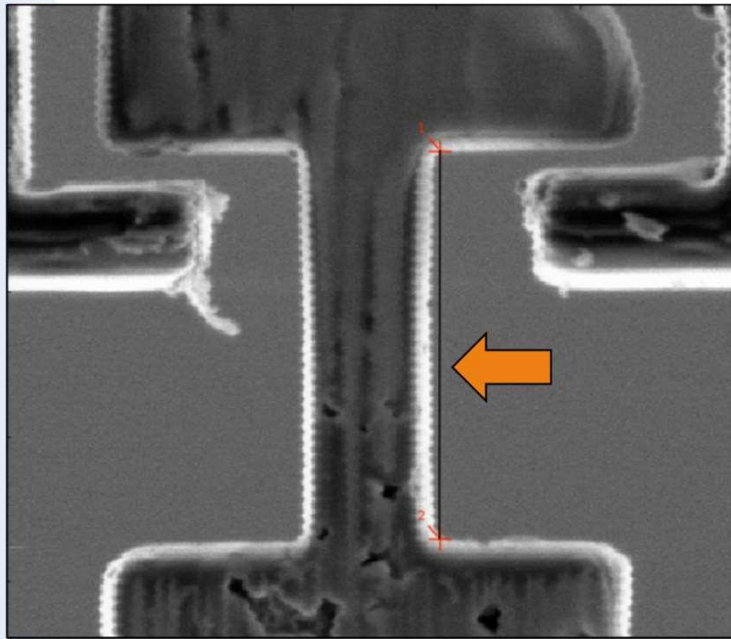


Before Test

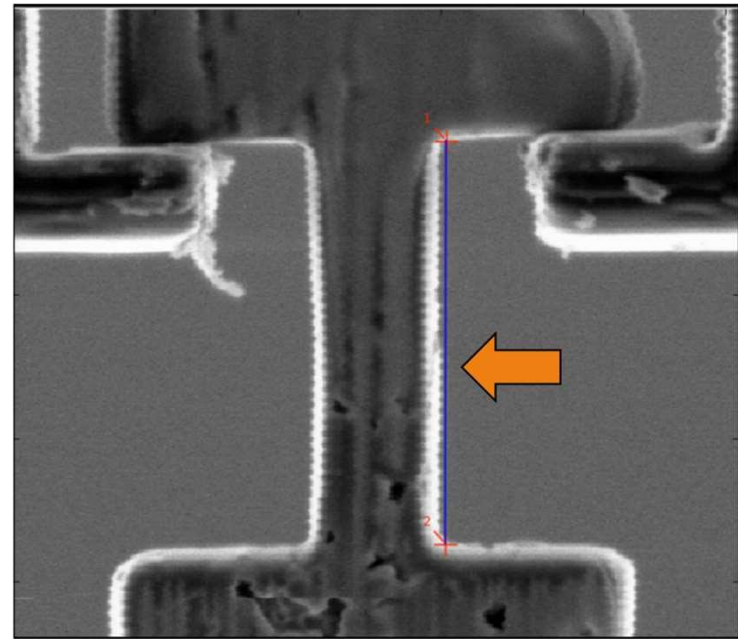


After Test

# Digital Image Correlation (DIC)



Before Test



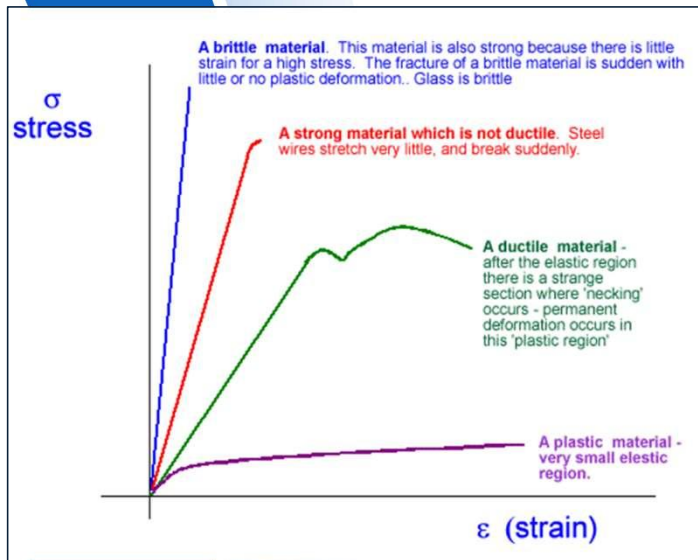
During Test

# Theory

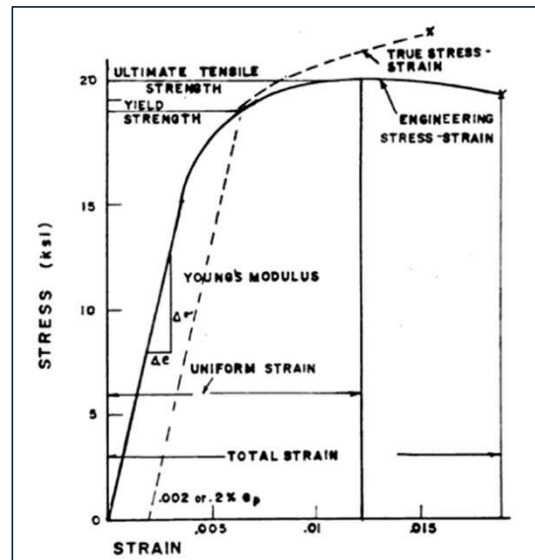
- Tensile Characteristics
- Weibull Statistics



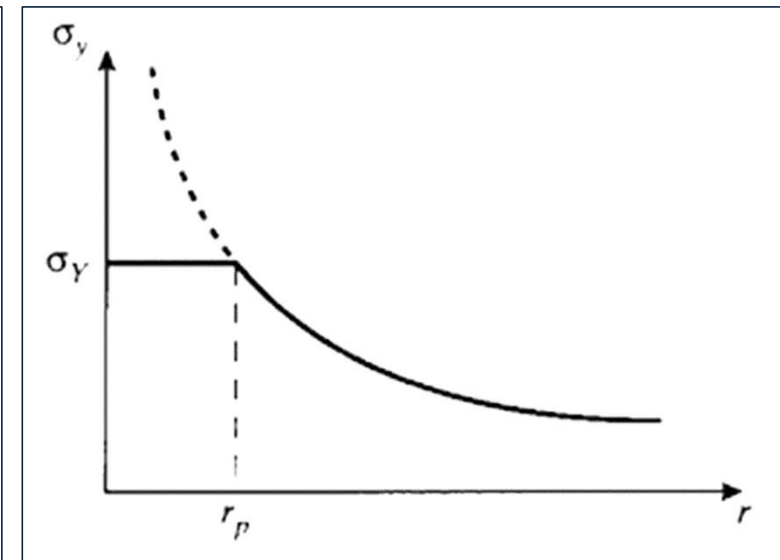
# Tensile Characteristics



Material Differences



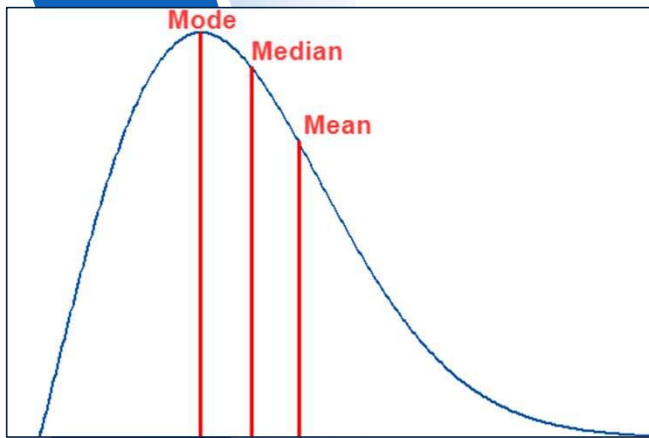
Stress-Strain Metrics



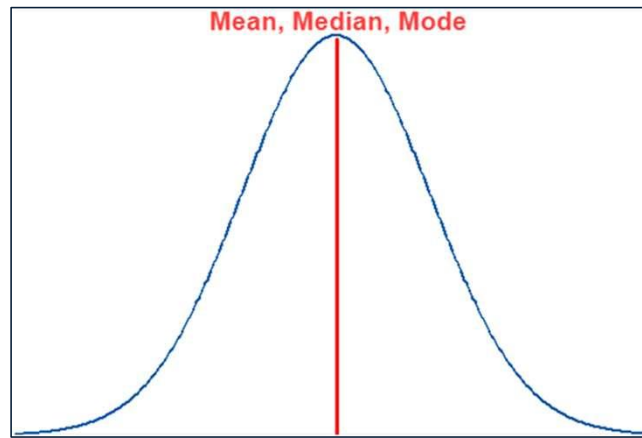
Fracture Stress vs Flaw Size



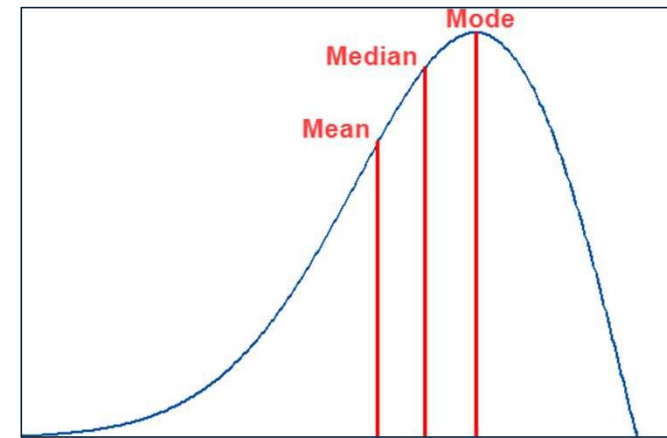
# Weibull Statistics



Right Skewed Curve



Normal Distribution



Left Skewed Curve



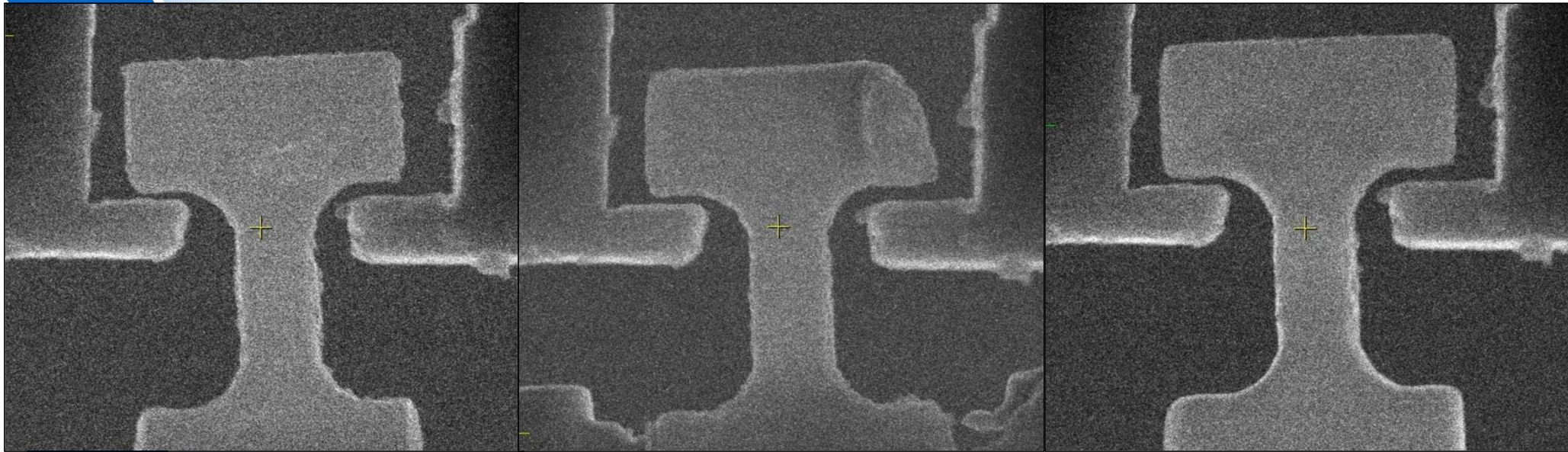
# Microtensile Results

- Six different AGR-2 UCO TRISO particles tested over four different compact samples (LEU09-F52, MNTD42, MNT64X, and MNT67X).
- Buffer, IPyC-Buffer, and IPyC layers were tested in every compact sample.
- Fractured Buffer, SiC-IPyC, and SiC layers were tested in MNT64X and MNT67X.





# Fracture Behavior



Brittle and Steady  
(Buffer, Fractured  
Buffer, IPyC-Buffer)

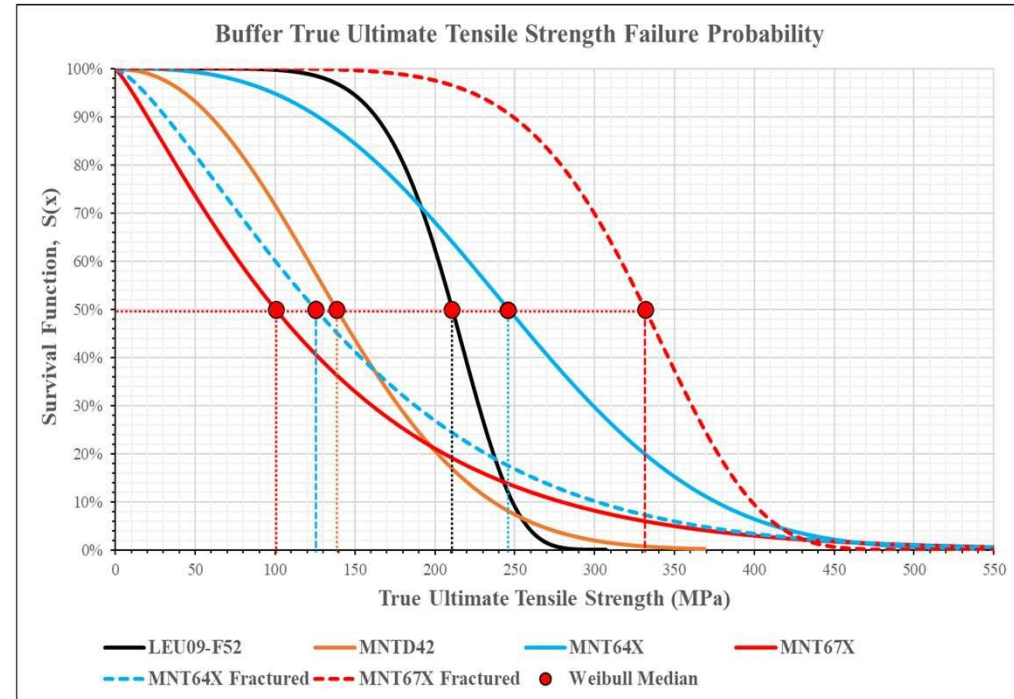
Brittle and Abrupt  
(IPyC, SiC, IPyC-  
Buffer)

Clean Delamination  
(SiC-IPyC)



# Buffer Comparison

| Mechanical Values                                     | LEU09-F52 | MNTD42 | MNT64X | MNT67X | MNT64X Fractured | MNT67X Fractured |
|---|-----------|--------|--------|--------|------------------|------------------|
| Number of samples                                     | 4         | 5      | 5      | 4      | 5                | 4                |
| Neutron Fluence (x10 <sup>25</sup> n/m <sup>2</sup> ) | None      | 2.14   | 3.03   | 2.88   | 3.03             | 2.88             |
| TAVA (°C)   | None      | 1060   | 1078   | 1194   | 1078             | 1194             |
| Weibull Median (MPa)                                  | 210.74    | 138.57 | 246.00 | 100.37 | 125.36           | 331.57           |
| Modulus (GPa)   | 19.35     | 22.83  | 14.55  | 13.65  | 8.46             | 27.43            |
| R <sup>2</sup>  | 0.92      | 0.98   | 0.90   | 0.94   | 0.94             | 0.91             |



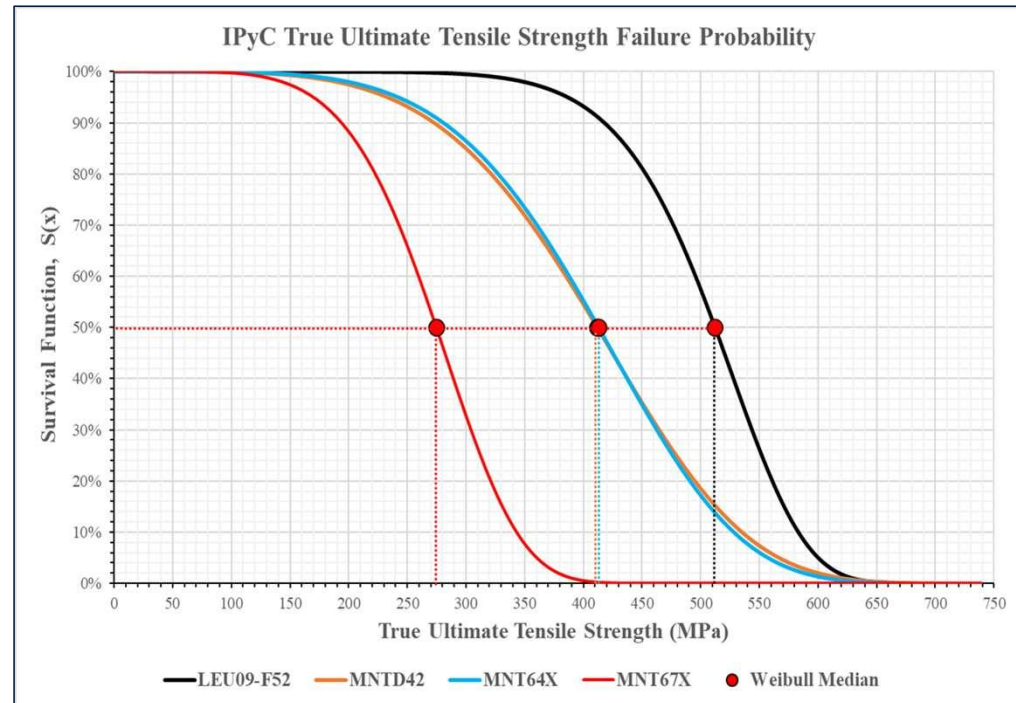
## Buffer Discussion

- MNT67X Fractured and MNT67X samples demonstrated both the highest and lowest strength values.
- Strength between buffer samples did not directly correlate well with irradiation and temperature, suggesting that differences in porosity and other microstructural features are the driving factor behind buffer layer sample fracture behavior.



# IPyC Comparison

| Mechanical Values                                     | LEU09-F52 | MNTD42 | MNT64X | MNT67X |
|---|-----------|--------|--------|--------|
| Number of samples                                     | 4         | 4      | 7      | 9      |
| Neutron Fluence ( $\times 10^{25}$ n/m <sup>2</sup> ) | None      | 2.14   | 3.03   | 2.88   |
| TAVA (°C)   | None      | 1060   | 1078   | 1194   |
| Weibull Median (MPa)                                  | 512.12    | 411.94 | 413.17 | 274.62 |
| Modulus (GPa)   | 31.69     | 27.11  | 27.21  | 30.81  |
| R <sup>2</sup>  | 0.89      | 0.94   | 0.99   | 0.93   |



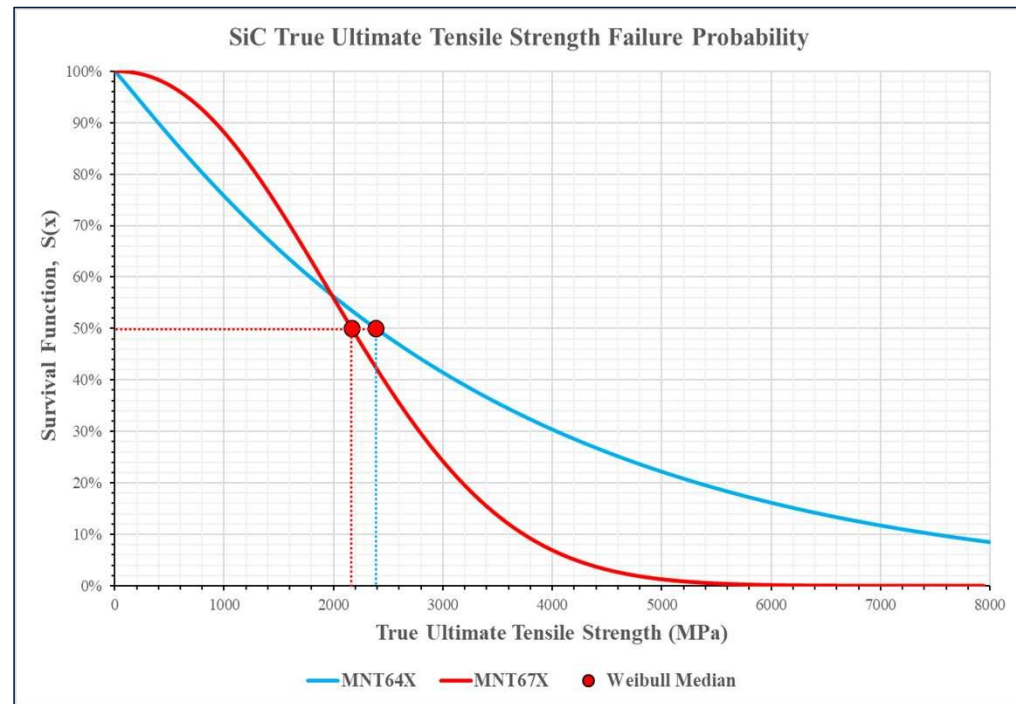
# IPyC Discussion

- Decrease in strength appears to correspond with increase in TAVA.
- Additionally, PARFUME metrics align well with experimental results:
  - PARFUME PyC Weibull Modulus: 9.5 with a density of 1.9 g/cm<sup>3</sup>
  - LEU09-F52 IPyC Weibull Modulus: 9.26 with a density of 1.85-1.95 g/cm<sup>3</sup>
  - PARFUME PyC Elastic Moduli: 26 - 62 GPa
  - Experimental IPyC Elastic Moduli: 27.11 – 31.69 GPa



# SiC Comparison

| Mechanical Values  | MNT64X  | MNT67X  |
|--|---------|---------|
| Number of samples  | 5       | 4       |
| Neutron Fluence<br>( $\times 10^{25}$ n/m <sup>2</sup> ) | 3.03    | 2.88    |
| TAVA (°C)  | 1078    | 1194    |
| Weibull Median<br>(MPa)                                  | 2388.16 | 2166.77 |
| Modulus (GPa)  | 335.94  | 154.64  |
| R <sup>2</sup>   | 0.94    | 0.98    |



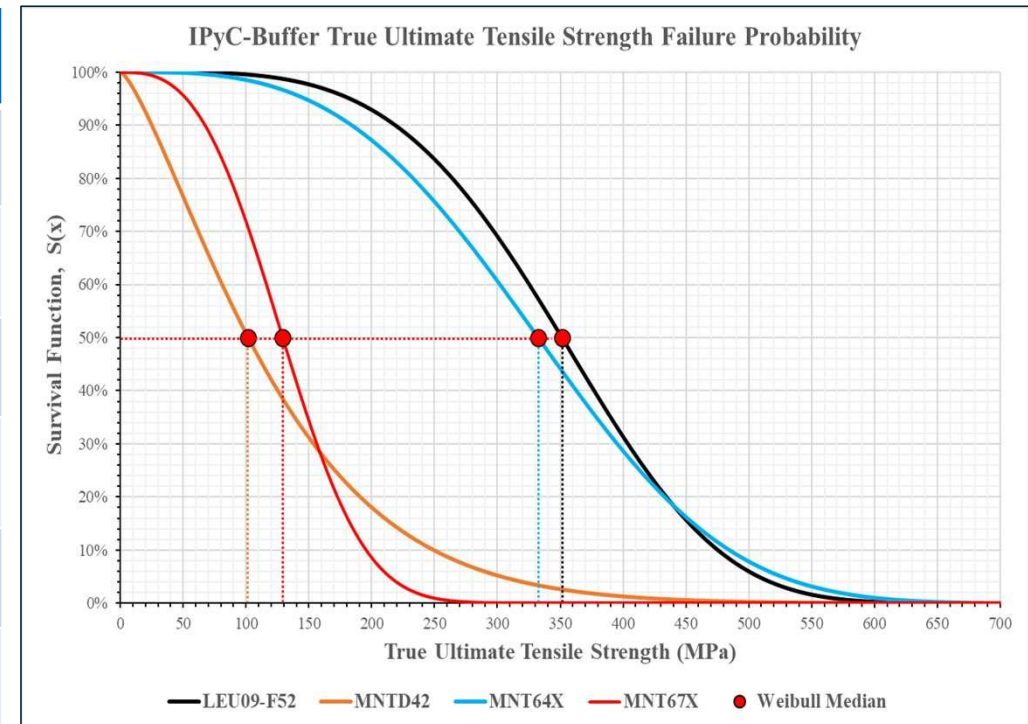
## SiC Discussion

- MNT64X appears to be moderately stronger than MNT67X.
- Unclear whether difference in strength between MNT64X and MNT67X is due to differences in irradiation and temperature or statistical variation.



# IPyC-Buffer Comparison

| Mechanical Values                                     | LEU09-F52 | MNTD42 | MNT64X | MNT67X |
|---|-----------|--------|--------|--------|
| Number of samples                                     | 12        | 9      | 4      | 5      |
| Neutron Fluence (x10 <sup>25</sup> n/m <sup>2</sup> ) | None      | 2.14   | 3.03   | 2.88   |
| TAVA (°C)   | None      | 1060   | 1078   | 1194   |
| Weibull Median (MPa)                                  | 351.33    | 101.60 | 332.39 | 129.25 |
| Modulus (GPa)   | 14.38     | 5.30   | 21.32  | 11.12  |
| R <sup>2</sup>  | 0.96      | 0.83   | 0.81   | 0.88   |





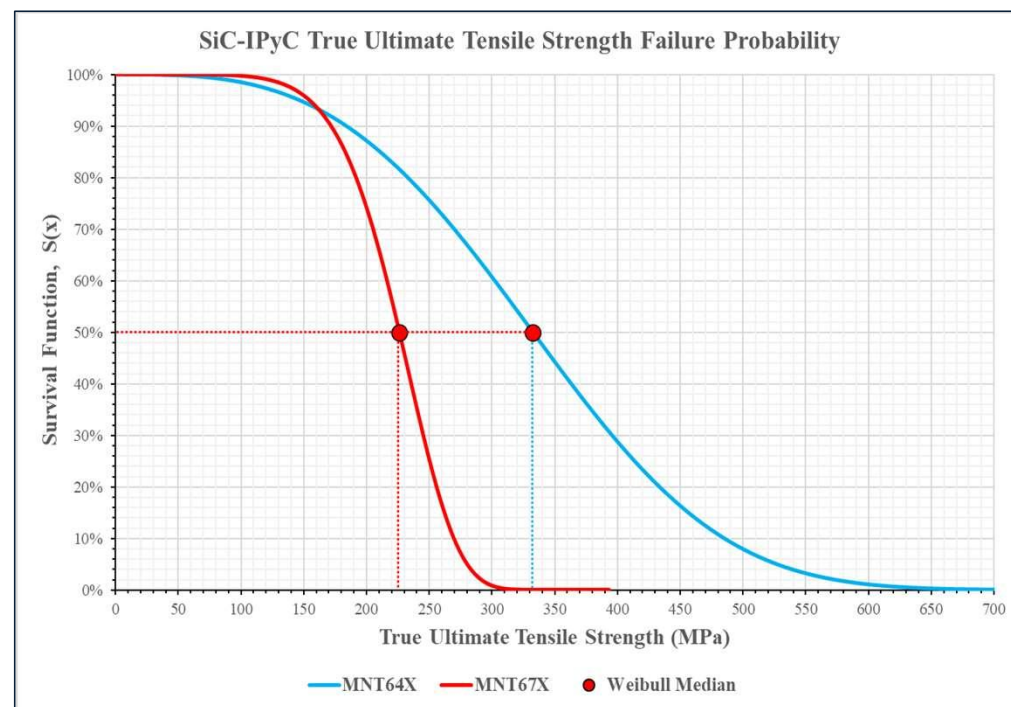
## IPyC-Buffer Discussion

- LEU09-F52 and MNT64X samples comparable in strength, MNTD42 and MNT67X significantly weaker.
- Buffer layer strength between individual particles may play a significant role in Buffer-IPyC debonding strength.



# SiC-IPyC Comparison

| Mechanical Values  | MNT64X | MNT67X |
|--|--------|--------|
| Number of samples  | 5      | 5      |
| Neutron Fluence<br>( $\times 10^{25}$ n/m <sup>2</sup> ) | 3.03   | 2.88   |
| TAVA (°C)  | 1078   | 1194   |
| Weibull Median<br>(MPa)                                  | 332.39 | 225.97 |
| Modulus (GPa)  | 21.32  | 25.42  |
| R <sup>2</sup>   | 0.93   | 0.89   |



## SiC-IPyC Discussion

- MNT64X appears to be stronger than MNT67X.
- SiC-IPyC interlayer samples from both MNT64X and MNT67X display lower strengths than SiC and IPyC layers from same parent particle.



# Summary and Future Considerations

- Buffer, Fractured Buffer, IPyC-Buffer, IPyC, SiC-IPyC, and SiC layers tested over multiple TRISO particles and irradiation conditions.
- Samples tested thus far paint a general picture, more testing needed to clarify properties of select layers:
  - Testing TRISO particles with higher neutron fluence and TAVA would help clarify correlated strength trends.
  - Different sized microtensile samples in different orientations in the buffer layer would help clarify any porosity-based size effects.
  - Other layers and layer interfaces (Kernel, Buffer-Kernel, OPyC-SiC, OPyC, Matrix-OPyC, etc.) have yet to be investigated.



# References

- Kiener, D., & Minor, A. M. (2011). Source truncation and exhaustion: Insights from quantitative in situ TEM tensile testing. *Nano Letters*, 11(9), 3816–3820. <https://doi.org/10.1021/nl201890s>
- Mauseth, T., Dunzik-Gougar, M. L., Meher, S., & van Rooyen, I. J. (2023). Determining the Tensile Strength of Fuel Surrogate TRISO-coated Particle Buffer, IPyC, and Buffer-IPyC Interlayer Regions. *Journal of Nuclear Materials*, 154540. <https://doi.org/10.1016/J.JNUCMAT.2023.154540>
- Mauseth, T. (2023). Micro-tensile Characterization of Select TRISO-coated Particle Layers and Interlayer Regions. Idaho State University.
- PI 88 SEM PicoIndenter ®. (2020). <https://airtable.com/shrrfypV1Wg7J9ULa>
- Wolff, A. (2020). Focused ion beams: An overview of the technology and its capabilities - 2020 - Wiley Analytical Science. <https://analyticalscience.wiley.com/do/10.1002/was.00070009>



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**Thank you for  
attending. Any  
questions?**

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