SUBSECTION HH, Subpart A

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Technical Seminar on Application of ASME Section III to New Materials for High Temperature Reactors
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TIM BURCHELL
BIOGRAPHICAL INFORMATION

Dr. Tim Burchell is Distinguished R&D staff member and Team Lead for Nuclear Graphite in the Nuclear Material Science and Technology Group within the Materials Science and Technology Division of the Oak Ridge National Laboratory (ORNL). He is engaged in the development and characterization of carbon and graphite materials for the U.S. Department of Energy. He was the Carbon Materials Technology (CMT) Group Leader and was manager of the Modular High Temperature Gas-Cooled Reactor Graphite Program responsible for the research project to acquire reactor graphite property design data. Currently, Dr. Burchell is the leader of the Next Generation Nuclear Plant graphite development tasks at ORNL. His current research interests include: fracture behavior and modeling of nuclear-grade graphite; the effects of neutron damage on the structure and properties of fission and fusion reactor relevant carbon materials, including isotropic and near isotropic graphite and carbon-carbon composites; radiation creep of graphites, the thermal physical properties of carbon materials. As a Research Officer at Berkeley Nuclear Laboratories in the U.K. he monitored the condition of graphite moderators in gas-cooled power reactors.

He is a Battelle Distinguished Inventor; received the Hsun Lee Lecture Award from the Chinese Academy of Science's Institute of Metals Research in 2006 and the ASTM D02 Committee Eagle Award in 2015. Dr. Burchell remains very active in both the ASTM and ASME. He is currently a member of the Committee on Construction of Nuclear Facility Components (BPV-III), member of the Subgroup on High Temperature Reactors, Chair of the Working Group on Graphite and Composite Materials, and a member of Subgroup on Materials, Fabrication and Examination and other related working groups and subgroups. Dr. Burchell is a Fellow of the American Carbon Society and a Fellow of ASME.

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Section III, Division 5

High Temperature Reactors – Subsection HH

SUBPART A
GRAPHITE MATERIALS

GRAPHITE I – Manufacture & Application

Overview of Presentation

I. Manufacture and Applications

II. Structure and Properties

- Single crystal and polycrystalline synthetic graphite
- Porosity and texture
- Physical Properties
  - Thermal
  - Electrical
- Mechanical Properties
  - Elastic constants
  - Strength and fracture

III. Reactor Environmental Effects

IV. ASME Code for Graphite

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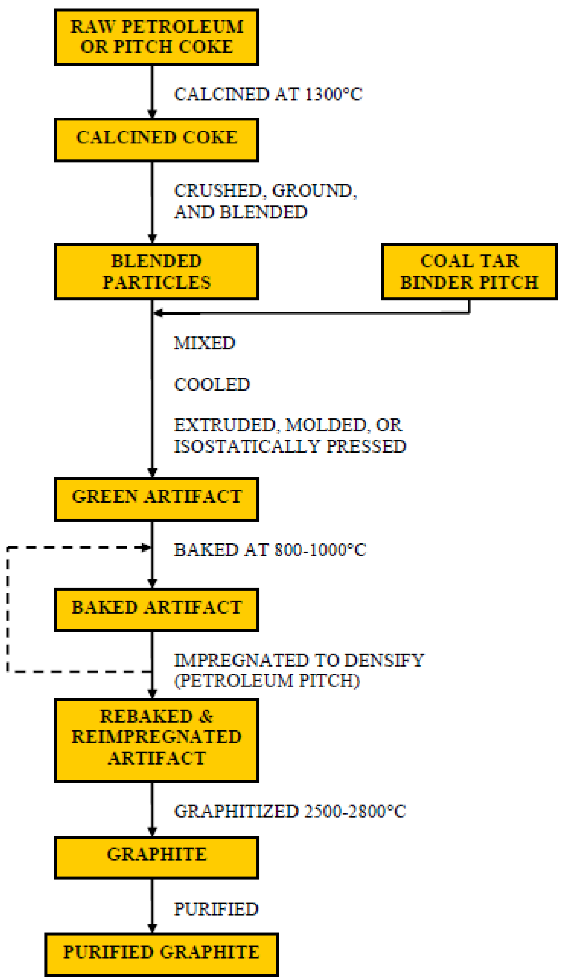
SYNTHETIC GRAPHITE MANUFACTURE



- Pitch coke from calcination of coal-tar pitch
- Coke filler particle morphology and green artifact forming method affect texture and properties
- Petroleum coke from calcination of heavy oil distillates
- First bake is a critical stage, - controlled binder pyrolysis
- Acheson or longitudinal graphitization
- Long cycle times ~ 9 months

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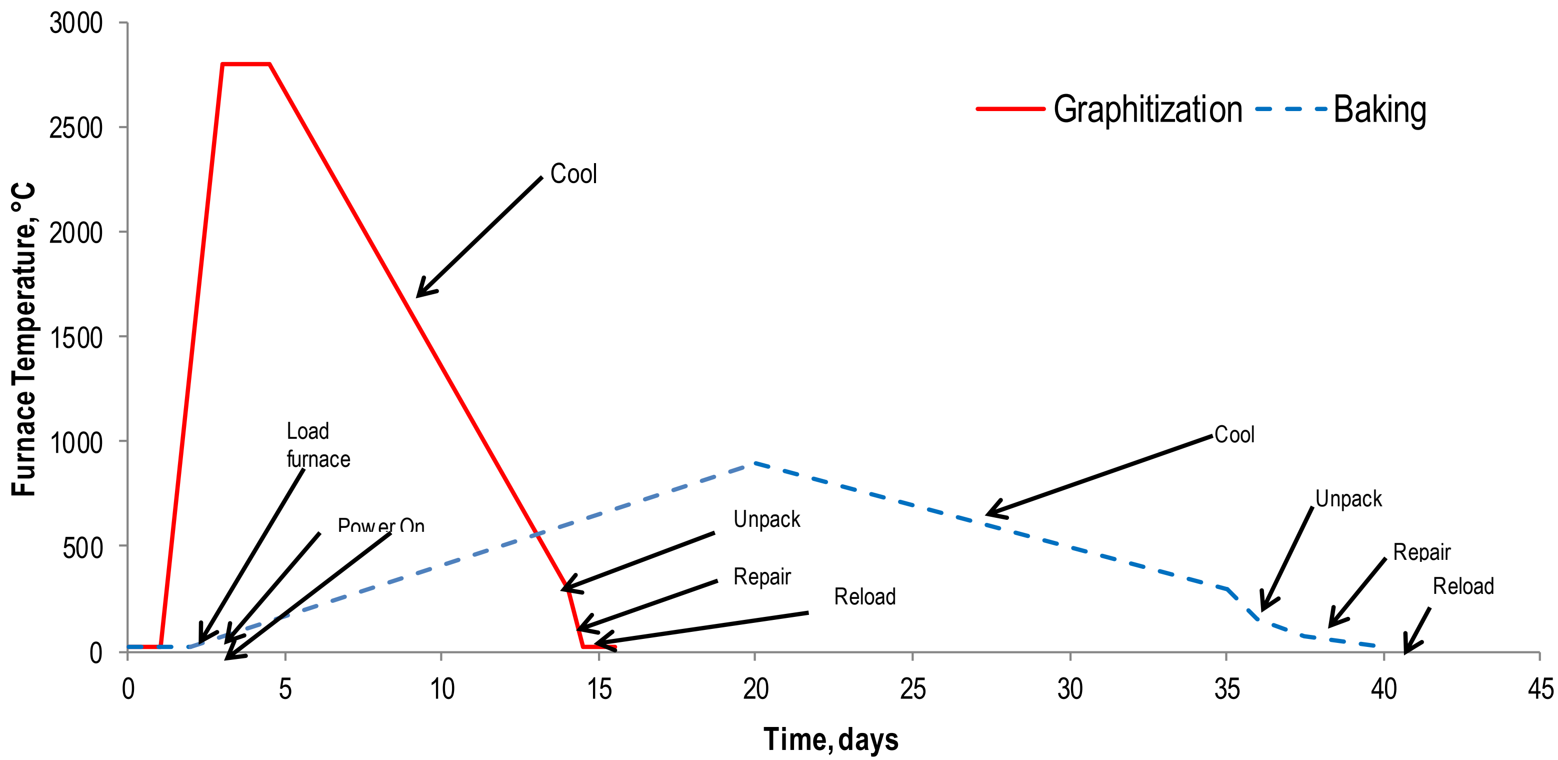
Manufacture – Baking and Graphitizing



Slow heating and cooling during baking allows escape of pyrolysis gasses and minimizes thermal gradients

Manufacture - Baking

Modern car bottom carbon/graphite baking furnace
Green bodies packed in coke and placed in steel saggers

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Manufacture – Acheson Graphitization

Baked artifacts surrounded by a coke pack and covered with sand to exclude air. Electric current flow through the coke-pack & artifacts

Manufacture – Longitudinal Graphitization

Furnace covered with sand to exclude air, current flows though the baked artifact
Carbon atoms migrate to thermodynamically more stable graphitic lattice structure and 3D ordering achieved (degree of ordering depends on feedstock type)

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Manufacture - Purification

Post graphitization halogen gas process
Can be performed with solid fluoride additives to Acheson graphitization furnace or solid fluoride additives to formulation

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Manufacture - Purification

- **Unpurified graphite**
  - "clean" raw materials
  - > 500 ppm total impurities
- **Thermally purified graphite**
  - Graphitized over 3000 °C
  - >100 ppm total impurities
- **Chlorine purified graphite**
  - High temperature chlorine treatment to remove impurities as volatile chlorides
  - < 5 ppm total impurities
  - not effective for Boron
- **Fluorine Purification**
  - Effective for Boron

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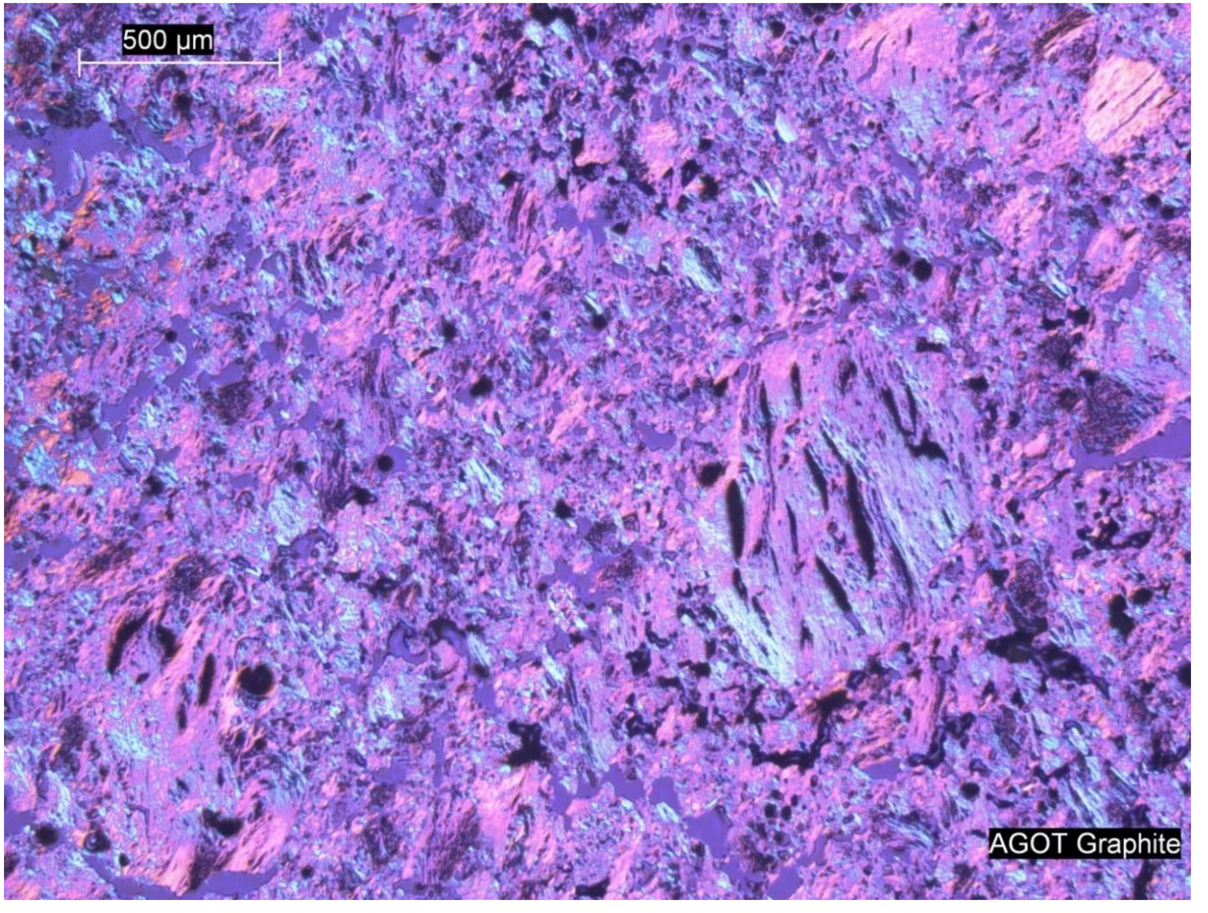
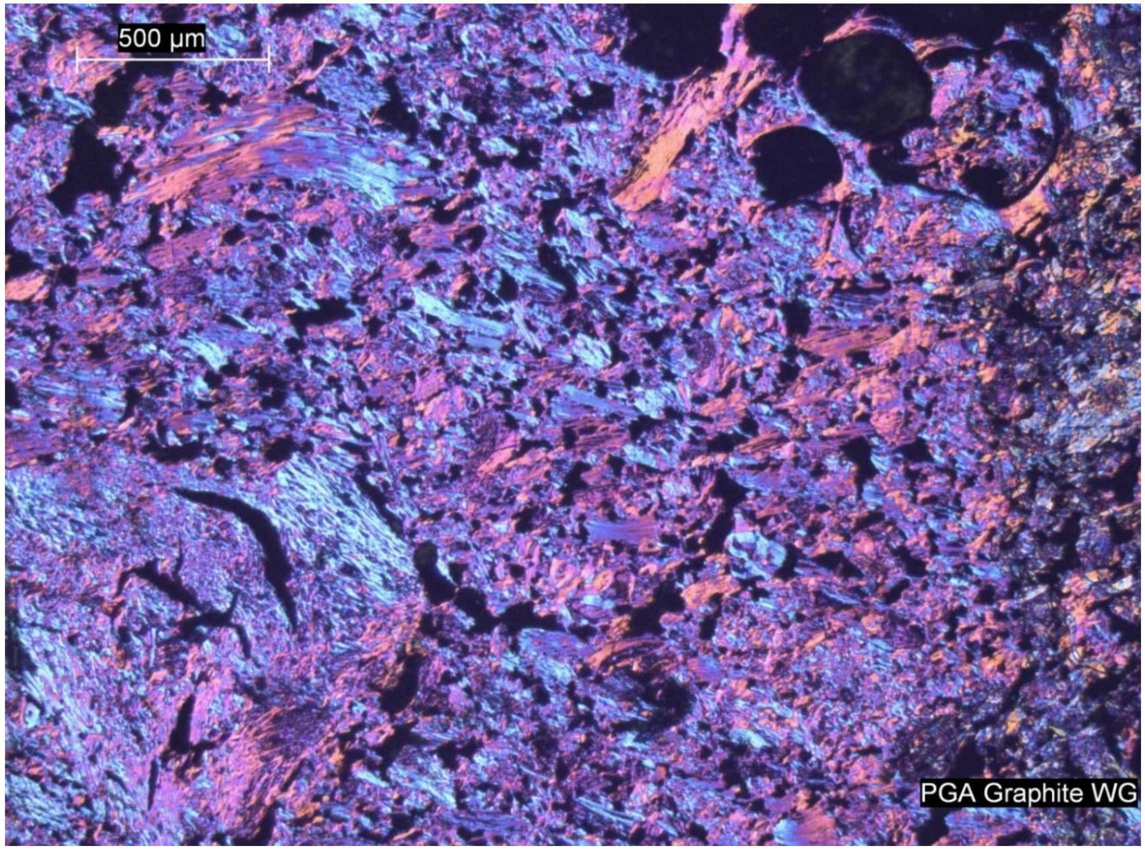
Synthetic Graphite Microstructure



Grade AGOT graphite microstructure (viewed under polarized light)

Synthetic Graphite Microstructure



Grade PGA graphite (with-grain) microstructure
(viewed under polarized light)

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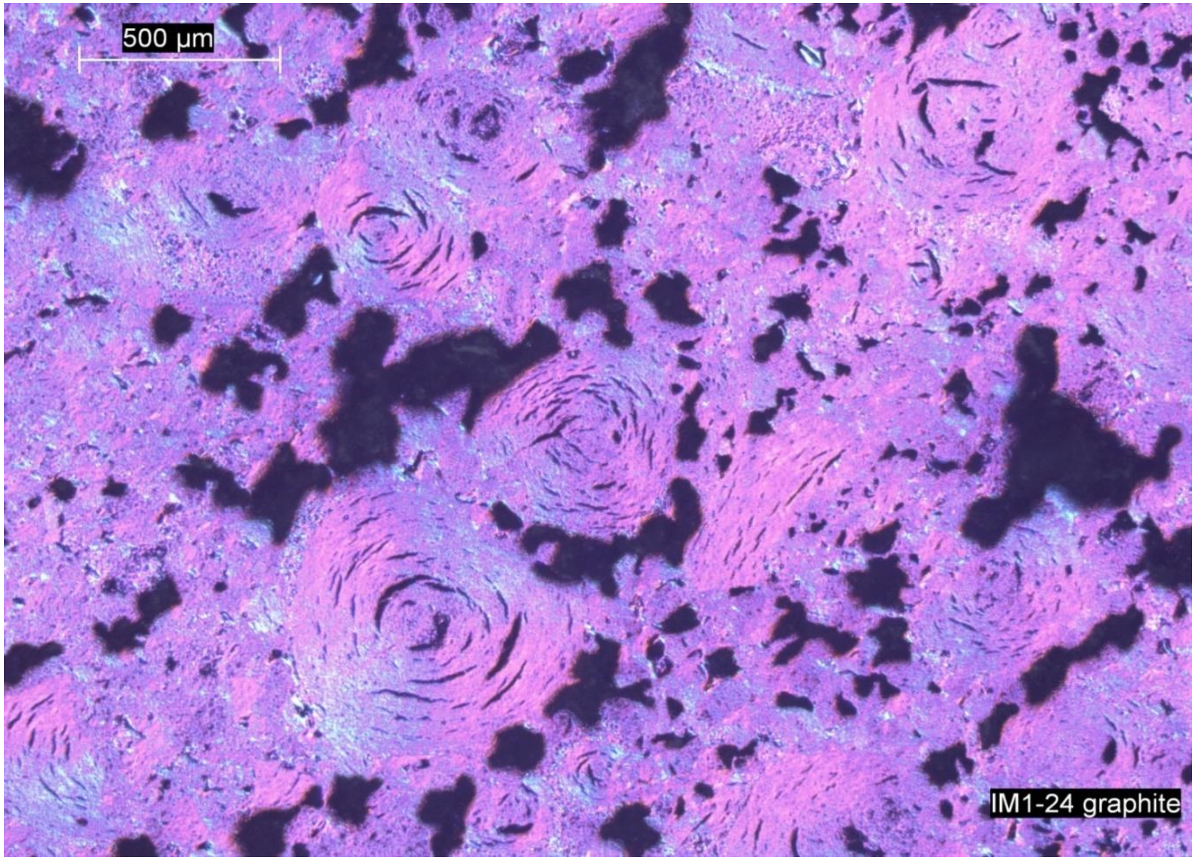
Synthetic Graphite Microstructure



Grade IM1-24 graphite microstructure (viewed under polarized light)

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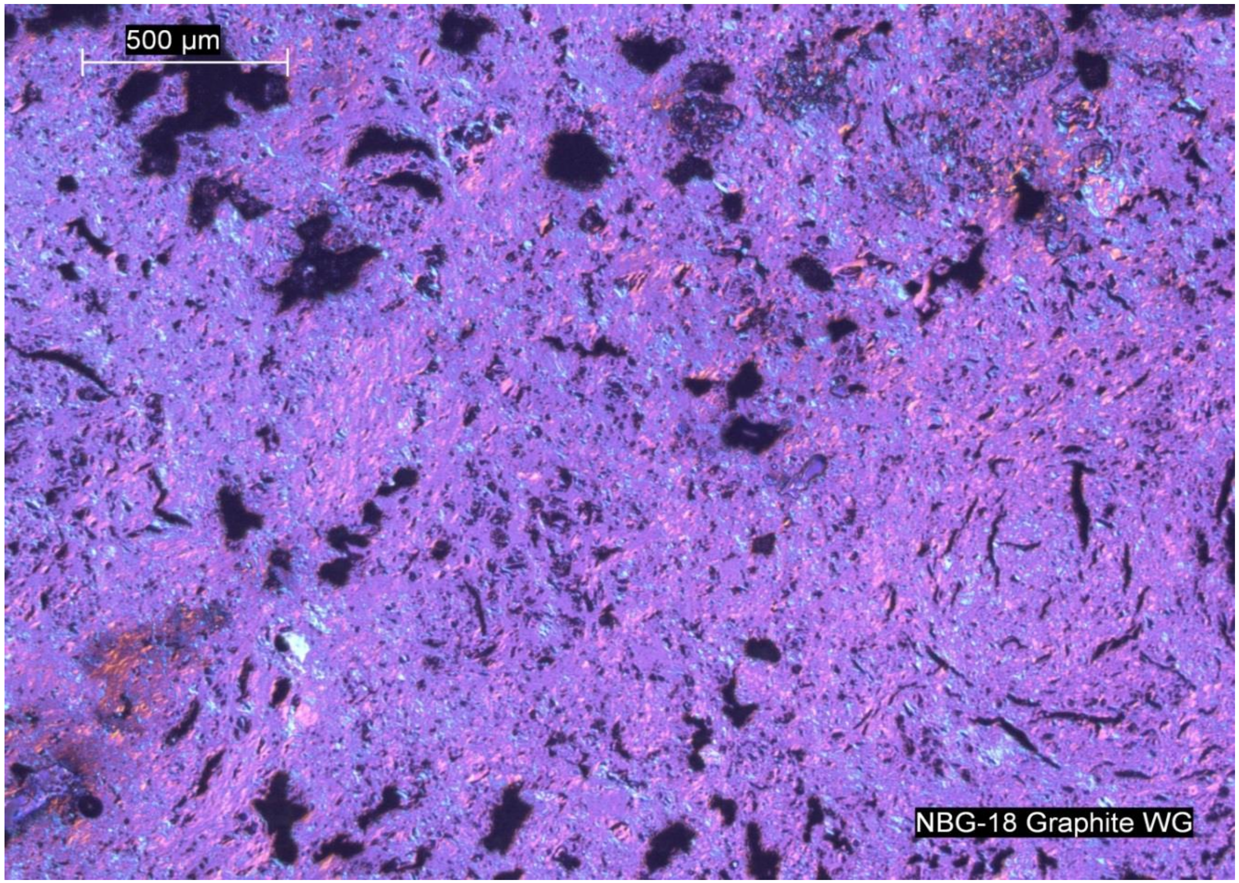
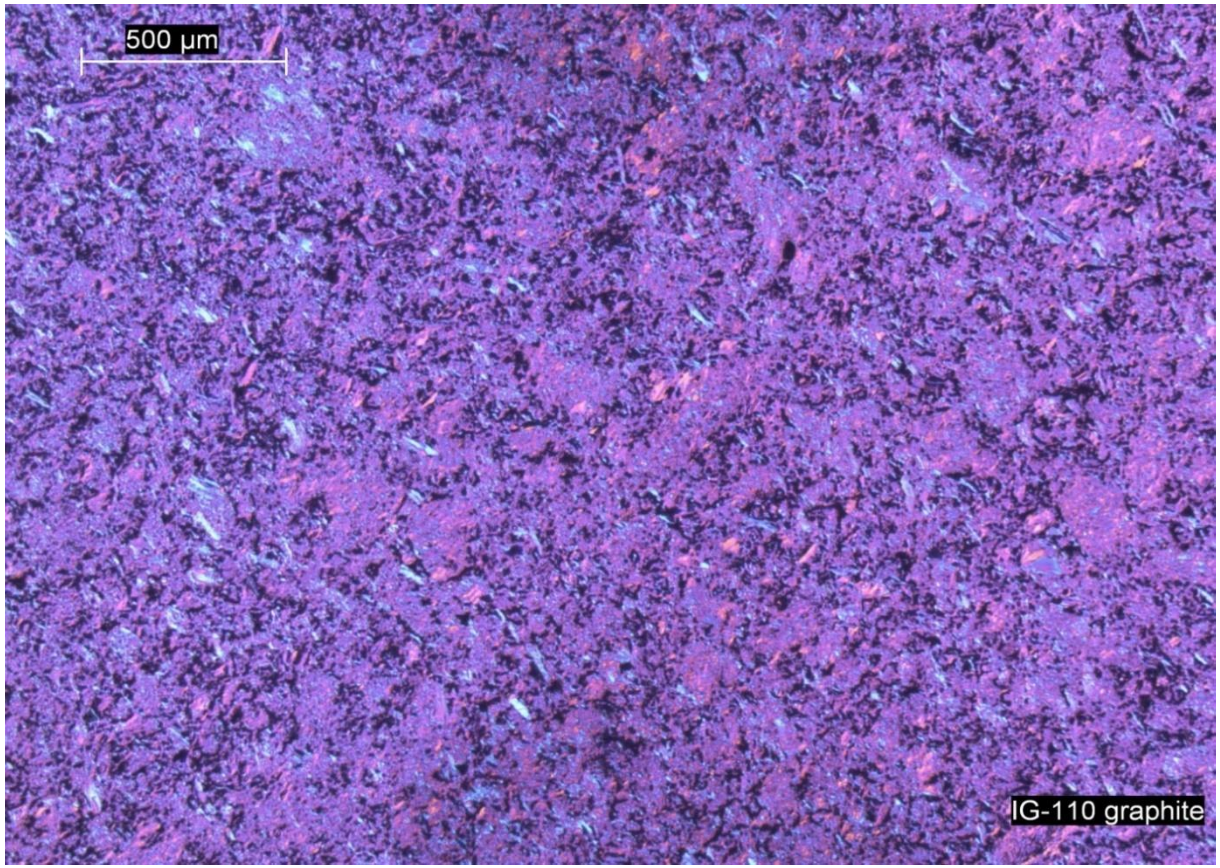
Synthetic Graphite Microstructure



Grade NBG-18 graphite (with-grain) microstructure (viewed under polarized light)

Synthetic Graphite Microstructure



Grade IG-110 graphite microstructure (viewed under polarized light)

Synthetic Graphite Texture

Texture in synthetic graphite arises because of:

- Crystal anisotropy, coke and binder domain size
- Filler cokes and binder cracks
- Size and shape distribution of filler particle
- Filler coke type
- Recycle fraction and morphology
- Porosity
- Forming method (preferential orientation of filler coke and binder porosity)

Texture imparts anisotropy!

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Applications

- Metal processing
  - Arc furnace electrodes (steel re-melting)
  - Hall-Heroult cell cathodes (aluminum electrolysis)
  - Casting dies
- Semiconductor manufacture
  - Si crystal processing, crucibles, boats
- Electrical and electronic
  - Electric motor brushes, resistance heating elements, EDM electrodes
  - Fuel cells (bipolar plates), lithium-ion rechargeable batteries
- Mechanical
  - Bearings, seals
- Aerospace
  - rocket motor throats and nozzles, missile nose cones, jet thrust plates and thermal protection systems
- Nuclear
  - Stay tuned!!

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APPLICATIONS

THE LARGEST MARKET FOR ARTIFICIAL GRAPHITE IS ARC FURNCE ELECTRODES (STEEL INDUSTRY) – ABOUT 1,000,000 TONS PER YEAR PRODUCED

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ASTM International

ASTM has a number of Specifications, Standards, guidelines and procedures under the jurisdiction of committee DO2.F and Published annually in Vol. 5.05

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ASTM Standards - Specifications (An ASME Requirement)

- D7219-08 Standard Specification for Isotropic and Near-isotropic Nuclear graphite
- D7301-08 Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose

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What is Specified by The ASTM?

- Coke type and isotropy (CTE)
- Method of determining coke CTE
- Maximum filler particle size
- Green mix recycle
- Graphitization temperature (2700^{o}C)
- Method of determining graphitization temperature
- Isotropy ratio and chemical purity
- Properties: density, strength (tensile, compressive, flexural), CTE, E
- Marking and traceability
- Quality assurance (NQA-1)

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ASTM Standard Practices

- C625 Reporting Irradiation Results on Graphite
- C781 Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components
- C783 Core Sampling of Graphite Electrodes
- C709 Standard Terminology Relating to Manufactured Carbon and Graphite

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ASTM Standard Test Methods

- C559 Bulk Density by Physical Measurement of Manufactures Carbon and Graphite Articles
- C560 Chemical Analysis of Graphite
- C561 Ash in a Graphite Sample
- C562 Moisture in a Graphite Sample
- C565 Tension testing of Carbon and Graphite Mechanical Materials
- C611 Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature

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ASTM Standard Test Methods (continued)

- C651 Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature
- C695 Compressive Strength of Carbon and Graphite
- C714 Thermal Diffusivity of Carbon and Graphite by Thermal Pulse Method
- C747 Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite by Sonic Resonance
- C748 Rockwell Hardness of Graphite Materials

ASTM Standard Test Methods (continued)

- C749 Tensile Stress Strain of Carbon and Graphite
- C769 Sonic Velocity in Manufactured Carbon and Graphite for Use in Obtaining Young's Modulus
- C816 Sulfur in Graphite by Combustion-Iodometric Titration Method
- C838 Bulk Density of As-Manufactured Carbon and Graphite Shapes
- C886 Scleroscope Hardness Testing of Carbon and Graphite Materials

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ASTM Standard Test Methods (continued)

- C1025 Modulus of Rupture in Bending of Electrode Graphite
- C1039 Apparent Porosity, Apparent Specific Gravity, and Bulk Density of Graphite Electrodes
- C1179 Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air
- D7542 Air Oxidation of Carbon and Graphite in the Kinetic Regime

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New ASTM Test Methods, Guidelines and Practices

- ASTM D02.F on Manufactured Carbons and Graphite has several (relatively) new test methods, Guidelines and Practices
  - **D7775-11** (2015), Standard Guide for Measurements on Small graphite specimens
  - **D7779-11** (2015), Standard Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature
  - **D7846-16**, Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Graphites

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New ASTM Test Methods, Guidelines and Practices (continued)

- **D7972-14**, Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles using Three Point Loading at Room Temperature
- **D8075-16**, Standard Guide for Categorization of Microstructural and Microtextural Features Observed in Optical Micrographs of Graphite
- **D8091-16**, Standard Guide for Impregnation of Graphite with Molten Salt
- **D8093-16**, Standard Guide for Nondestructive Evaluation of Nuclear Grade Graphite

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New ASTM Test Methods Currently in Development

- ASTM D02.F on manufactured carbons and graphite has several Test Methods and Guides in development:
  - Sonic Elastic Constants
  - Chemical purity by
    - ICP-OES
    - GDMS
  - Fluorine, chlorine and sulfur in graphite by Combustion Ion Chromatography
  - Tensile Strength using a Brazilian Disc Specimen Geometry
  - Standard Guide for Use of Gas Adsorption Method for the Evaluation of Surface Area and Porosity in Nuclear Graphites

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ASTM Committee D02.F has also Published an STP

- STP 1578, Graphite Testing for Nuclear Applications:

  The Significance of Test Specimen Volume and Geometry and the Statistical Significance of Test Specimen Population

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Thank You!

Feel free to contact **Richard Barnes** email at **rwbarnes@anric.com** or by phone at **(416) 727-3653 (cell)** or **(416) 255-9459 (office).**

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High Temperature Reactors – Subsection HH

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GRAPHITE MATERIALS

GRAPHITE II – Structure & Properties

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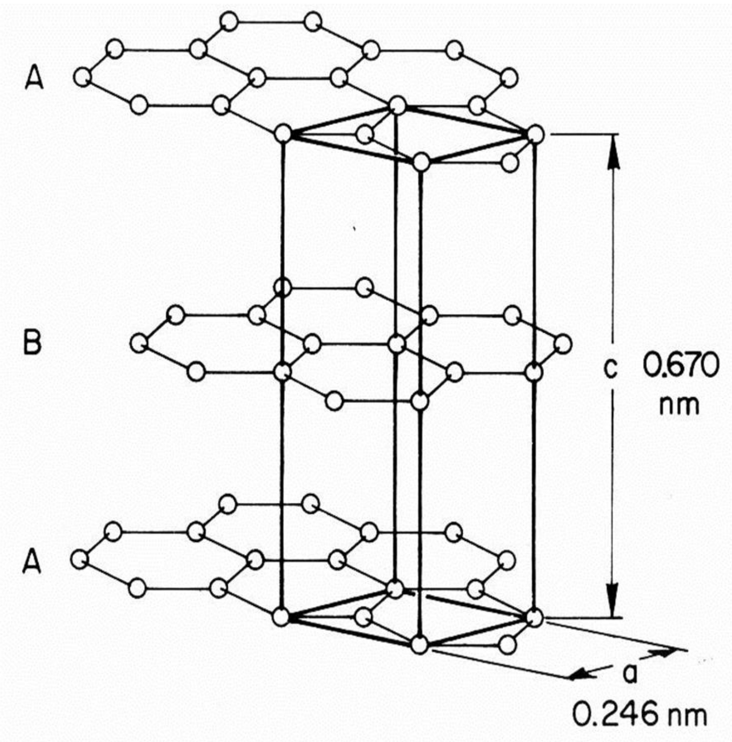
Graphite Single Crystal Structure



BOND ANISOTROPY

- Strong, stiff covalent bond in-plane
- Weak bonds of attraction between graphene planes
- ABA repeat stacking (can get ABC…)
- Crystal unit cell size:
- <a> = 0.246 nm
- <c> = 0.670 nm
- Coherence lengths, l_a and l_c are measures of crystal size

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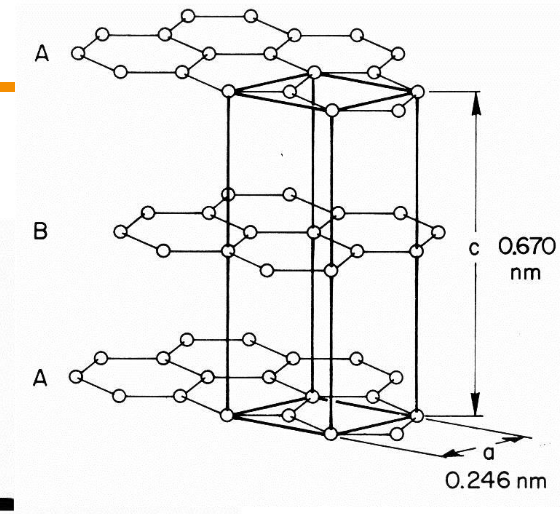
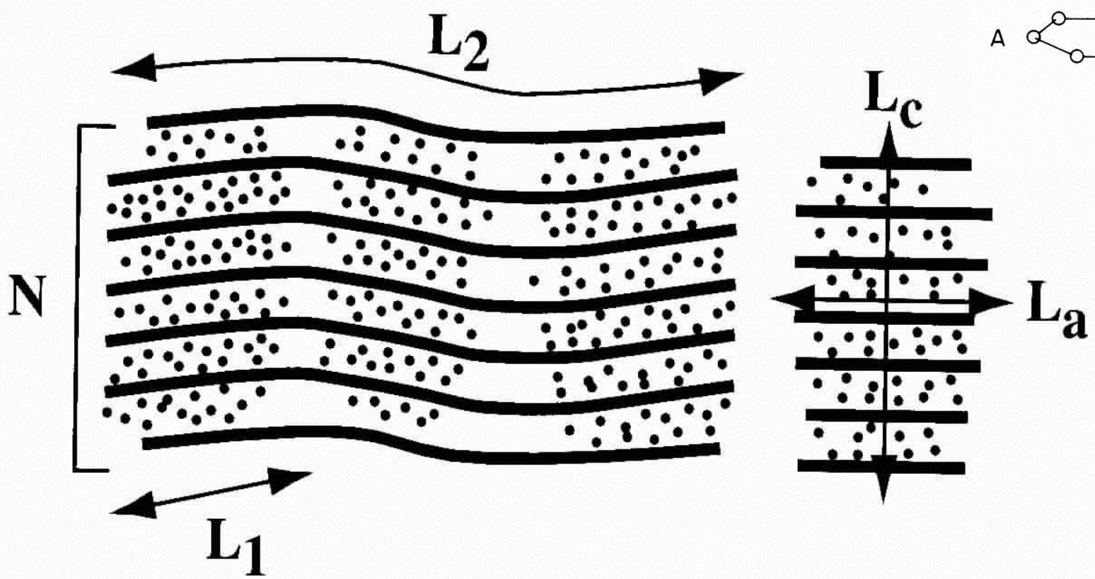
Crystallites & Optical Domain





Sciences of Carbon Materials, Marsh & Reinoso

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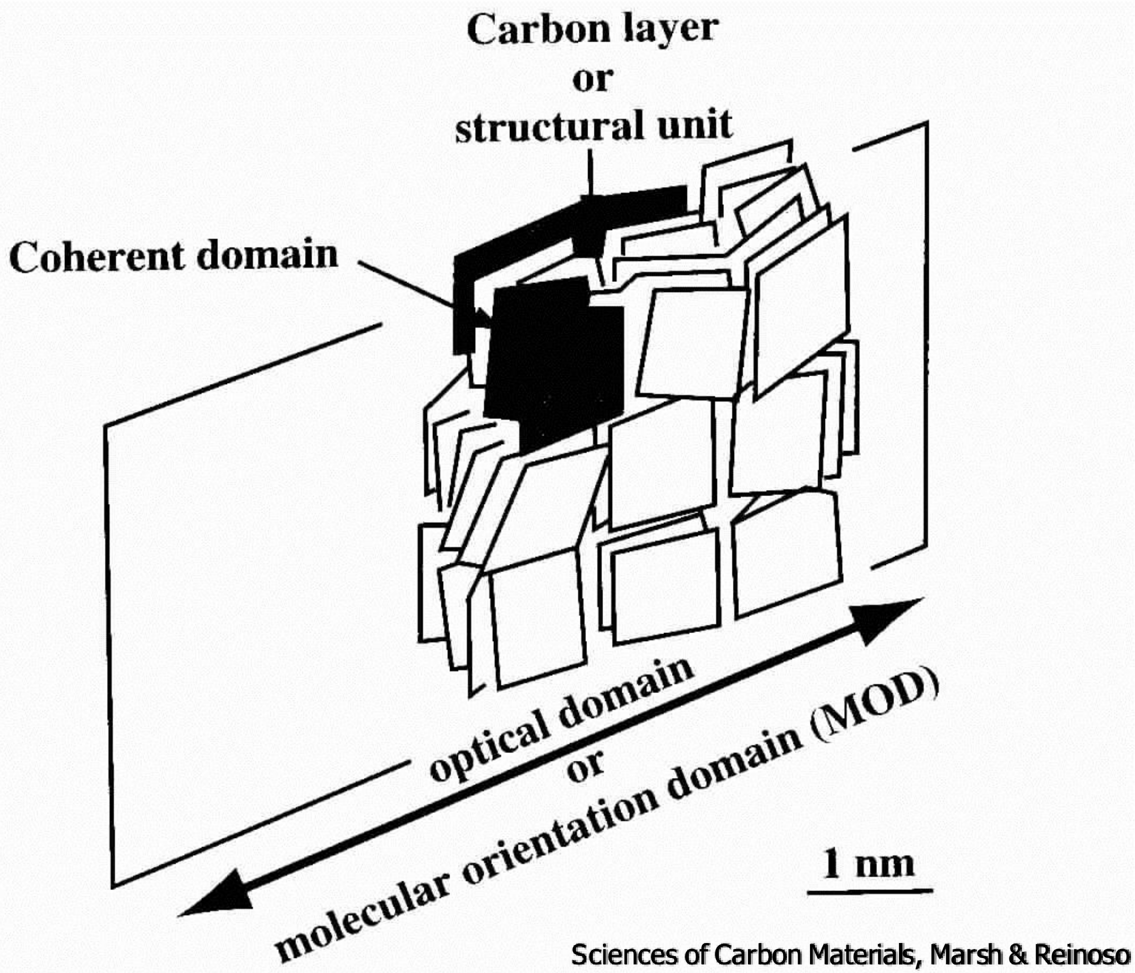
Crystallites & Optical Domain



Sciences of Carbon Materials, Marsh & Reinoso

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Porosity in Graphite

- Graphite single crystal density = 2.26 g/cc
- Synthetic graphite bulk density = 1.6-1.9 g/cc
- Most graphite contains ~>20% porosity
- > 60% of porosity is open

- **Three classes of porosity may be identified in synthetic graphite:**

1. Those formed by incomplete filling of voids in the green body by the impregnant pitch, the voids originally occur during mixing and forming;
2. Gas entrapment pores formed from binder phase pyrolysis gases during the baking stage of manufacture;
3. Thermal cracks formed by the anisotropic shrinkage of the crystals in the filler coke and binder.

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Graphite Structural Features: Nuclear Graphite

- Lattice (a= 2.45 A, c= 6.7 Å)
- Crystallite "Coherent Domain"
  - Extent of 3D order
  - Lc = Stack Height, La = Stack Width (250-600 Å)
- Micro-crack (between planes, about the size of crystallite)
- Optical Domain (extended orientation of crystallites)
  - Carbonization chemistry + Coking transport phenomena
  - Scale is 5-100 microns
  - Controls Isotropy of Synthetic Graphite
- Grain Size
  - Usually refers to largest filler particles (10-1000 microns)
- Pore Size
  - Pores could be within filler or binder phases
  - Different for isomolded graphite
  - Scale of largest pores (10 - 350 microns)

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Synthetic graphite properties @ RT

Typical Properties	Graphite Grade and Manufacturer					
	AXF-5Q	IG-43	2020	ATJ	NBG-18	AGR
	POCO	Toyo-Tanso	Mercen	GTI	SGL Carbon	GTI
Forming Method	Isomolded	Isomolded	Isomolded	Isomolded	vibro-molded	Extruded
Maximum Particle Size, µm	5	10 (mean)	15	25 (mean)	1600	3000
Bulk Density, g/cm^3	1.8	1.82	1.77	1.76	1.88	1.6
Thermal Conductivity, W/m.K (Measured at ambient temperature)	85	140	85	125(WG) 112 (AG)	156 (WG) 150 (AG)	152 (WG) 107 (AG)
Coefficient of Thermal Expansion, 10^{-6}/K (over given temperature range)	7.4 (20-500°C)	4.8 (350-450°C)	4.3 (20-500°C)	3.0 (WG) 3.6 (AG) (@500°C)	4.5 (WG) 4.7 (AG) (20-200°C)	2.1 (WG) 3.2 (AG) (@500°C)
Electrical Resistivity, µΩ.m	14	9.2	15.5	10.1 (WG) 11.7 (AG)	8.9 (WG) 9.0 (AG)	8.5 (WG) 12.1 (AG)
Young's Modulus, GPa	11	10.8	9.3	9.7 (WG) 9.7 (AG)	11.2 (WG) 11.0 (AG)	6.9 (WG) 4.1 (AG)
Tensile Strength, MPa	65	37	30	27.2 (WG) 23.1 (AG)	21.5 (WG) 20.5 (AG)	4.9 (WG) 4.3 (AG)
Compressive Strength, MPa	145	90	80	66.4 (WG) 67.4 (AG)	72 (WG) 72.5 (AG)	19.8(WG) 19.3 (AG)
Flexural Strength, MPa	90	54	45	30.8 (WG) 27.9 (AG)	28 (WG) 26(AG)	8.9 (WG) 6.9 (AG)

WG-with grain, AG-against grain

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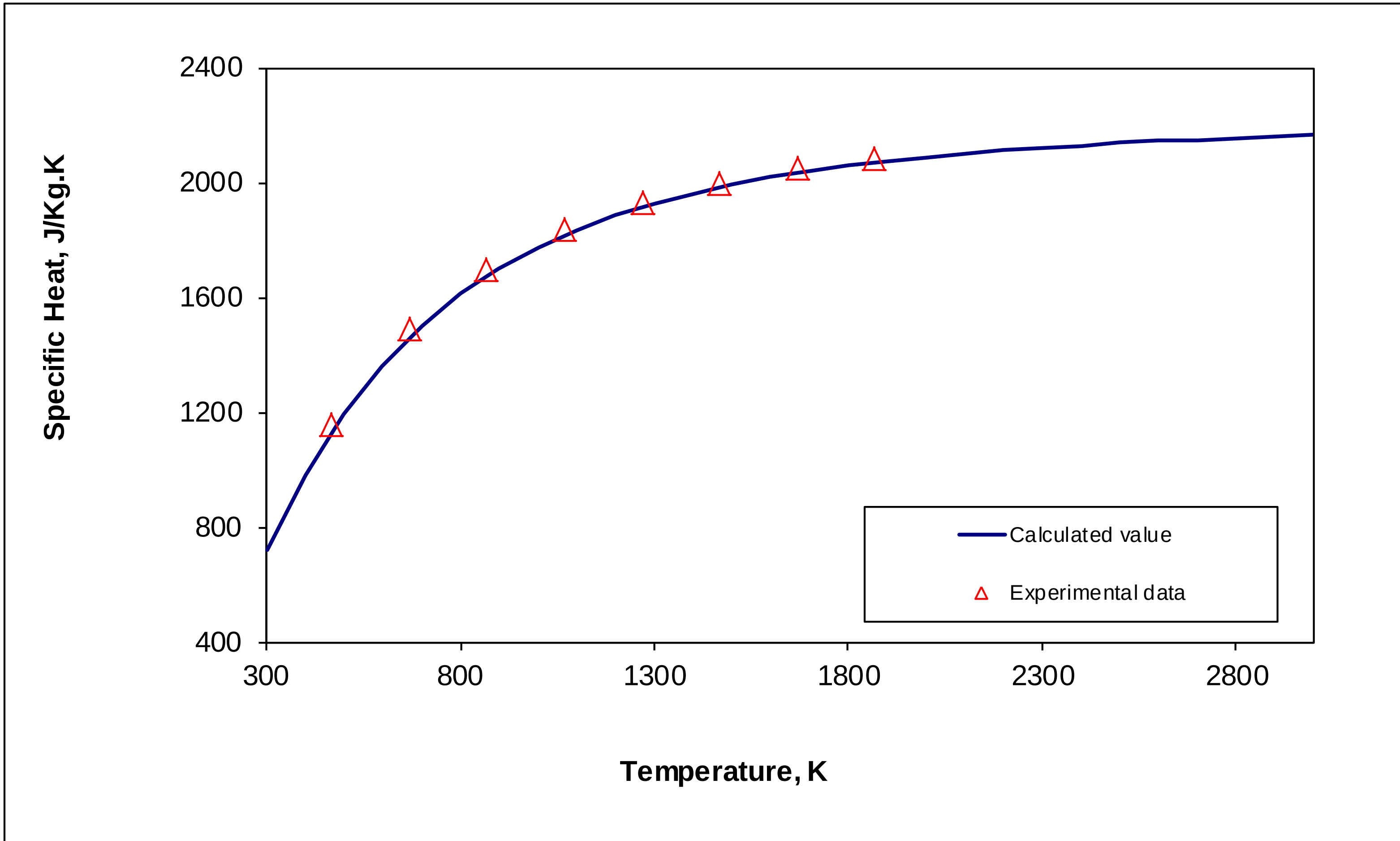
Temperature Dependence of Specific Heat



Calculated from ASTM C781

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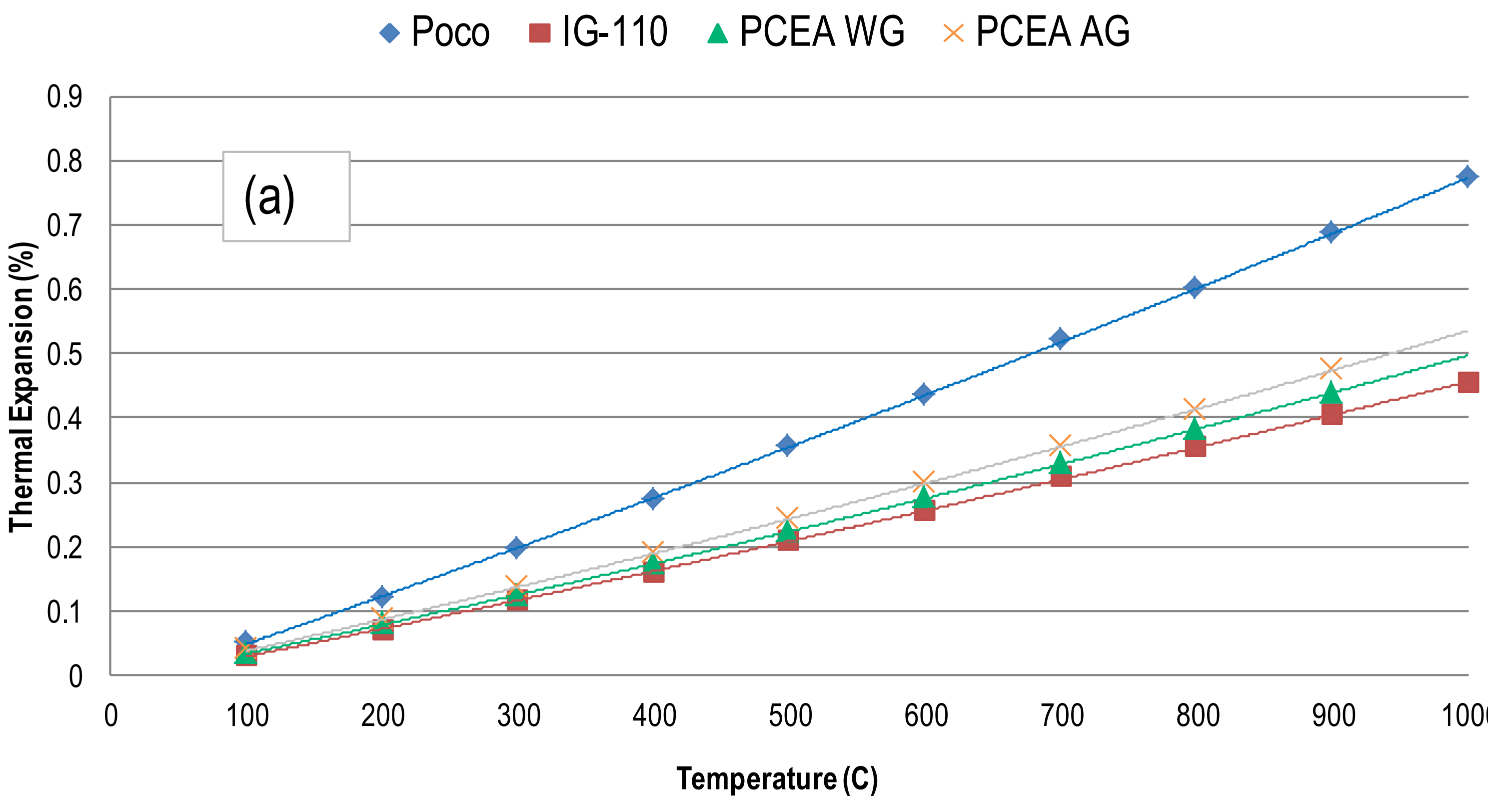
Single Crystal Thermal Expansion Behavior

Hexagonal graphite lattice has two principal thermal expansion coefficients; α_c, the thermal expansion coefficient parallel to the hexagonal <c>-axis and α_a, the thermal expansion coefficient of the crystal parallel to the basal plane (<a>-axis). The thermal expansion coefficient in any direction at an angle φ to the <c> axis of the crystal given by:

$$\alpha(\varphi) = \alpha_c cos^2\varphi + \alpha_a sin^2\varphi$$

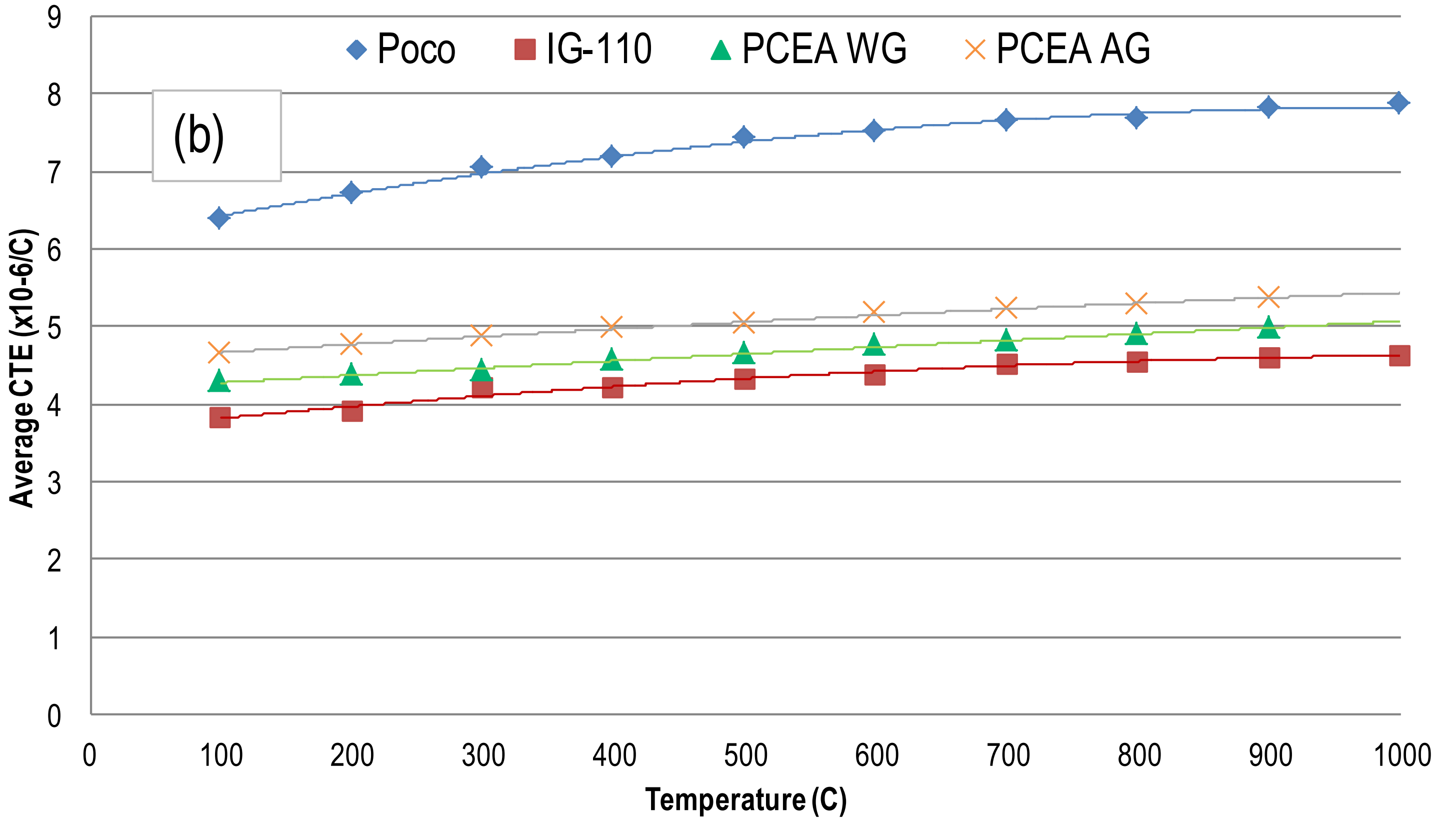
- α_c varies linearly with temperature from ~25 × $10^{-6}K^{-1}$ at 300K to ~35 × 10^{-6} K^{-1} at 2500K.
- α_a is much smaller and increases rapidly from -1.5 × 10^{-6} K^{-1} at ~300K to approximately 1 × 10^{-6} K^{-1} at 1000K, and remains relatively constant at temperatures up to 2500K.

Temperature Dependence of Thermal Expansion



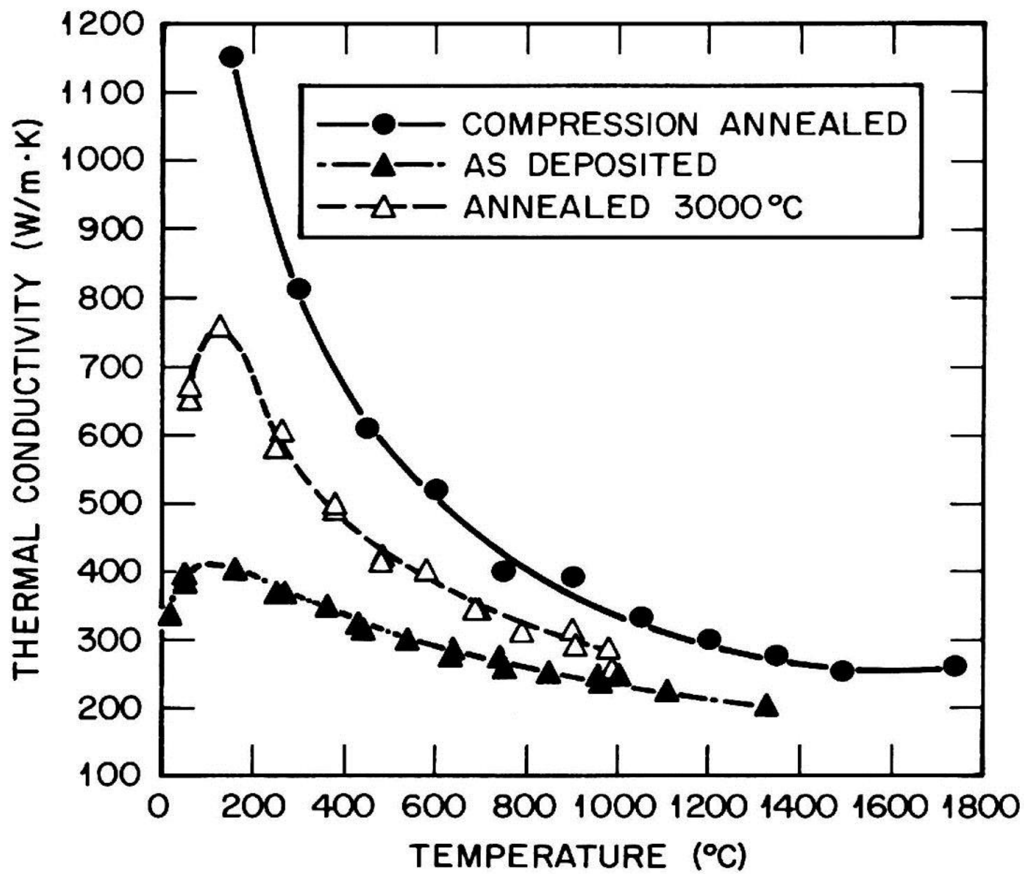
Thermal closure of aligned porosity

Temperature Dependence of Coefficient of Thermal Expansion



Thermal closure of aligned porosity

Temperature Dependence of HOPG Thermal Conductivity



Phonon scattering, effect of intrinsic defects

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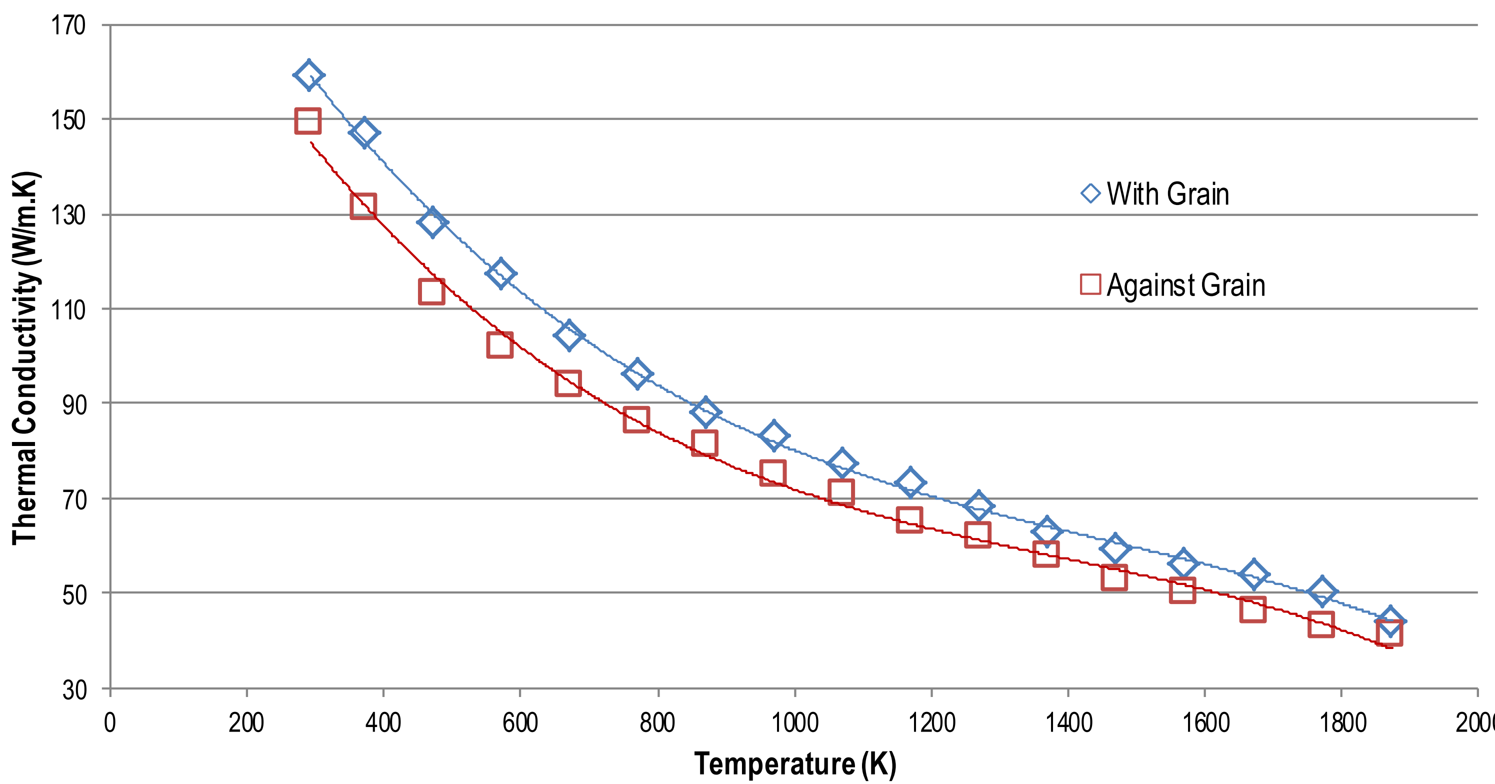
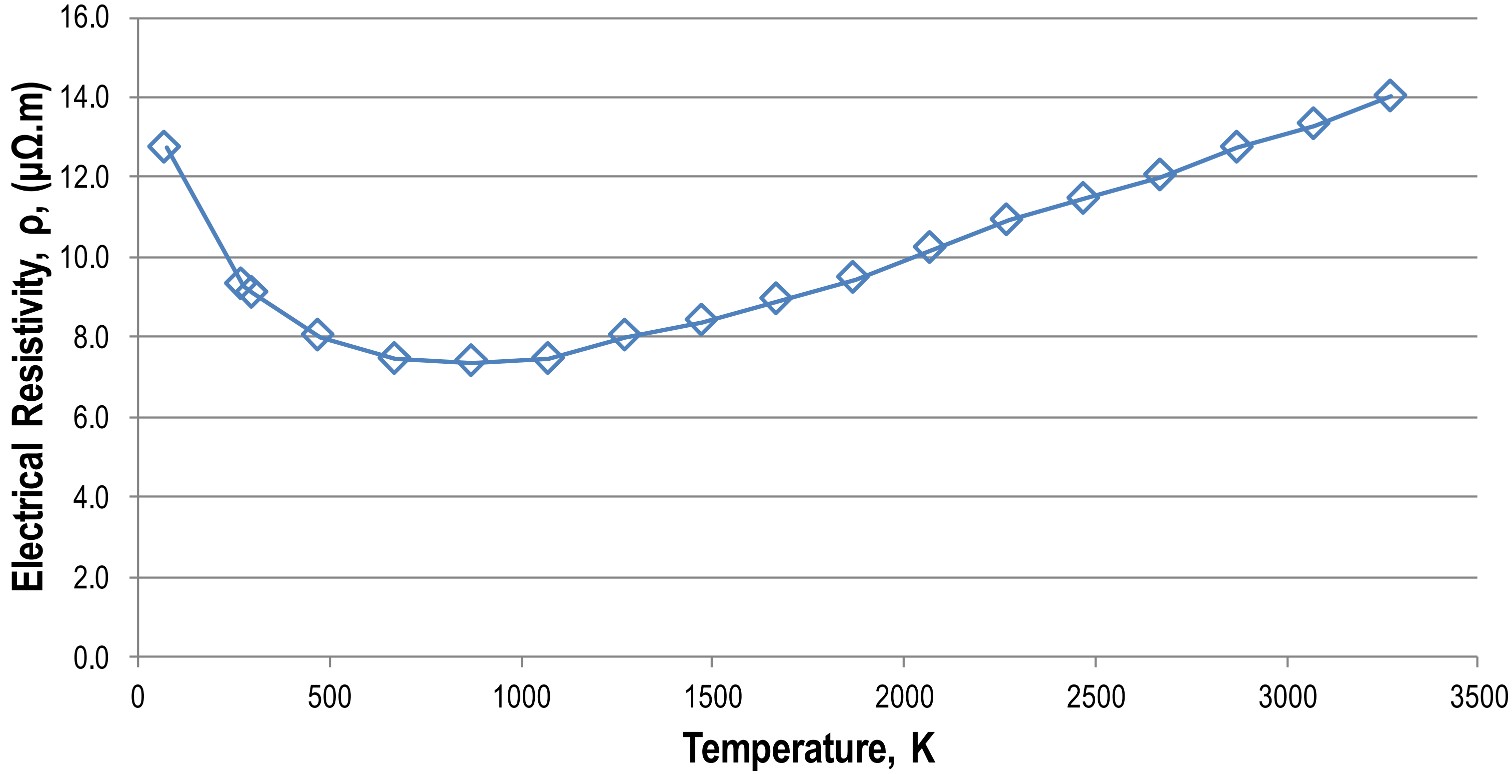
Temperature Dependence of the Thermal Conductivity



Anisotropy in extruded thermal conductivity

Temperature Dependence of the Electrical Resistivity



Electron transport mechanism

Elastic Constants Of Single Crystal Graphite (Kelly, 1981)

Elastic moduli, GPa		Elastic compliances, 10^{-13} Pa^{-1}	
C_{11}	1060 ± 20	S_{11}	9.8 ± 0.3
C_{12}	180 ± 20	S_{12}	-1.6 ± 0.6
C_{13}	15 ± 5	S_{13}	-3.3 ± 0.8
C_{33}	36.5 ± 1	S_{33}	275 ± 10
C_{44}	4.0 - 4.5	S_{44}	2222 - 2500

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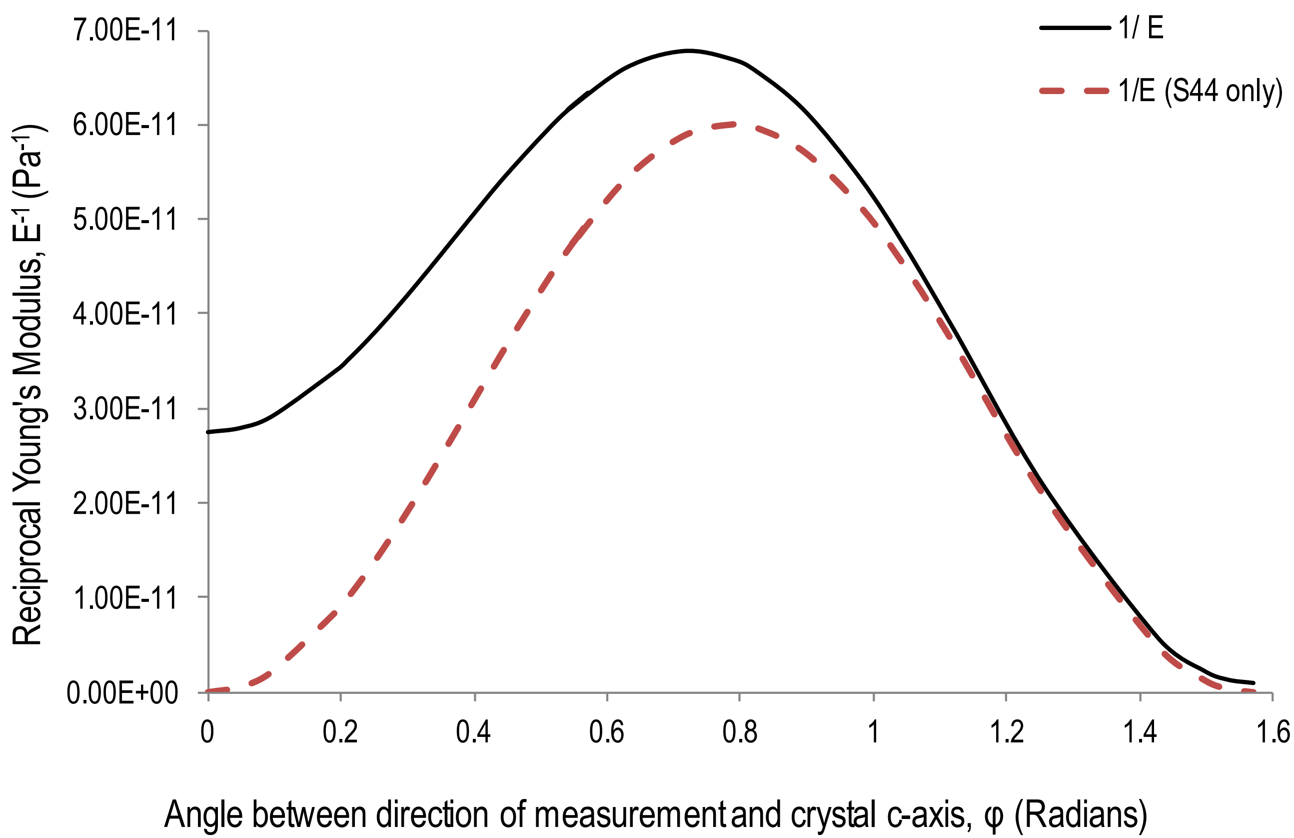
Variation Of The Reciprocal Young's Modulus with Angle Of Miss-orientation Between The c-axis and Measurement Axis



$$E^{-1} = S_{11}(1-\gamma^2)^2 + S_{33}\gamma^4 + (2S_{13} + S_{44})\gamma^2(1-\gamma^2)$$

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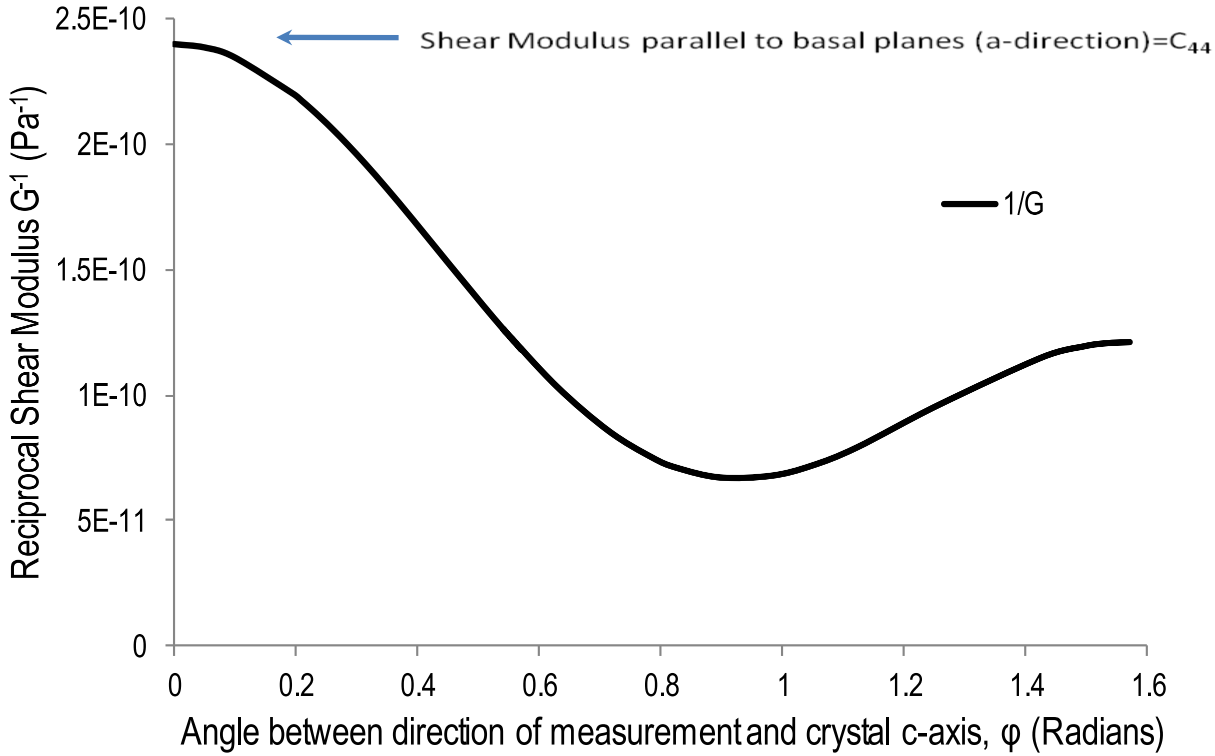
Variation of the reciprocal Shear modulus with angle of miss-orientation between the c-axis and measurement axis



$$G^{-1} = S_{44} + \left(S_{11} - S_{12} - \frac{S_{44}}{2}\right)(1-\gamma^2) + 2(S_{11} + S_{33} - 2S_{13} - S_{44})\gamma^2(1-\gamma^2)$$

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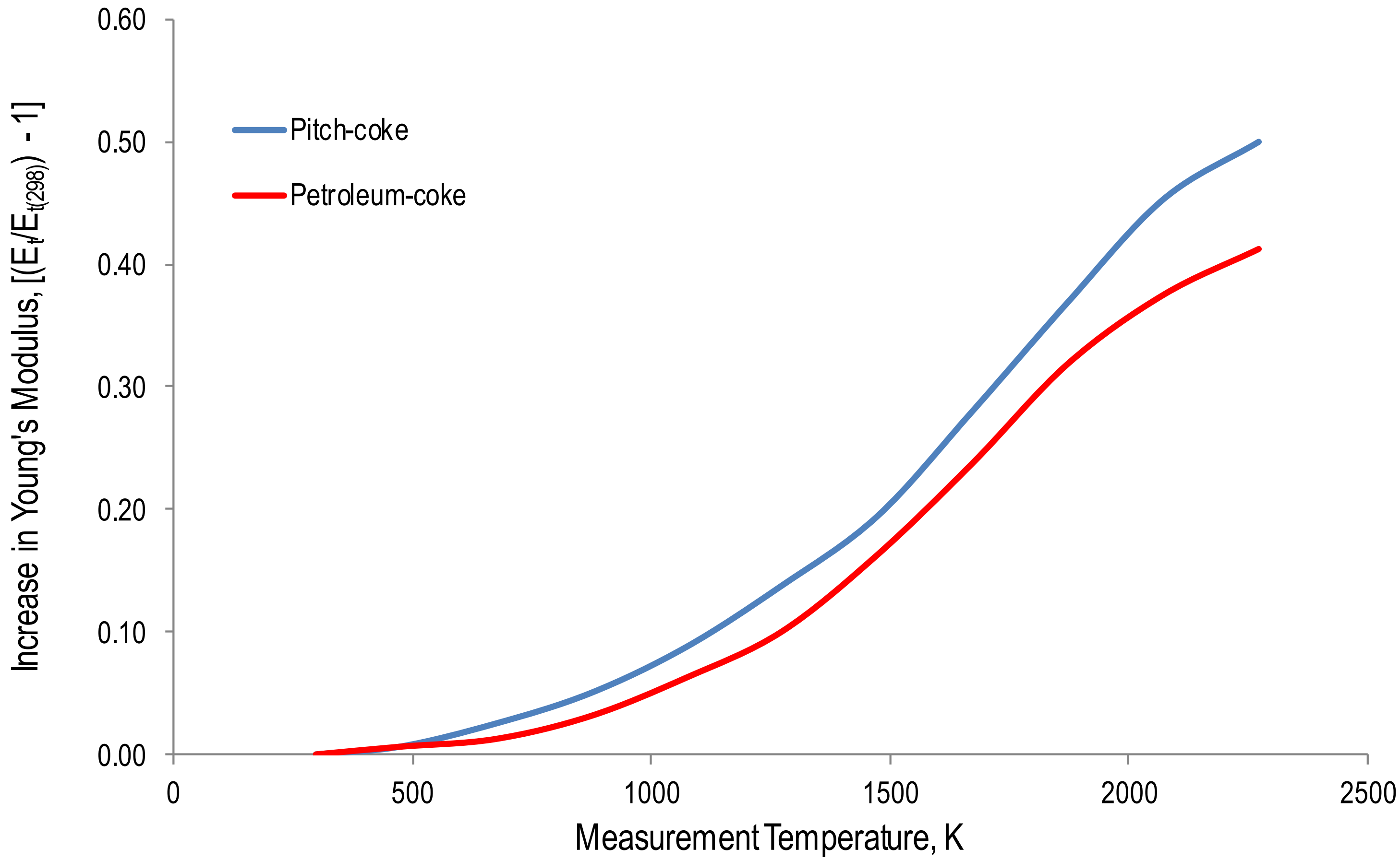
Typical Young's Modulus increase with temperature for pitch-coke and petroleum coke synthetic graphite



Stress-strain behavior of synthetic graphite

There are two major factors that control the stress-strain behavior of synthetic graphite:

- The magnitude of the constant C_{44}, which dictates how the crystals respond to an applied stress,
- The defect/crack morphology and distribution, which controls the distribution of stresses within the body and thus the stress that each crystallite experiences.

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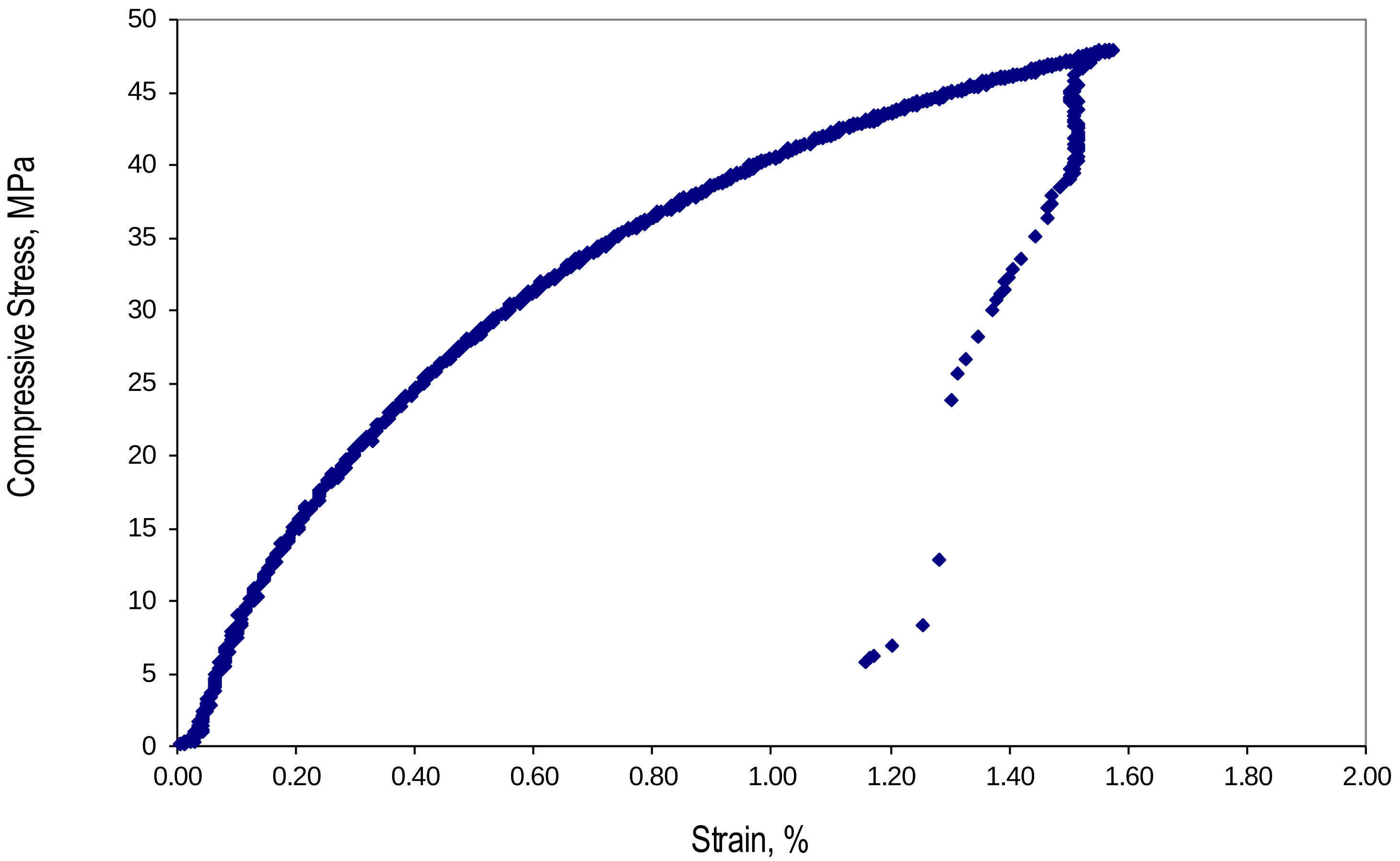
Typical compressive stress-strain curve for medium-grain extruded graphite (WG)



Non-linear stress strain curve

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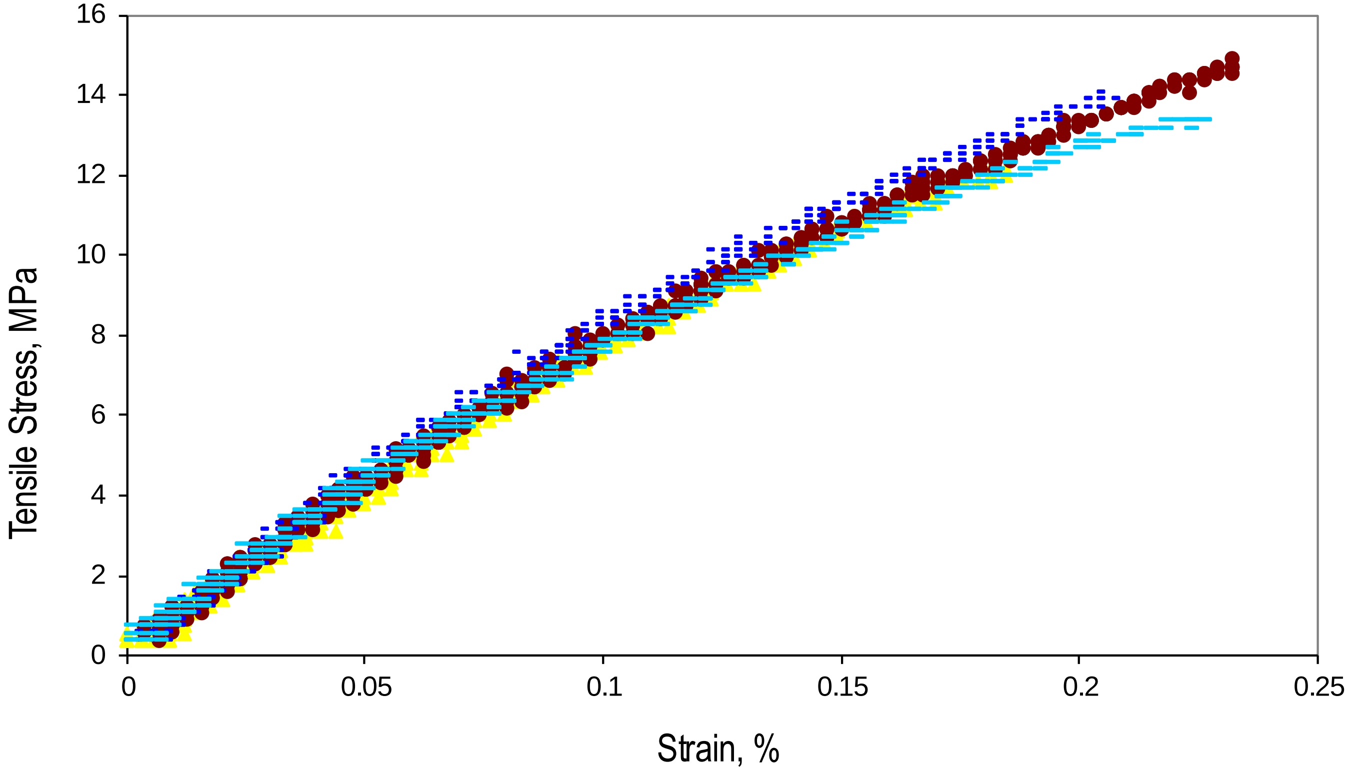
Typical tensile stress-strain curves for medium-grain extruded graphite (WG)



Non-linear stress strain curve

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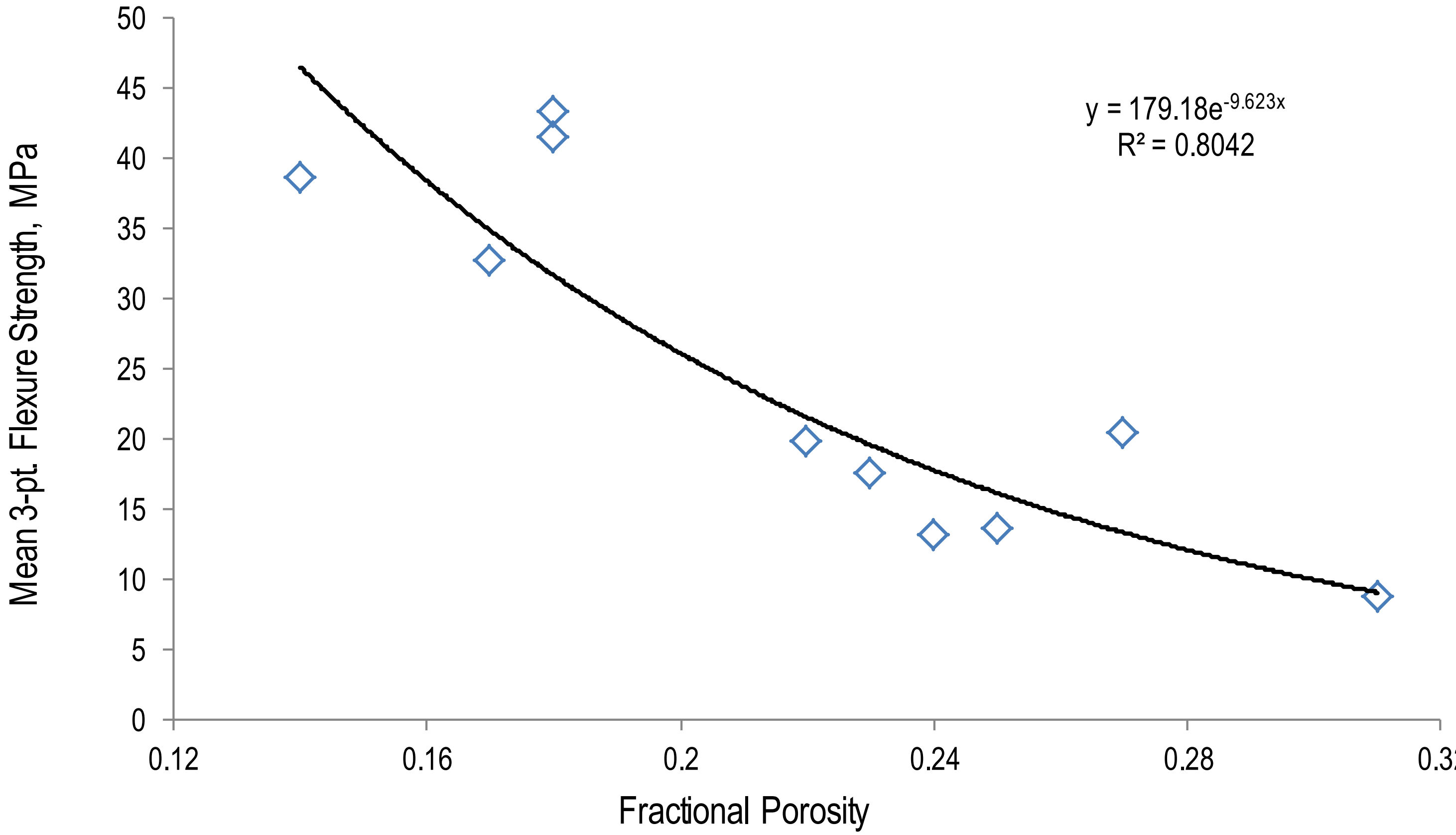
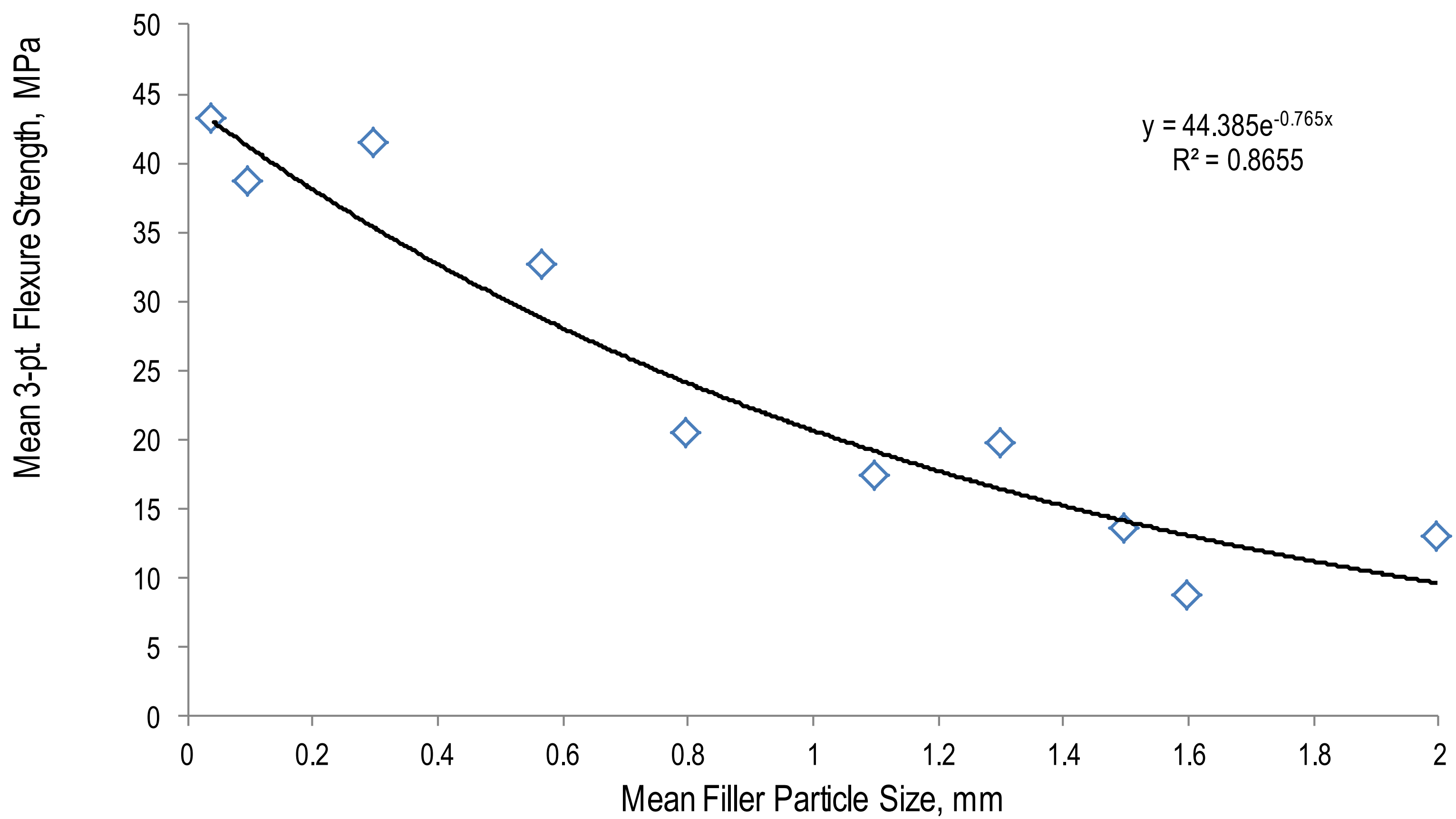
The correlation between mean 3-pt flexure strength and fractional porosity for a wide range of synthetic graphite representing the variation of textures



The correlation between mean 3-pt flexure strength and mean filler coke particle size for a wide range of synthetic graphite representing the variation of textures



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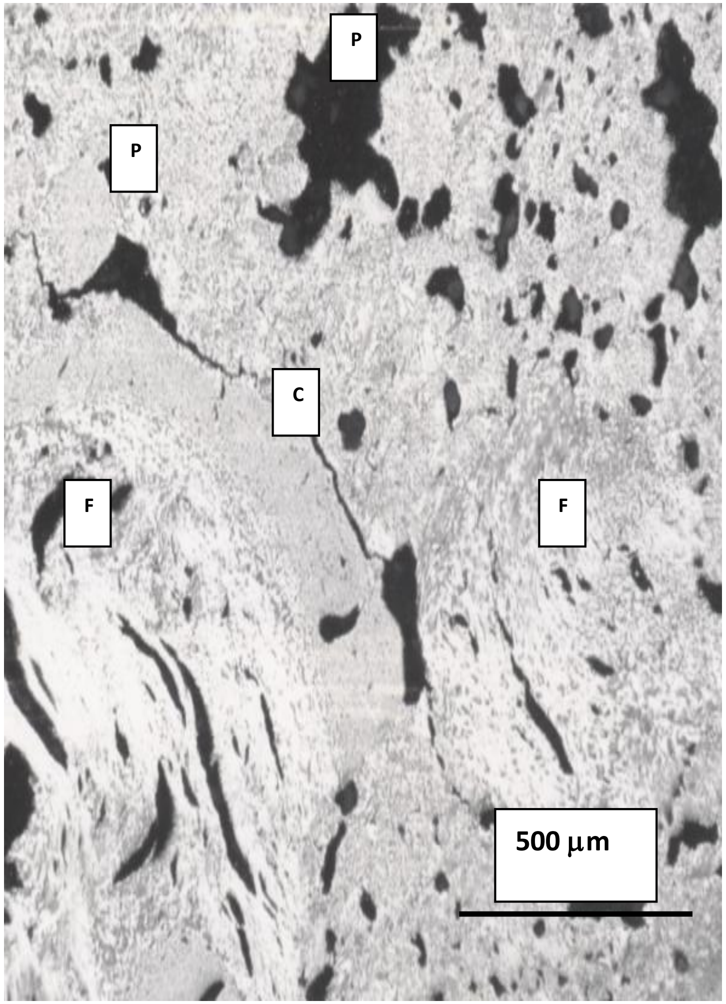
Crack Propagation in Synthetic Graphite



An optical photomicrograph of the microstructure of grade H-451 graphite revealing the presence of pores [P], coke filler particles [F] and crack [C] which have propagated through the pores presumably under the influence of their stress fields

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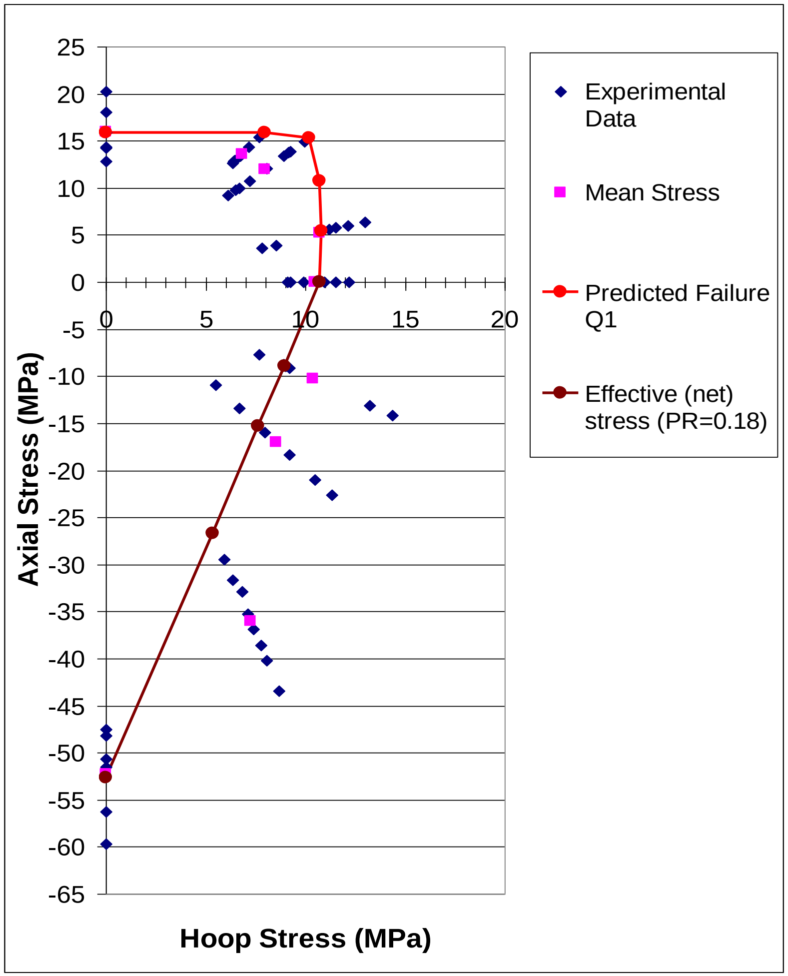
Graphite multiaxial strength behavior & model predictions



- Previous 1st and 2nd stress quadrant testing of H-451 & IG-110
- Extended to NBG-18 1st & 2nd stress quadrants
- NBG-18 modeled with Burchell model incorporating Shetty mixed mode fracture criterion
- NBG-18 3rd and 4th stress quadrant testing initiated at ORNL

Graphite thermal shock resistance

- Thermal Shock FOM, $\Delta_{th} = \frac{K\sigma}{\alpha E(1-\upsilon)}$

K is the thermal conductivity, σ the yield strength, α the thermal expansion coefficient, E the Young's modulus, and υ is Poisson's ratio

Material	FOM
Graphite, AXF-5Q	124,904
Graphite, IG-110	84,844
Wrought beryllium	$\sim 1\text{x}10^4$
Pure tungsten	$\sim 0.5\text{x}10^5$
Carbon-carbon composite	$\sim 1\text{x}10^6$

Graphite does not melt but rather sublimes at T>3300K

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Thank You!

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Section III, Division 5

High Temperature Reactors – Subsection HH

SUBPART A
GRAPHITE MATERIALS

GRAPHITE III – Environmental Effects

Overview of Presentation

I. Manufacture and Applications

II. Structure and Properties

- Single crystal and polycrystalline synthetic graphite
- Porosity and texture
- Physical Properties
  - Thermal
  - Electrical
- Mechanical Properties
  - Elastic constants
  - Strength and fracture

III. Reactor Environmental Effects

IV. ASME Code for Graphite

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Reactor Environmental Effects

- The following reactor environmental effects are considered:
  - Gas-coolant/graphite interactions (oxidation)
    - Acute (Air) oxidation: Chronic (moisture) oxidation: radiolytic oxidation
    - Primarily of concern in GCR
  - Salt coolant-graphite interactions
  - Radiation damage
    - Of concern to all graphite moderated HTRs

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Thermal Oxidation (Air and Moisture)

- Air/steam oxidation can occur in all graphite moderated reactors and will cause property degradation
- Air ingress accident
  - $C+O_2 \rightarrow CO_2$
  - $CO_2+C \rightarrow 2CO$
- Steam in Helium Coolant
  - $C + H_2O \rightarrow CO + H_2$
  - $C + 2H_2 \rightarrow CH_4$
- Oxidation = Loss of solid Carbon (Graphite)

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Thermal Oxidation (Air and Moisture)

- Properties degrade as a function of oxidative weight loss (burn-off)
- To predict burn-off we need to know:
  - Kinetics of oxidation reactions over the appropriate range of temperature and partial pressure (or concentration) of oxidizing species
  - Local partial pressure (or concentration) of oxidizing species within core/graphite block (Effective Diffusivity)
- Graphite purity also has an effect since some impurities act as oxidation catalysts

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Graphite Wear/Abrasion

- Tribological data are needed to establish wear of components
- Friction coefficients (in Helium, effect of pressure and temperature)
  - Graphite on graphite
  - Pebble on Pebble
  - Pebble on Graphite
- Wear rates need to be established
- Wear products (dust) represents a possible fission product transport mechanism

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Radiolytic Oxidation is not a Problem In He Cooled HTRGs

- $CO_2 + \gamma = CO_2^*$, an activated species that can oxidize carbon at reactor temperatures
- Radiolytic weight loss can degrade physical properties
- Special measures include gaseous phase inhibitors
- Helium cooled reactors are immune from radiolytic oxidation
- Air/steam oxidation can occur in all graphite moderated reactors and will cause property degradation

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Graphite For Molten Salt Reactors

What are the "issues" for salt cooled graphite moderated reactors?

- Salt permeation (into pores)
- Fission Gas build up
  - Xe^{135},Tritium
- Not challenged by damage dose or high temperature
- TRISO fuel retains fission products
- Tritium build-up in graphite if salt permeates graphite
- Hot spots if fueled salt is retained in pores
- What graphite should a Salt Cooled Reactor use?
  - fine Grained Generally < 50 μm
- Finer grains ≡ finer pores ≡ lower permeability

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Factors Controlling The Neutron Irradiation Damage Response Of Graphite

- Crystallinity (degree of graphitization): More graphitic crystals retain less displacement damage. Crystallinity is a function of precursor (pitch/coke) and graphitization temperature.
- Small crystallite sizes promotes higher strength and retardation of pore generation.
- Structural isotropy (both coke isotropy and final product isotropy). Isotropic irradiation behavior is much preferred. CTE ratio is used as an indication of isotropy. Higher coke CTE and graphite CTE preferred.
- Forming technique – structural and property anisotropy is introduced by extrusion and molding. Isostatic molding produces an isotropic graphite.

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Graphite – Physics and History

- In the beginning:- Manhattan Project - 1940s
- Needed a good nuclear moderator (C, H_2O, D_2O, Be) for experimental reactor
- Graphite available to Enrico Fermi & team for assembly of experimental piles to determine criticality
- Neutron moderator
  - **Thermalize fast neutrons to sufficiently low energies that they can efficiently fission $_{92}U^{235}$**
- Neutron reflector – returns neutrons to the active core
- Graphite (nuclear grade) has a low neutron capture cross section
- First "piles" at Pupin Laboratory, Columbia University, New York

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Graphite as a Nuclear Moderator

- Graphite for first reactors needed to be:
- Relatively low cost
- Sufficiently low neutron absorption cross section (high purity required)
- Available with low Boron content (ultimately < 0.5 ppm)
- Sufficient density (> 1.70 g/cc)
- Engineering material, adequate strength, available in sufficient quantity, readily machined

What was learned over the years flowed down to improved graphites:-

- Halogen purification (allowed alternate feedstock sources)
- Understanding of damage mechanism and role of graphite crystallite size
- Need for isotropic cokes - high CTE which yield isotropic properties in the final artifact
- **Thus second generation graphites were born**
  - **USA, H-451 – extruded, isotropic pet coke**
  - **UK, IM1-24 – molded, Gilsonite coke**

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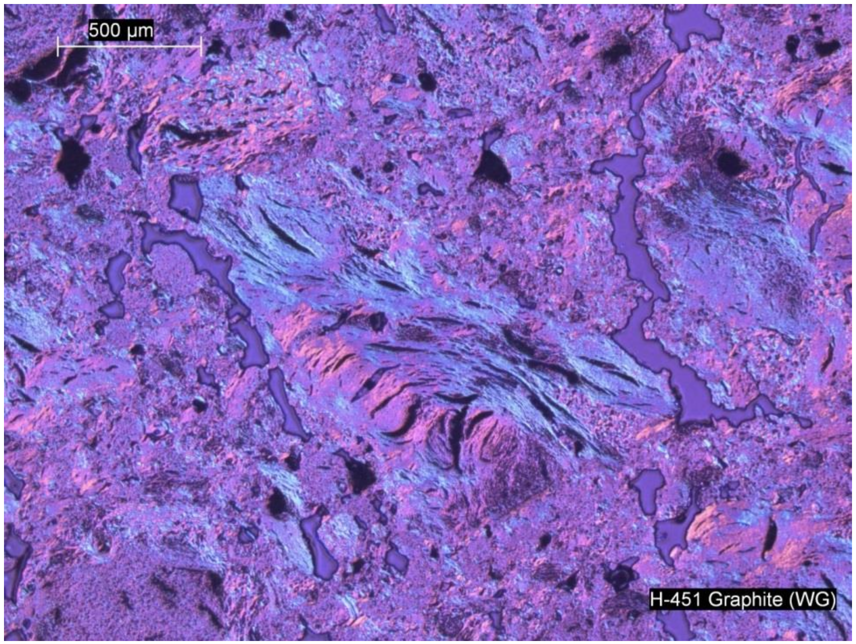
Near-isotropic graphites H-451



- Extruded, isotropic petroleum coke
- 500 µm mean filler particle size
- Near-isotropic physical properties
- High CTE & reasonable strength

Fuel elements and replaceable reflectors in the GA designed HTGR (FSV) and GT-MHR

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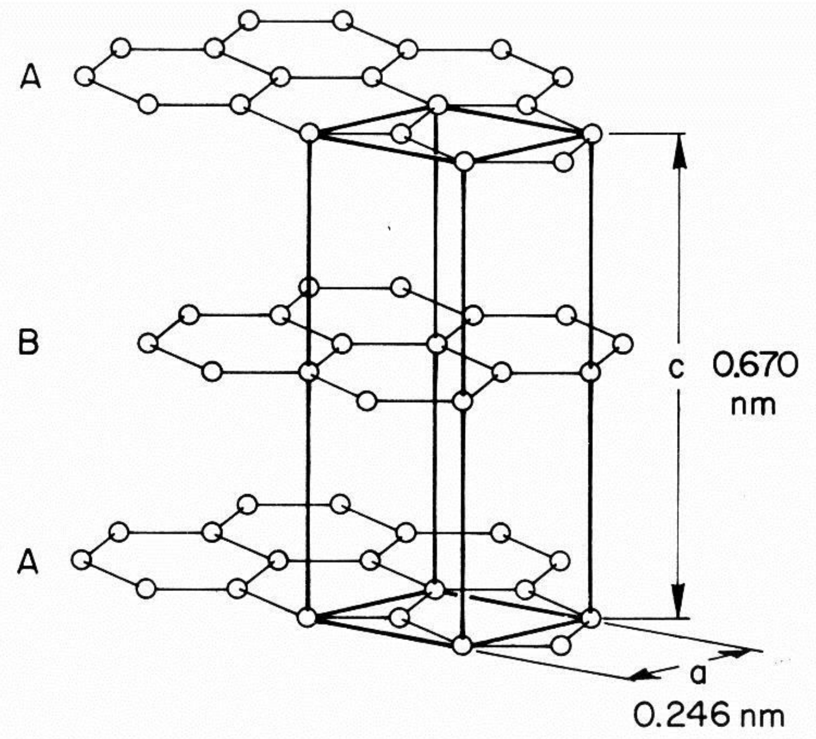
Graphite Crystal Structure



- Carbon atoms in a hexagonal structure
- Covalent (strong) bonding in basal plane
- Van der Waals (weak) bonding between the planes
- ABAB stacking sequence
- Bond anisotropy and crystal perfection impart unique combination of properties
- In-plane conductor
- Cross-plane insulator

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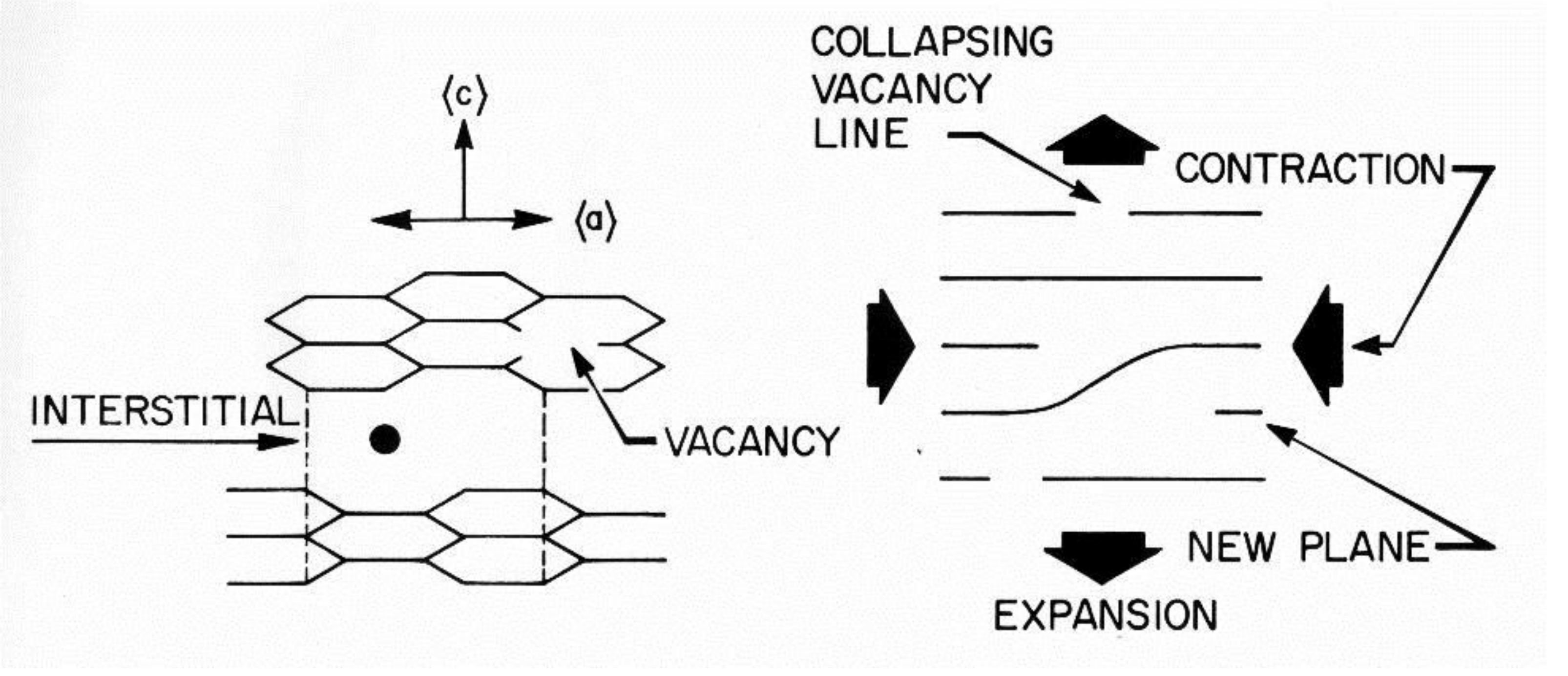
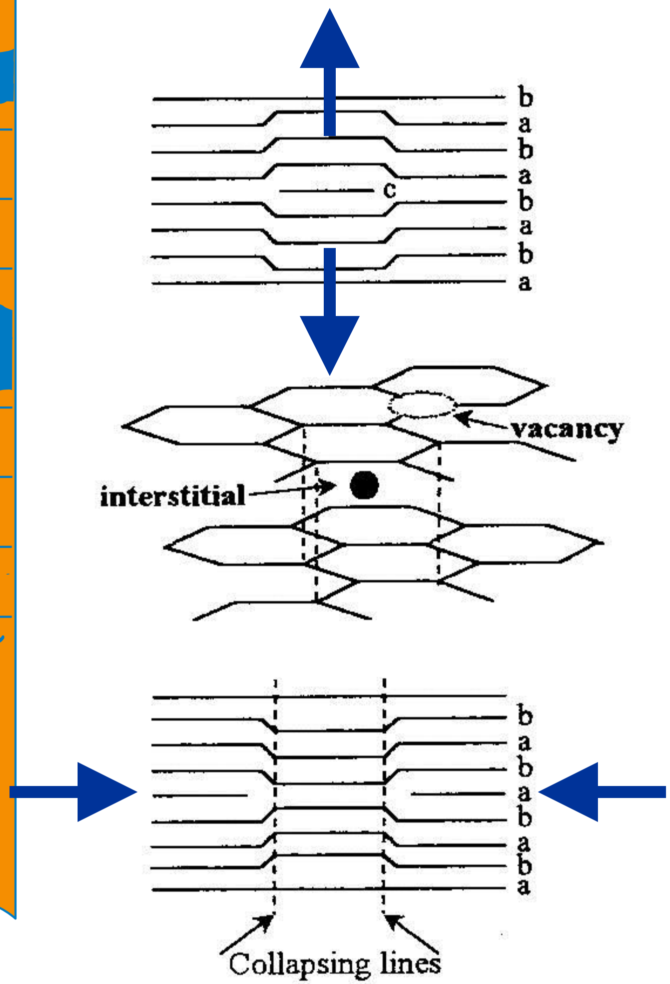
The Radiation Damage Mechanism In Graphite



Displacement energy for carbon atom is approx. 30 eV

Radiation Damage In Graphite Is Temperature Dependent



- INTERSTITIALS
- Mobile at room temperature.
- Above 100^{o}C form into clusters of 2 to 4 interstitials.
- Above 300^{o}C form new basal planes which continue to grow at temperatures up to 1400^{o}C.

- VACANCIES
- Immobile below 300^{o}C.
- 300-400^{o}C formation of clusters of 2-4 vacancies which diffuse in the basal planes and can be annihilated at crystallite boundaries (function of lattice strain and crystal perfection).
- Above 650^{o}C formation of vacancy loops.
- Above 900^{o}C loops induce collapsing vacancy lines.

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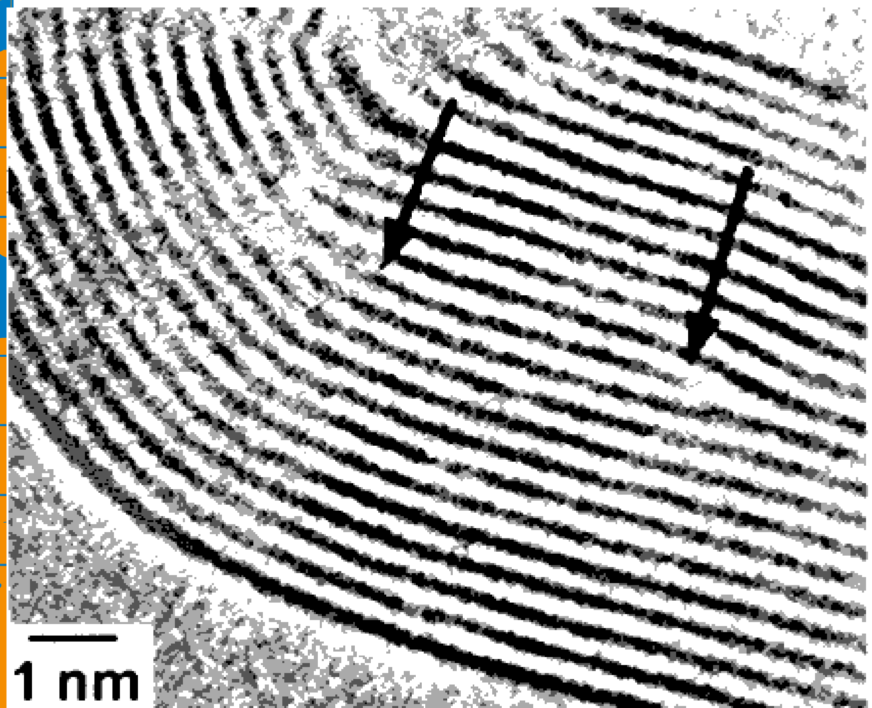
Basal Planes in Layered Graphitic Structures



A high-resolution electron micrograph showing the basal planes of a graphitic nano-particle with an interstitial loop between two basal planes, the ends of the inserted plane are indicated with arrows.

Banhart, F. *Rep. Prog. Phys.* **1999**, 62, 1181–1221.

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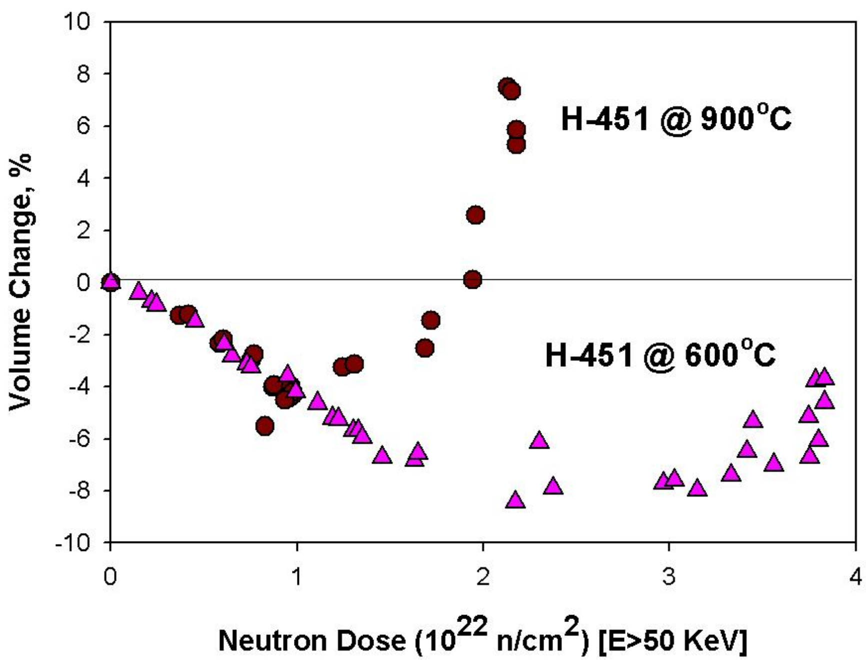
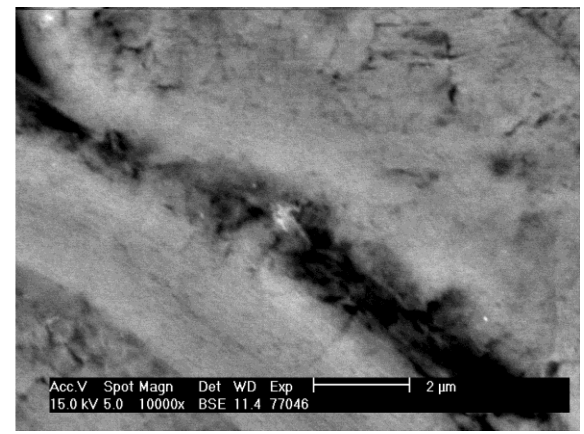
Volume Changes In H-451 (Effect Of Temperature)





Interplaner cracks and porosity accommodate thermal expansion and c-axis irradiation induced swelling

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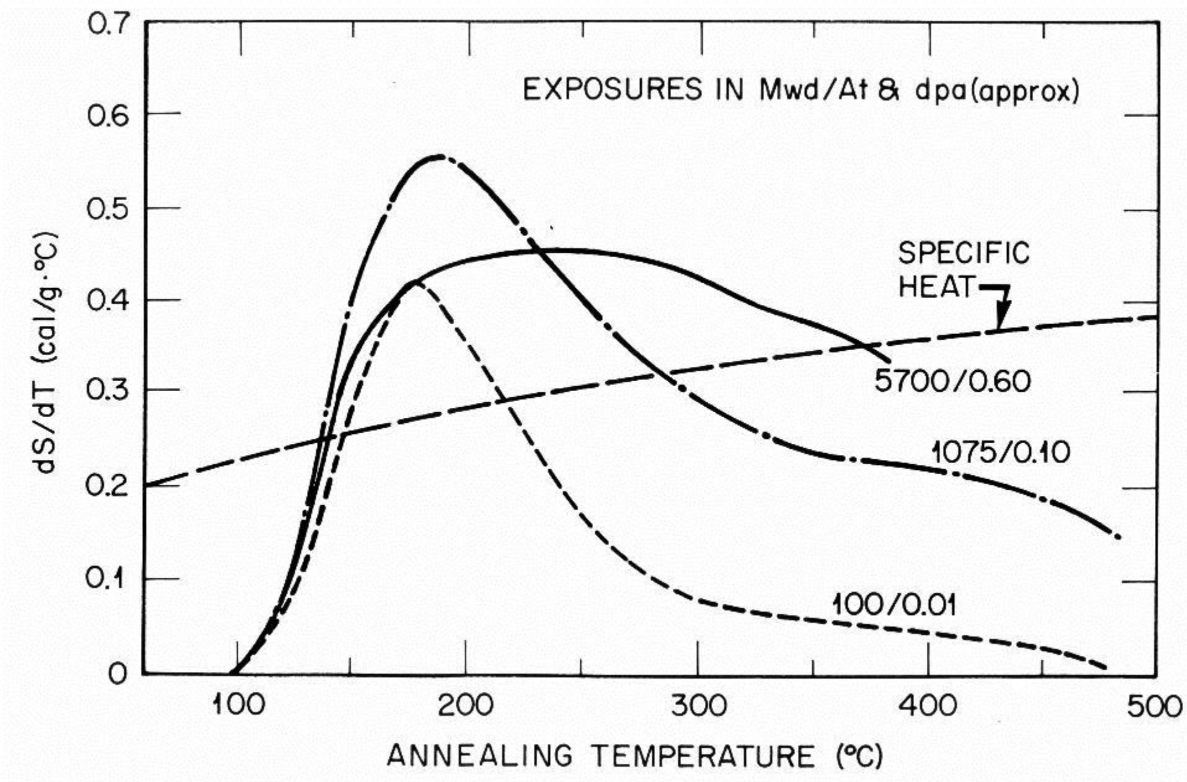
Low Temperature Stored Energy Release

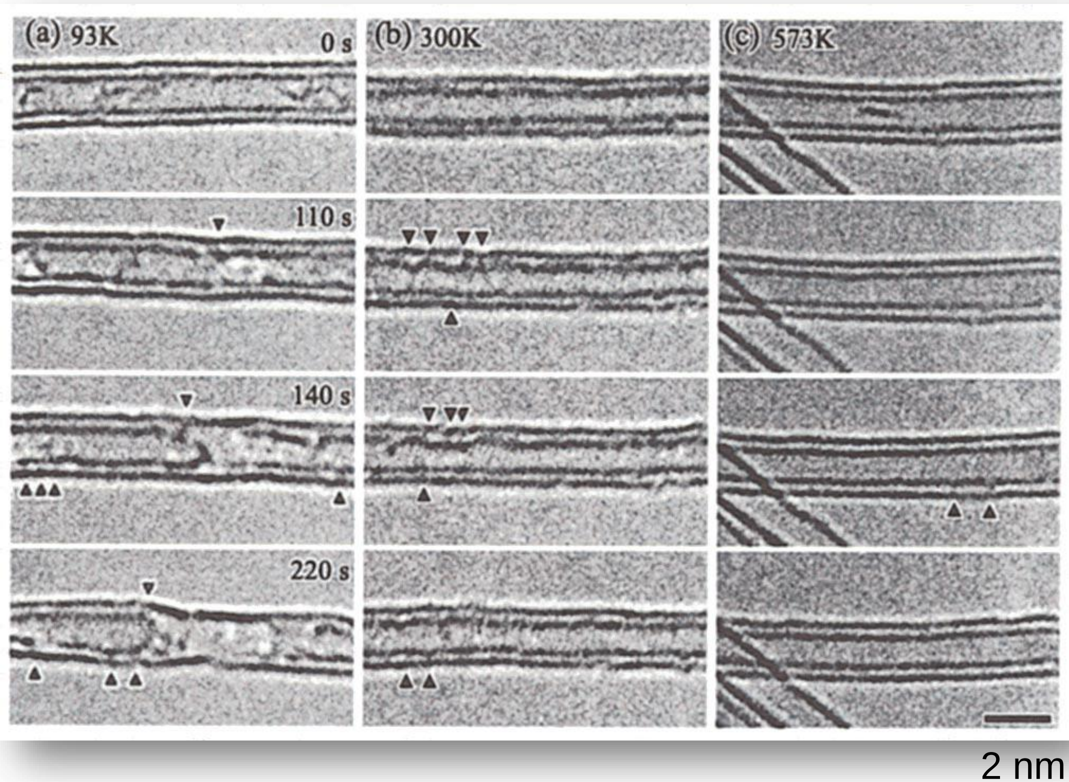


- T_{irr} ~ 30°C
- Hanford K
- Reactor test
- Data
- Traditionally associated with Frenkel pair recombination
- *New evidence?*

Burchell T, Carbon Materials for Advanced Technologies, Chpt. 13 (1999) p. 429

[Adapted from Nightingale, Nuclear Graphite (1962)]

Displacement Damage in Layered Graphitic Structures



Urita, K.; Suenaga, K.; Sugai, T.; Shinohara, H.; Iijima, S. *Physical Review Letters* **2005**, 94, 155502.

Sequential high resolution transmission electron microscope images illustrating the formation rates of interlayer defects at different temperatures with the same electron irradiation flux & time scale (0 to 220 seconds). (a) 93K, (b) 300K, (c) 573K, in double-wall carbon nanotubes.

The arrows indicate possible interlayer defects.

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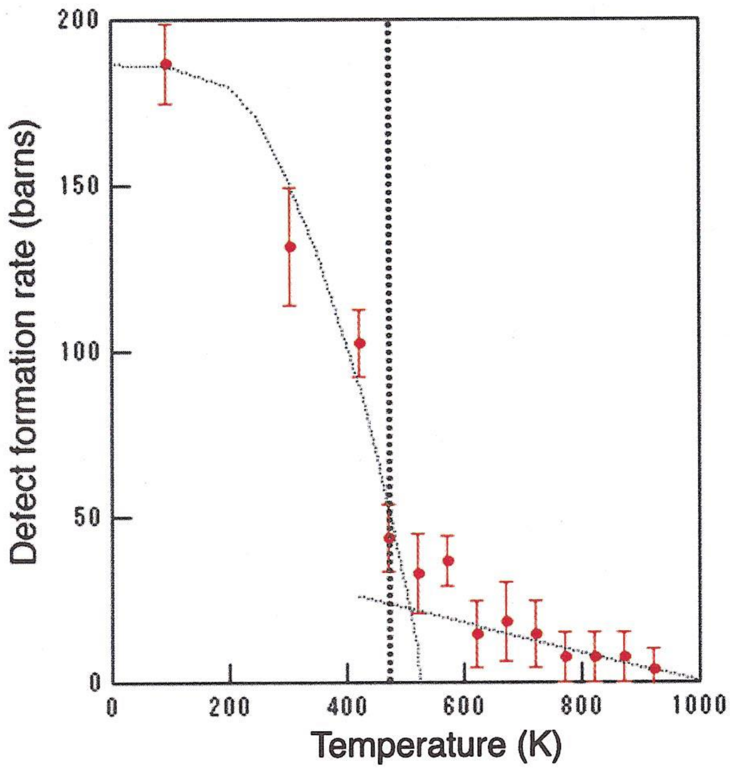
Displacement Damage in Layered Graphitic Structures



- Normalized formation rate of the clusters of *I-V* pair defects per unit area of bilayer estimated in HRTEM images recorded at different temperatures
- The dotted line shows the known temperature for Wigner-energy release (~473 K)
- Heggie & Telling, University of Sussex, UK: Simulations of spiro-interstitial

Urita, K.; Suenaga, K.; Sugai, T.; Shinohara, H.; Iijima, S. *Physical Review Letters* **2005**, 94, 155502.

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Neutron Irradiation Induced Dimensional Change

- Graphite dimensional changes are a result of crystallite dimensional change and graphite texture.
- Swelling in c-direction is initially accommodated by aligned microcracks that form on cooling during manufacture.
- Therefore, the a-axis shrinkage initially dominates and the bulk graphite exhibits net volume shrinkage.
- With further irradiation, incompatibilities in crystallite strains causes the generation of new porosity and the volume shrinkage rate falls eventually reaching zero.

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Neutron Irradiation Induced Dimensional Change (continued)

- The graphite begins to swell at an increasing rate with increasing damage dose due to c-axis growth and new pore generation.
- The graphite thus exhibits volume “turnaround” behavior from initial shrinkage to growth.
- Eventually loss of mechanical integrity occurs due to excessive pore/crack generation.

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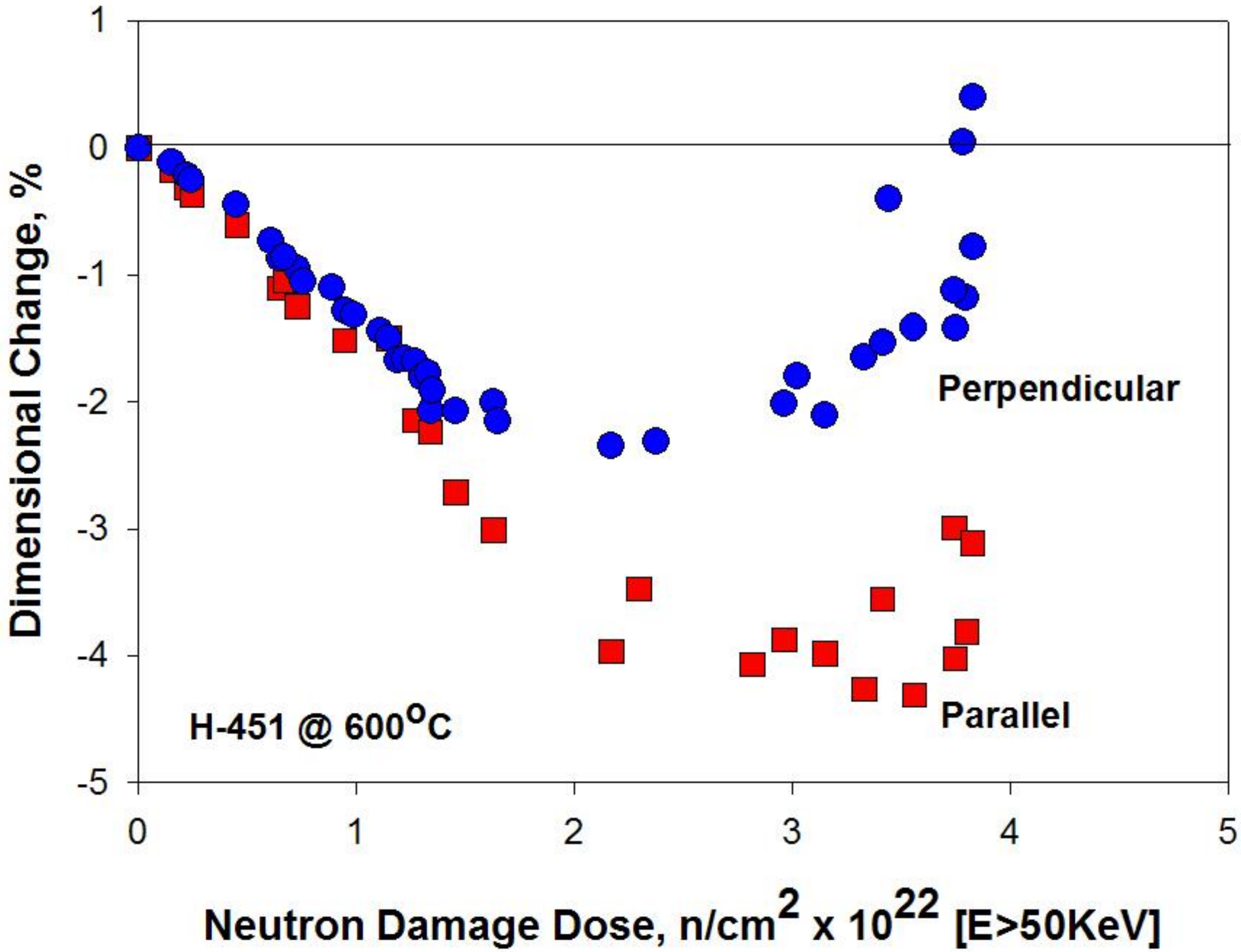
Radiation Induced Dimensional Changes in H-451 (Effect of Texture)



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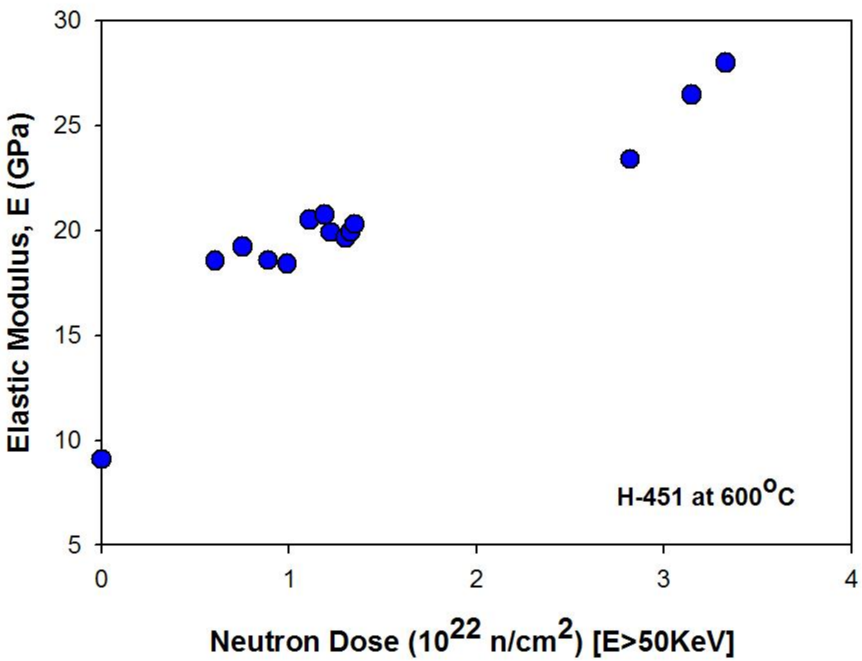
Neutron Irradiation Induced Changes in Young's Modulus



- Initial rise due to dislocation pinning
- Subsequent increase due to volume shrinkage (densification)
- Eventual turnover and reduction due to pore/crack generation and volume expansion
- $\sigma \alpha (E)^{1/2}$

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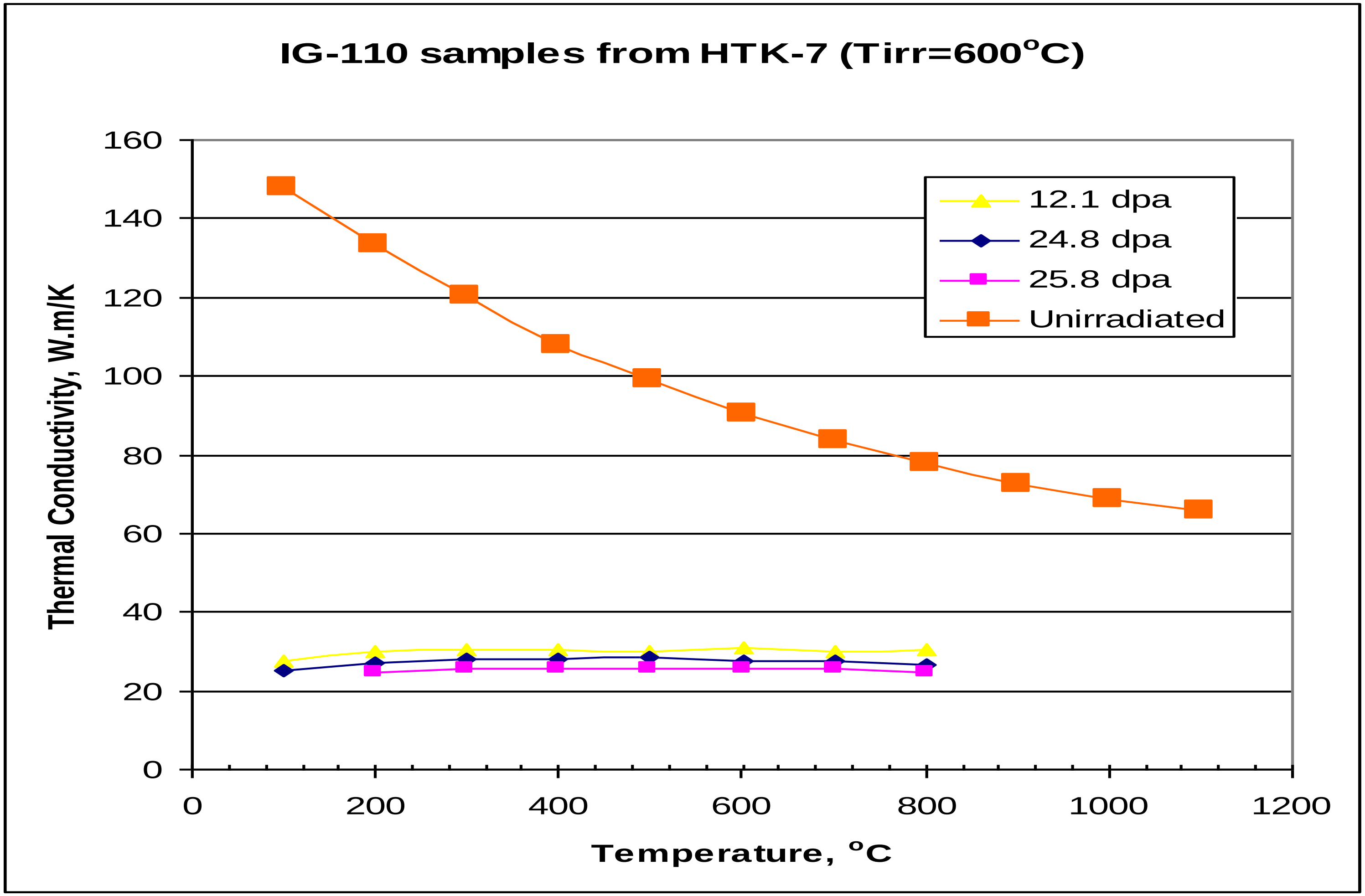
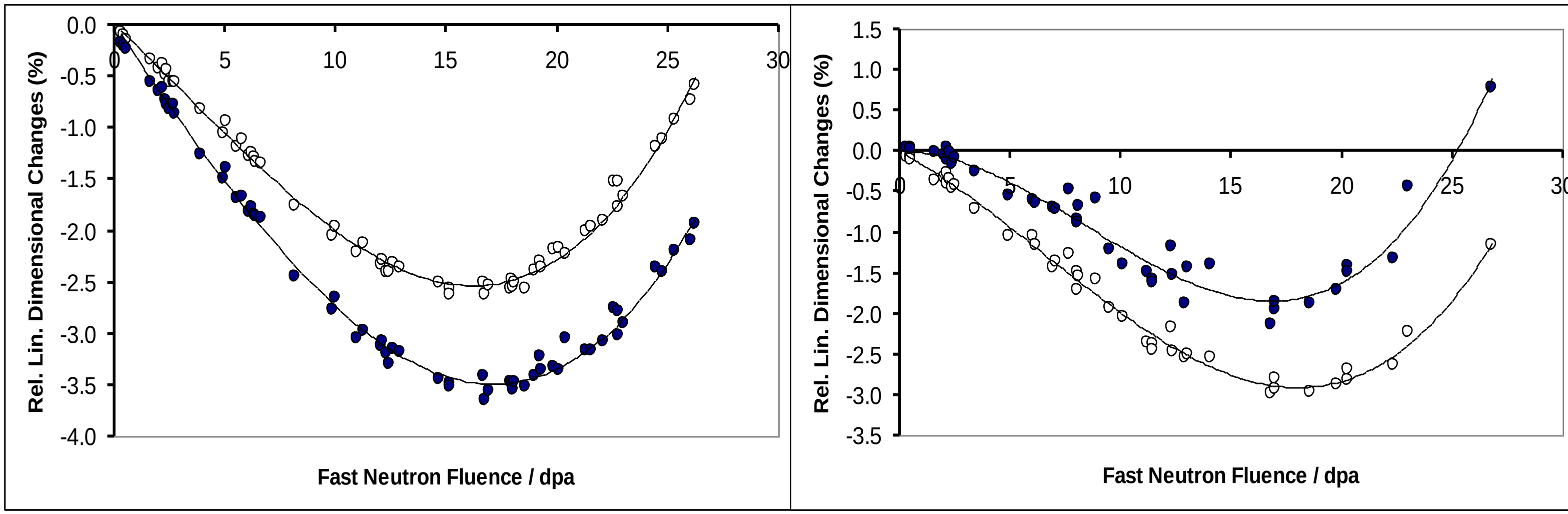
Thermal Conductivity Changes Umklapp and Defect Scattering



Radiation Damage in Nuclear Graphite –*inelastic deformation*

Irradiation Induced Creep in Graphite



ATR-2E Graphite (WG), T_{irr} = 550°C, 5 MPa compressive stress

ATR-2E Graphite (WG), T_{irr} = 500°C, 5 MPa tensile stress

Graphite dimensional change behavior is modified by the application of stress. Tensile stress hastens turnaround and compressive stress delays turnaround

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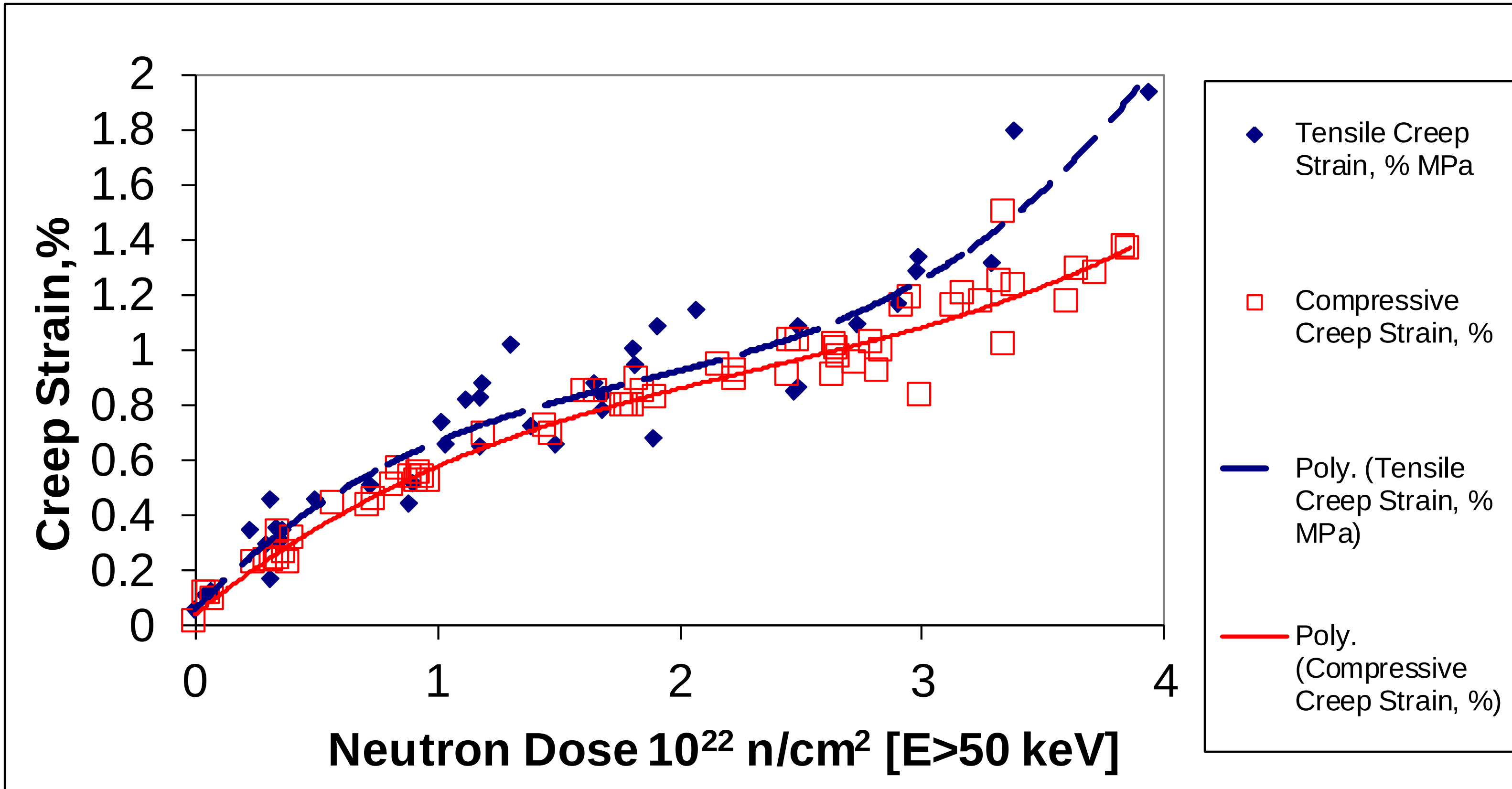
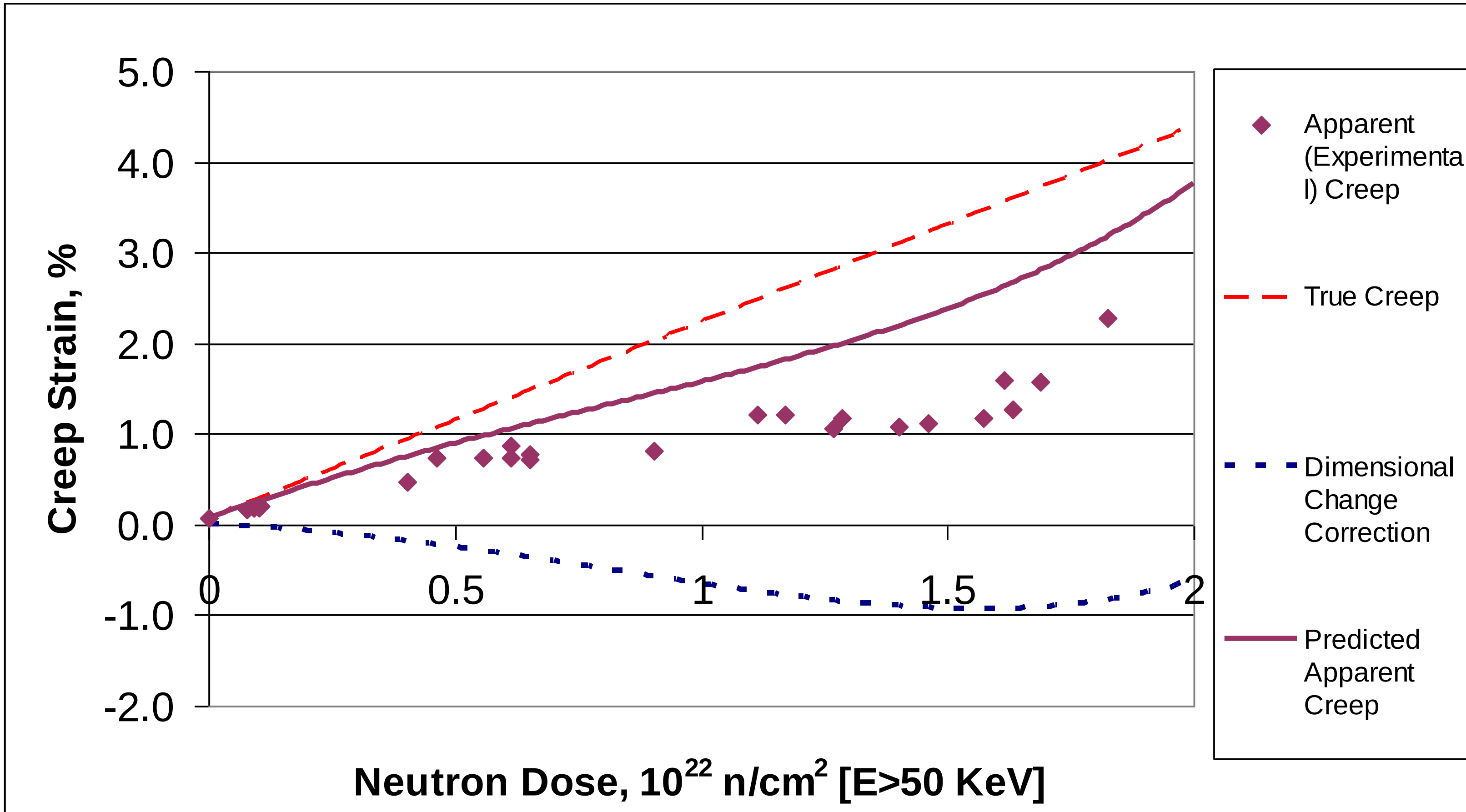
A Comparison of Compressive and Tensile Creep Strain Behavior for ATR-2E Graphite



G. Haag. Report No. Jul-4183, FZ-J Germany

Irradiation Temperature =500-550^{o}C

Comparison of predicted apparent and the experimental creep strain data for irradiation creep at 900°C under a tensile stress of 6 MPa.



T.D. Burchell, J. Nucl. Mater. 381 (2008) 46-54

New Revision to Kelly-Burchell Model – Pore Generation Recognized

- Need to account for pore generation at higher doses
- Pores that affect CTE (already accounted for in the Kelly-Burchell model)
- Pores that do not affect CTE
- Fx and Fx′ needs to be evaluated
- The sign of the ΔFx′ term changes with the sign of the applied creep stress

$$\varepsilon_c' = \left(\frac{\sigma}{E_0} + k\sigma\gamma\right) + \int_0^{\gamma}\left(\frac{\alpha_x' - \alpha_x}{\alpha_c - \alpha_a}\right)\left(\frac{dX_T}{d\gamma}\right)d\gamma - \int_0^{\gamma}\left[\Delta F_x\right]d\gamma$$

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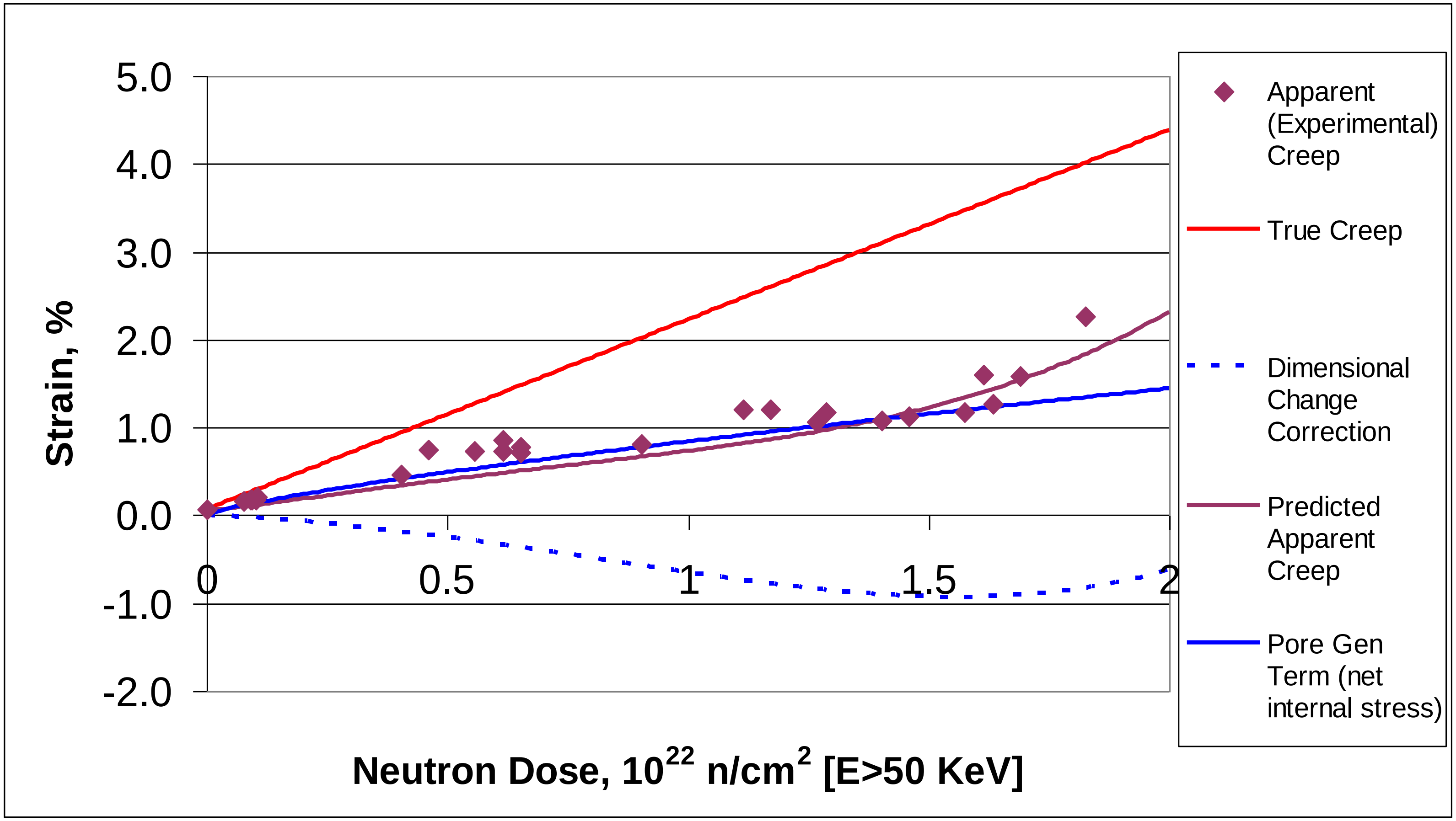
Revised Model with ΔFx' term evaluated



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Nuclear Graphite - The Future

- Fine filler particles with well ordered crystal structure (choice of precursor pitch, graphitization temperature)
- Green coke technology (iso-molding)
- Secondary cokes (BAN – GrafTech Inc.)
- Possible increased use of recycled graphite to minimize disposal and storage
- Irradiated graphite crushed and annealed then reformed and reused (super BAN)
- High dose irradiation experiments (irradiation creep)
- Fundamental studies of damage & creep mechanism

It is time for some Materials Science

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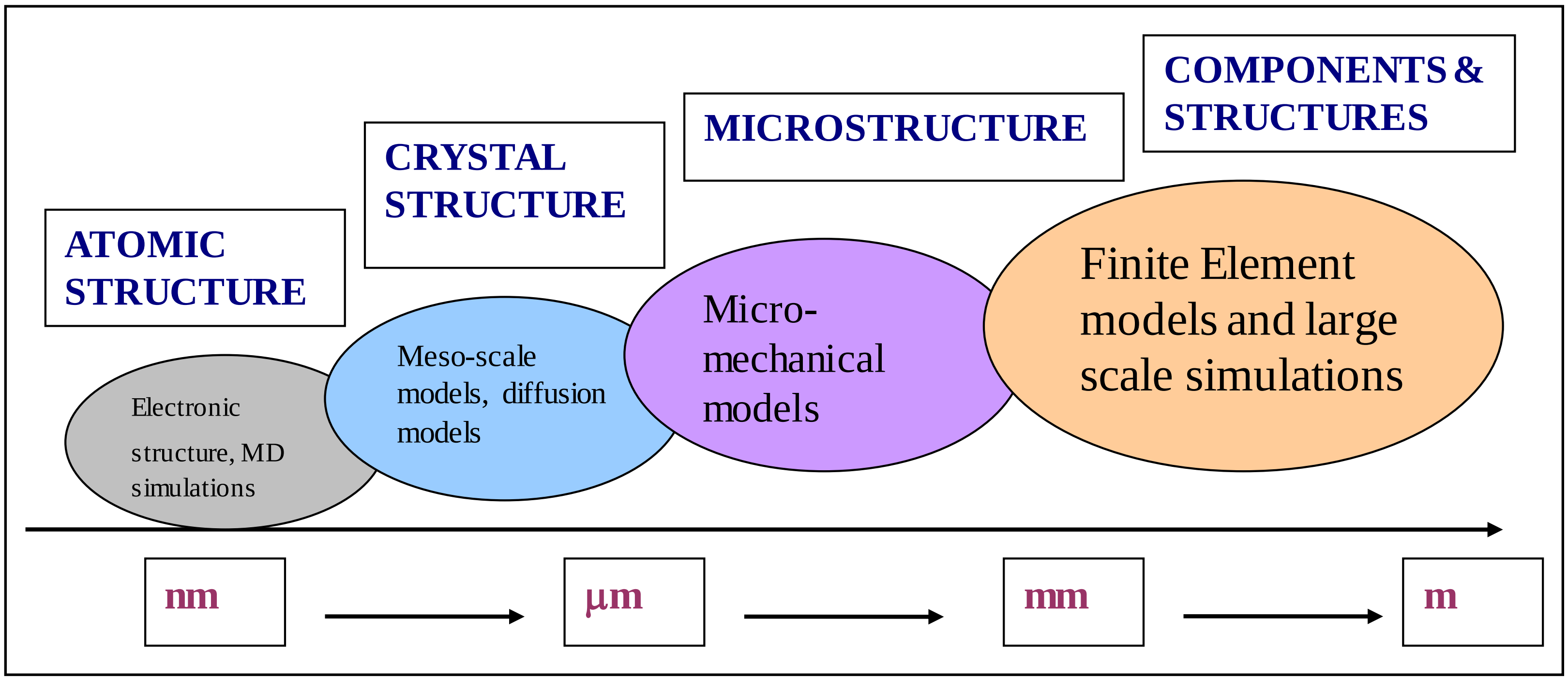
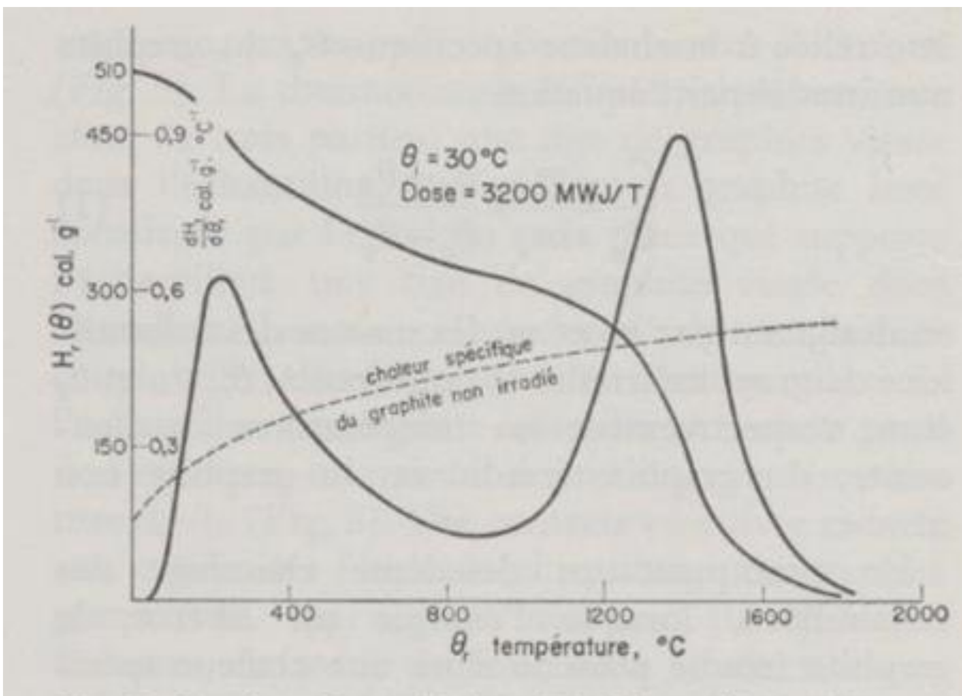
Knowledge and multiscale models linking to describe complex materials behavior from the sub-nanoscale to the millimetric (component) scale



High Temperature Stored Energy Release

Stored Energy Release Curve for Graphite Irradiated at 30^{o}C Compared with Unirradiated Graphite Cp Curve



Rappeneau et al, CARBON 9 (1966) 115-124

- A second release peak is observed at ~1400^{o}C in graphite irradiated at LOW temperatures
- Associated with annealing of small interstitial clusters
- Immobile vacancies can coalesce at high temperature
- Release rates > Cp NOT reported in graphite irradiated at higher temperatures

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Section III, Division 5

High Temperature Reactors – Subsection HH

SUBPART A
GRAPHITE MATERIALS

GRAPHITE IV – ASME Code for Graphite

Overview of Presentation

I. Manufacture and Applications

II. Structure and Properties

- Single crystal and polycrystalline synthetic graphite
- Porosity and texture
- Physical Properties
  - Thermal
  - Electrical
- Mechanical Properties
  - Elastic constants
  - Strength and fracture

III. Reactor Environmental Effects

IV. ASME Code for Graphite

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ASME Code for Graphite Introduction & Contents

- Presents information on the rules for the design and construction of graphite core component of a High Temperature Reactor
- Contents
  - HTRs and their Graphite Core Components (GCC)
  - Structure of the Code
  - Criteria for design of Graphite Core Components
    - GCC & Reactor Safety Case
    - Modes of failure, stress categories ands tress limits
    - Comparison of design margins
    - Verification of design method

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Role of Graphite in a Nuclear Reactor

- Neutron moderator (carbon & graphite)
  - Thermalize fast neutrons to sufficiently low energies that the can efficiently fission ${}_{92}U^{235}$
- Neutron reflector – returns neutrons to the active core
- Graphite (nuclear grade) has a low neutron capture cross section
- High temperature material

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Role of Graphite in a Nuclear Reactor

- Graphite is the reactor core structural material
- HTGR cores are constructed from graphite blocks and do not form a pressure boundary
- In prismatic cores the graphite fuel elements retain the nuclear fuel
- In a pebble bed the graphite structure retains the fuel pebbles
- The graphite reflector structure contains vertical penetrations for reactivity control
- Reactivity control is also in graphite fuel elements

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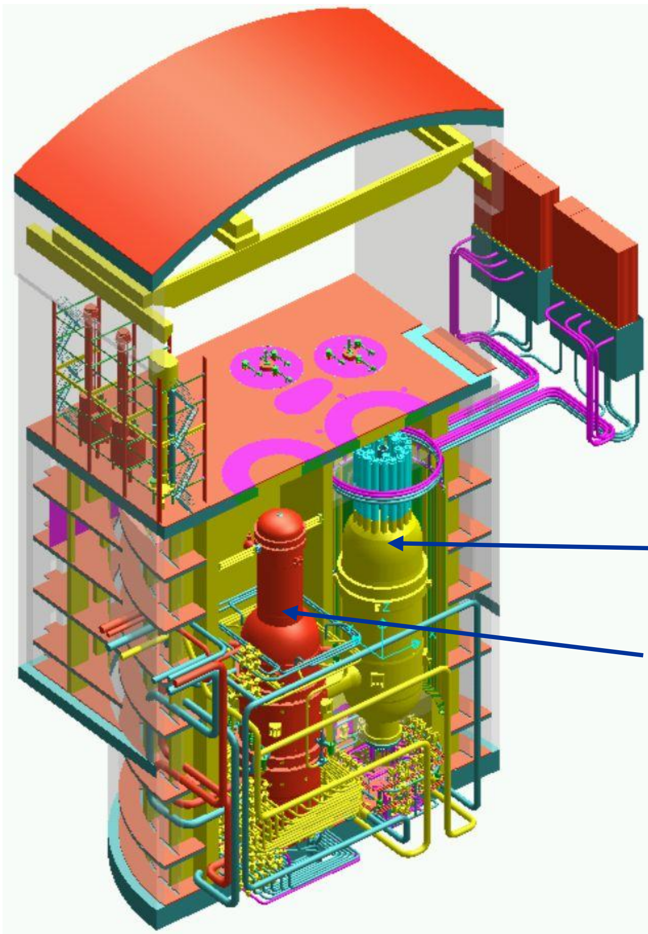
Gas Turbine-Modular Helium Reactor (GT-MHR)

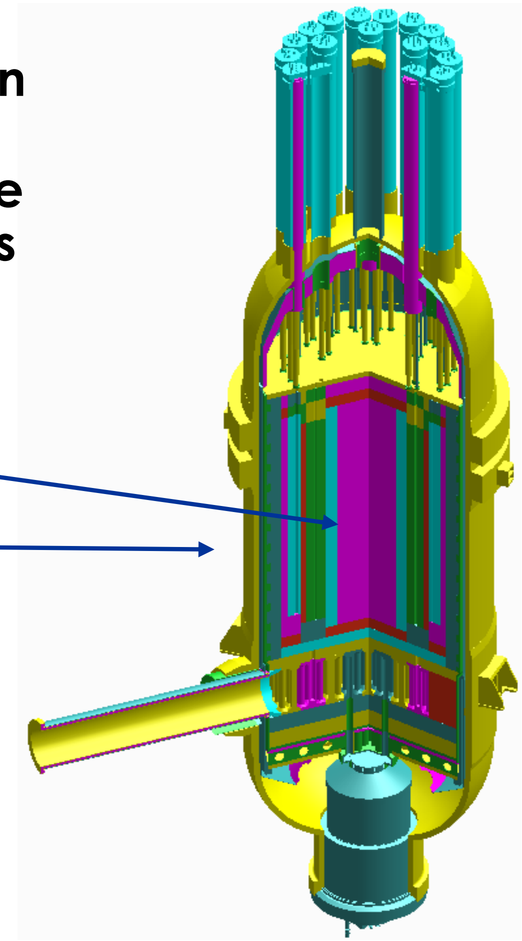


> 50% conversion efficiency (Brayton Cycle) or 1000°C outlet temperature Helium for process heat applications (H_2 production)

Graphite core

Reactor

Helium Gas Turbine-Generator or Intermediate Heat Exchanger



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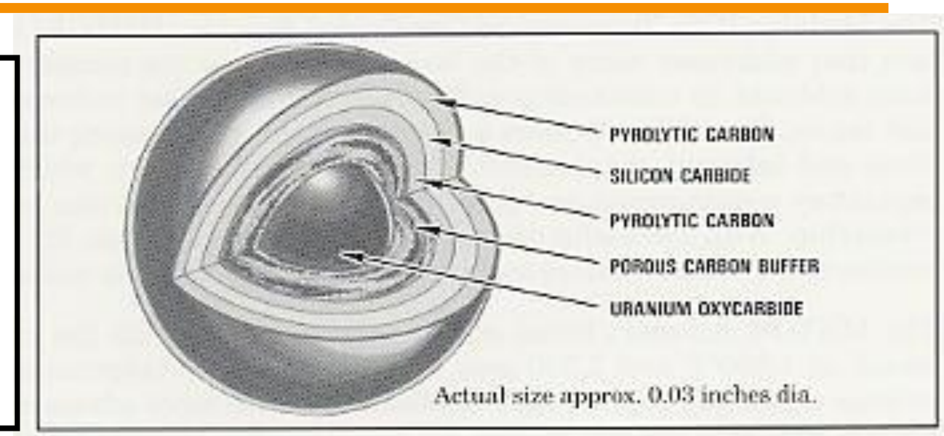
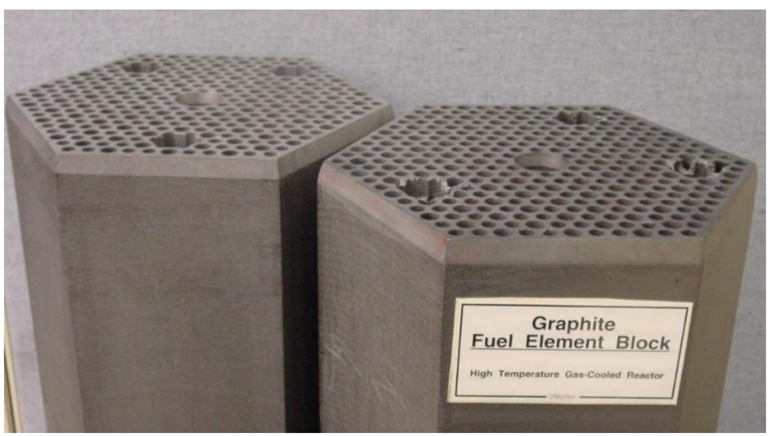
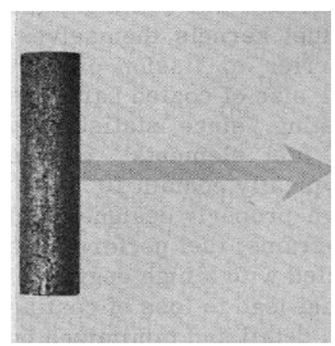
ASME Code for Graphite

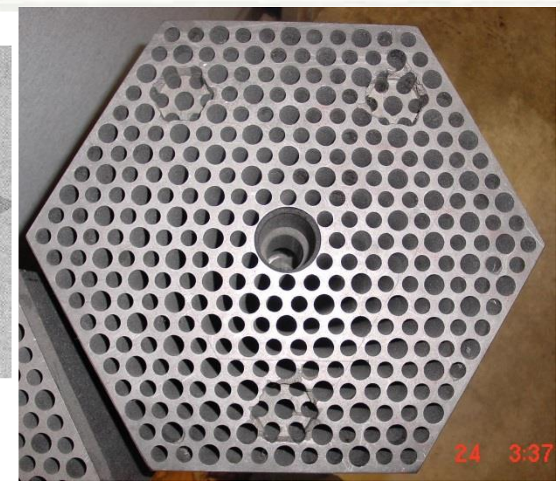
The GT-MHR Utilizes Ceramic Coated Particle Fuel

The TRISO fuel particles are formed into 12 mm diameter graphite (carbon) fuel sticks and inserted into graphite fuel blocks



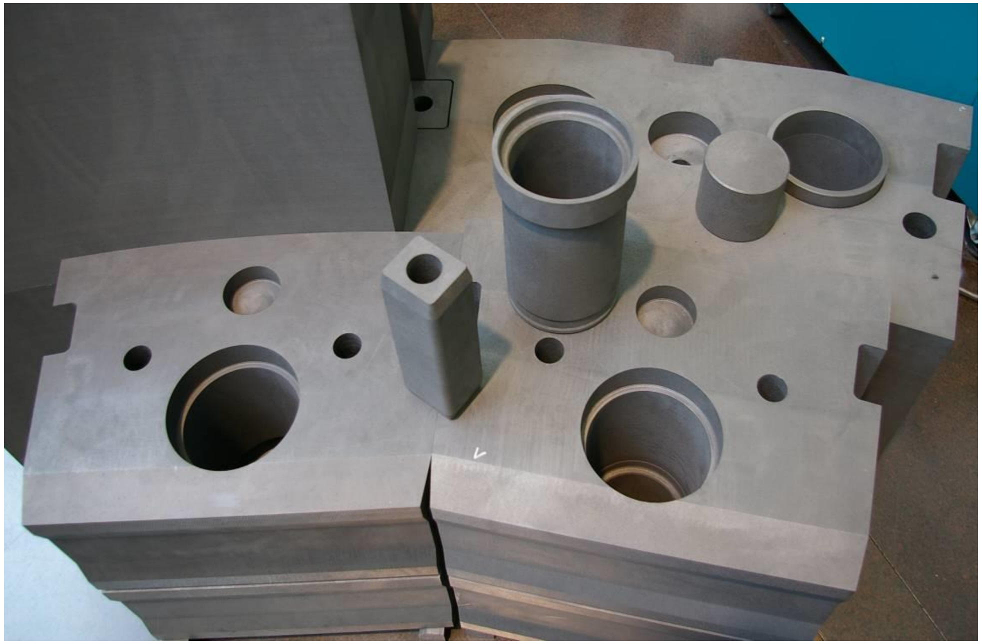


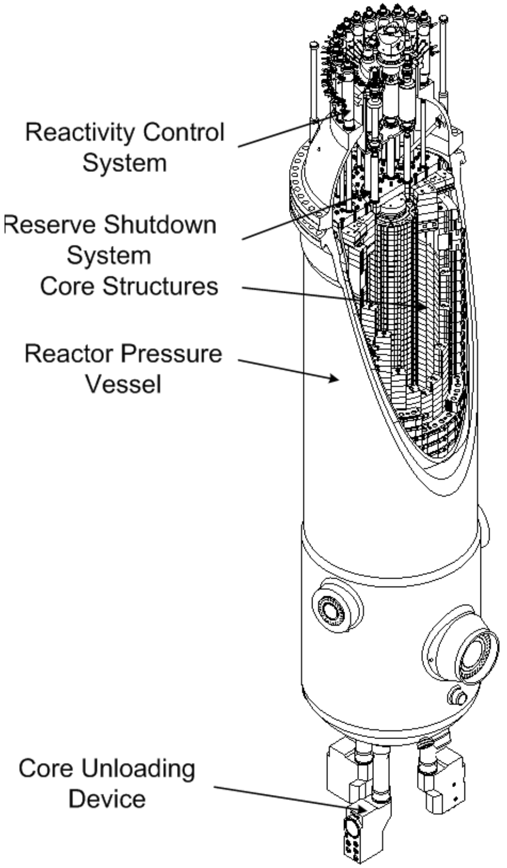




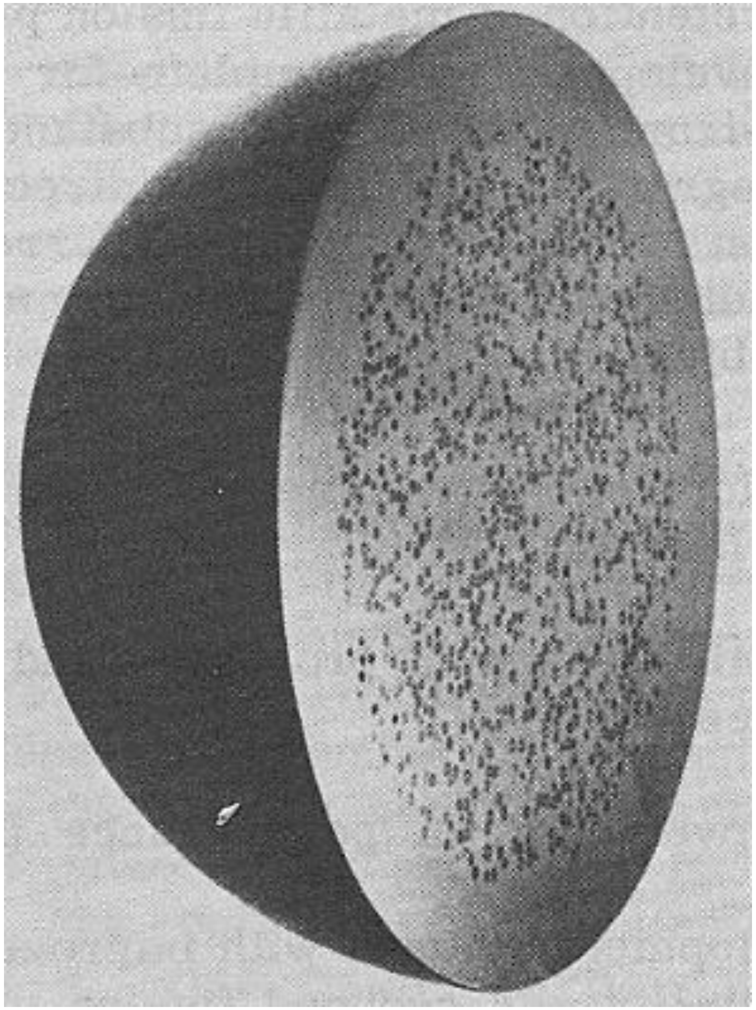
ASME Code for Graphite

- Graphite Core Components – Pebble Type HTR (PBMR)

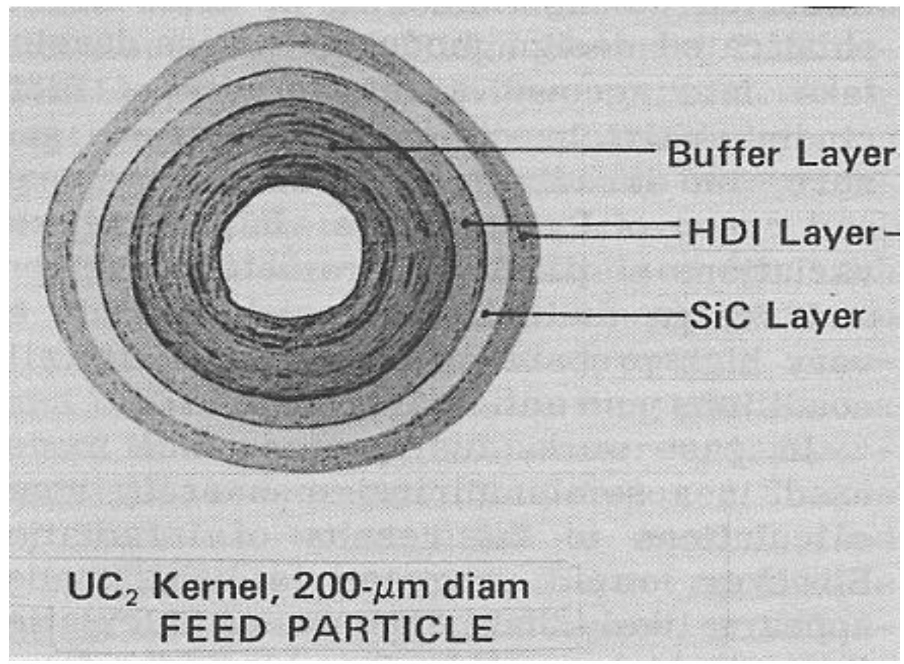




The Pebble Type HTR Utilizes Ceramic Coated Particle Fuel



The TRISO fuel particles are combined into a graphite (carbon) fuel ball (pebble) 6 cm in diameter



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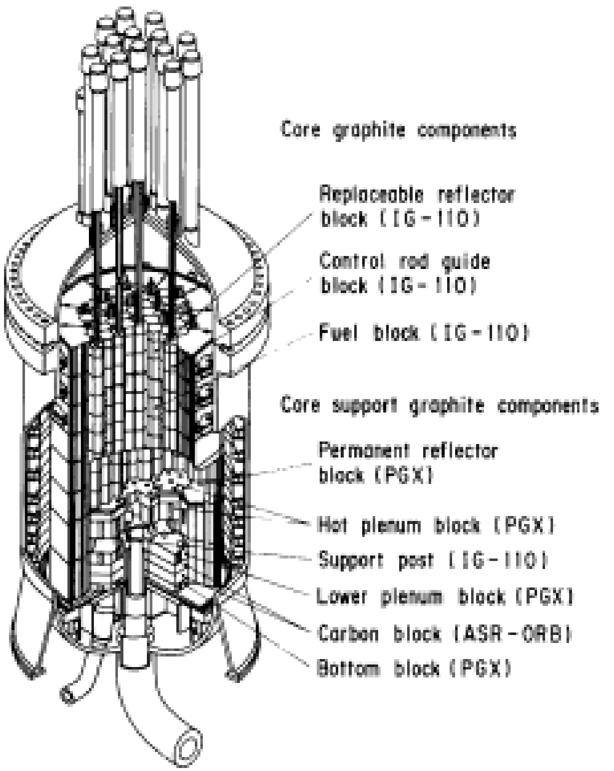
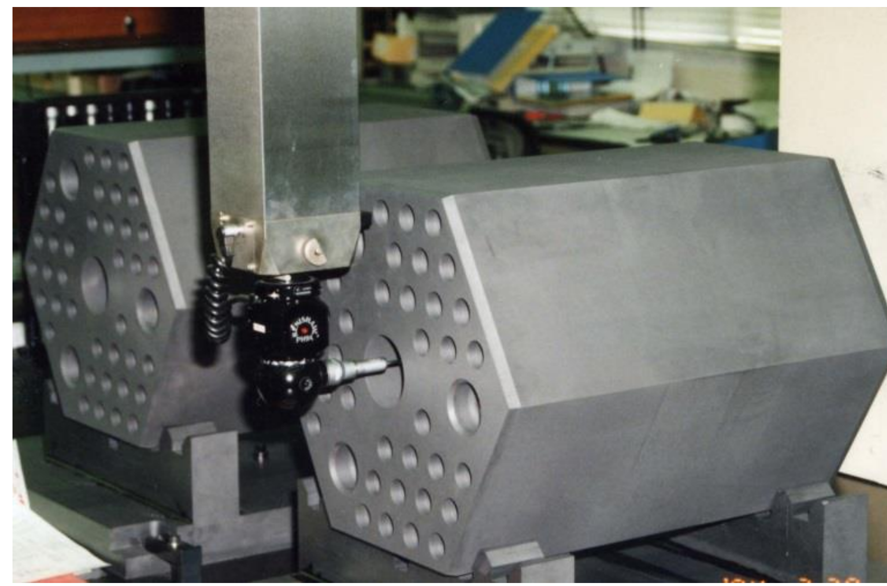
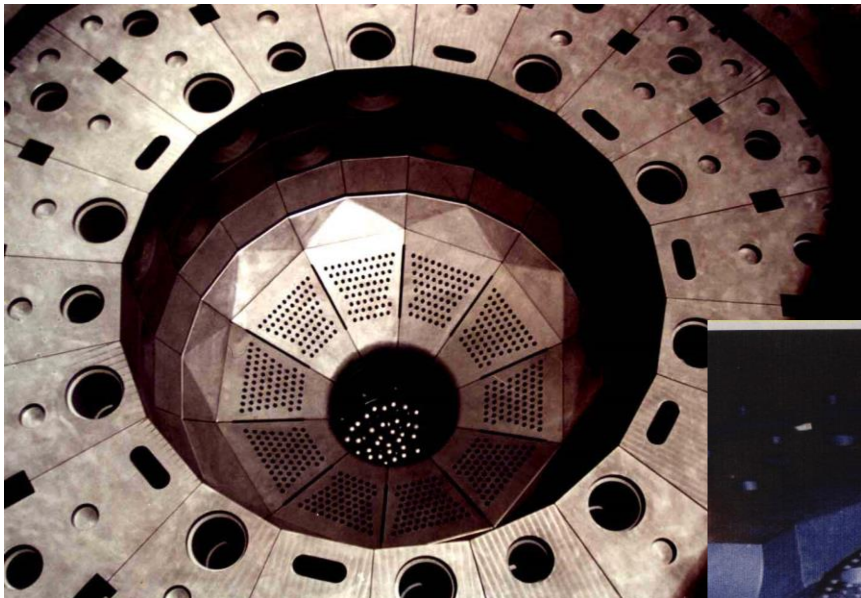
ASME Code for Graphite

- Graphite Core Components – Prismatic Type HTR (HTTR)

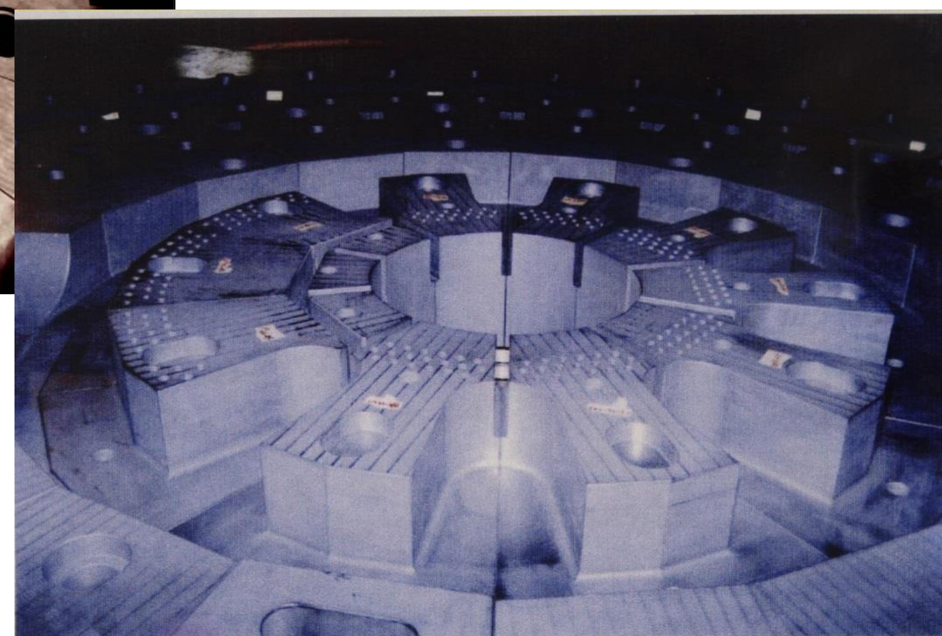




HTR-10 Graphite Reactor Internal Structures (Grade IG-110)



Top of the graphite core of HTR-10



Core bottom of the HTR-10 showing the fuel pebble collection area

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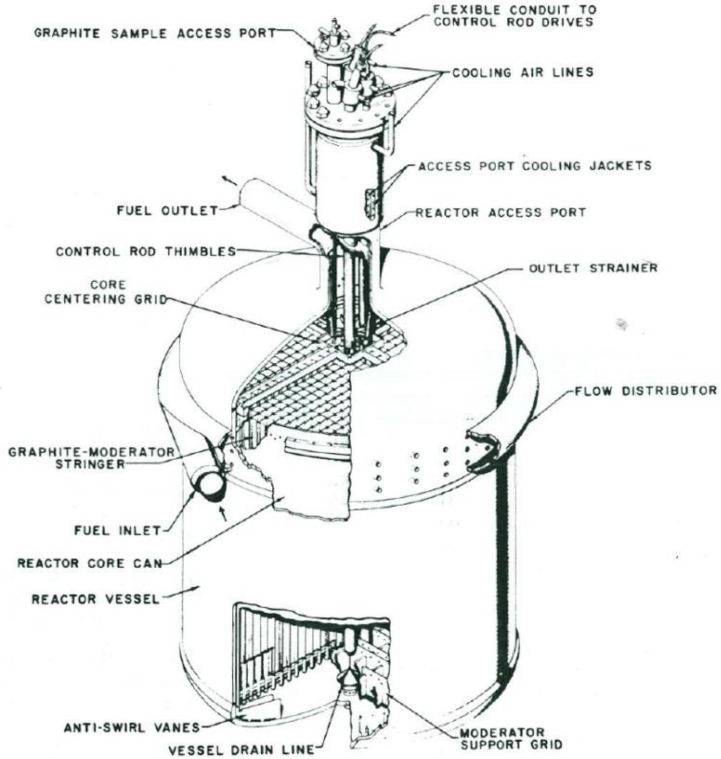
ASME Code for Graphite

Graphite Core Components - Molten Salt HTR (MSRE, Oak Ridge)



THE MSRE

- **Uranium fuel dissolved in the coolant salt**
- **Salt mixture flows down outside of vessel and up through graphite core**
- **Core dimensions 54-in diameter X 64 in tall**
- **CGB Graphite (National Carbon Company)(UCC)(GrafTech)**
- **2 inch square section with ½ in slot X 64 ins-tall. 3.7 tons in core**

REF: P.N Haubenreich and J.R.Engel. NUCLEAR APPLICATIONS & TECHNOLOGY, VOL. 8, pp.118-136, Feb. 1970.

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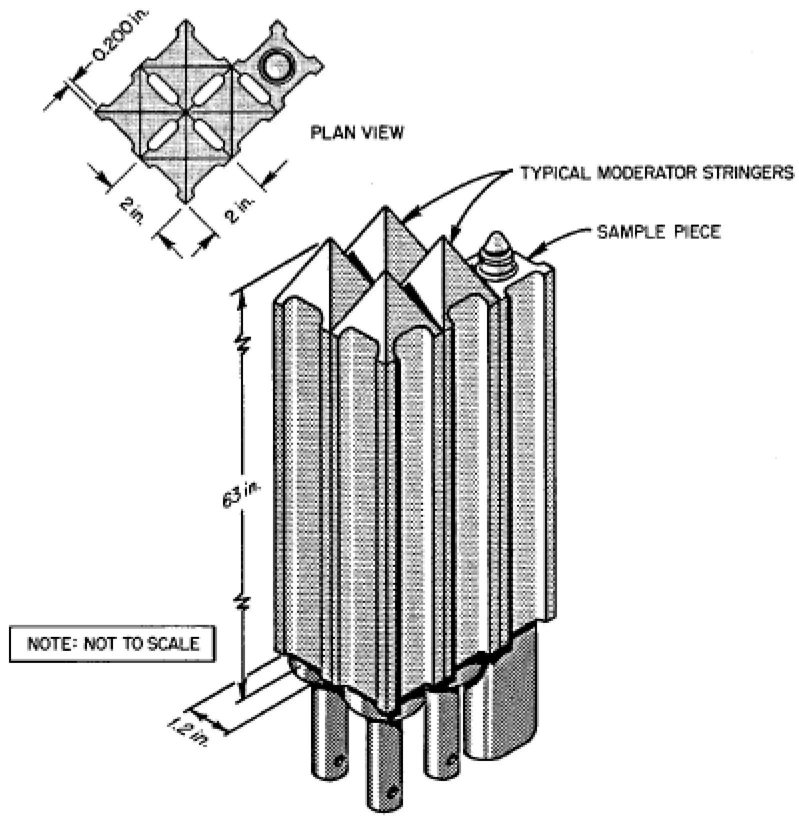
MSRE Graphite Stringer Arrangement



REF: R.C. Robertson. ORNL TM 728, "Development and Operation Report pt 1: Description of Reactor Design, p. 80, Pub. Oak Ridge National Laboratory, Jan 1965.

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ASME Code for Graphite

OVERVIEW OF HHB CODE (2017)

- **SUBSECTION HA GENERAL REQUIREMENTS, SUBPART B: GRAPHITE MATERIALS**
  - HAB-1000 Introduction
  - HAB-2000 Classification of Graphite Core Components
  - HAB-3000 Responsibilities and Duties
  - HAB-4000 Quality Assurance
  - HAB-5000 Authorized Inspection
  - HAB-7000 Reference Standards
  - HAB-8000 Certificates and Data Reports
  - HAB-9000 Glossary
  - Mandatory Appendix HAB-I Certificate Holder's Data Report Forms, Instructions, and Application Forms for Certificates of Authorization

- **SUBSECTION HH CLASS A NONMETALLIC CORE SUPPORT STRUCTURES, SUBPART A: GRAPHITE MATERIALS**
  - HHA-1000 Introduction
  - HHA-2000 Materials
  - HHA-3000 Design
  - HHA-4000 Machining, Examination, and Testing
  - HHA-5000 Installation and Examination
  - HHA-8000 Nameplates, Stamping, and Reports
  - Mandatory Appendix HHA-I Graphite Material Specifications
  - Mandatory Appendix HHA-II Requirements for Preparation of a Material Data Sheet
  - Mandatory Appendix HHA-III Requirements for Generation of Design Data for Graphite Grades
  - Nonmandatory Appendix HHA-A Graphite as a Structural Material
  - Nonmandatory Appendix HHA-B Environmental Effects In Graphite
  - Nonmandatory Appendix HHA-D Guidance on Defects and Flaws in Graphite (in course of preparation)

ASME Code for Graphite – HHB IOU's

- Make the HHA code inclusive of all graphite moderated HTRs
- HHB 3144 **Fatigue** (in course of preparation)
- HHB 3217 FEM analysis volumes (change from Grain Size (GS) to Process Zone (PZ) Size relationship)
  - GS varies from a few microns to a tens mm, PZ (derived from σ_t and K_{Ic})
  - Code case and code change pending
- Code alignment
  - Article HHA-4000 Machining, Examination, and Testing
  - Article HHA-5000 Installation and Examination
  - **To become**
  - **Article HHA-4000 Fabrication and Installation**
  - **Article HHA-5000 Examination**
  - **ARTICLE HHA 6000 Testing**
- Appendix HHA-IX CLAEANLINESS
- Appendix HHA-D GUIDANCE ON DEFECTS AND FLAWS IN GRAPHITE

(These Appendices are in the course of preparation)

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Graphite Design Code Differences

- Design code methodology – PROBABILISTIC:
  - Design margin related to materials uncertainty
  - Man. App III defines materials qualification
- Core component vs. assembly design, catering for damage tolerant assessment:
  - Designer selection and classification of parts for structural reliability
- Design for effects of reactor environment over core lifetime:
  - Irradiation
  - Oxidation
  - Chemical attack

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ASME Code for Graphite HHA-2000 MATERIALS

Graphite Material Issues

- Graphite is nuclear moderator and core structural material, but is not pressure retaining
- The following material issues had to be considered when drafting code:
  - Differences between nuclear graphite and steel
  - Manufacture of graphite (see Graphite I)
  - Effect of reactor environment on the nuclear graphite (see Graphite III)

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ASME Code for Graphite: HHA-2000 MATERIALS

Properties & behavior of graphite are fundamentally different from steel

Steel	Nuclear Graphite (Ceramic)
Region of linear elastic behavior	Always non-linear behavior
Yield stress can be defined	Yield stress is not definable
High tensile strength, fracture strain, and fracture toughness	Low tensile strength, fracture strain and fracture toughness
Small scatter of the strength data	Large scatter of the strength data
Strength decreases with increasing temperature	Strength increases with increasing temperature
Relief of peak stresses due to plasticity	Relief of peak stresses by micro-cracking
Local peak stresses are non-critical	Local peak stresses can cause damage
Crack initiation depends on the primary stress	Crack initiation depends on the total stress
Material properties are thermal neutron flux dependent	Material properties are thermal neutron flux independent
Fast neutron flux influences the material properties (increases the NDT)	Fast neutron flux changes all material properties, and induces dimensional change and irradiation creep

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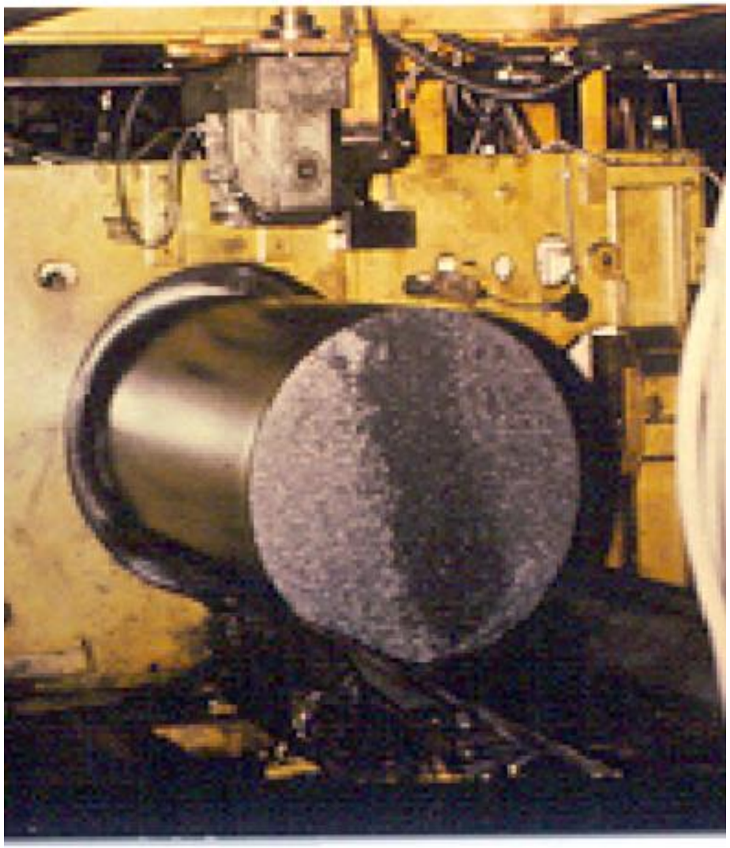
Nuclear Graphite Manufacture



- ASTM Nuclear Graphite Specifications provides minimum requirements for properties and QA
- ASME code adopts these
- Current production grades requires complete characterization for reactor design (materials data Sheet)
- Grades are supplier specific.

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HHA-3000 DESIGN: Table of Contents

DESIGN CRITERIA FOR GCC

- Brief overview of the design criteria for GCC (supporting Article HHA-3000)
  - Role of GCC in a HTR Safety Case
  - Modes of Failure addressed
  - Determination of Limits
  - Material Reliability Curve
  - Probabilistic Method – Simplified Assessment
  - Probabilistic Method – Full Assessment
  - Comparison of Margins
  - Verification

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GCC in the HTR safety case

- Graphite is quasi brittle
- Graphite Strength shows high variability
- It is not necessarily possible to ensure against cracking of graphite components
- A Graphite Core Assembly (GCA) design shall ensure that the **failure** (cracking) of a **GCC** does not result in the loss of **Functional Integrity** of the **Graphite Core Assembly**
- As opposed to a pressure vessel, damage tolerance in a GCA is ensured by limiting the consequences of failure of a single GCC, thus damage tolerance is assured by assemblies of many components where no single failure is critical to the functional integrity of the assembly

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MODES OF FAILURE

- The identified modes of failure for graphite are:
  - **Brittle fracture**
    - **Based on small number of parts cracking. Related to loss of function. Materials dependent.**
  - Fatigue
  - Buckling (Elastic Instability)
  - Environmental effects
    - Oxidation
      - Air ingress (acute oxidation)
      - Helium impurities (H_2O, CO, H)
    - Neutron Irradiation

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DETERMINATION OF LIMITS

- Key Code Assumptions:
  - It is possible to design parts by comparing **calculated stresses** to **strength limits** based on **specimen test results** incorporating adequate **Design Margin**.
  - For graphite, fixed **Design Margins** do not ensure uniform reliability, variability in the graphite grade must be accounted for.
  - It is possible to characterize the materials variability statistically and from this determine the design margin.

DETERMINATION OF LIMITS

- Probabilistic approach selected.
- Design Margins to be provided by means of reliability targets, allocated for stress categories based on part classification.

Table GB-3000-1

	Load category			
SRC	A	B	C	D
SRC-1	10^{-4}	10^{-4}	10^{-4}	10^{-3}
SRC-2	10^{-4} (10^{-2} EOL)	10^{-4} (10-2)	5.10^{-2}	5.10^{-2}
SRC-3	10^{-2}	10^{-2}	5.10^{-2}	10^{-2}

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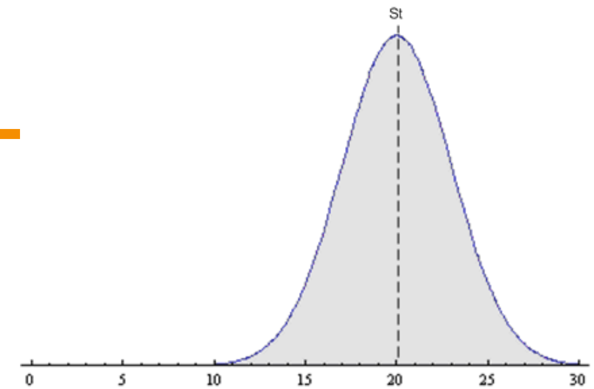
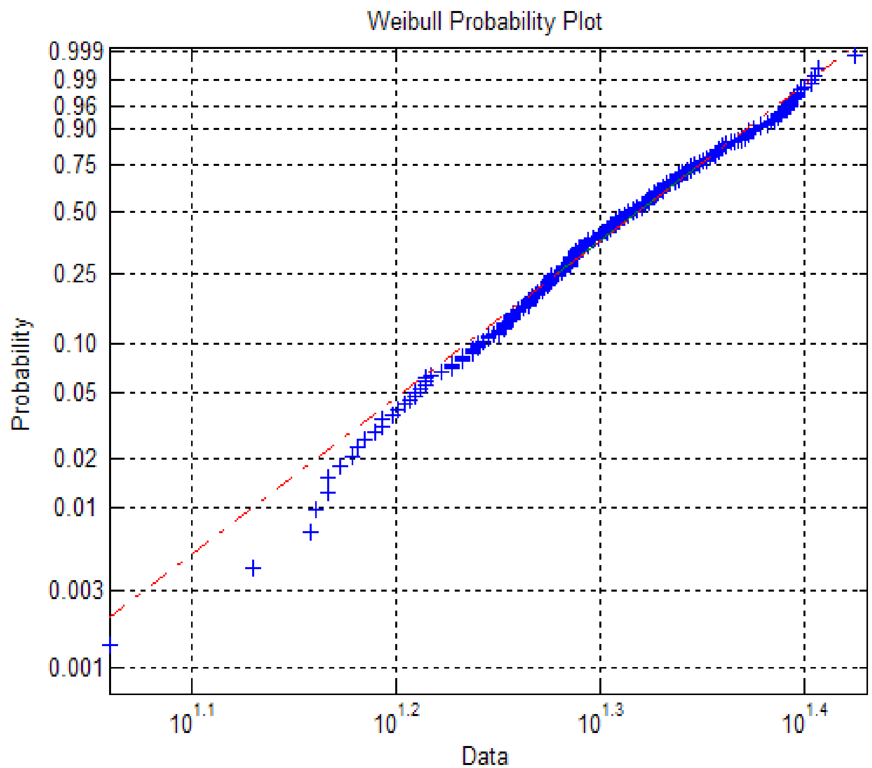
MATERIALS RELIABILITY CURVE

- The variability in material strength is characterised by the material reliability curve.
  - –Use a Weibull Distribution to characterise the material strength (Ho, Schmidt, Nemeth & Bratton)
  - –Conservatism introduced using 95% confidence limits.

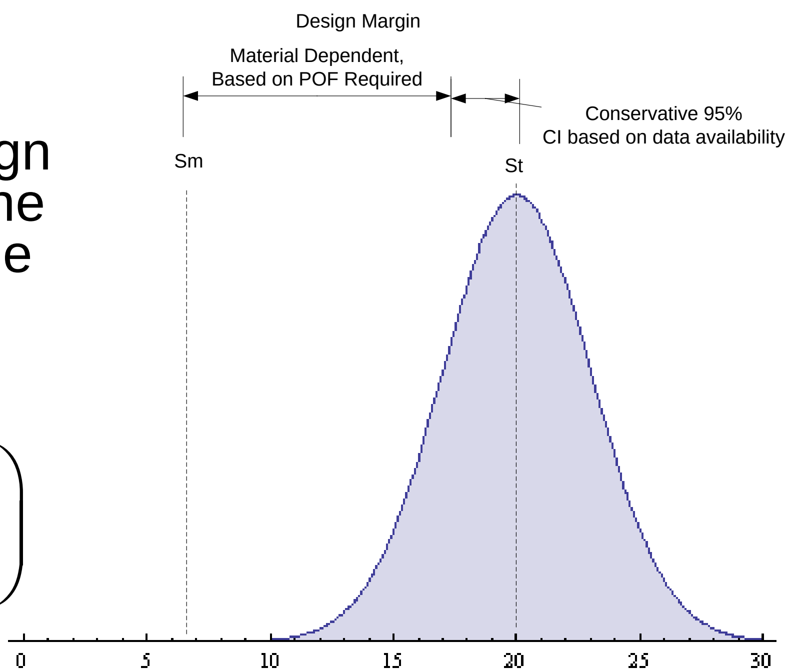




SIMPLIED ASSESSMENT

- Simplified assessment:
  - Compare the highest stress calculated in the part to a design stress value, calculated from the Material reliability curve and the target POF for the part for this service level.
- Using Weibull:

$$S_{allow} = Sc\left(-\ln\left(1-POF\right)^{\frac{1}{m}}\right)$$



		Sallow		Smean/Sallow
	POF	MPa	ksi	MPa
SRC 1	1.00E-04	2.6	0.38	7.3
SRC 2	1.00E-03	4.2	0.61	4.5

Note: The Allowable stress is now a function of material quality.

SIMPLIFIED ASSESSMENT (Contd.)

- The Design margin can be calculated as a function of required POF and material variability (m)

	Design Margin for Probability of Failure					
Weibull Modulus	10^{-2}	10^{-3}	10^{-4}	10^{-5}	10^{-6}	10^{-7}
2	12.0	38.0	120.1	379.8	1201.1	3798.3
5	2.7	4.3	6.8	10.8	17.1	27.0
10	1.6	2.1	2.6	3.3	4.1	5.2
15	1.4	1.6	1.9	2.2	2.6	3.0
20	1.3	1.4	1.6	1.8	2.0	2.3

- Note: For a typical grade, $5 < m < 15$ are typical.

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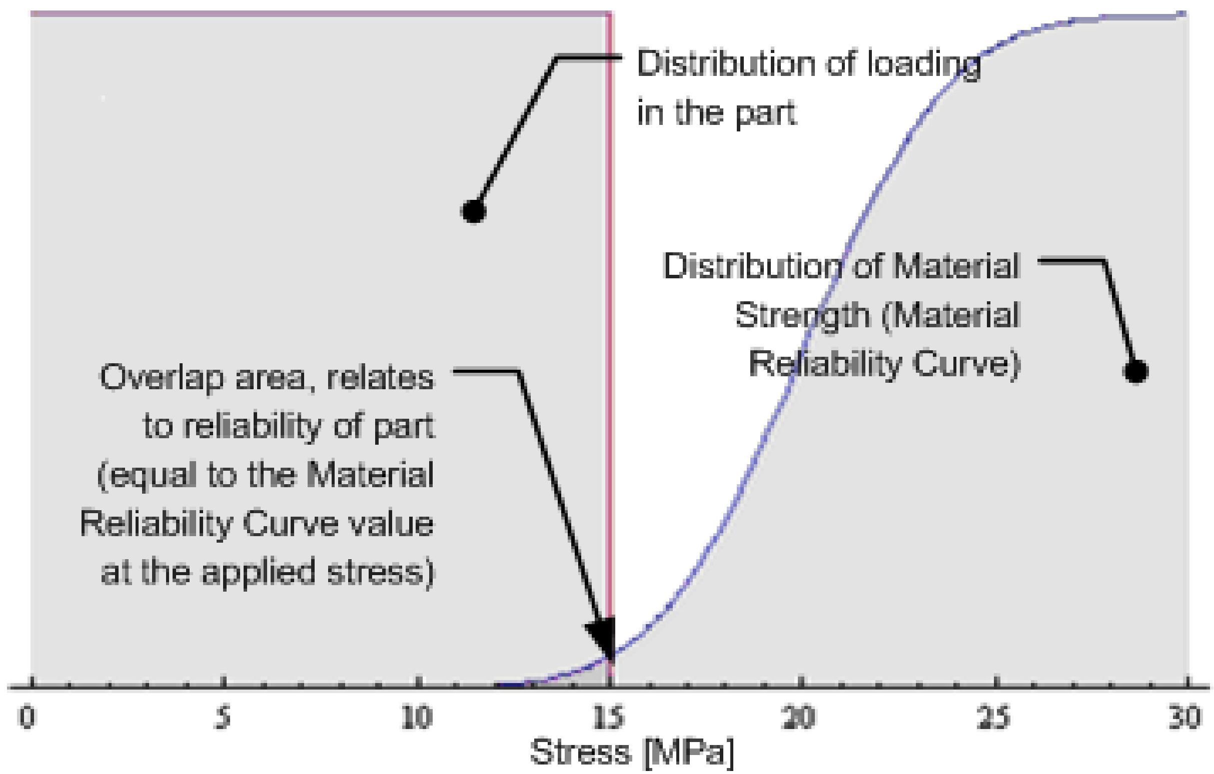
SIMPLIFIED ASSESSMENT (Contd.)

- Schematic of simplified assessment.



HHA-3000 DESIGN: FULL ASSESSMENT

- Note that the simplified assessment assumes that the entire part is at the same stress (or at least in a simple stress distribution in the case of bending).
- Full assessment takes account of the actual distribution of stress in the part.
  - Smaller volumes of material at the same stress level will result in a lower probability of failure of the part.

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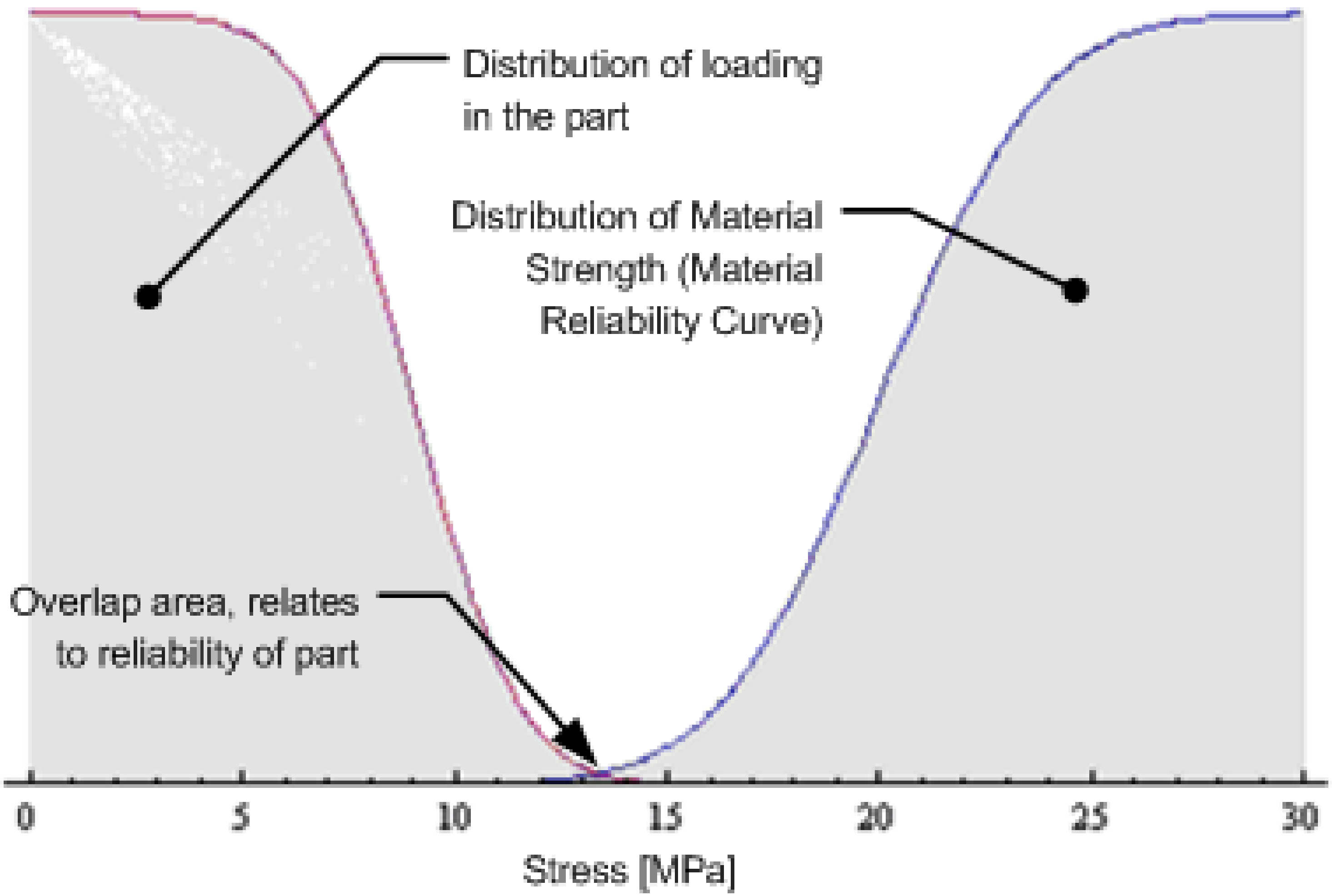
HHA-3000 DESIGN :FULL ASSESSMENT



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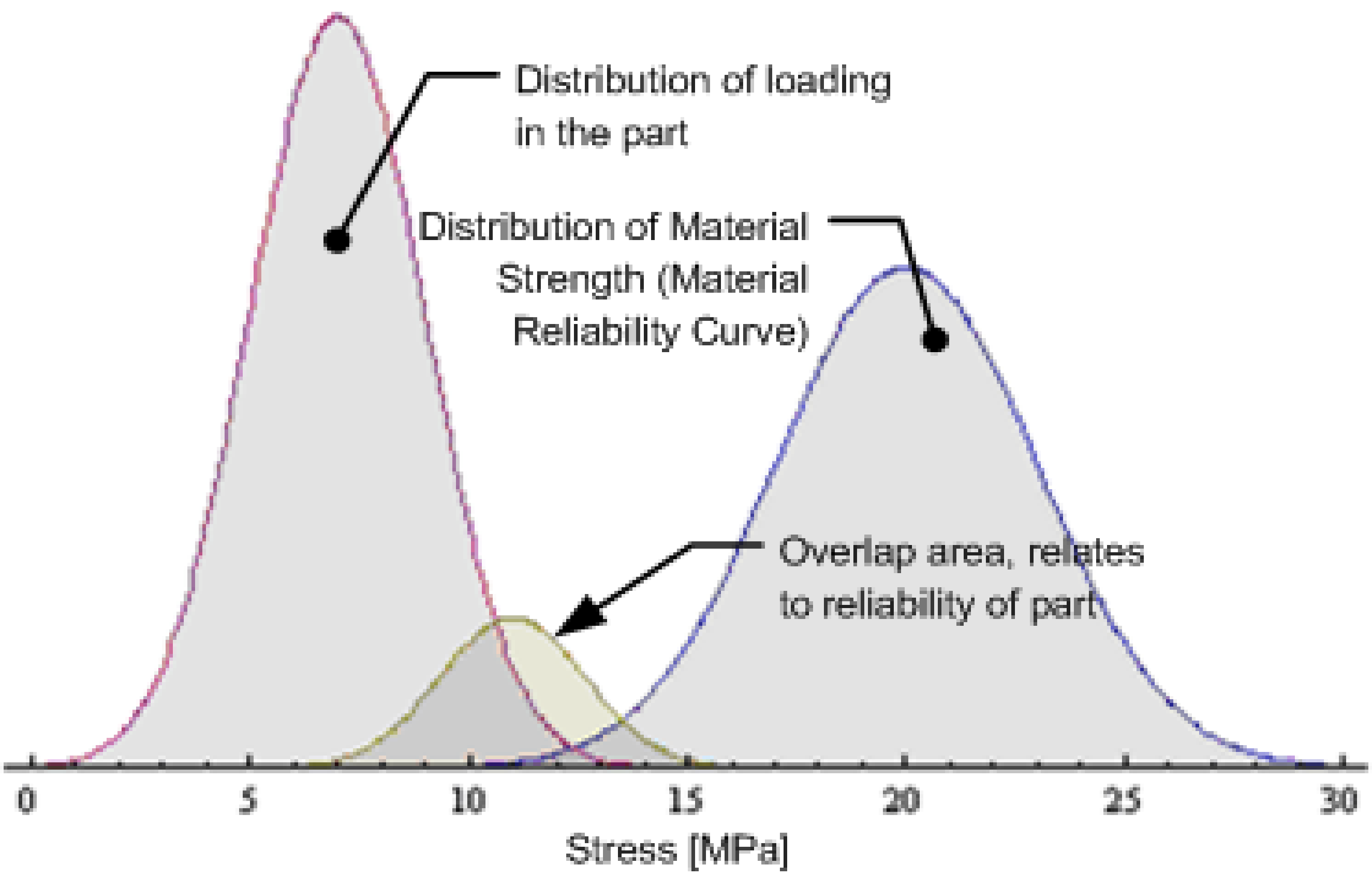
HHA-3000 DESIGN :FULL ASSESSMENT



HHA-3000 DESIGN :FULL ASSESSMENT
How is this achieved?

- Typically be means of some integral such as Weibull's weakest link.

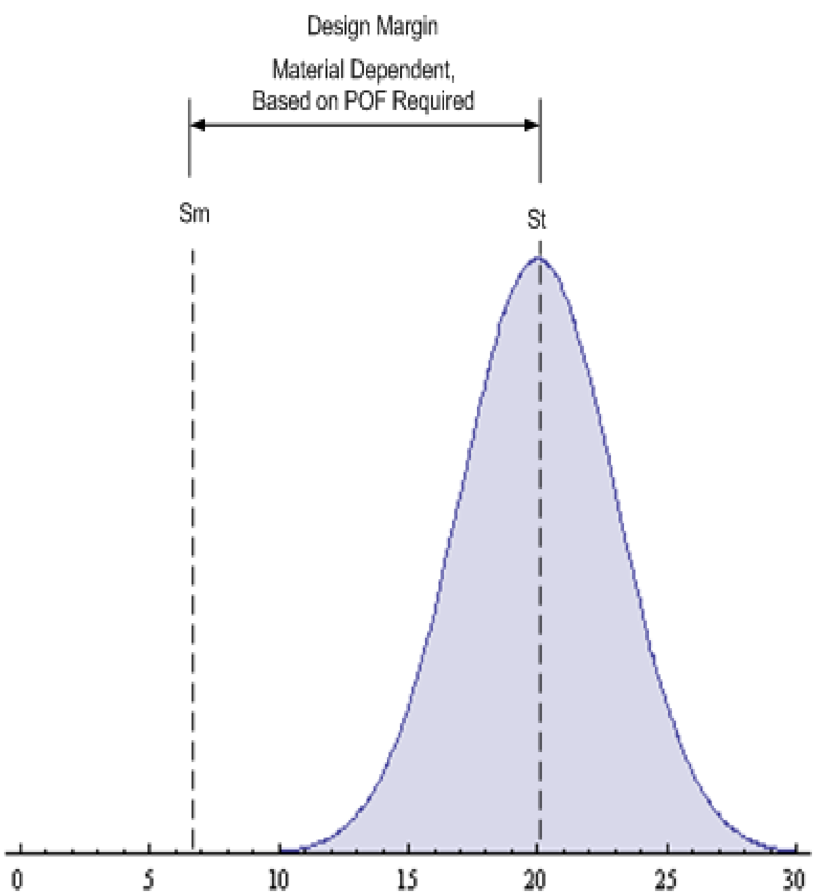
$$P_{fV} = 1 - \exp\left[-\int_V \left(\frac{\sigma(x,y,z)}{\sigma_o} \right)^m \mathrm{d}V \right]$$

- Or, a modified Weibull approach

$$L = \exp\left[-\sum_{j=1}^{n} \left(\frac{\sigma_j}{S_c} \right)^m \cdot \frac{V_j}{V} \right]$$

HHA-3000 DESIGN :FULL ASSESSMENT
Comparison to other margins

- Incorporation of design margin guards against failure by backing off from the load at which failure is anticipated.
- The design margin can be compared to design margin values that are in use internationally today.
- Sources
  - ASME CE Draft
  - JAEA Design Methodology
  - RDMCI Methodology (South Africa)
  - UK Methodology
- Assessed for Both Core Blocks and Core Supports for materials with different levels of variability (Weibull moduli from 5 to 15).
- Converted to the same units (distance from the mean tensile strength (St) to the Design Stress (Sm)



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HHA-3000 DESIGN :FULL ASSESSMENT
Comparison to other margins

Table 12: For Core support structures, in tensile loading, for Weibull moduli from m=5 to m=15

	ASME CE	JAEA	KTA	UK AGR
Level A	5.3 -9.3	5.3 -9.3	1.8 - 5.9 (3)	3 (RSF=5.0)
Level B	5.3 -9.3	5.3 -9.3	1.8 - 5.9 (3)	2 (RSF=3)
Level C	2.7 - 4.7	2.7 - 4.7	1.8 - 5.9 (3)	1.5 (RSF=2.0) [1.3 – 1.5]
Level D	2.2 - 3.9	2.2 - 3.9	1.6 - 3.7 (2.25)	1.25 (RSF=1.5)

Table 13: For Core blocks, in tensile loading, for Weibull moduli from m=5 to m=15

	ASME CE	JAEA	KTA	UK AGR
Level A	N/A	4.0 - 7.1	1.3 - 2.3 (1.7)	3 (RSF=5.0)
Level B	N/A	4.0 - 7.1	1.3 - 2.3 (1.7)	2 (RSF=3)
Level C	N/A	2.7 - 4.7	1.3 - 2.3 (1.7)	1.5 (RSF=2.0) [1.3 – 1.5]
Level D	N/A	1.9 - 3.3	1.2 - 1.7 (1.4)	1.25 (RSF=1.5)

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HHA-3000 DESIGN :FULL ASSESSMENT
Comparison to other margins

- Verification of Methods
  - Work completed by volunteers to integrate the verification case into a criteria document.
- Test of the methods over a range of problems.
  - Demonstrate accuracy or conservatism.

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Verification - Acceptance Criteria

- What is a suitable basis for acceptance?
- Material variability and experimental bias:
  - Material data for all material of the same grade
  - Typical part tests from few billets
- Analysis of typical (NBG-18) billet data provides the following: (Billet mean tensile strength values of 24 billets)
  - 50% of billets fall within +/- 6% of the material mean
  - 95% of billets fall within +/-18% of the material mean
- No additional uncertainties included in the acceptance criteria.
  - Analysis accuracy / convergence
  - Experimental accuracy (confidence in mean prediction)

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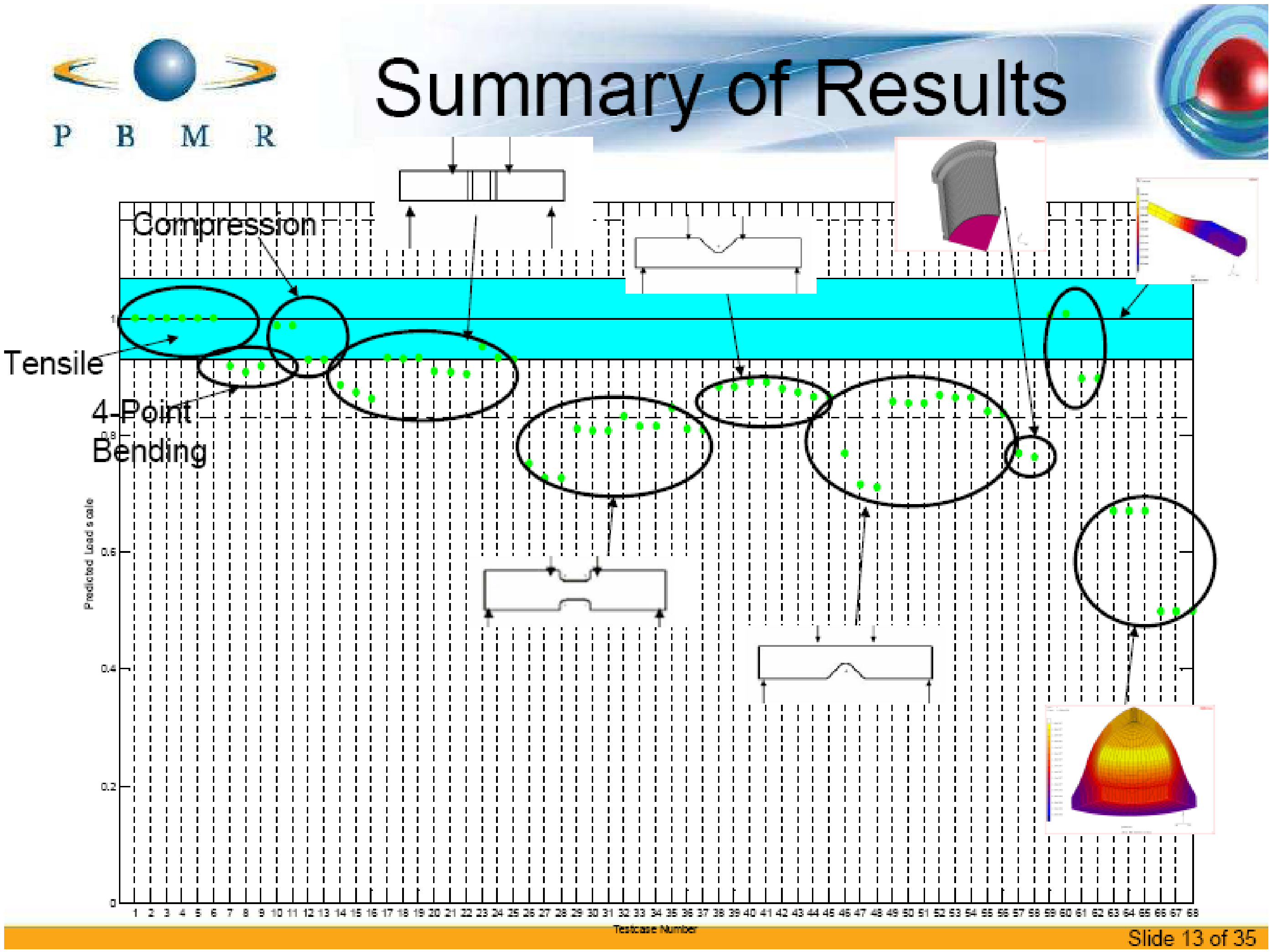
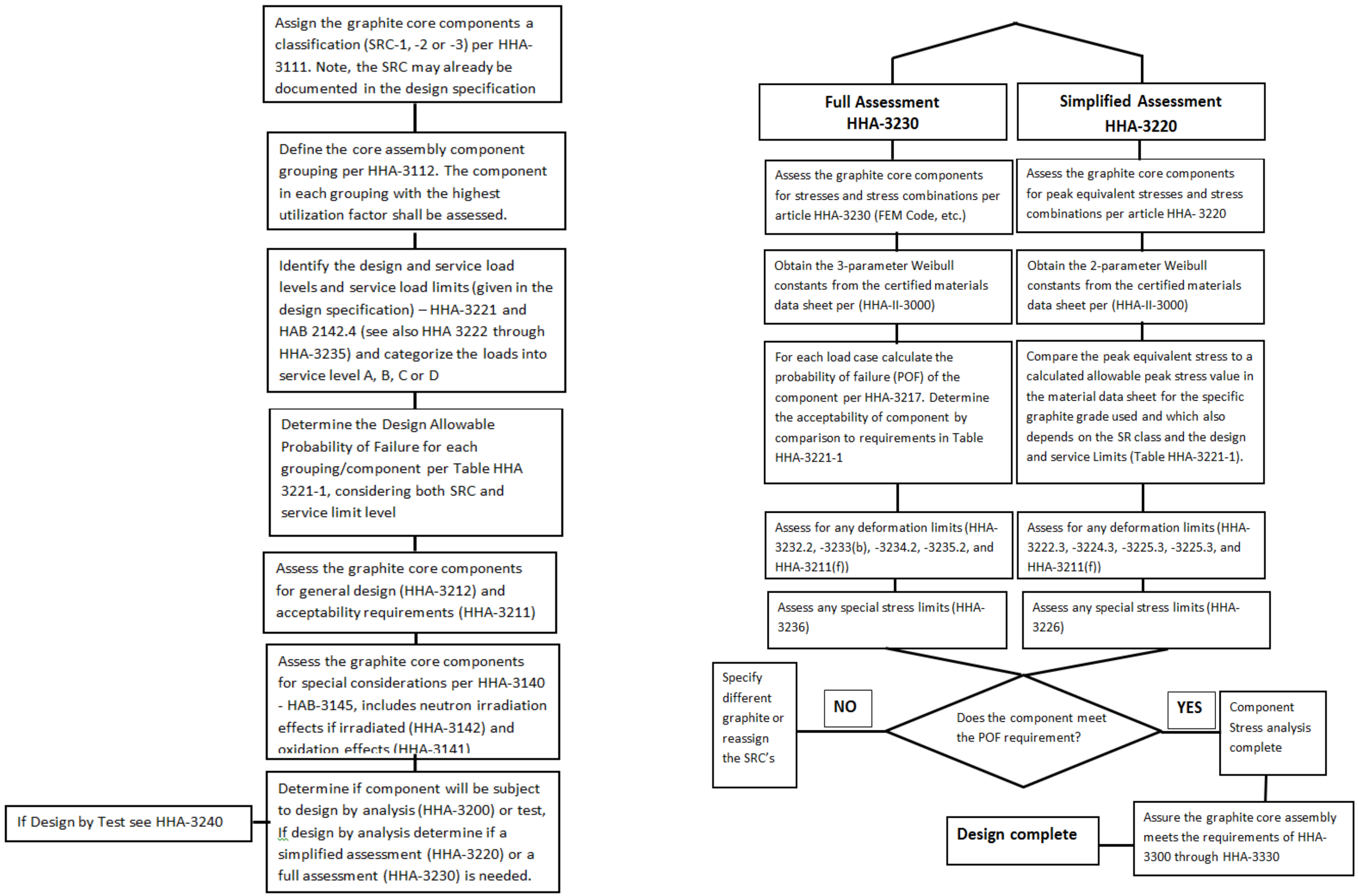
VERIFICATION – TYPICAL RESULTS



HHA-3000 DESIGN :ASSESSMENT
Design Process Steps



Thank You!

Feel free to contact **Richard Barnes** email at **rwbarnes@anric.com** or by phone at **(416) 727-3653 (cell)** or **(416) 255-9459 (office).**

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