OAK RIDGE National Laboratory

GAS-COOLED REACTOR
ADVANCED REACTOR TECHNOLOGIES PROGRAM

Effect of Sample Thickness on the Tensile Strength of Small Graphite Discs

Lianshan Lin[1], Nidia C. Gallego[2]

1. Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN, 37831, USA

2. Chemical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, TN, 37831, USA

U.S. DEPARTMENT OF ENERGY

July 17, 2024

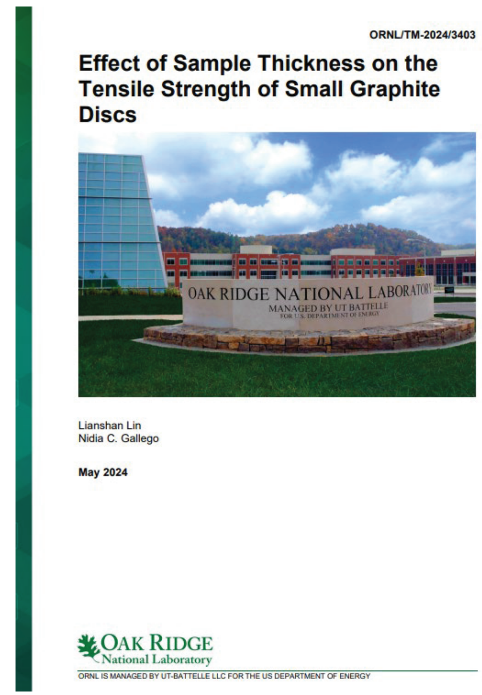
DOE ART GCR Review Meeting
Hybrid Meeting at INL
July 16–18, 2024

Summary

Objectives:
Investigate the factors limiting the applicability of ASTM D8289 for a true measurement of tensile strength of graphite and developed an improved standard.

Approach:
Incorporated DIC technique and studied the effect of sample thickness on splitting tensile strength when applying the ASTM D8289 standard.

Accomplishments:
- Implemented the 3D DIC technique while performing split disc testing (ASTM D8289)
- Measured split disc tensile strength of two fine grain graphites of various thicknesses
- Completed Milestone M3TG-24OR0501054: ORNL/TM-2024/3403


ORNL/TM-2024/3403

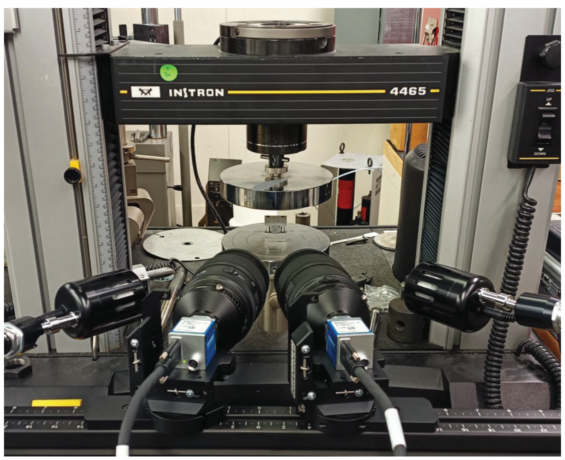
Effect of Sample Thickness on the Tensile Strength of Small Graphite Discs

Lianshan Lin
Nidia C. Gallego

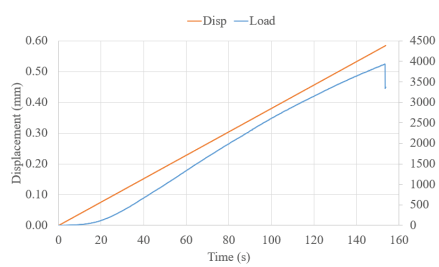
May 2024

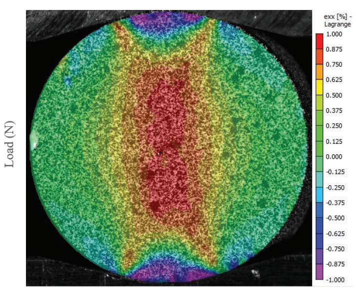
OAK RIDGE National Laboratory
ORNL IS MANAGED BY UT-BATTELLE LLC FOR THE US DEPARTMENT OF ENERGY

Test Facility & Experimental Setup



Experimental Results



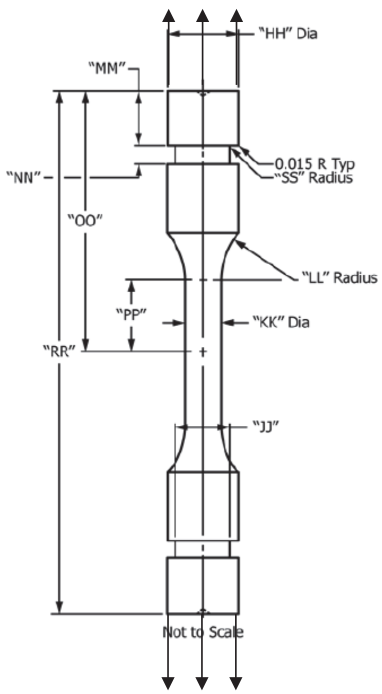


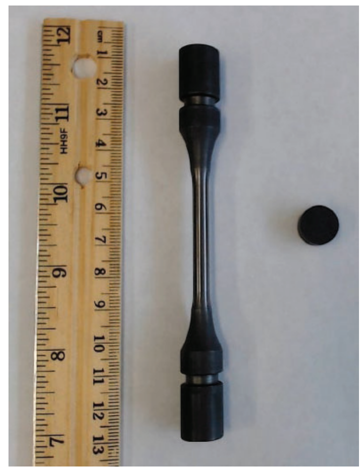
OAK RIDGE National Laboratory

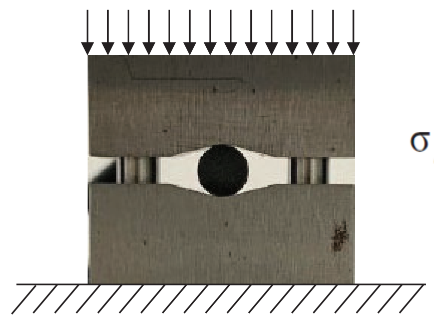
GAS-COOLED REACTOR
ADVANCED REACTOR TECHNOLOGIES PROGRAM

Standards for graphite tensile strength

C749







$$\sigma_{sts} \approx 0.931 \frac{P}{\pi L R}$$

D8289

Ultimate strength, the **strain to failure**, and the **elastic moduli** can be calculated, sample size ≥ 0.51"x4$\frac{3}{4}$" (12.95mm x 120.65 mm).[1]

Determining the **splitting tensile strength** of graphite, the small specimen geometry (minimum Ø6mm x 3mm, maximum Ø12.7mm x 6.35mm) is specifically intended for irradiation capsule use.[1,2,3]

1. ASTM D8289-19, Standard Test Method for Tensile Strength Estimate by Disc Comparison of Manufactured Graphite, Annual Book of ASTM Standards, ASTM International, West Conshohocken, Pennsylvania, 2020.
2. ASTM C749, Standard Test Method for Tensile Stress-Strain of Carbon and Graphite, Annual Book of ASTM Standards, ASTM International, West Conshohocken, Pennsylvania, 1973.
3. Awaji, H. and Sato, S., "Diametral Compressive Stress Considering the Hertzian Contact," Journal of the Society of Materials Science, Japan, Vol 27, 1978, pp. 336–341.

Comparison between Standard C749 and D8289

ASTM C749 Uniaxial Tensile

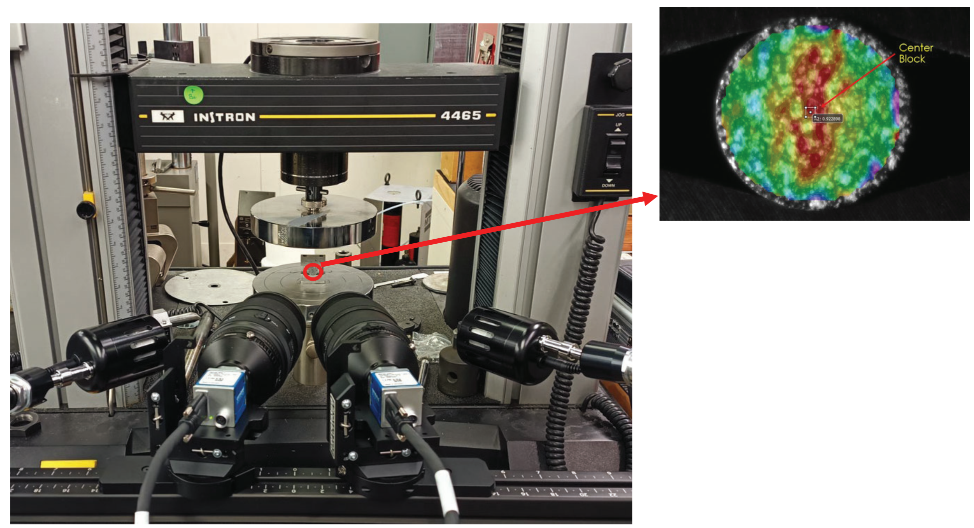
- Pros
  - Traditional standard
  - A straightforward method
  - Simple calculation of tensile strength
  - Ultimate strength, strain to failure, and elastic moduli can be calculated
- Cons
  - Large sample size
  - Not ideal for irradiation samples
  - More procedures to manufacture the sample
  - Grip problem

ASTM D8289 Splitting Disc

- Pros
  - The small sample size fit with irradiation capsule applications
  - Simple sample geometry
  - Easy to manufacture
- Cons
  - Standard provides only an "**estimation**" of tensile strength
  - Special loading fixture needed
  - Deformation and strain information is not available

DIC technique can be incorporated with D8289 to extract additional information

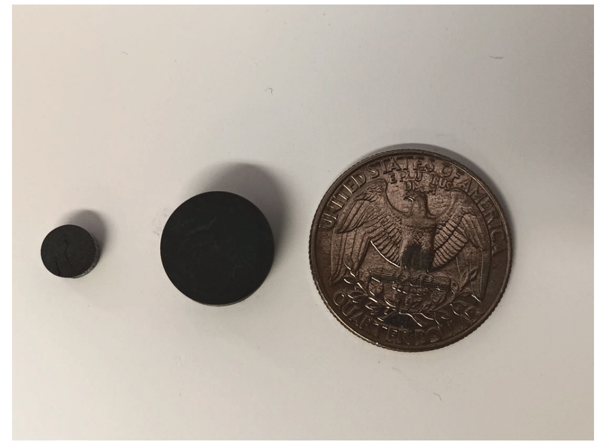
- DIC can be used to extract displacement/strain from loaded specimen without any accessory attached on specimen surface
- No change in standard disk splitting test
- Solve the difficulty of using traditional strain gage
- The measured displacement/strain field can help interpret the loading history and mechanical behavior before crack[4]

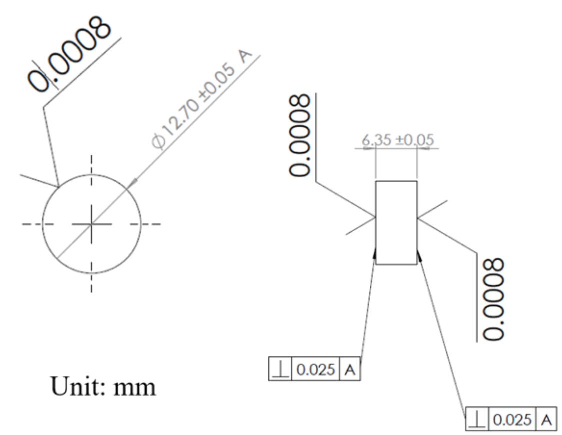


4. Lin L, Paul R, Hawkins C, Gallego NC. Mechanical behavior analysis using small-size graphite disc compression testing with digital image correlation methods. Journal of Nuclear Materials. 2023 Dec 15;587:154731.

Specimen geometry and dimensions in ASTM D8289

- Determining the splitting tensile strength of graphite, the small specimen geometry (minimum Ø6mm x 3mm, maximum Ø12.7mm x 6.35mm) is specifically intended for irradiation capsule use.





7. Test Specimens

7.1 The minimum specimen diameter is 6 mm. Specimen geometries used in the intra-laboratory study are given in Table 2. Note the thickness values represent the maximum allowed thickness for the corresponding diameter.

TABLE 2 Acceptable Specimen Geometries

Specimen Dimensions	
Diameter, mm	Thickness, mm
6	3
8	4
10	5
12.7	6.35

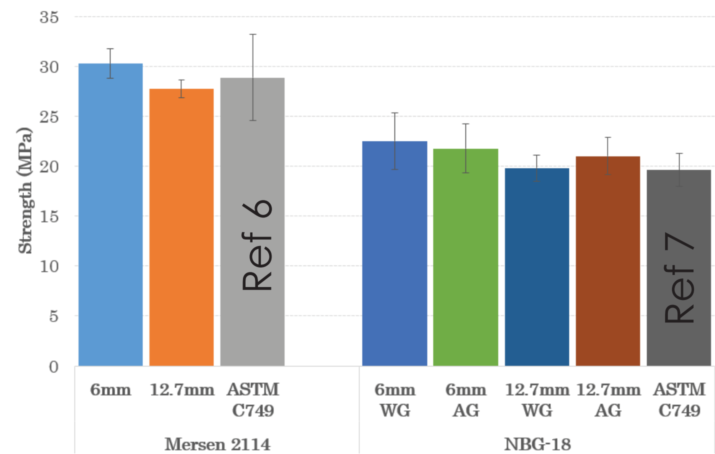
Interlaboratory study for ASTM D8289

- **Used the same thickness—maximum allowable thickness**

7 laboratories, 6 samples for each specified graphite[5]
Sample diameters Ø6 and Ø12.7 mm, thickness = radius

Material	Average $\bar{x}$	Repeatability Standard Deviation sr	Reproducibility Standard Deviation sR	Repeatability Limit r	Reproducibility Limit R
Graphite A, 12.7mm	27.738	0.896	1.478	2.510	4.139
Graphite B WG, 12.7 mm	19.807	1.287	1.474	3.603	4.127
Graphite B AG, 12.7mm	21.022	1.866	2.078	5.224	5.819
Graphite A, 6mm dia	30.263	1.498	4.297	4.195	12.033
Graphite B WG, 6mm dia.	22.510	2.848	4.373	7.973	12.244
Graphite B AG, 6mm dia.	21.761	2.463	3.318	6.898	9.291

Graphite A: Mersen 2114
Graphite B: NBG-18



5. W. David Swank, Interlaboratory Study to Establish Precision Statements for ASTM D8289-19, Test Method for Tensile Strength Estimate by Disk Compression of Manufactured Graphite, ASTM, Research Report: D02-1910, 2019.
6. T. D. Burchell, Mersen Grade 2114: Tensile Strength from Brazilian Disc Testing, ORNL/TM-2020/1485, March 2020.
7. NUMARK Associates Inc, Appendices to the Assessment of Graphite Properties and Degradation, Including Source Dependence, U.S. NRC, TLR/RES/DE/REB-2021-03, 2021.

Sample thickness consideration

- ASTM D8289 requires specimen thickness ≤ diameter/2.

7. Test Specimens

7.1 The minimum specimen diameter is 6 mm. Specimen geometries used in the intra-laboratory study are given in Table 2. Note the thickness values represent the maximum allowed thickness for the corresponding diameter.

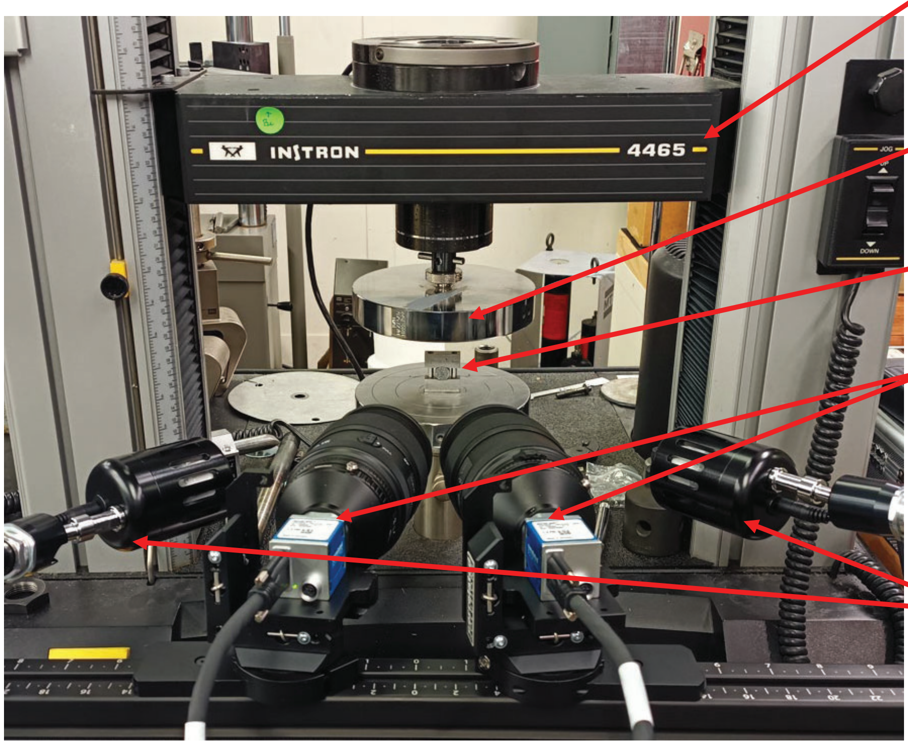
- Equation to calculate splitting tensile strength is based on 2D plane stress assumption.
- In engineering world, plane stress state assumes thickness < diameter/3, plane strain state assumes thickness > diameter *3.
- What's the effect of sample thickness on tensile strength?

Test plan – ASTM D8289 tests

Graphite grade	Graphite properties[8]			Sample diameter (mm)	Sample thickness (mm)	Number of samples tested
	Grain size (μm)	Bulk density (g·cm^{-3})	Porosity (%)			
2114	13	1.81	19	12.7	6.35	8
					5	8
					4	8
					3	8
IG-110	10	1.76	21	12.7	6.35	8
					5	8
					4	8
					3	8

8. Gallego NC, Contescu CI, Keiser J. Progress report on graphite-salt intrusion studies. Oak Ridge National Lab.(ORNL), Oak Ridge, TN (United States); 2020 Jul 1.

Test facilities and setup



Load frame

Platen

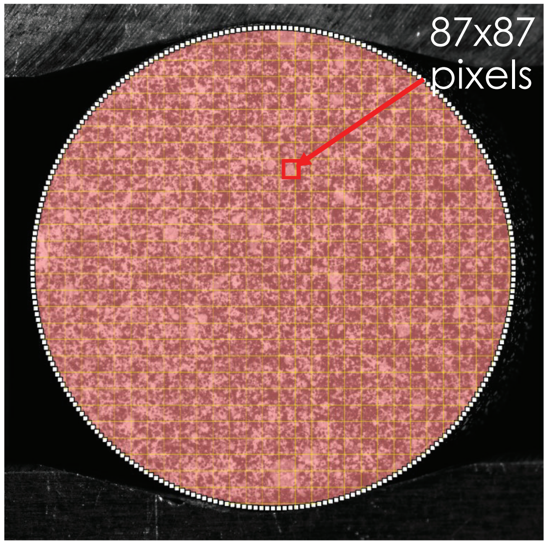
Fixture and specimen

Two cameras

Two lights

The loading force is applied via displacement control mode in a rate of 0.2286 mm/min (0.009 in./min) to satisfy an average rupture time greater than 30 s required by Standard.

The DIC help evaluate the thickness's impact on splitting tensile strength by measuring the surface displacement/strain.

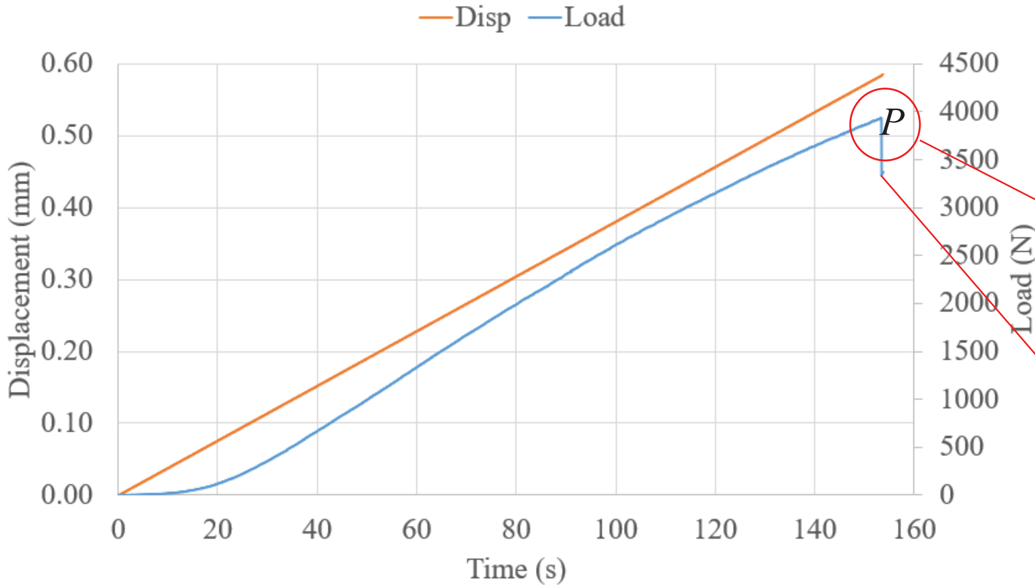


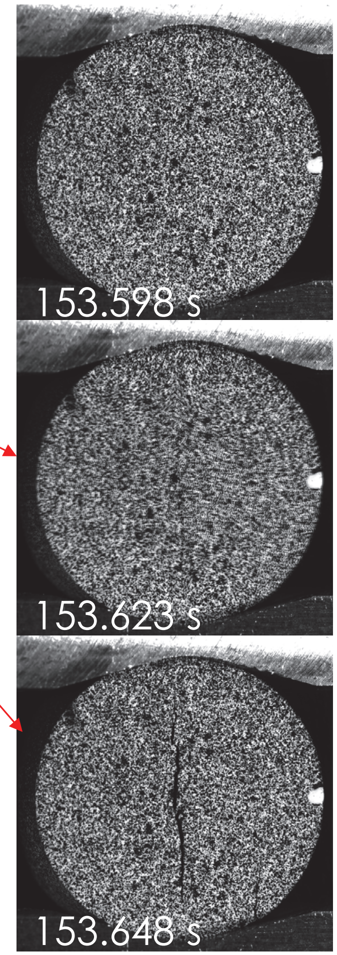
correlated SOLUTIONS VIC-3D

Full resolution of 2840 × 2840 pixels
Optical resolution ~203 pixel/mm
Camera shutter speed: 40 Hz

Load and displacement

Mersen 2114 Ø12.7 mm disc, thickness 6.35 mm sample 1





Frame 6144

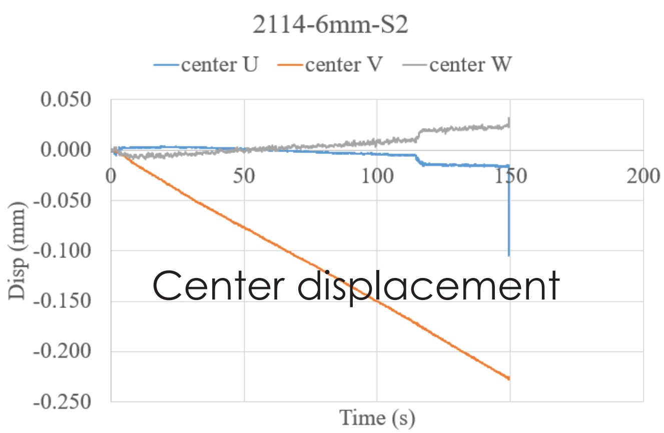
Frame 6145

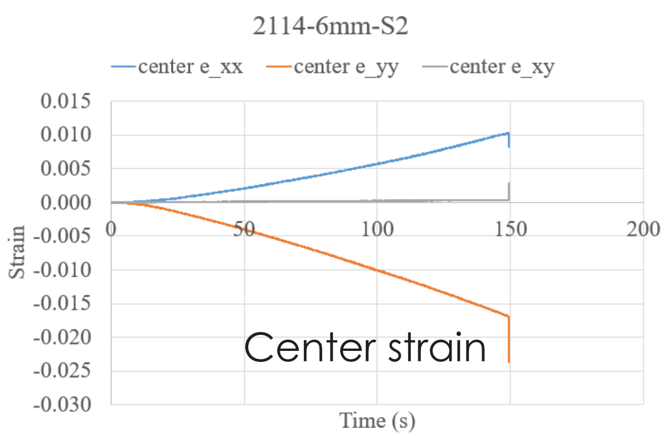
Frame 6146
Center crack

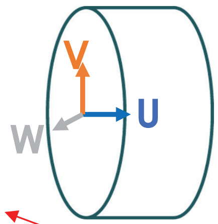
σ_{sts} = splitting tensile strength, MPa (psi),
P = maximum applied load indicated by the testing machine, N (or lbf),
L = thickness of the specimen, mm (or in.),
D = diameter of the specimen, mm (or in.),
b = circumference of contact length, mm (or in.), and
R = specimen radius, mm (or in.).

$$\sigma_{sts} \approx 0.931 \frac{P}{\pi L R}$$

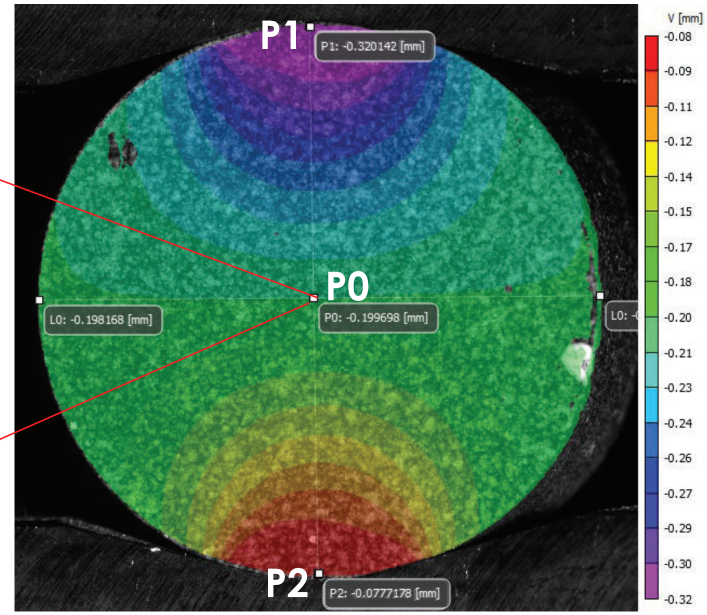
Measurements from DIC



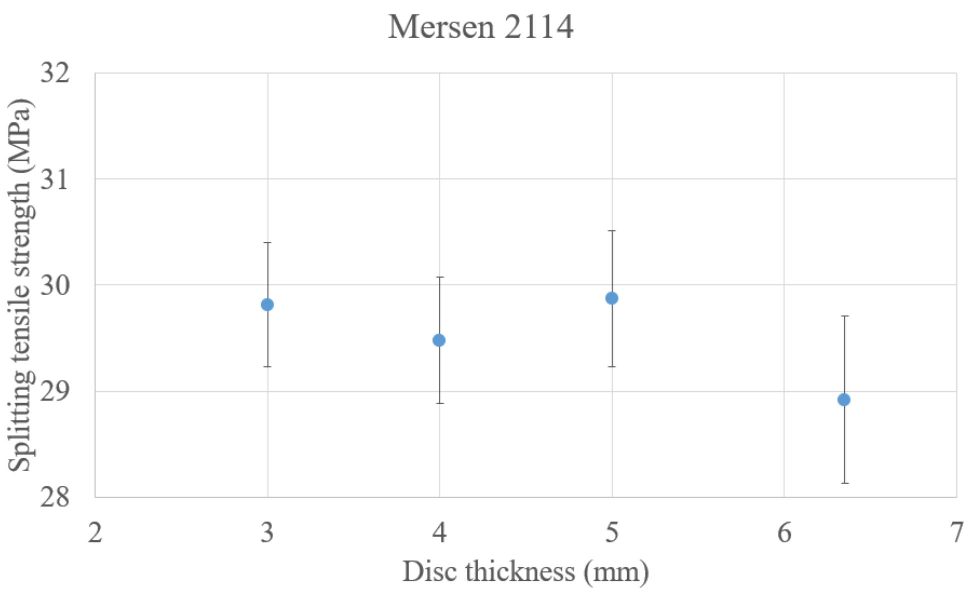


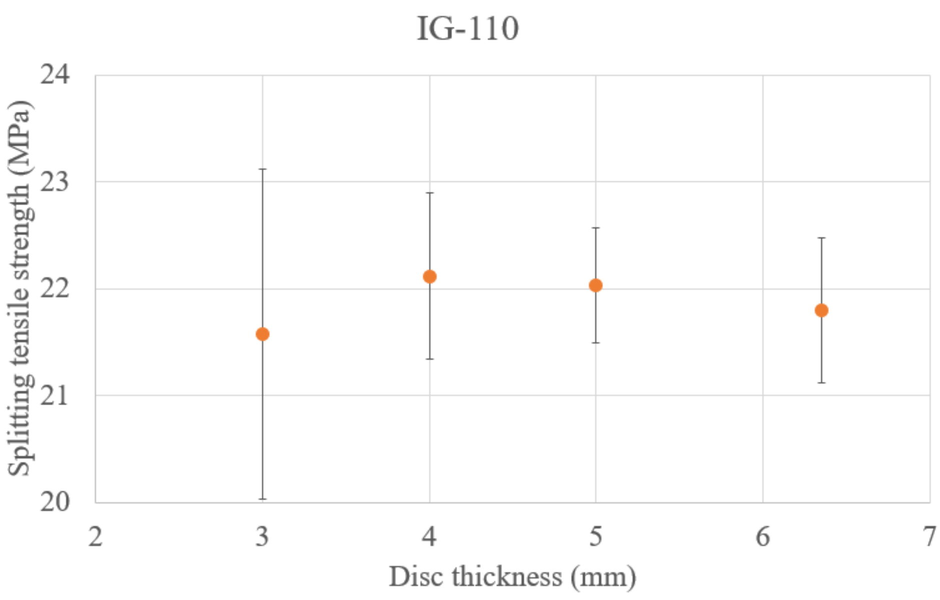


Cross lines to allocate points **P0** (center), **P1** (top) and **P2** (bottom)

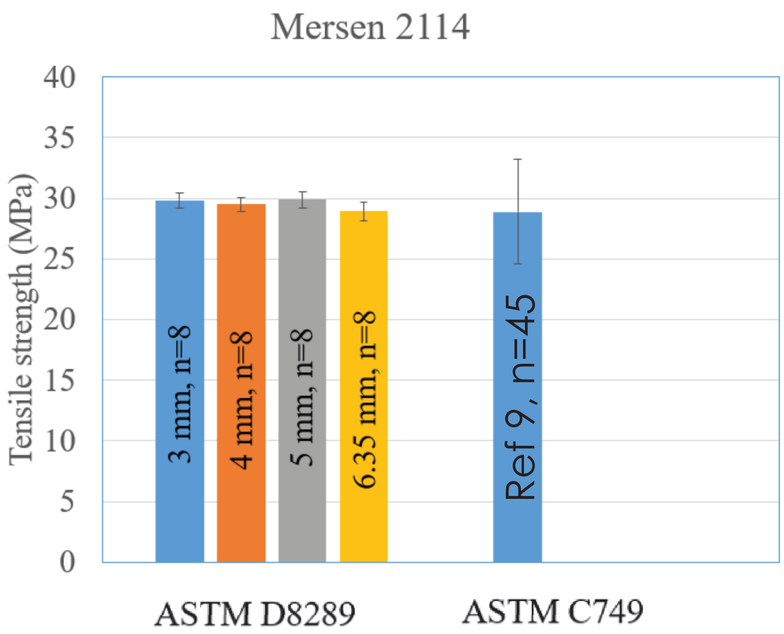


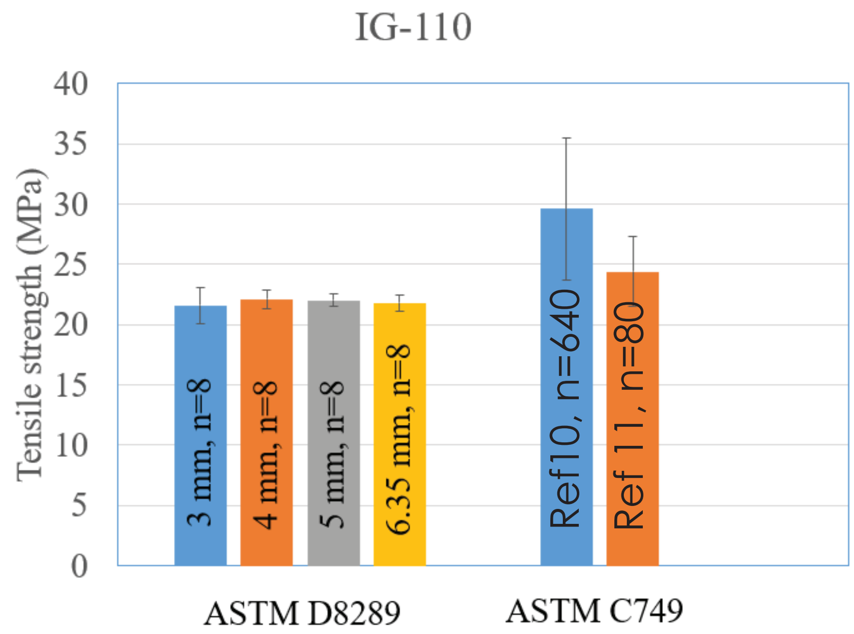
Sample thickness and tensile strength





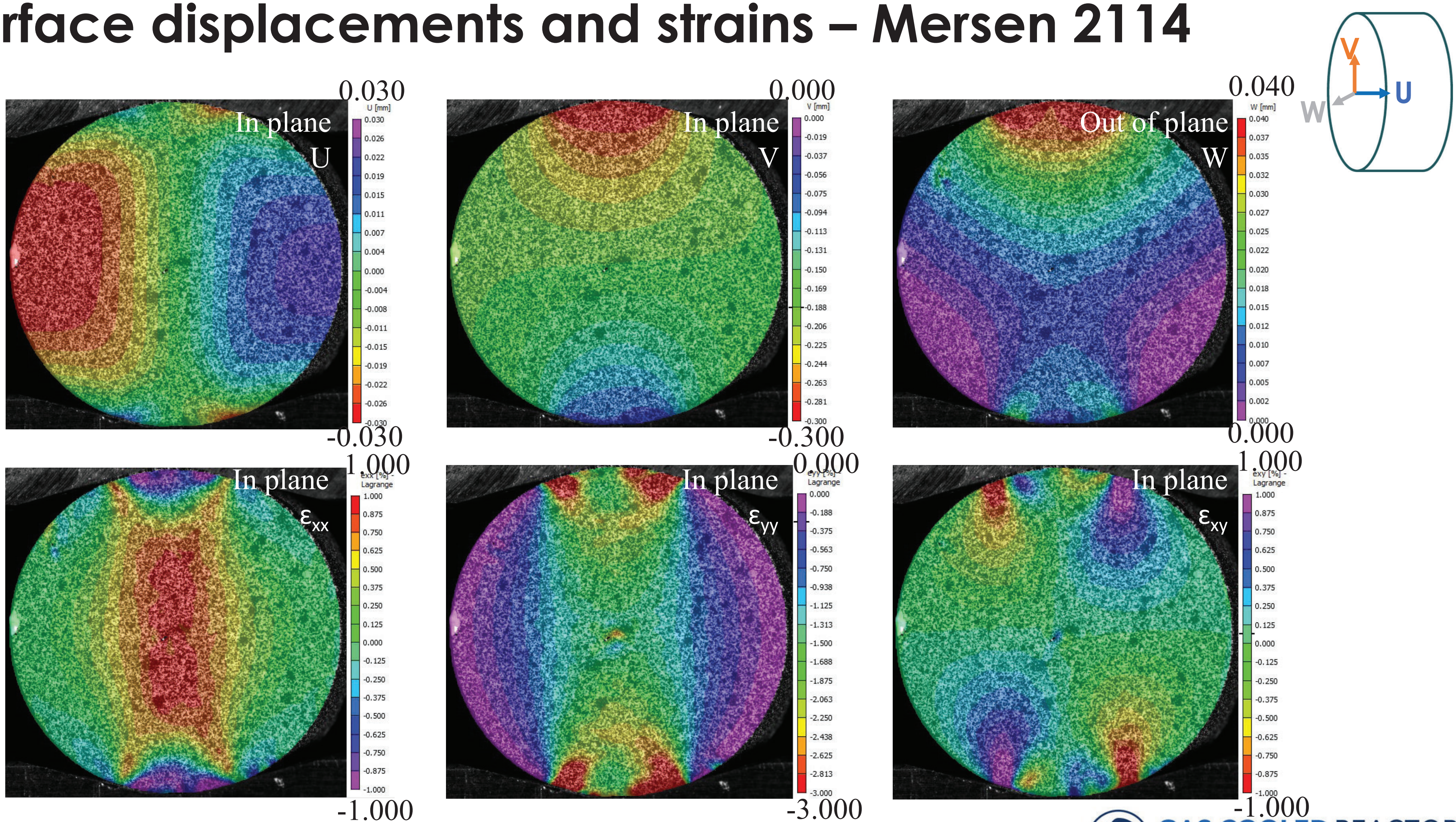
Comparison with uniaxial tensile strength



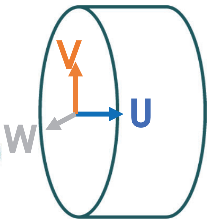


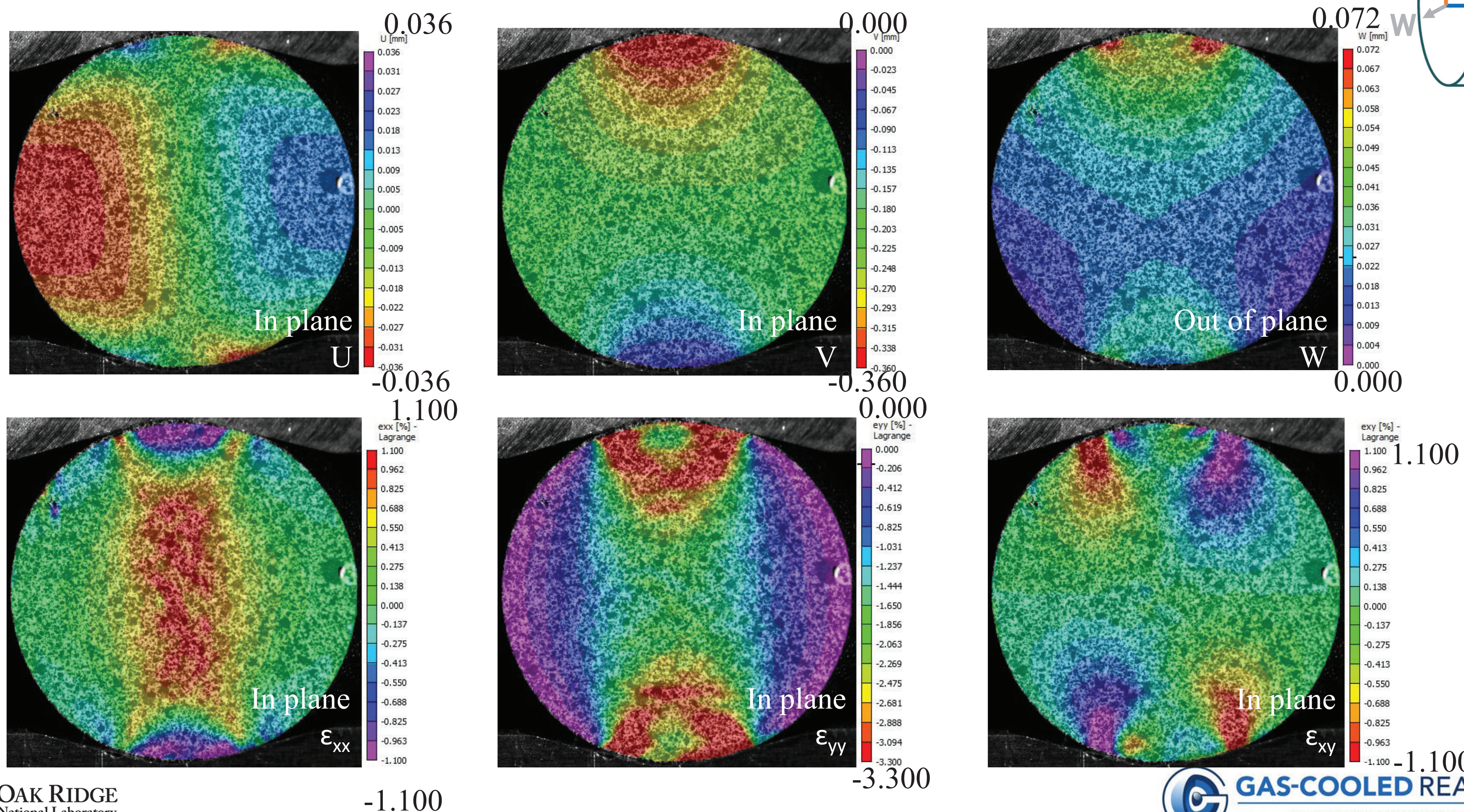
9. T. D. Burchell, Mersen Grade 2114: Tensile Strength from Brazilian Disc Testing, ORNL/TM-2020/1485, March 2020.
10. Sumita J, Shibata T, Iyoku T, Sawa K, Hanawa S, Ishihara M. Characteristics of first loaded ig-110 graphite in httr core. Japan Atomic Energy Agency; 2006.
11. Ishihara M, Mogi H, Ioka I, Arai T, Oku T. Statistical considerations of graphite strength for assessing design allowable stresses. 1987.

Surface displacements and strains – Mersen 2114



Surface displacements and strains – IG-110





Conclusions

- A small increase in splitting tensile strength when the disc thickness decreased in both Mersen 2114 (2~3%) and IG-110 (~1%) samples. No significant effect of thickness on splitting tensile strength was observed for samples with thickness smaller than half diameter.
- The 3 mm thick IG-110 samples showed a reverse trend and a larger standard deviation in splitting tensile strength, indicating that these discs were too thin to obtain a statistically high confidence result compared with the other thicknesses.
- The splitting tensile strengths of Mersen 2114 samples agreed with uniaxial tensile strength tested from ASTM C749, while the IG-110 samples had lower splitting tensile strengths than the referenced uniaxial tensile test values.
- The DIC results confirmed the high tensile strain region around the disc center that eventually led to the major vertical center crack. Significant out-of-plane displacement and nonlinearity were measured at disc top and bottom compared with the disc center.

Acknowledgement

- This research was sponsored by the US Department of Energy, Office of Nuclear Energy's Advanced Reactor Technologies Program.

Lianshan Lin
linl@ornl.gov/1-865-241-4531