



INL Carbon Characterization Lab

October 2022

Changing the World's Energy Future

Austin C Matthews



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**Idaho National Laboratory
Idaho Falls, Idaho 83415**

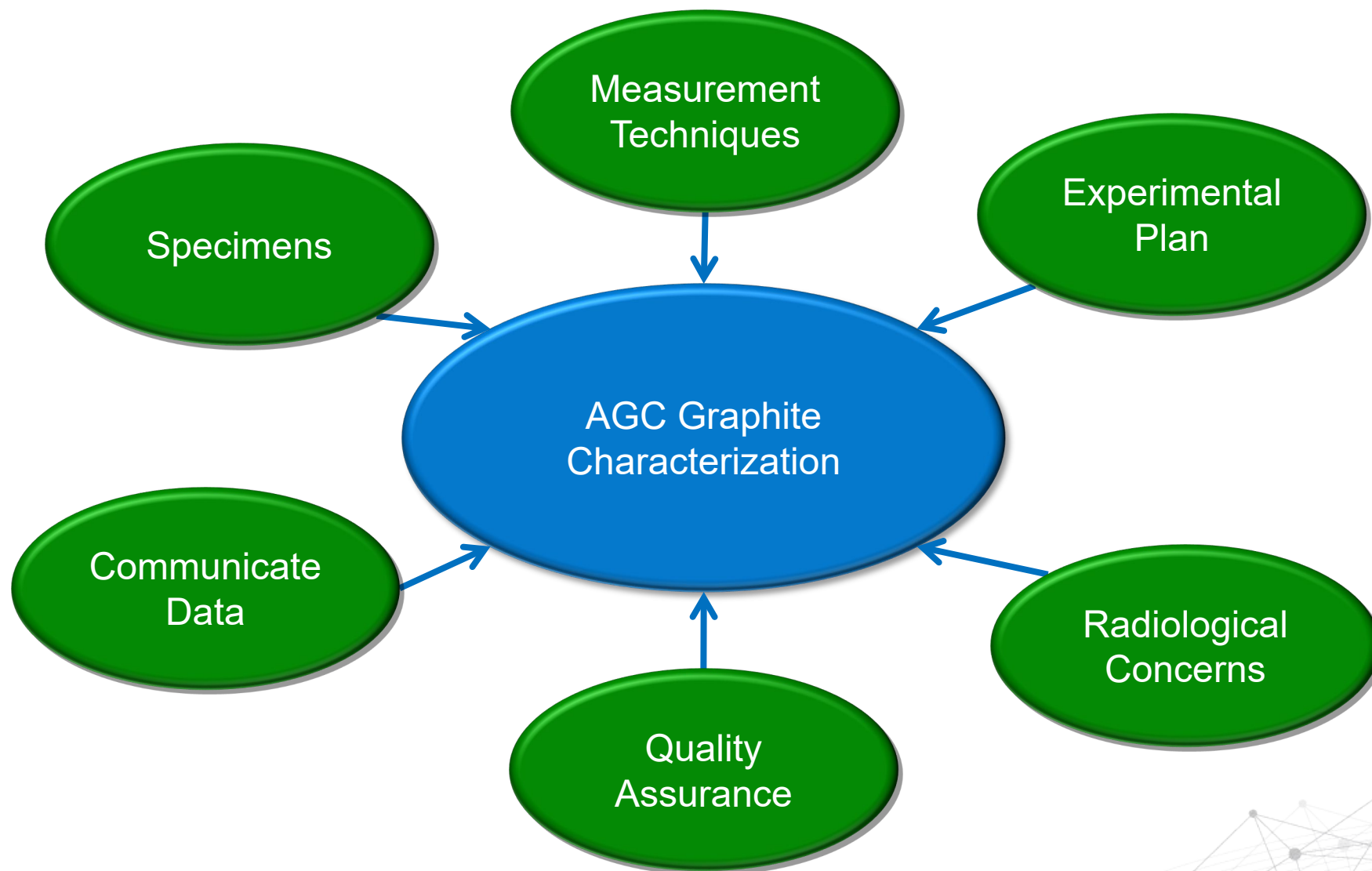
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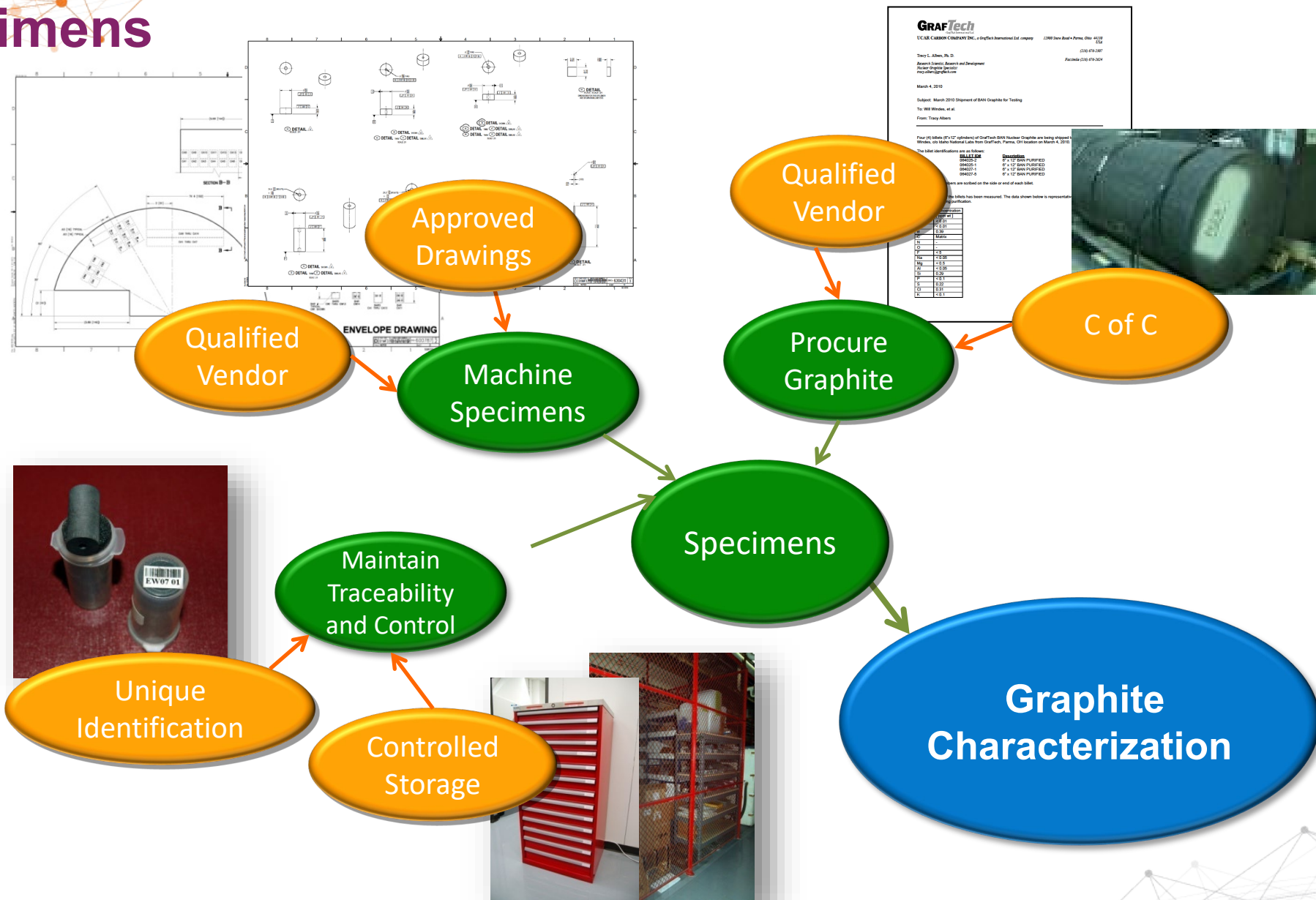
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What goes into AGC Graphite Characterization



Specimens



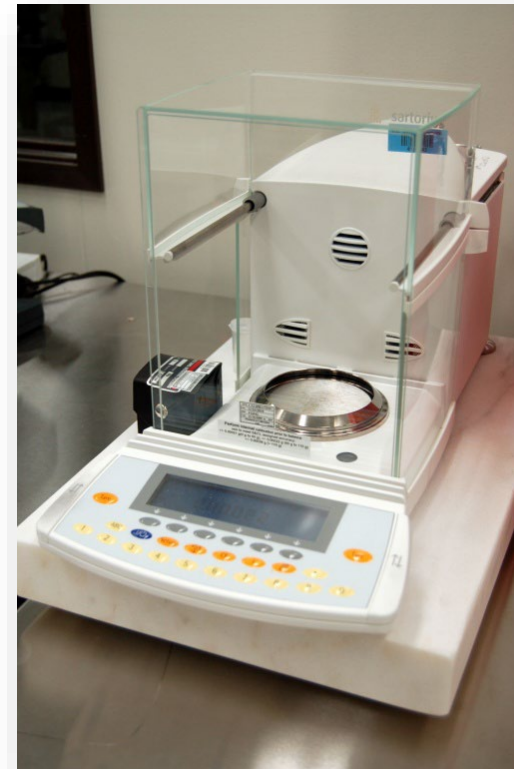
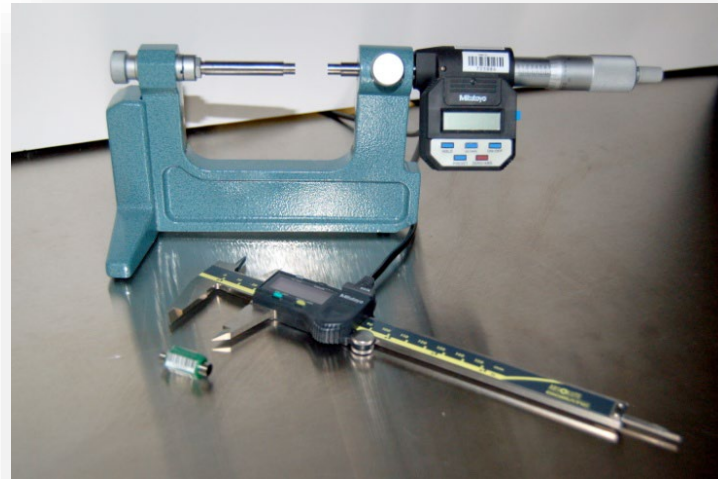
Measurement Techniques



- Bulk Density
- Modulus of Elasticity
- Electrical Resistivity
- Coefficient of Thermal Expansion
- Thermal Diffusivity

Mass and Dimensional Measurements

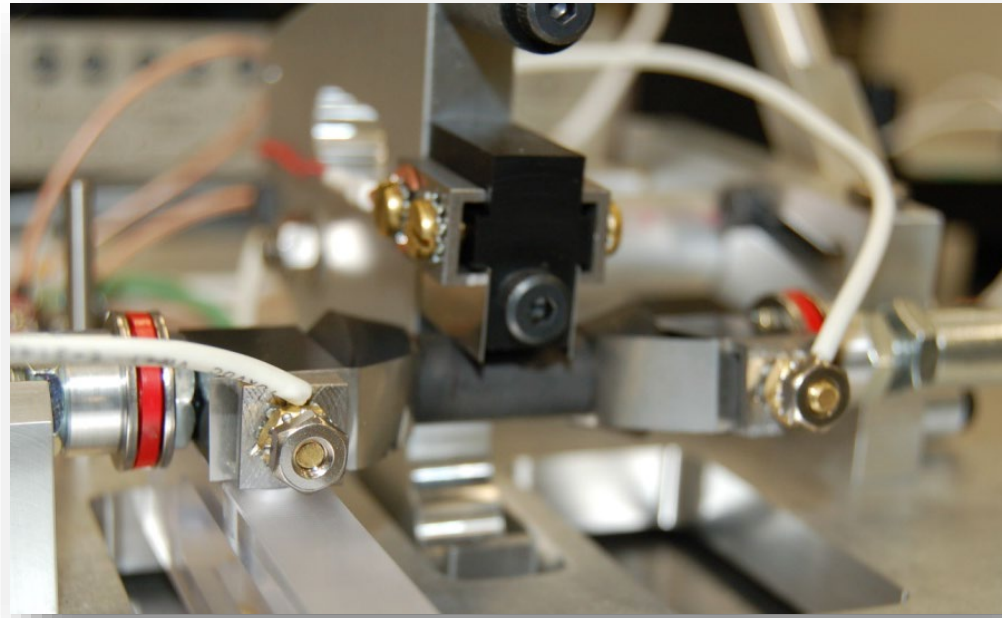
- Dimensional change is one of the key issues affecting the performance of graphite in a neutron environment.
 - Defines irradiation creep
- Mass and Dimensional measurements are utilized in the calculation of all other properties measured.



Electrical Resistivity

- Electrical resistivity is used as a rapid, simple means to determine grain orientation, structure, and crystallinity of the graphite
- 4 point probe technique
- Pass a known current through the sample while measuring the voltage across the sample
- From Ohms law the resistance is determined and the resistivity is calculated from,

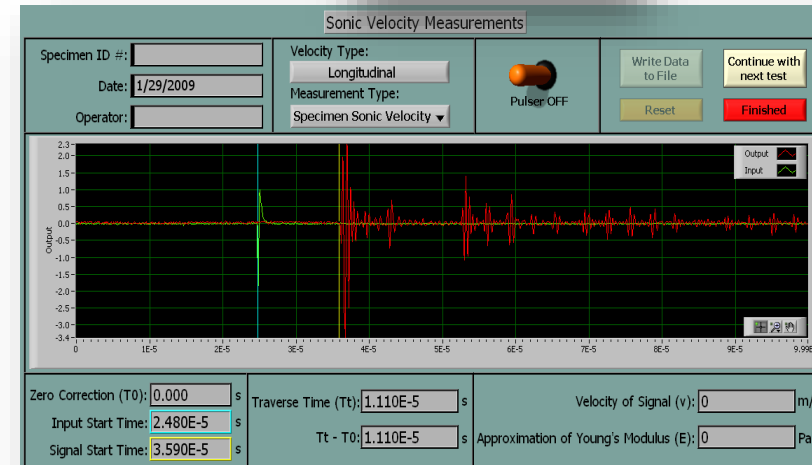
$$\rho = R \cdot A / L$$



Elastic Modulus from the Measurement of Sonic Velocity

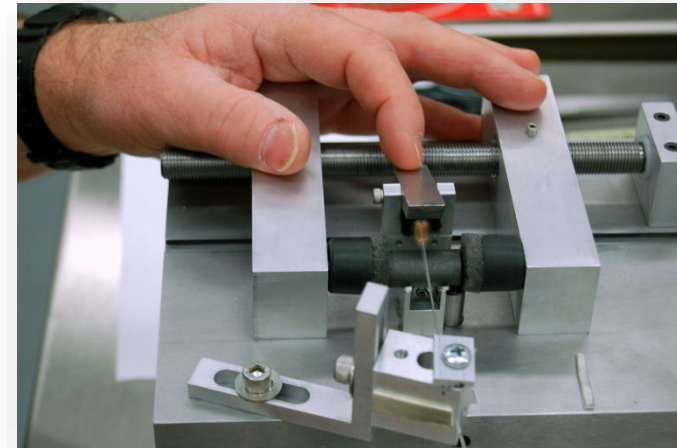
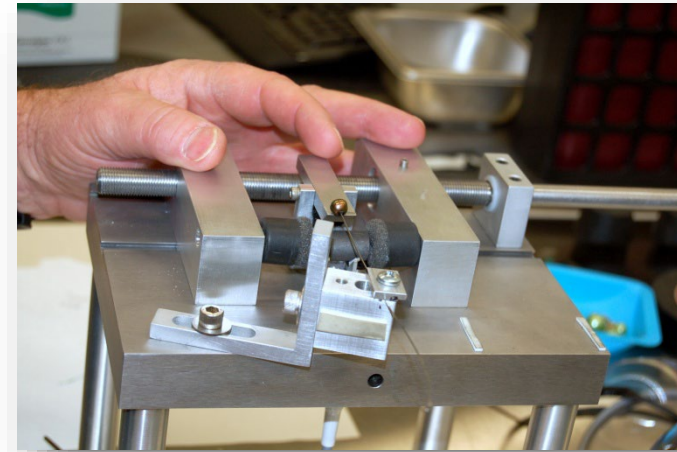
- Mechanical properties of graphite are needed to determine strength and integrity of the reactor core.
- The sonic velocity is determined from the ratio of the sample length to the time lapse of an acoustic wave between transducers at each end (time-of-flight)
- An approximate value for Young's Modulus, E can be obtained from,

$$E = C_v \rho V^2$$



Modulus of Elasticity by Impulse Excitation

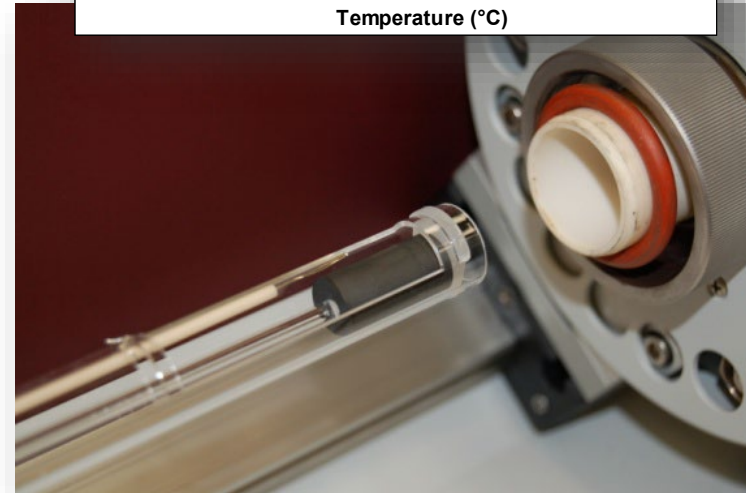
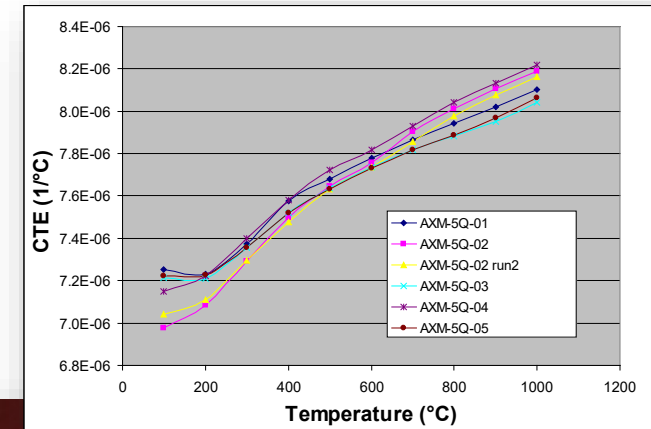
- The fundamental resonant frequency of a test specimen is measured by exciting it mechanically with a singular elastic strike.
- The measured fundamental resonant frequency, specimen dimensions and mass are used to calculate dynamic Young's modulus.



Coefficient of Thermal Expansion

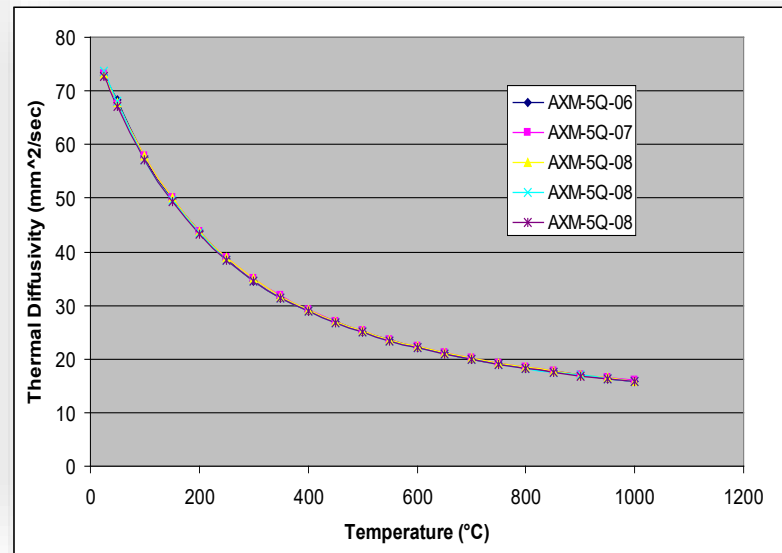
- Thermal dimensional changes
 - As a function of irradiation dose
 - Residual stresses within graphite components due to thermal gradients
- Push-rod dilatometry
 - Change in length as a function of temperature

$$\alpha = \frac{1}{L_0} \frac{\Delta L}{\Delta T}$$



Thermal Diffusivity

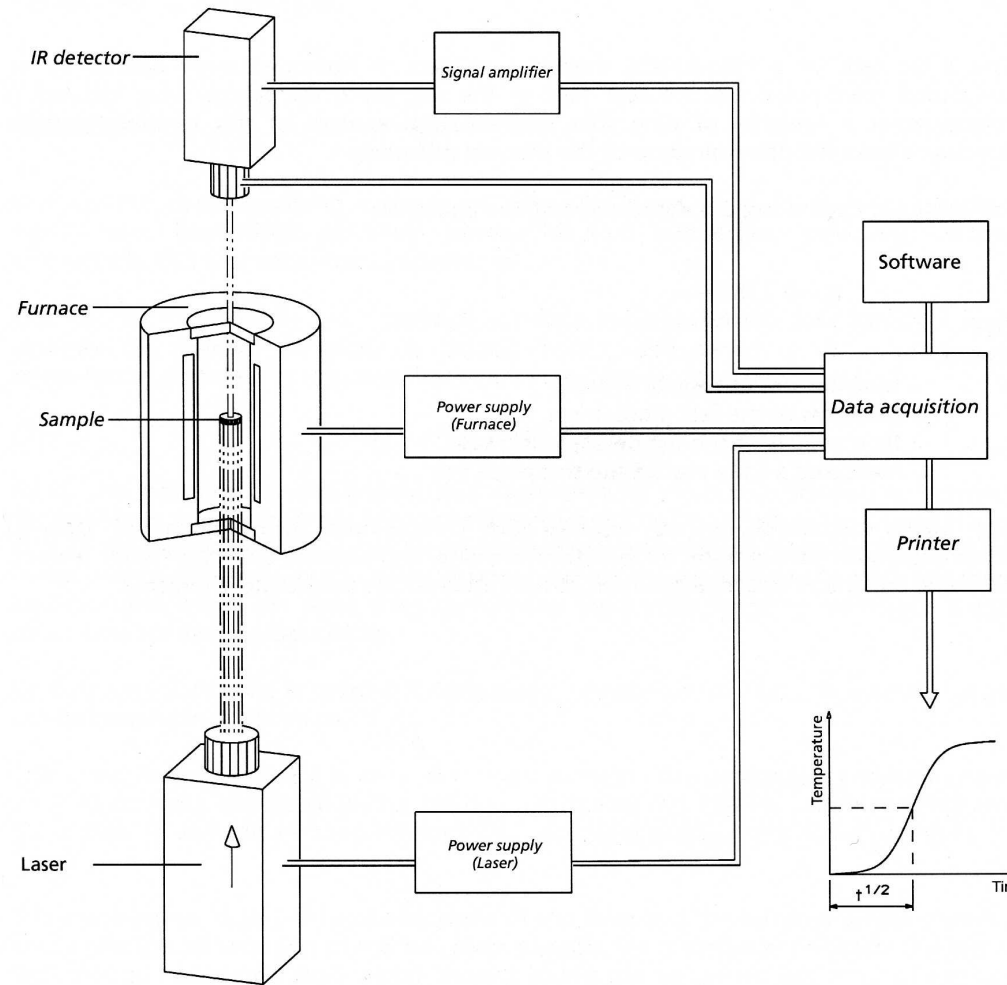
- Changes in Thermal conductivity
 - Extent of changes as a function of irradiation dose and temperature
 - Passive safety of system – gets the heat out
- Thermal conductivity is related to thermal diffusivity through the density and specific heat.



$$k = \alpha \cdot \rho \cdot C_P$$

Laser Flash Apparatus (LFA) Operation

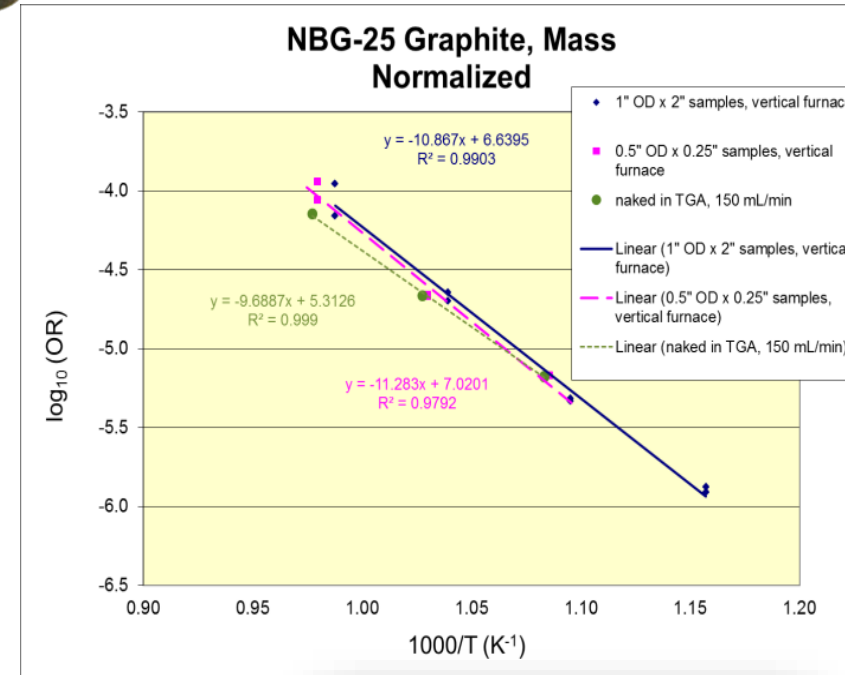
- Pulsed Nd-YAG laser radiates bottom side of specimen.
- Controlled atmosphere furnace 1200°C max.
- LN cooled InSb detector observes temperature rise of specimen top surface.
- Data acquisition and control computer.



Oxidation

Vertical Furnace

- Variations with specimen scale

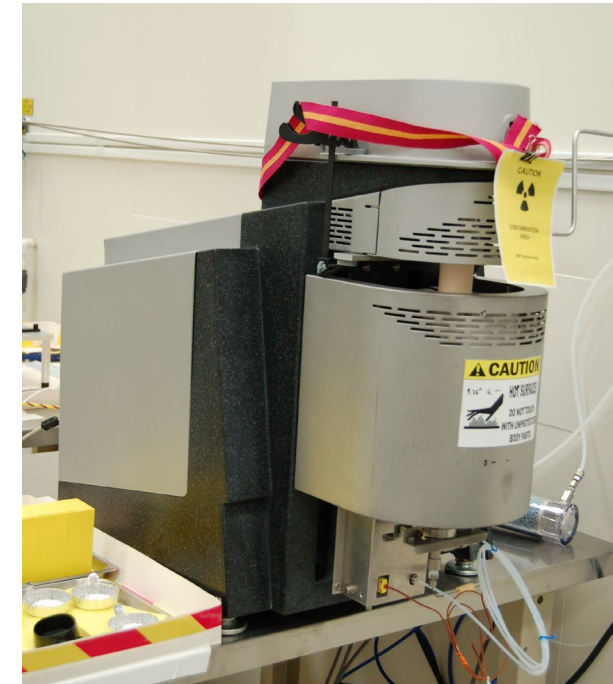


Compressive strength after oxidation



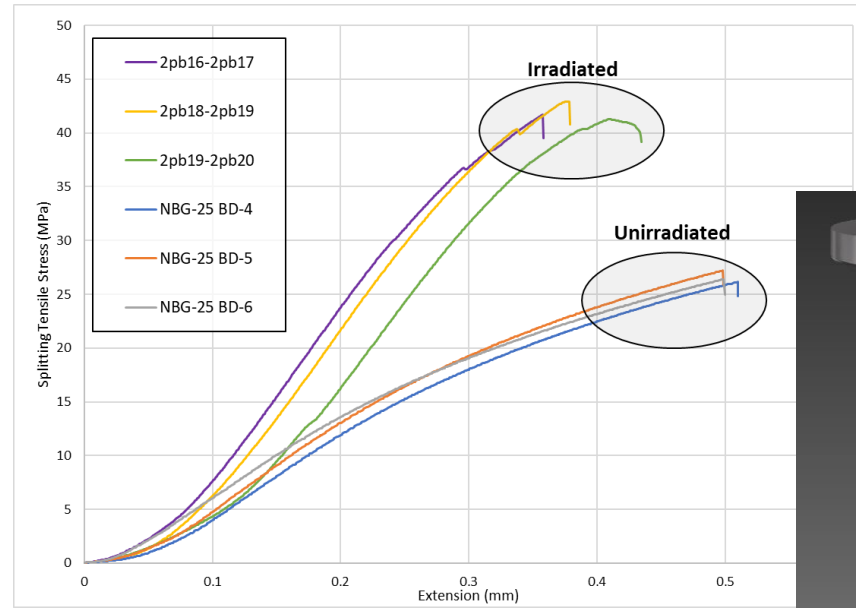
TGA

- Precision testing of small specimens



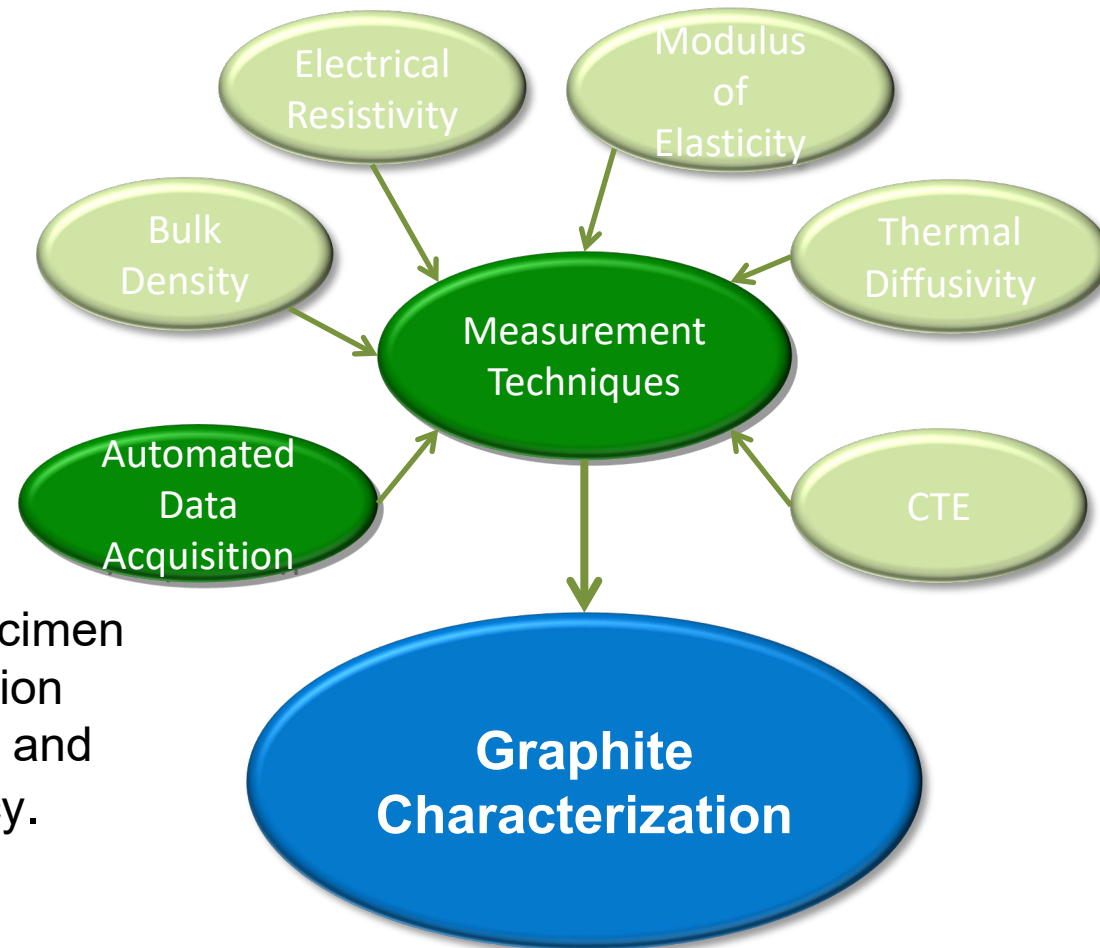
Mechanical Testing

- Tensile
- Flexure
- Compression
- Split Disc
 - DIC for irradiated samples
- All per ASTM Standards



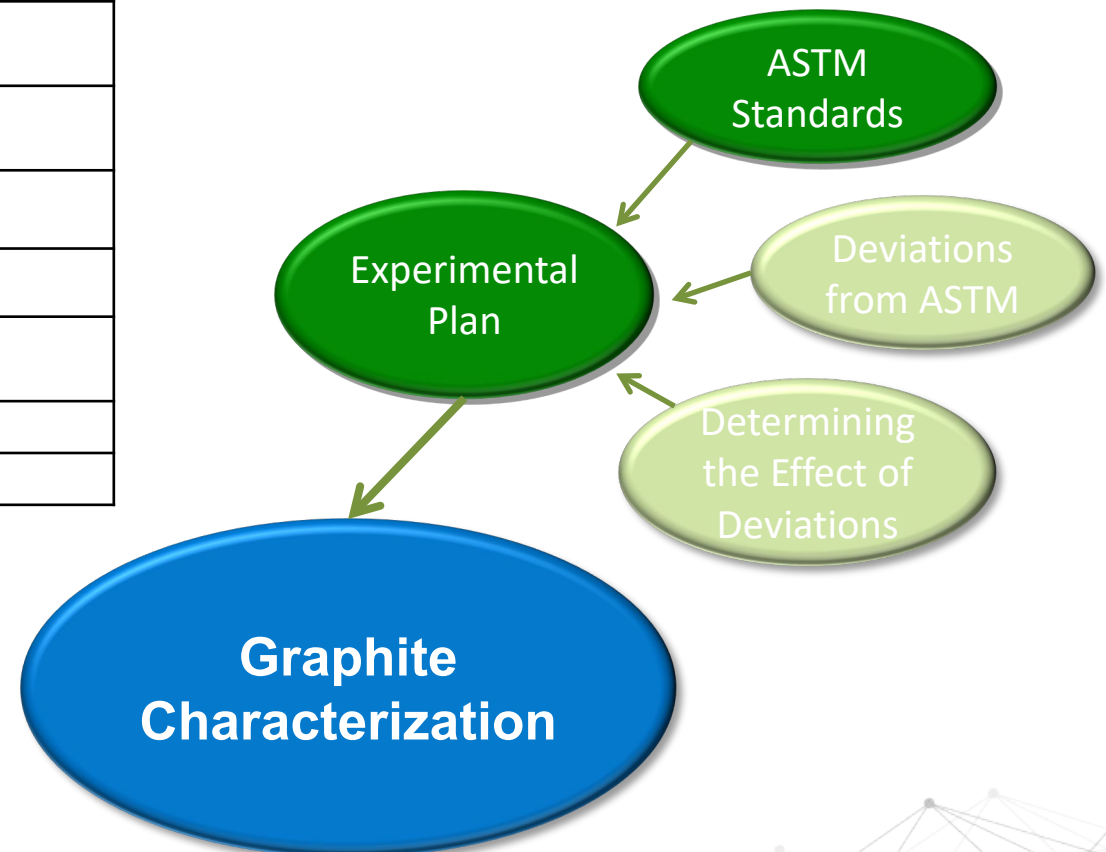
Automated data acquisition

- Specimens have a unique identity (*bar code*)
- Data acquisition and parsing software (*Labview® system*)
 - Output interfaces to data (i.e., spread sheets) that are easily incorporated into a database
- An **automated** system for specimen identification and data acquisition eliminates all data entry errors and significantly improves efficiency.



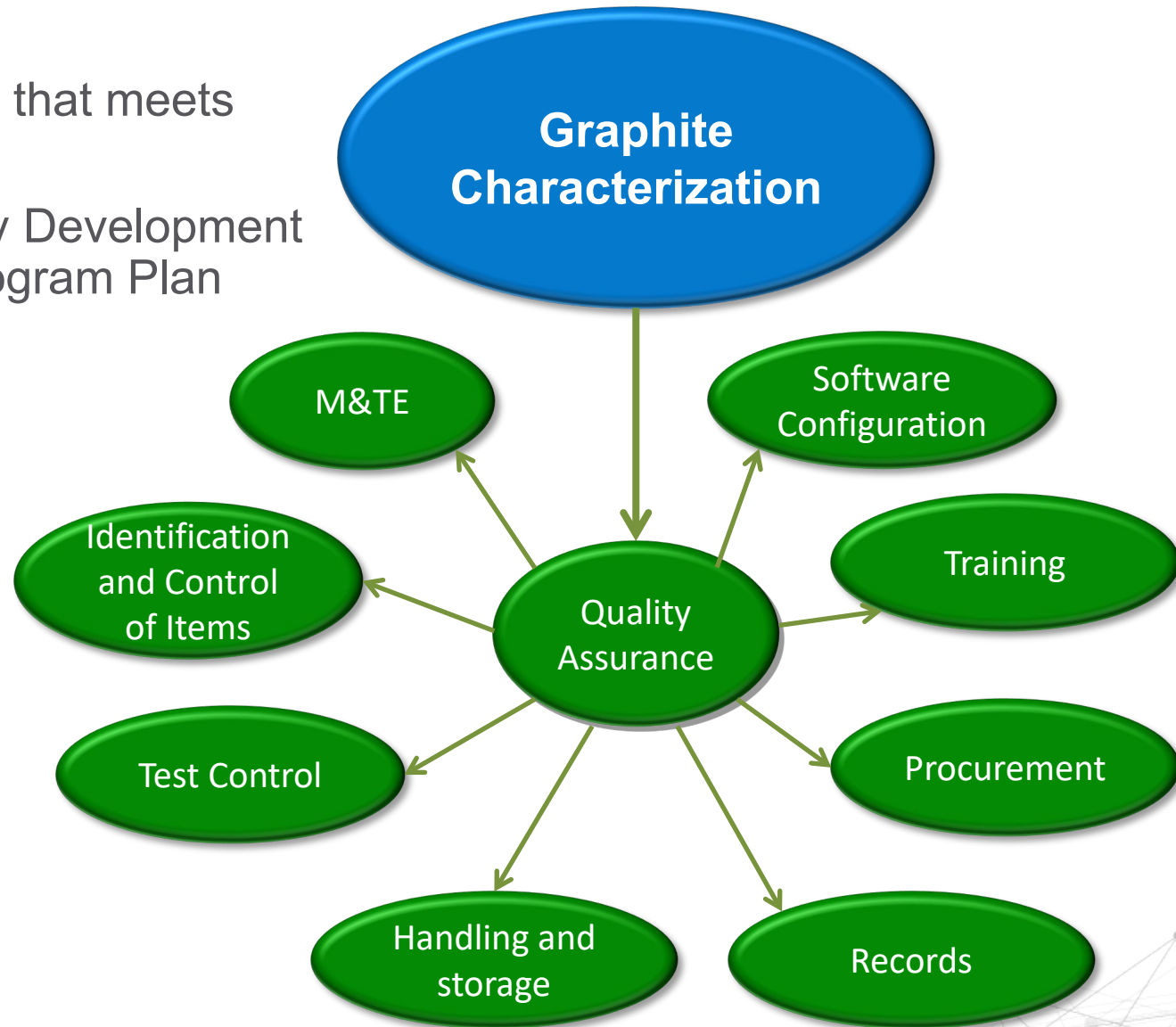
ASTM Standards Used

Measurement	Standard	Result
Physical Dimensions	ASTM C559	Creep
Mass	ASTM C559	Bulk Density
Sonic Resonance	ASTM C747 ASTM C1259	Elastic Modulus
Sonic Velocity	ASTM C769	Young's Modulus, Shear modulus, Poisson ratio
4 point Electrical Resistivity	ASTM C611	Electrical Resistivity
Laser Flash Diffusivity	ASTM E1461	Thermal Diffusivity
Push Rod Dilatometry	ASTM E228	Coefficient of Thermal Expansion
Tension	ASTM C749	Tensile strength
Compression	ASTM C695	Compressive strength



Quality Assurance

- INL Quality program in place that meets **NQA-1**
- PLN-2690 VHTR Technology Development Office Quality Assurance Program Plan



Conclusion

- INL has systems and processes in place to:
 - Produce traceable specimens from large graphite billets
 - Perform a multitude of tests and gather quality data
 - Prove the quality of the data

