July 26, 2023

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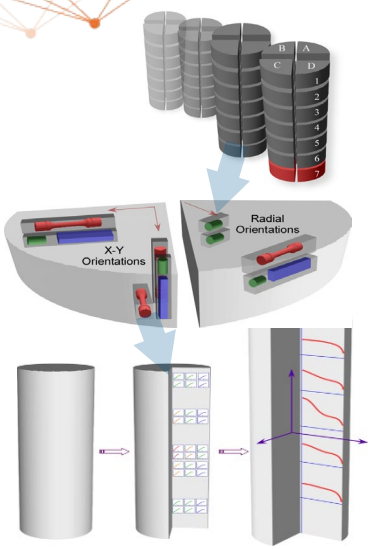
Baseline Graphite Characterization

DOE ART Gas-Cooled Reactor (GCR) Review Meeting
Virtual Meeting
July 25 – 27, 2023

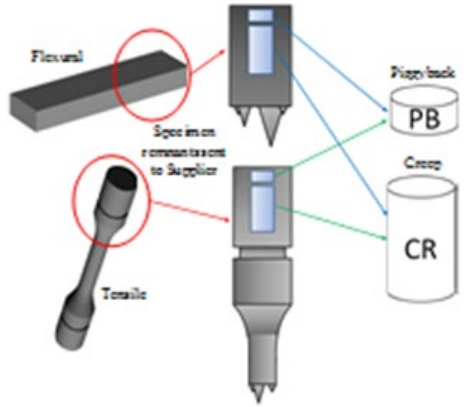
ART
Advanced Reactor Technologies

Baseline Graphite Characterization Purpose and Results



Establish the physical and mechanical properties of nuclear grade graphite and their variability…

- Intra Billet
- Billet to Billet
- Batch to Batch
- Grade to Grade



ASME BPVC code development

- Largest database of unirradiated material properties
- Design, Grade Qualification, and structural material code rules

Development of measurement techniques, standards and design code

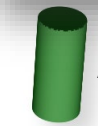
- Split disc tensile strength
- ASME BPVC.III.5
- ASTM D02.F0

Baseline of un-irradiated properties for comparison to AGC irradiated properties

- Statistically valid
- Scalar value
- Distribution

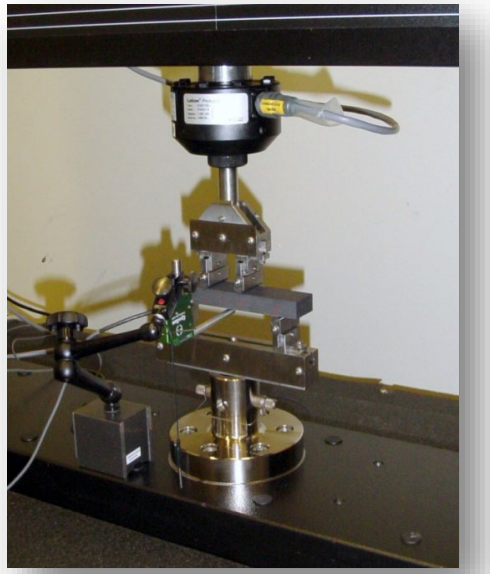
Baseline Property Measurements

Compressive Strength



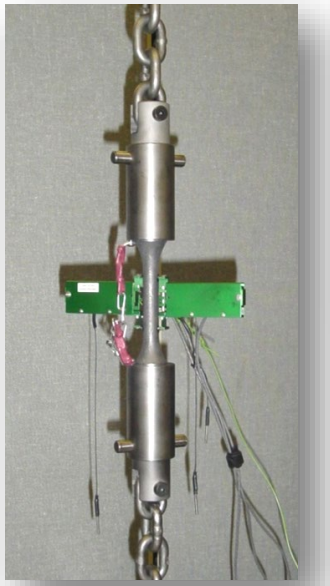
ASTM C695

Flexural Strength



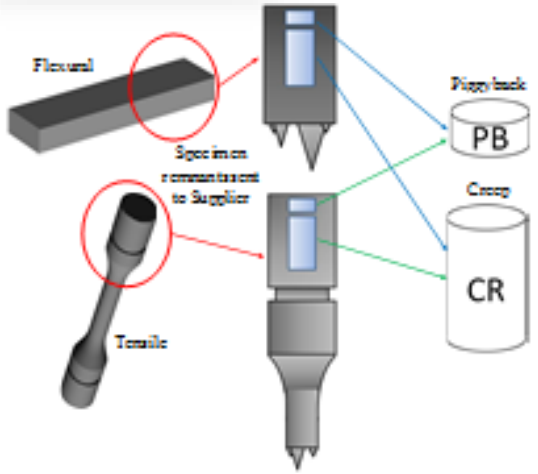
ASTM C651

Tensile Strength



ASTM C749

Brazilian Disc



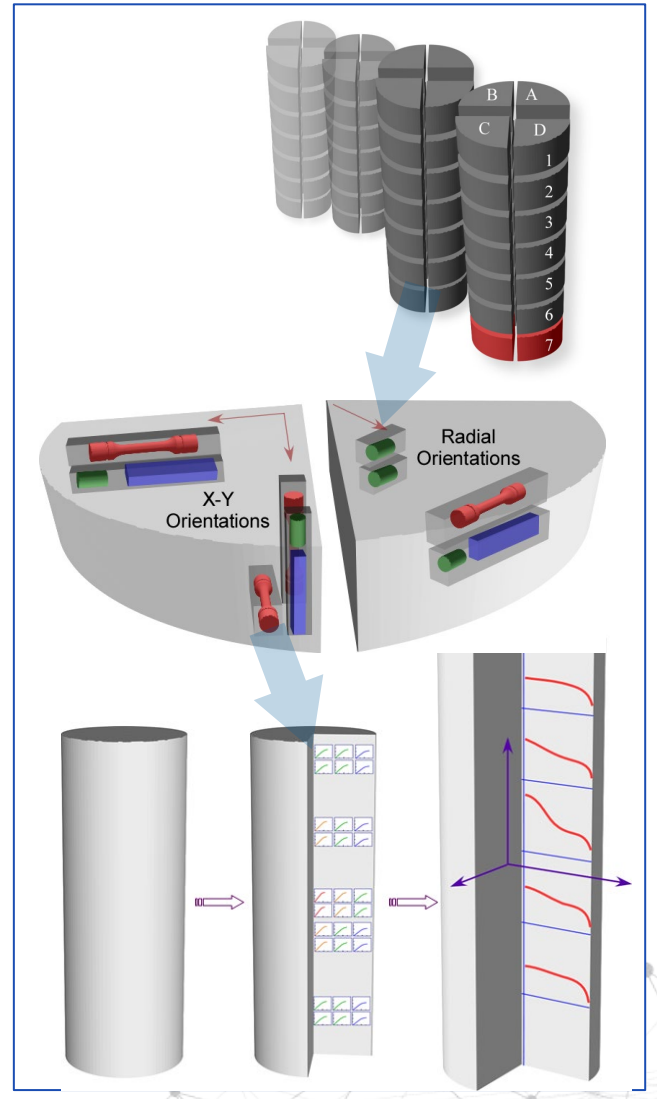
Physical Properties Testing

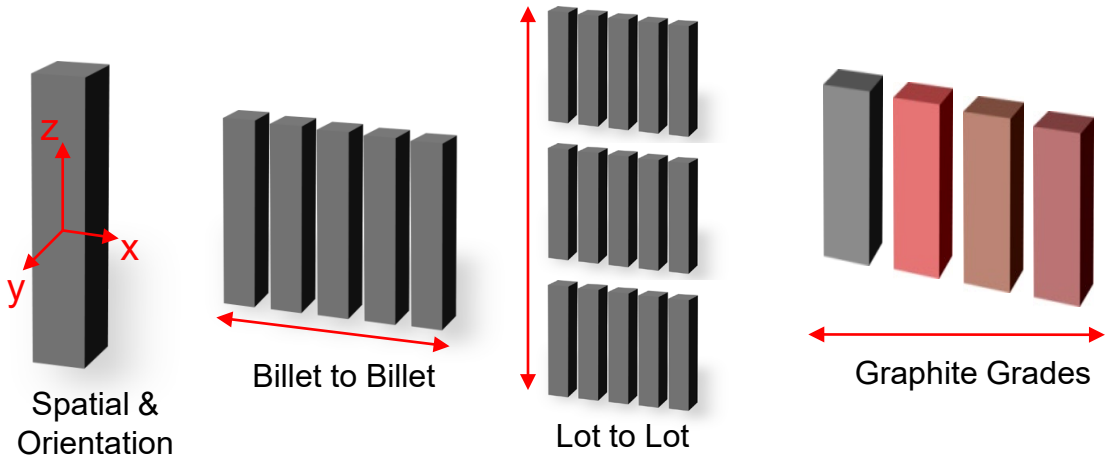
- Density
- Coefficient of Thermal Expansion
- Thermal Diffusivity
- Electrical Resistivity
- Elastic Modulus
  - Young's
  - Shear

Baseline Graphite Characterization Method

- Select necessary material properties
- Apply sampling plan
- Perform standardized testing
- Evaluate/compare properties
- Build NQA-1 qualified database
- Apply the “system” and database to the evaluation and qualification of future grades of graphite

- **Current Grades**
  - NBG-18
  - PCEA
  - IG-110
  - 2114
  - NBG-17
- **Additional Grades**
  - IG-430





Baseline Progress

- Billet XPC01D-35, of PCEA is currently being machined
- The second billet of NBG-17 is complete in machining and a third the way done in testing
- Over 23,800 NQA-1 qualified measurements thus far
- PCEA Grade
  - 1st batch (XPC01S8)
  - 2nd batch (XPC02S8)

  } Low density

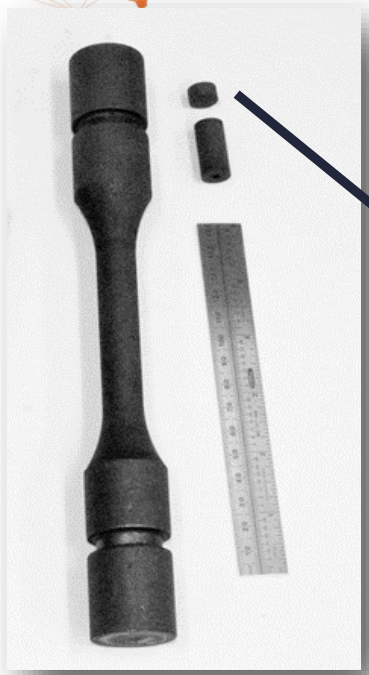
  - 3rd batch (XPC01D3)

  } High density

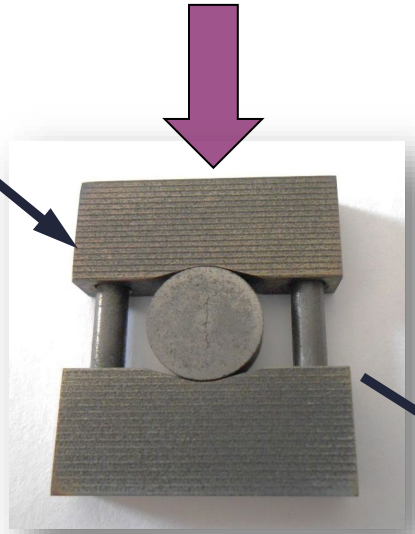
High importance to ASME code development due to high variably

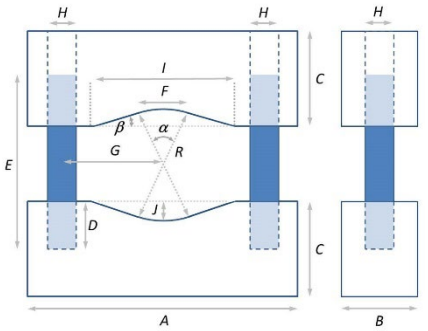
Graphite	Laboratory	Billet #	Percent Complete					Data Report	Analysis Reports	Data In NDMAS?	Notes
			Machining	Mass and Density	Elastic Testing	Mechanical Testing	Thermal Testing				
PCEA	ORNL	XPC01S8-11	100%	100%	100%	100%	100%	ORNL/TM-2015/765	ORNL/TM-2015/765	NO	Data spreadsheets were requested from ORNL
PCEA	INL	XPC02S8-7	100%	100%	100%	100%	100%	ECAR-3725	INL/EXT-13-30011	YES	
PCEA	INL	XPC01S8-9	100%	100%	100%	100%	100%	ECAR-6111	INL/MIS-23-70949	NO	
PCEA	INL	XPC02S8-5	100%	100%	100%	100%	100%	ECAR-6110	INL/MIS-23-70951	NO	
PCEA	INL	XPC01D3-35	66%							NO	
PCEA	INL	XPC01D3-36	100%	100%	100%	100%	100%	ECAR-3677	INL/EXT-16-39604	YES	
PCEA		Multiple Other Billets Available									
NBG-18	INL	635-4	100%	100%	100%	100%	100%	ECAR-3726	INL/EXT-14-33120, INL/EXT-13-30011	YES	
NBG-18	INL	635-14	100%	100%	100%	100%	100%	ECAR-1930	INL/EXT-10-19910, INL/EXT-13-30011	YES	
NBG-18	ORNL	635-6	100%	100%	100%	100%	100%	ORNL/TM-2010/219	ORNL/TM-2010/219	NO	Data spreadsheets were requested from ORNL
NBG-18		Multiple Other Billets Available									
2114	INL	A20568	100%	100%	100%	100%	100%	ECAR-5798	INL/MIS-22-65680	NO	
2114	INL	A20570	100%	100%	100%	100%	100%	ECAR-4322	INL/EXT-14-33120	YES	
2114	ORNL	116310	100%	100%	100%	100%	100%	2018/1038, 2019/1256	ORNL/TM-2018/1038, ORNL/TM-2019/1256	YES*	Data spreadsheets were requested from ORNL
2114		Multiple Other Billets Available									
NBG-17	INL	830-3	100%	100%	100%	100%	100%	ECAR-3727	INL/EXT-14-33120	YES	
NBG-17	INL	V104	100%			33%				NO	
IG-110	INL	089052-7	100%	100%	100%	100%	100%	ECAR-3621	INL/EXT-14-33120	YES	
IG-110	INL	10X69	100%	100%	100%	100%	100%	ECAR-4182	ECAR-4182	NO	

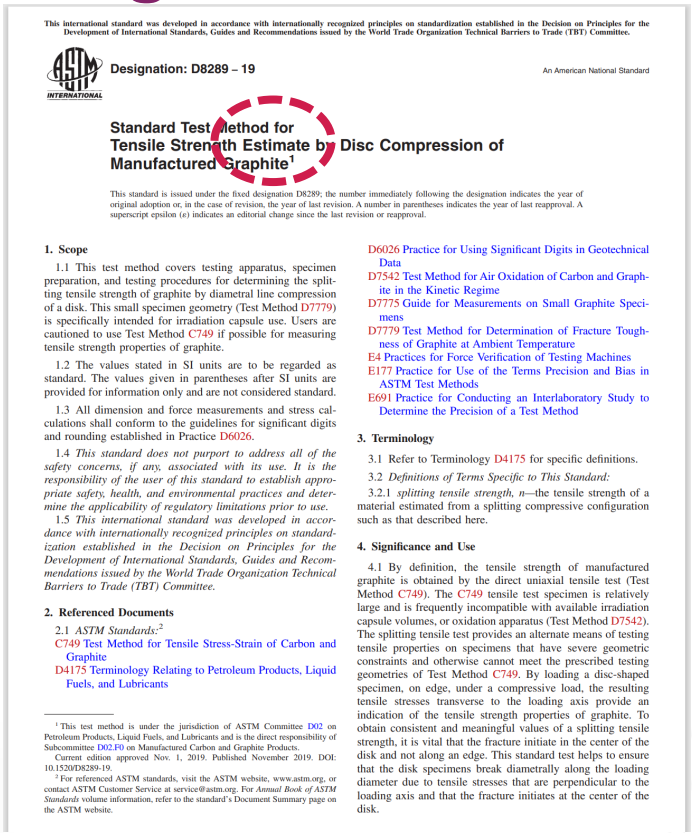
Split Disc Tensile Strength: ASTM Tensile Strength Alternative



First miniaturized test standard for graphite







This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D8289 – 19

An American National Standard

Standard Test Method for Tensile Strength Estimate by Disc Compression of Manufactured Graphite[1]

This standard is issued under the fixed designation D8289; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers testing apparatus, specimen preparation, and testing procedures for determining the splitting tensile strength of graphite by diametral line compression of a disk. This small specimen geometry (Test Method D7779) is specifically intended for irradiation capsule use. Users are cautioned to use Test Method C749 if possible for measuring tensile strength properties of graphite.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 All dimension and force measurements and stress calculations shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C749 Test Method for Tensile Stress-Strain of Carbon and Graphite

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D6026 Practice for Using Significant Digits in Geotechnical Data

D7542 Test Method for Air Oxidation of Carbon and Graphite in the Kinetic Regime

D7775 Guide for Measurements on Small Graphite Specimens

D7779 Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature

E4 Practices for Force Verification of Testing Machines

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Refer to Terminology D4175 for specific definitions.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *splitting tensile strength, n*—the tensile strength of a material estimated from a splitting compressive configuration such as that described here.

4. Significance and Use

4.1 By definition, the tensile strength of manufactured graphite is obtained by the direct uniaxial tensile test (Test Method C749). The C749 tensile test specimen is relatively large and is frequently incompatible with available irradiation capsule volumes, or oxidation apparatus (Test Method D7542). The splitting tensile test provides an alternate means of testing tensile properties on specimens that have severe geometric constraints and otherwise cannot meet the prescribed testing geometries of Test Method C749. By loading a disc-shaped specimen, on edge, under a compressive load, the resulting tensile stresses transverse to the loading axis provide an indication of the tensile strength properties of graphite. To obtain consistent and meaningful values of a splitting tensile strength, it is vital that the fracture initiate in the center of the disk and not along an edge. This standard test helps to ensure that the disk specimens break diametrally along the loading diameter due to tensile stresses that are perpendicular to the loading axis and that the fracture initiates at the center of the disk.

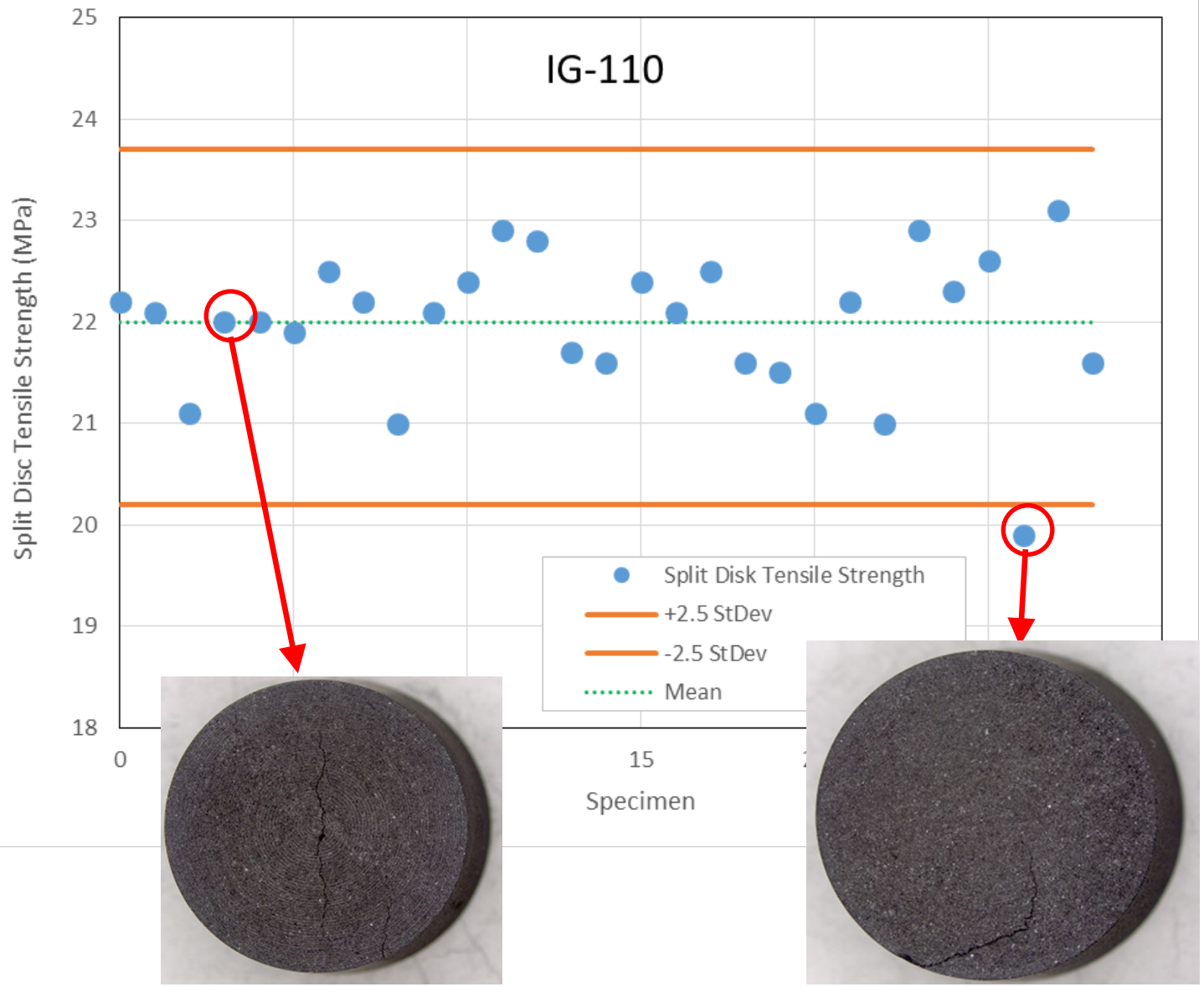
[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.
Current edition approved Nov. 1, 2019. Published November 2019. DOI: 10.1520/D8289-19.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

Compressive load is applied to a disc-shaped specimen on edge, resulting in tensile stress transverse to the loading axis. The load at failure, P, and geometry of the specimen provide an indication of the tensile strength.

$$\sigma_{sts} \approx \frac{P}{\pi LR}\left[1-\left(\frac{b}{R}\right)^2\right]$$

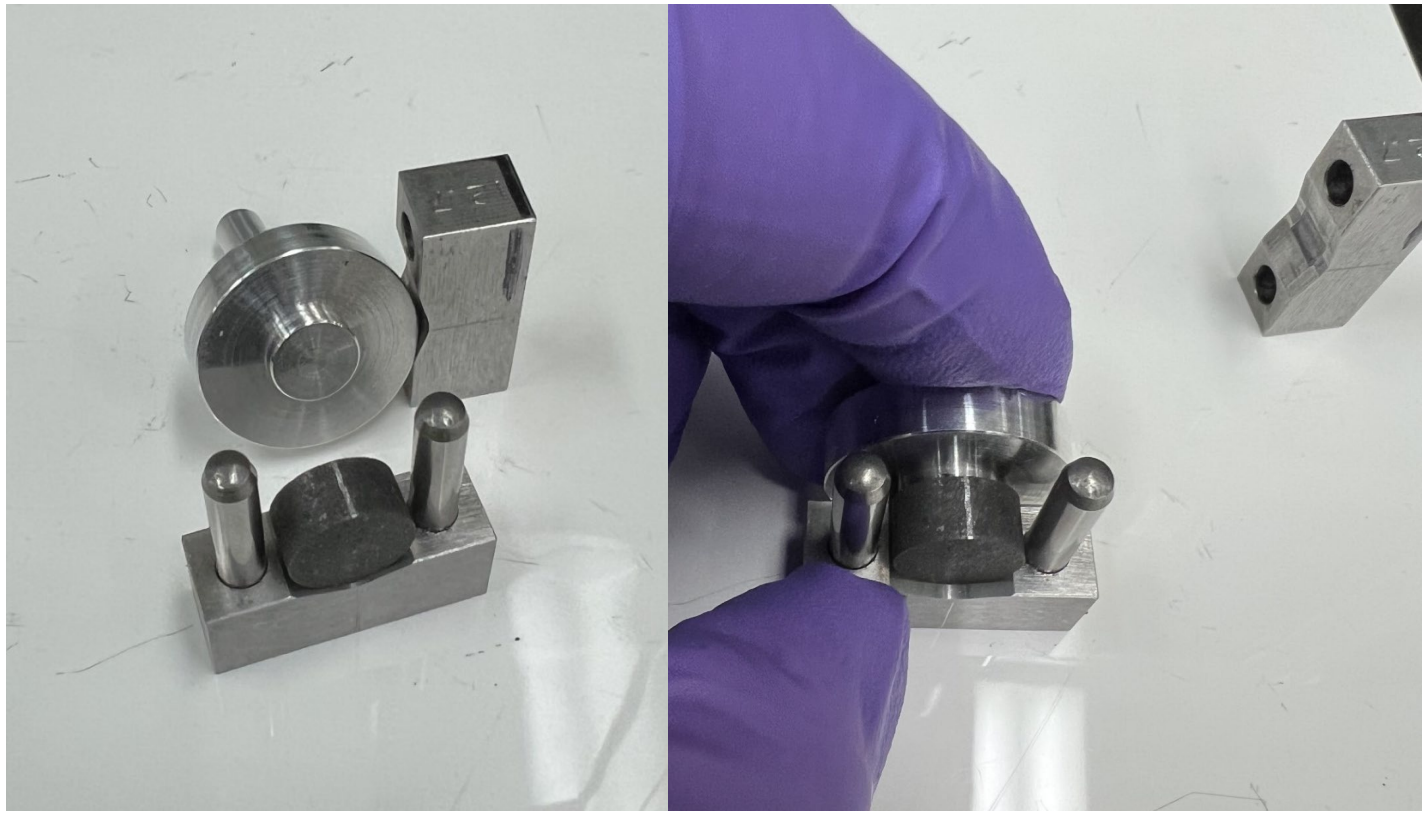
Identification of Proper Split Disk Fracture



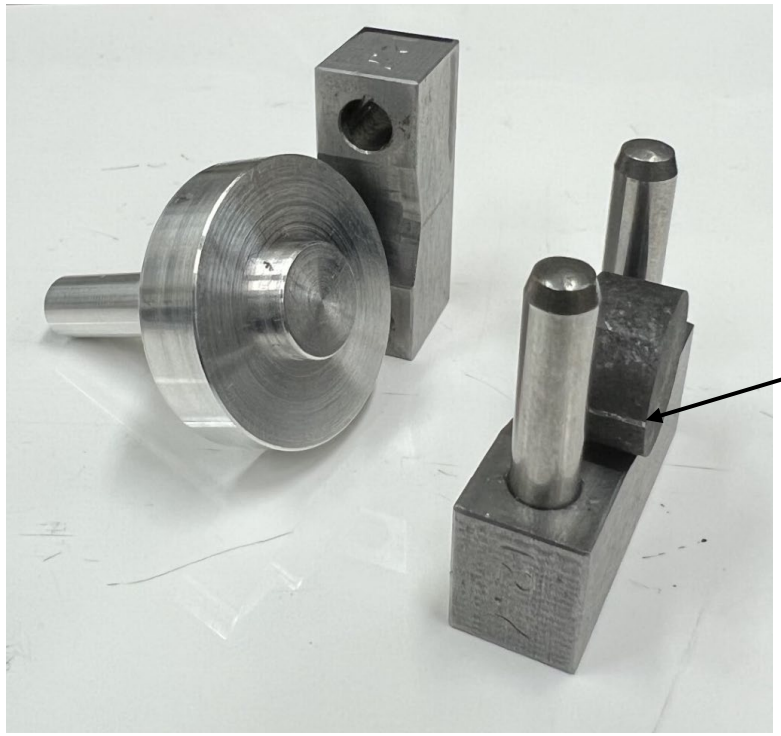
- This measurement technique originated in rock and concrete where the ratio of compressive to tensile strength is ~10. (Graphite ~3-4).
- Calculation of tensile stress in a compressed disc requires the fracture to initiate from the center of the disc.
- This occurs when the compressive strength is much higher than the tensile strength.
- **Proper crack/fracture initiation is easily identifiable.**

Baseline Graphite ASTM D8289

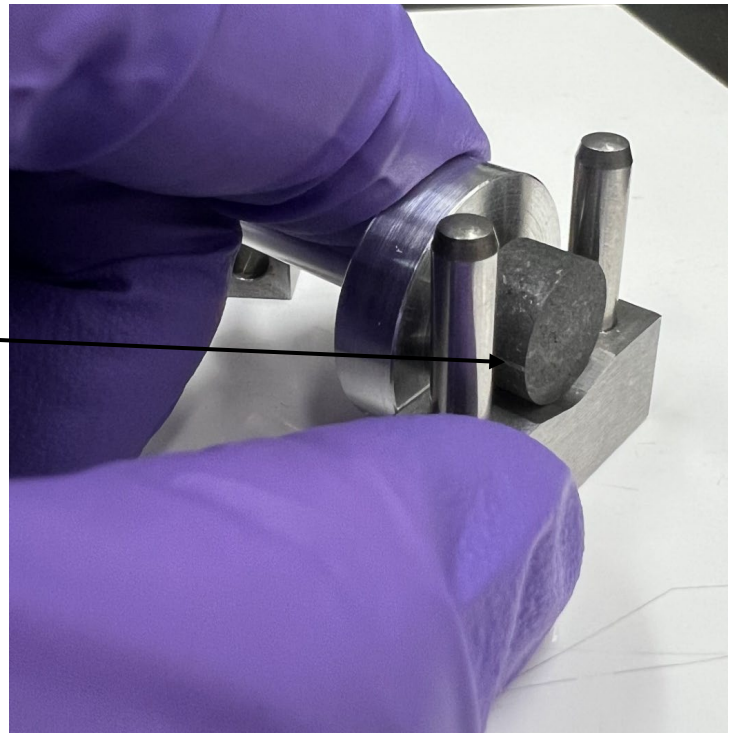
- Loading samples for split disc test ASTM D8289-19
- The sample is loaded in the vertical orientation as to the grain direction
- Samples can be positioned With Grain (WG) or Against Grain (AG)
- The samples are shown without speckle pattern for clarity



Horizontal Orientation

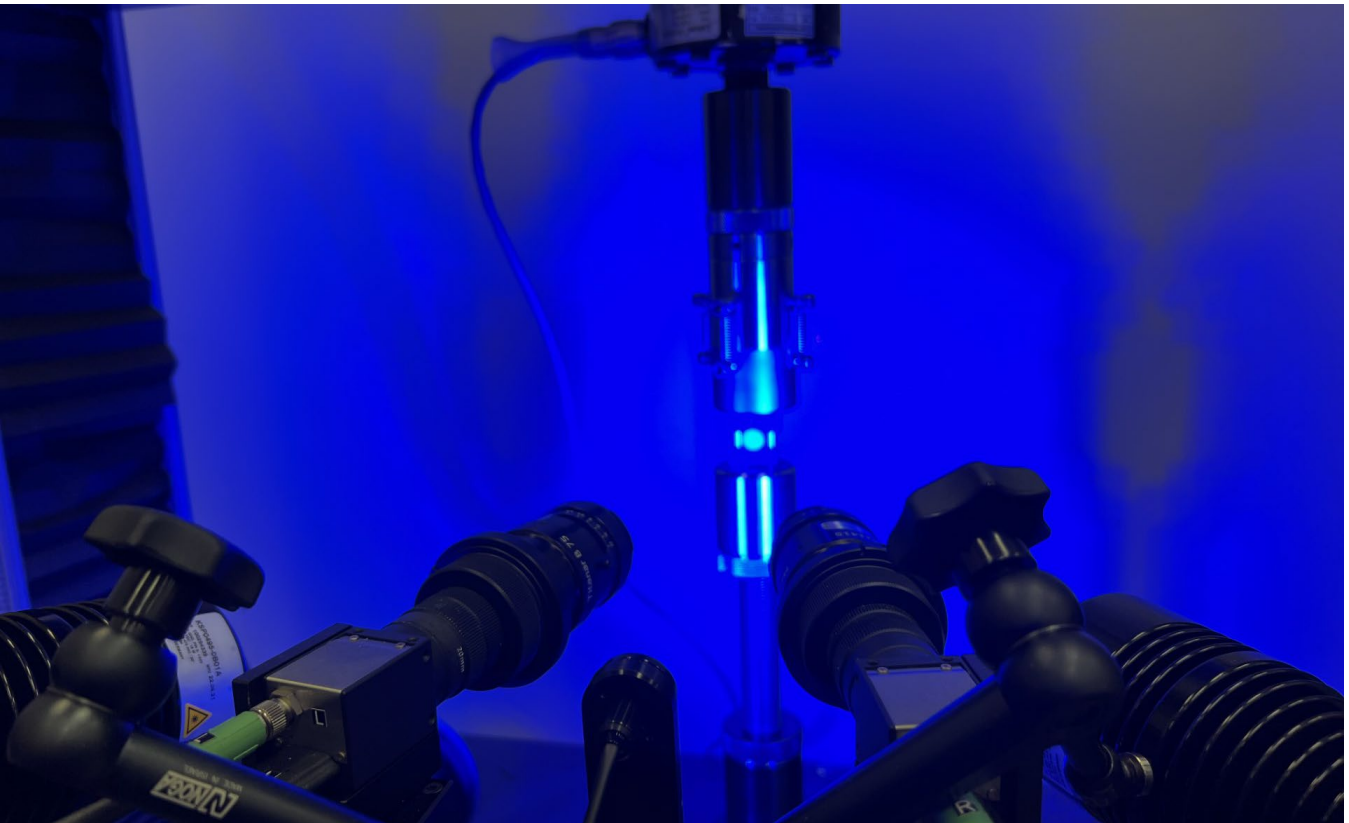


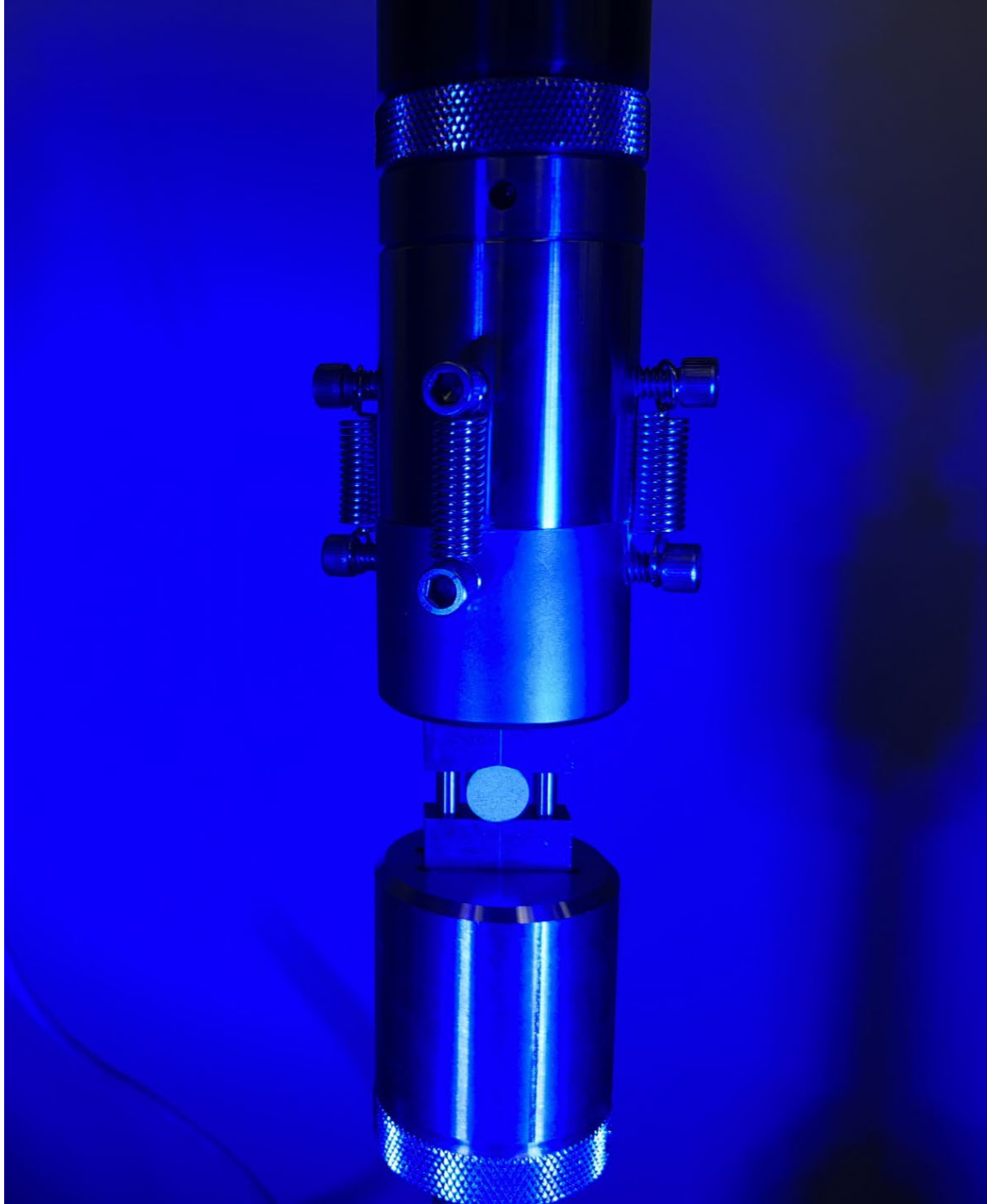
Fiducial mark



Digital Image Correlation

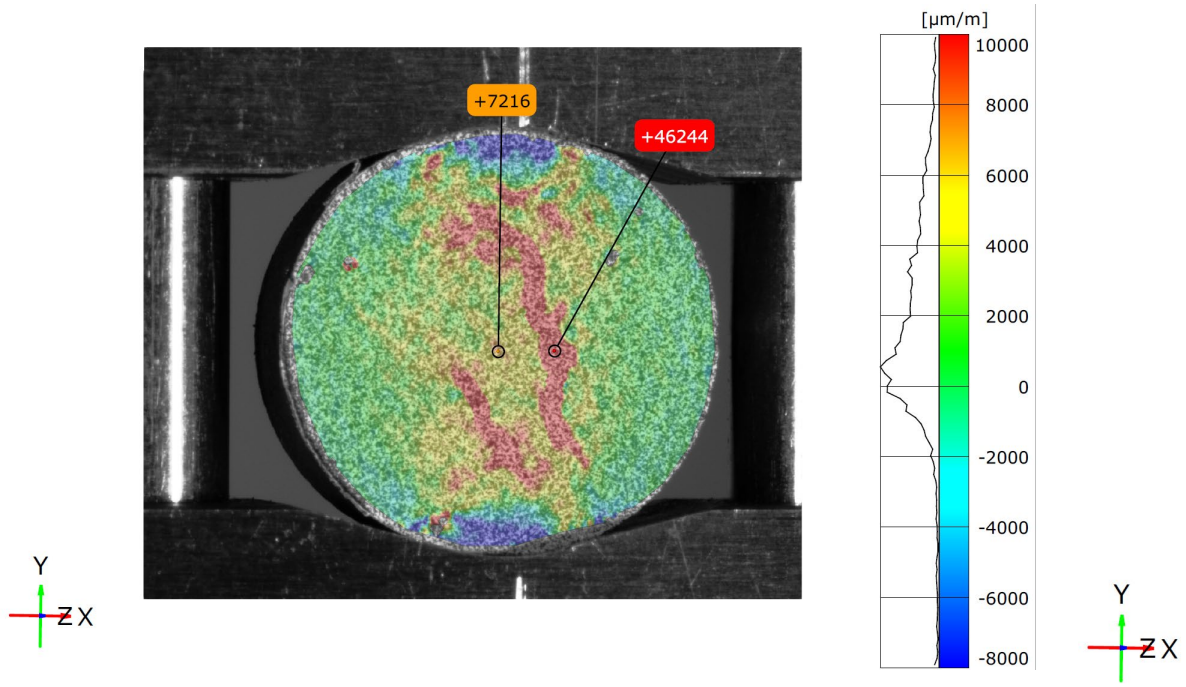
- Use of DIC to corroborate current baseline data



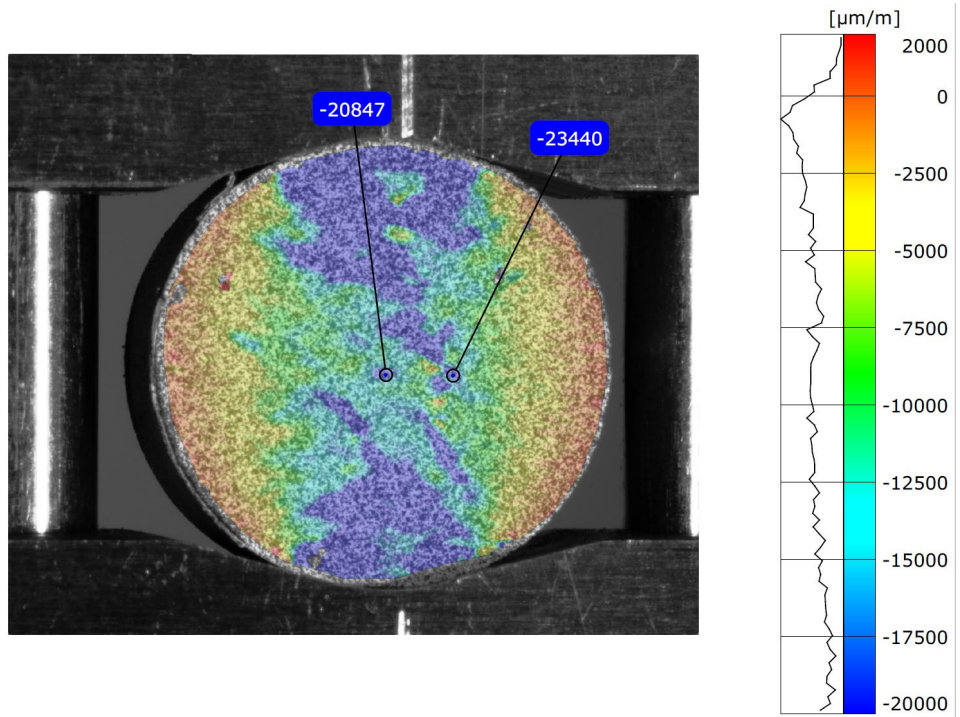


DIC Analyzation

ε_{xx} Point Analysis



ε_{yy} Point Analysis



Additional considerations

- Because it uses ASTM approved testing standards, Baseline data is invaluable for:
  - Development of accurate **new** test standards handling degraded material
    - ***Strength after oxidation, molten salt mechanical strength effects, irradiated strength/modulus***
  - ASME code rules,
  - As well as quantification of effects from degradation: irradiation, oxidation, molten salt, etc.
- Developing new High Temperature ASTM test standards
  - Room temperature data will provide a “Baseline” for high temperature testing results
- Split Disc results will be primary method for determining irradiation strength
  - We must perfect the test method **to obtain real tensile strength** – not estimated strength
- New areas being considered for addition to Baseline data
  - High temperature mechanical strength and modulus
  - Probability of failure calculations
  - Specimen locations within billet – to provide comparison with ASME qualification code rules
  - Unirradiated microstructure database (David Arregui-Mena)

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Thank You