

Nuclear Graphite Components

William E Windes

April 2019



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

<http://www.inl.gov>

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Nuclear Graphite Components

Advanced Reactor Technologies

Idaho National Laboratory

Will Windes

ART Graphite R&D Technical Lead

NRC HTGR Training April 16-17, 2019

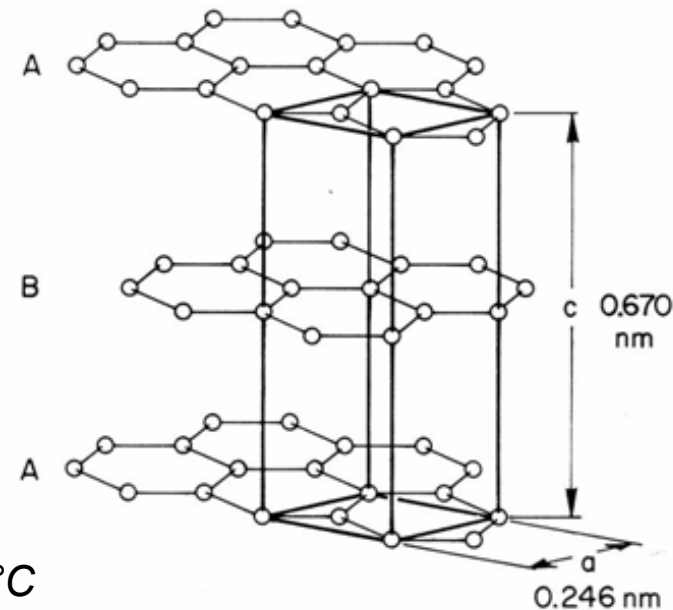


Idaho National Laboratory



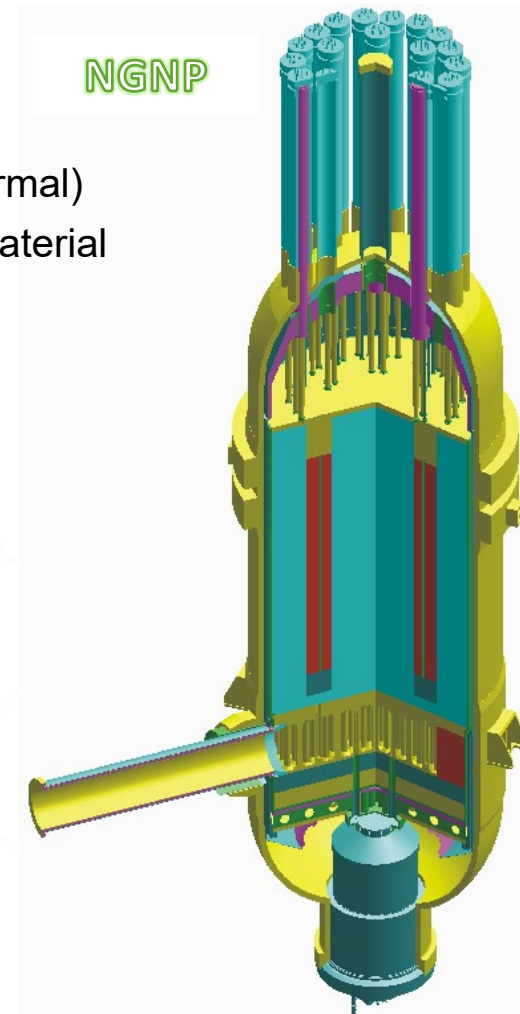
Graphite Outline

- Functions and Requirements
 - Normal and off-normal component functions
 - Key safety requirements of core components
- Graphite Manufacture
 - Unique material properties of graphite
 - Ideal unirradiated material properties – it's not metal
- Environmental effects on nuclear graphite
 - Effects of oxidation
 - ***It doesn't burn!***
 - Effects of irradiation of graphite
 - ***No Wigner*** (stored) energy if operated above 300°C
 - *Physical, thermal, and mechanical properties*
 - *Turnaround and creep significance explained*
- ASME Code for Graphite Core Components
 - New ASME code: probabilistic (ceramics) vs. deterministic (metals)
 - How environmental effects are accounted for in design requirements
- Operating considerations (prismatic vs. pebble vs. molten salt)
 - Differences between different graphite core designs

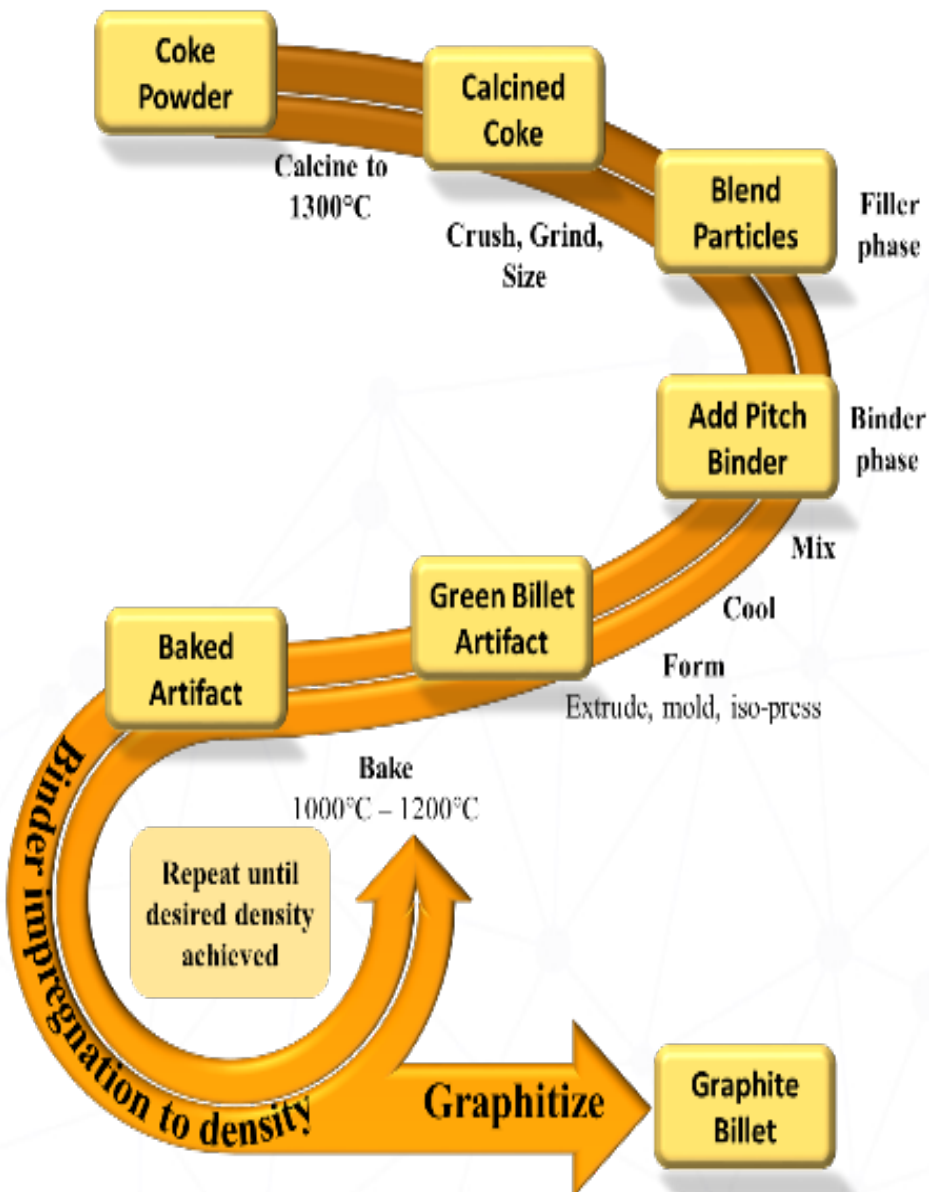


Critical Safety Requirements

- Maintain core geometry and structural integrity
 - Maintain fuel configuration during all operations (normal and off-normal)
 - Maintain undisturbed access for the insertion of reactivity control material
 - Maintain proper core coolant configuration
 - *No blockage of coolant pathway*
 - *No gaps between graphite components*
- Protection of fuel
 - Compacts within the prismatic fuel elements
 - Pebbles within the core center
- Passively remove core heat during off-normal events
 - Rapidly absorb large thermal transients
 - Primarily by radial conduction from the fuel to the core barrel
 - *During off-normal events when forced cooling is not available*
- How does it do this?:
 - Graphite does **NOT** melt or burn
 - Graphite **DOES** have high thermal conductivity and thermal stability
 - Relatively strong in compression, weak in tension.



Graphite Manufacture



- All graphite grades **are proprietary**. Only limited/general fabrication data is known.
- Unique manufacturing processes for graphite must be understood to appreciate graphite behavior
 - Graphite is a porous material (15-20%) - **By design!**
 - Porosity provides thermal and irradiation stability
- Graphite is manufactured from calcined coke and a pitch binder.
 - Multiple pitch impregnations to increase density
- Green forming technique influences the final microstructure
 - Desire isotropic (or near isotropic) material response
- Properties and performance of graphite are significantly influenced by both raw materials and processing
 - Nuclear graphite undergoes further purification steps

Graphite Material Properties of Interest

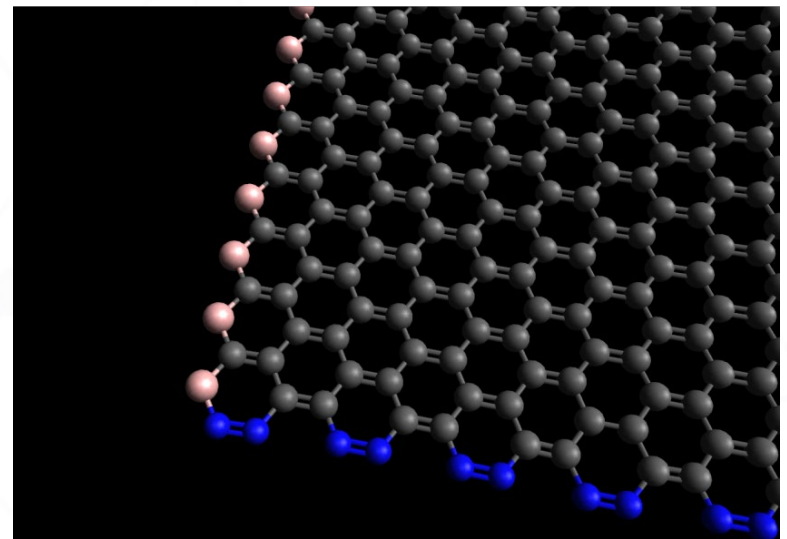
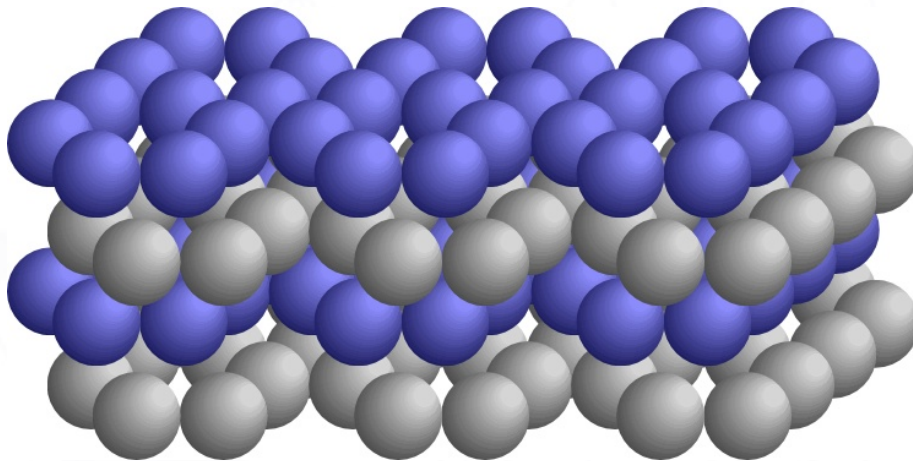
Property	Nominal Range	Performance Attributes
Density	1.7 - 1.9 g/cm ³	Neutron efficiency, Structural integrity, Thermal efficiency
Thermal Conductivity (at Room Temperature)	> 90 W/m/K	Heat transport
Purity (Total Ash Content)	< 300 ppm	Reduced component activity levels during replacement and/or disposal Reduced graphite oxidation under normal and accident conditions.
Tensile Strength	> 15 MPa	Structural integrity
Compressive Strength	> 45 MPa	Structural integrity
Flexural Strength	> 20 MPa	Structural integrity
CTE (20°C to 500°C)	3.5 to 5.5 x 10 ⁻⁶ K ⁻¹	High value is indication of isotropy = dimensional stability under irradiation Lower value potentially beneficial in terms of thermal stress
CTE Isotropy Ratio	< 1.10	Irradiation dimensional stability Structural integrity
Dynamic Elastic Modulus	8 – 15 GPa	Structural integrity Irradiation creep
Dimensional Changes with Irradiation	Minimal shrinkage Minimal differences in with-grain and against-grain directions	Structural integrity (lower internal stresses)

From ASTM D7219 : *Standard Specification for Isotropic and Near-isotropic Nuclear Graphites*

- **Density**
 - Higher = Stronger
 - Lower = Better irradiation performance
- **Conductivity**
 - Nearly a 70% drop almost immediately after reactor startup
- **CTE (Coefficient of Thermal Expansion)**
 - Indicates isotropy and needed for gas gap analysis
- **Purity**
 - Requires additional heat treatment
- **Dimensional changes**
 - Affects structural integrity
 - If internal stress exceeds inherent strength of graphite = *cracks*

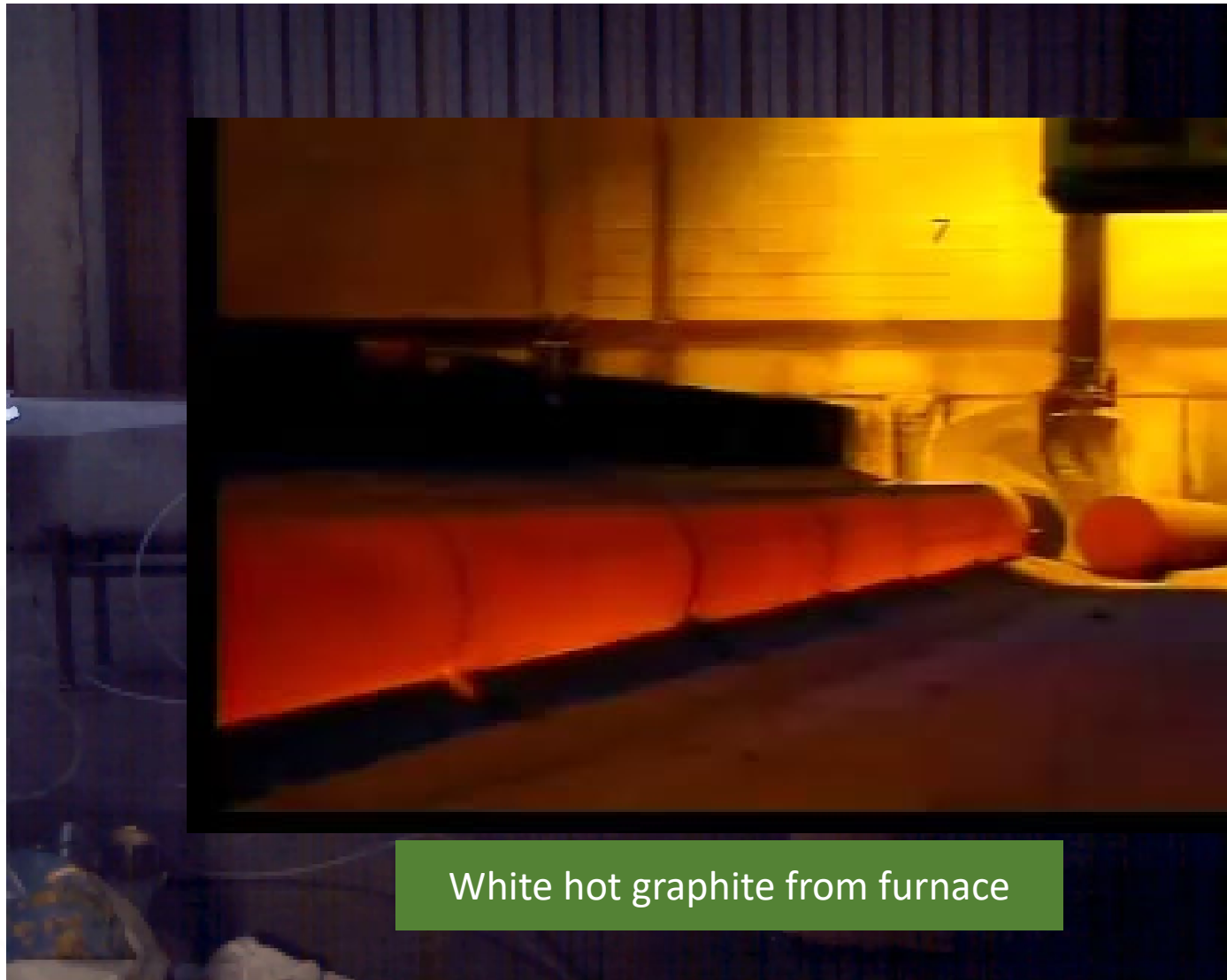
Graphite “Burning” and dust “Explosions”

- Graphite **can not** burn – just physically can not sustain self oxidation
 - Fire needs ➡ Heat, fuel, and oxygen
 - Fuel (carbon) is restricted to only the edges. Oxygen is restricted by the crystallography.
 - Self-sustained oxidation (better definition than simple burning) can not be sustained.



- Graphite dust **can not** explode
 - It does rapidly react but it self-suppresses. Similar mechanisms for “burning”
 - Initial flare up of surface layer on dust particles – but then nothing.
 - *No chain reaction*

Graphite “Burning” and dust “Explosions”



Corn

Graphite

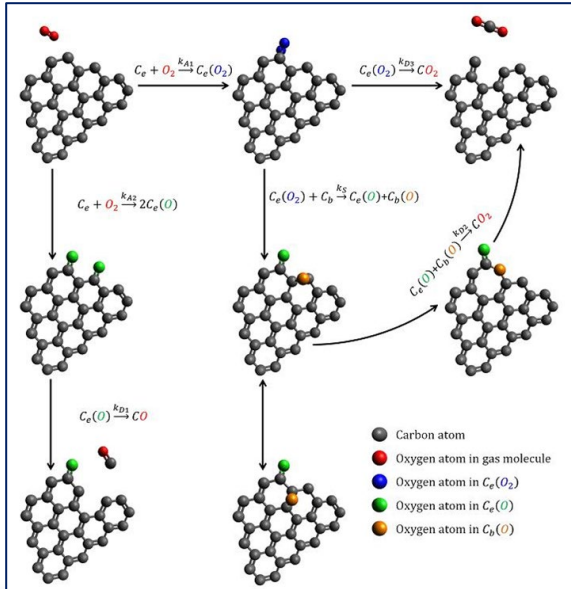
Acheson

White hot graphite from furnace

Corn (Maize) Dust

Graphite Oxidation and “Burning”

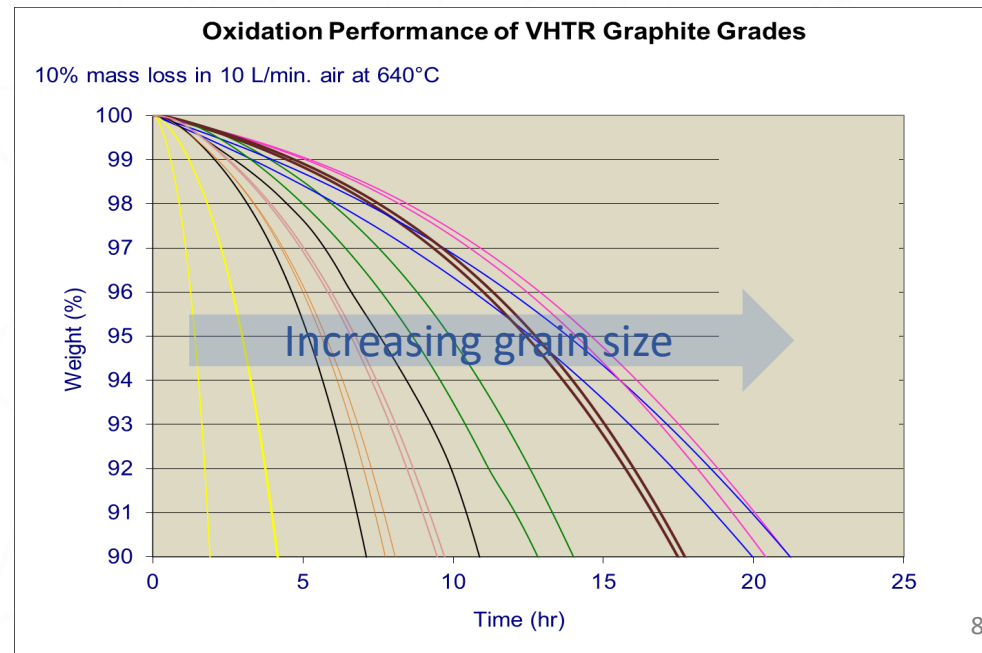
- Graphite **can** and **does** oxidize – high temperatures



- Needs continuous oxygen and temperatures above 200°C – 300°C
 - Temperatures > 400°C needed for more rapid acute oxidation (accidents)
 - Temperatures < 400°C can still oxidize but at very slow rates (chronic oxidation)
- Oxidation still restricted to edges of crystallites with porosity dictating oxygen transport into component

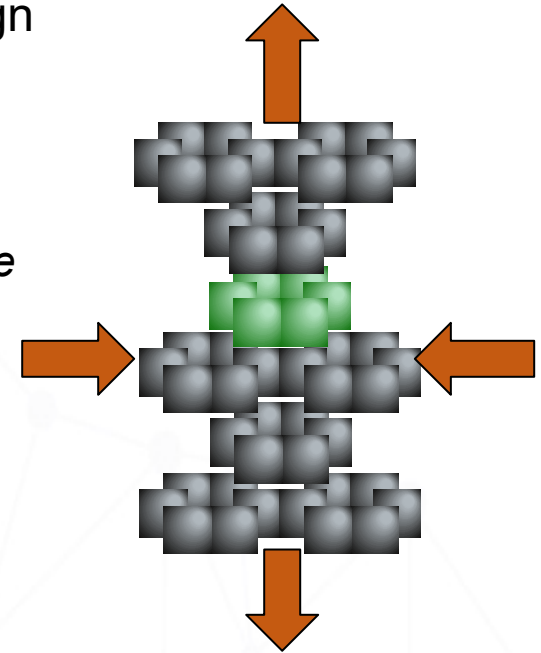
- Oxidation rates of different grades can be compared using ASTM D7542 standard, “Air Oxidation of Manufactured Carbon and Graphite in Kinetic Regime”

- Grain size dependent
- Oxidation of small grain grade >> than large grain size

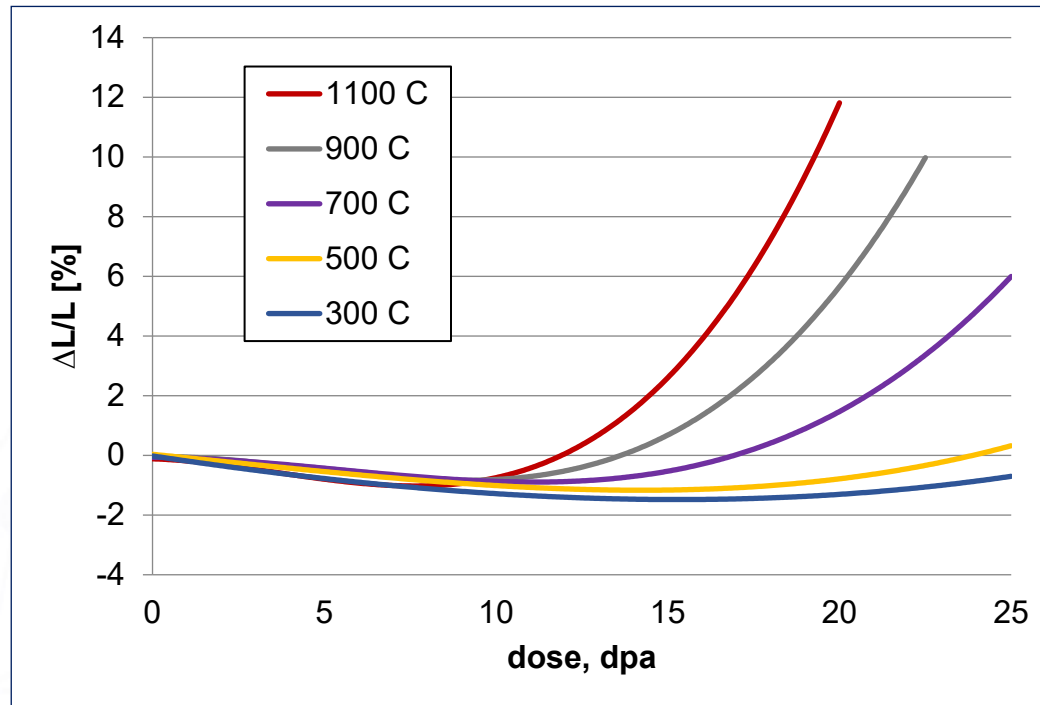


Irradiation Effects on Graphite Properties

- Irradiation induced changes **must** be considered in design
- Significant changes occur during normal operation in:
 - Component dimensions
 - *Components actually shrink ...*
 - *Until **Turnaround** when they begin to expand until failure*
 - Density
 - *Components become more dense ...*
 - *After **Turnaround** dose they decrease in density*
 - Strength and modulus
 - *Graphite gets stronger with irradiation ...*
 - *Until **Turnaround** dose is achieved. It then decreases*
 - Thermal conductivity
 - *Decreases almost immediately to ~30% of unirradiated values*
 - Coefficient of thermal expansion
 - *Initially increases but then reduces after **Turnaround** until saturation*
- Significant changes do not typically occur in the following properties:
 - Oxidation rate, neutron moderation, specific heat capacity, emissivity
- No Wigner energy release **if** components irradiated above 300°C.



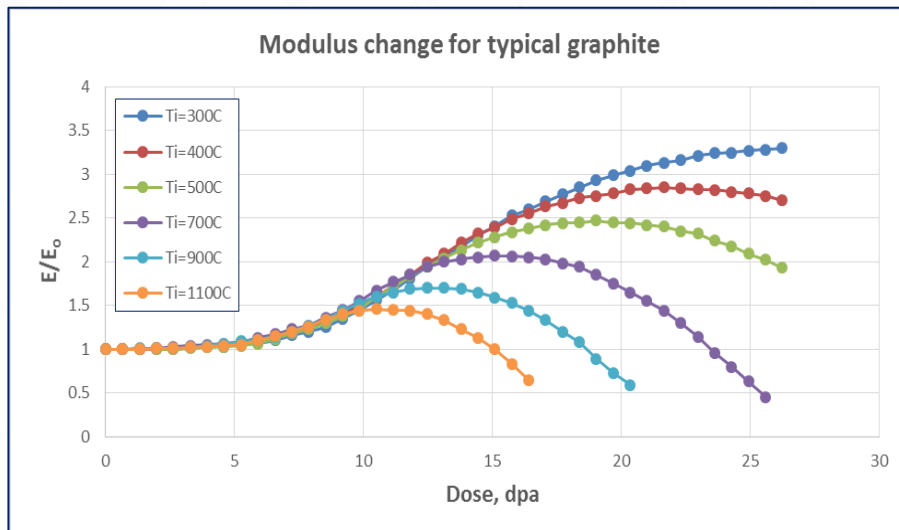
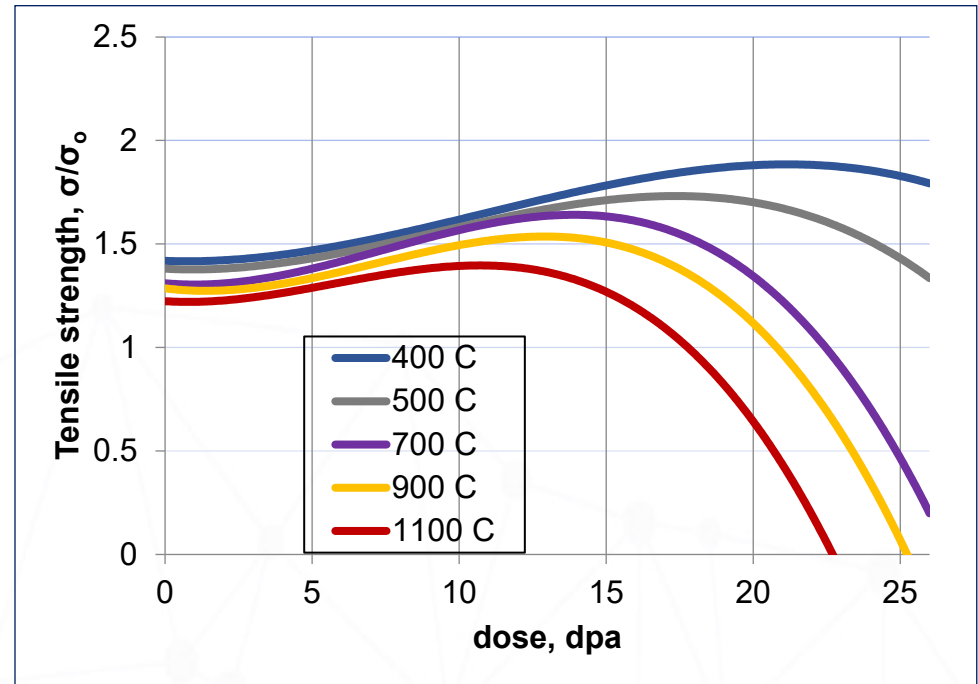
Irradiation-Induced Dimensional Changes



- Under neutron irradiation graphite components shrink (densify) – stop at **Turnaround** – then begin to expand (crack formation)
 - Change is dose dependent: Higher doses = larger change
 - Rate of change is highly temperature dependent
 - Rate and amount of change is grade specific
- Results in tremendous internal stresses formed within graphite
 - Crack formation and component failure – usually after Turnaround
 - Isotropic response is desired to assist in prediction of stresses and dimensional changes

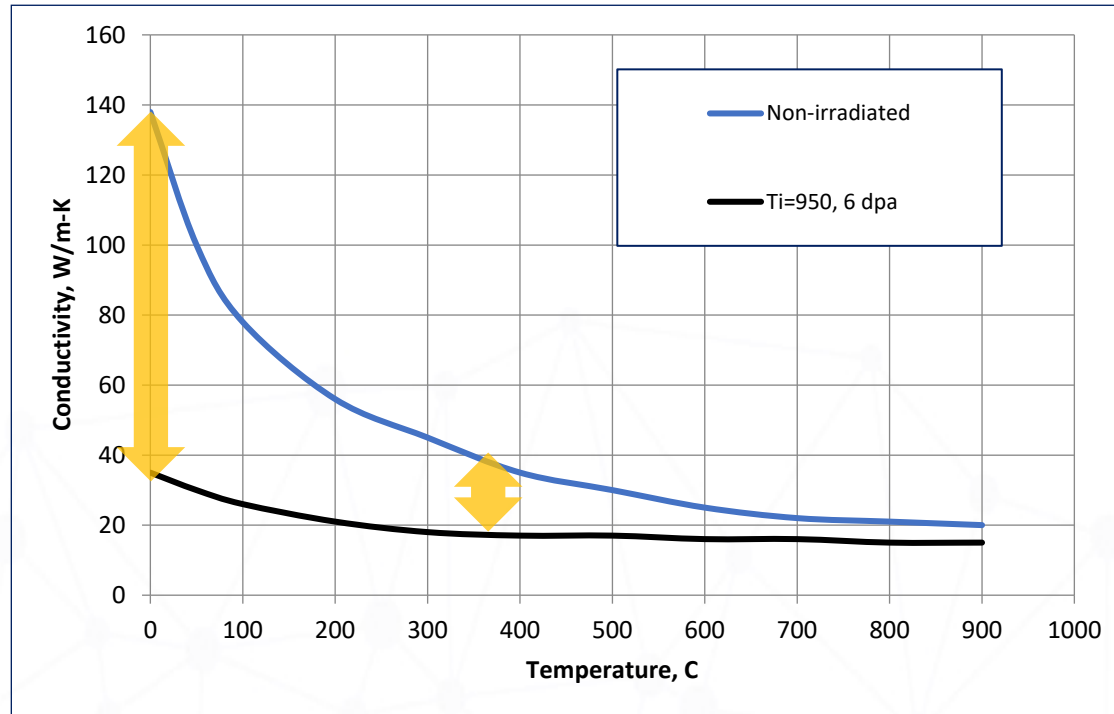
Irradiation-Induced Strength/Modulus Changes

- Changes in strength and modulus somewhat parallel dimensional changes
- Strength/modulus initially increase
 - Maximum value is reached at approximately the **Turnaround** dose



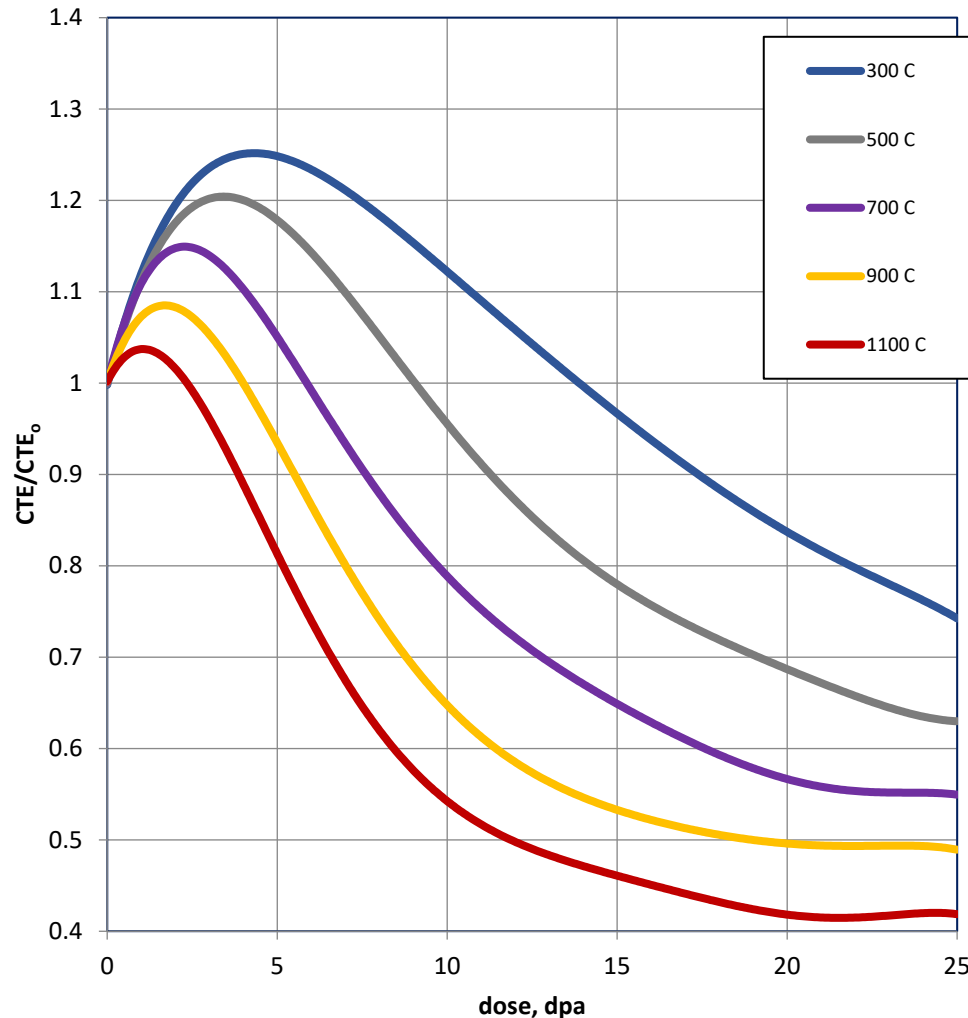
- **After Turnaround** pores start to form in microstructure
 - As porosity forms, strength and modulus fall at increasing rate
- As with dimensional changes, strong dependence on irradiation temperature

Irradiation-Induced Thermal Conductivity Changes



- Initial steep drop in conductivity followed by a saturation level
 - Point defects interrupt thermal diffusivity/conductance
 - Efficiency of recombination rate of point defects is dependent upon irradiation temperature = saturation
 - Further degradation of conductivity due to larger microstructure defects
 - Pore generation after turnaround
- At high operating temperatures irradiated and non-irradiated thermal diffusivity differences are small

Irradiation-Induced CTE Changes

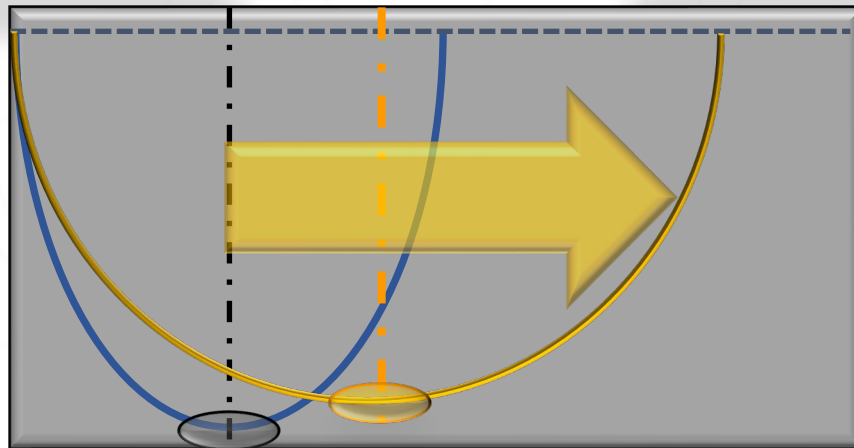


- Overall, graphite CTE is low compared to other structural materials, e.g., metals
 - Implies excellent shock resistance
- Along with dimensional changes, must be accounted for in the design
- Initial increase with dose as manufacturing-related microcracks are closed
 - Limited dependence on **Turnaround**
- Subsequent reduction of CTE at increased dose rate

Irradiation Creep – Life Limiting Mechanism

Irradiation
Dimensional
Change

Irradiation
Creep
(Residual stress
remover)

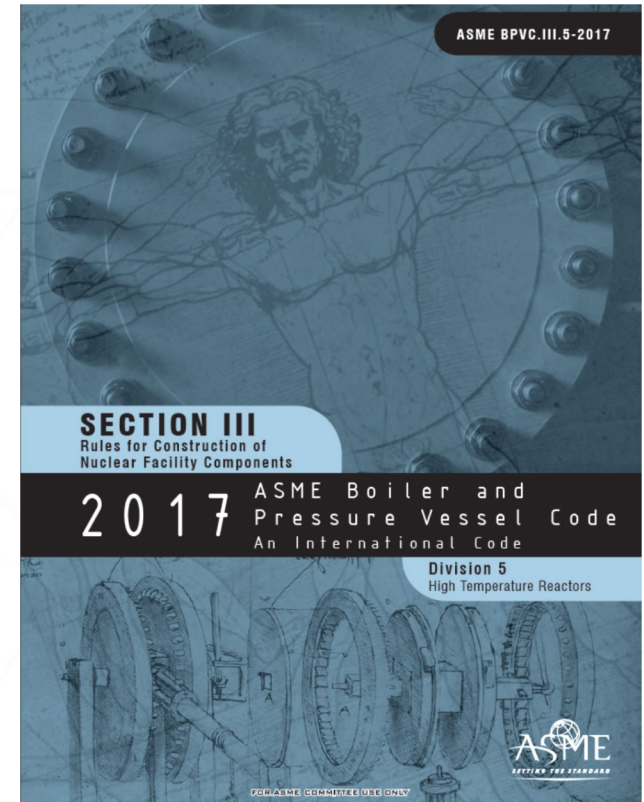


Dose, dpa

- **Reduces internal stresses** resulting from dimensional changes
- Creep strain rate generally increases with temperature
- The net effect is positive in that stresses associated with dimensional changes and differential thermal expansion under irradiation are reduced
- As the total fluence (dose) is increased, this effect becomes increasingly important in attaining acceptable design lifetimes.

ASME Code for Graphite Core Components

- ASME Code for Graphite Core approved by ASME BNCS in early-2010
 - Developed by Section III Subgroup on Graphite Core Components
 - First published in 2012 under Section III, Division 5 (High-Temperature Reactors)
- Key features:
 - Applies to fuel, reflector and shielding blocks, plus interconnecting dowels and keys;
 - *Excludes fuel compacts and pebbles*
 - Rules apply to both individual components and assemblies
 - Applies probabilistic design methods
 - Design must account for statistical variations in graphite properties within billets and for different production runs
 - Design must account for irradiation effects on graphite properties
 - Allowance of cracks in graphite components, provided that safety functions are retained



ASME Code for Graphite Core Components

Three methods are provided for assessing structural integrity

1. Deterministic

- Simplified conservative method based on ultimate strength derived from Weibull statistics

2. Full Analysis Method

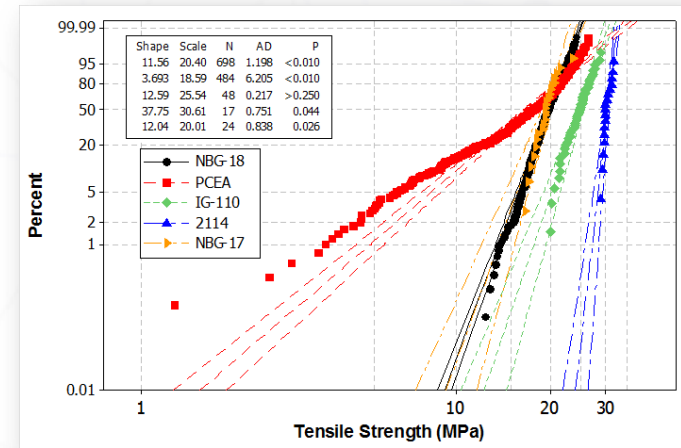
- Detailed structural analysis taking into account loads, temperatures and irradiation history
- Weibull statistics used to predict probability of failure
- Maximum allowable probability of failure defined for three Structural Reliability Classes (**SRCs**), which relate to safety function

3. Qualification by Testing

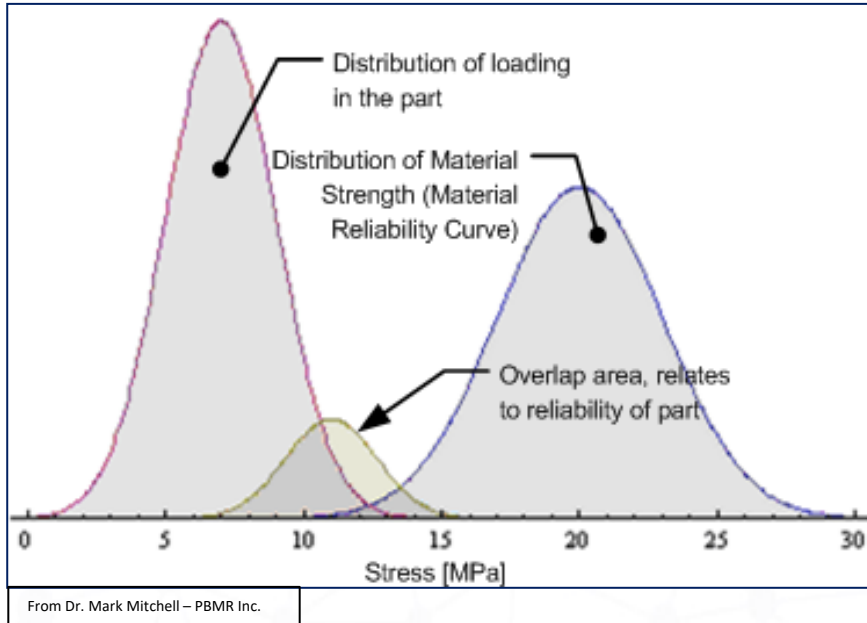
- Full-scale testing to demonstrate that failure probabilities meet criteria of full-analysis method

All methods must consider changes from irradiation and oxidation

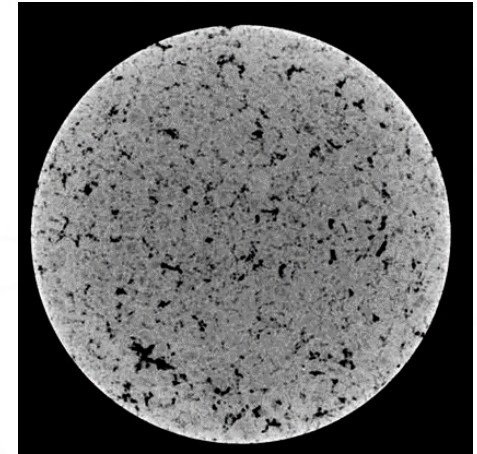
Structural Reliability Class	Maximum Probability of Failure
SRC-1	1.00E-04
SRC-2	1.00E-02
SRC-3	1.00E-01



ASME Code for Graphite Core Components



- New grades (third generation) are consistent and ready for codification
 - Lack of quantitative data on graphite behavior at higher temperature and dose applications
 - Test data is needed to define how precursor material changes, fabrication, and microstructure changes will affect performance
- **Probabilistic** verses **deterministic** design approach
 - Deterministic is too limiting for a brittle material
 - A distribution of possible strengths in a material is needed for quasi-brittle materials (i.e., flaw size for graphite)
- Some amount of failure (i.e., a crack) is certain – graphite is porous
 - The core needs to be designed to accept some amount of failure
 - Probability of failure based upon overlap of applied stresses and graphite strength
 - Irradiation and oxidation effects must be addressed



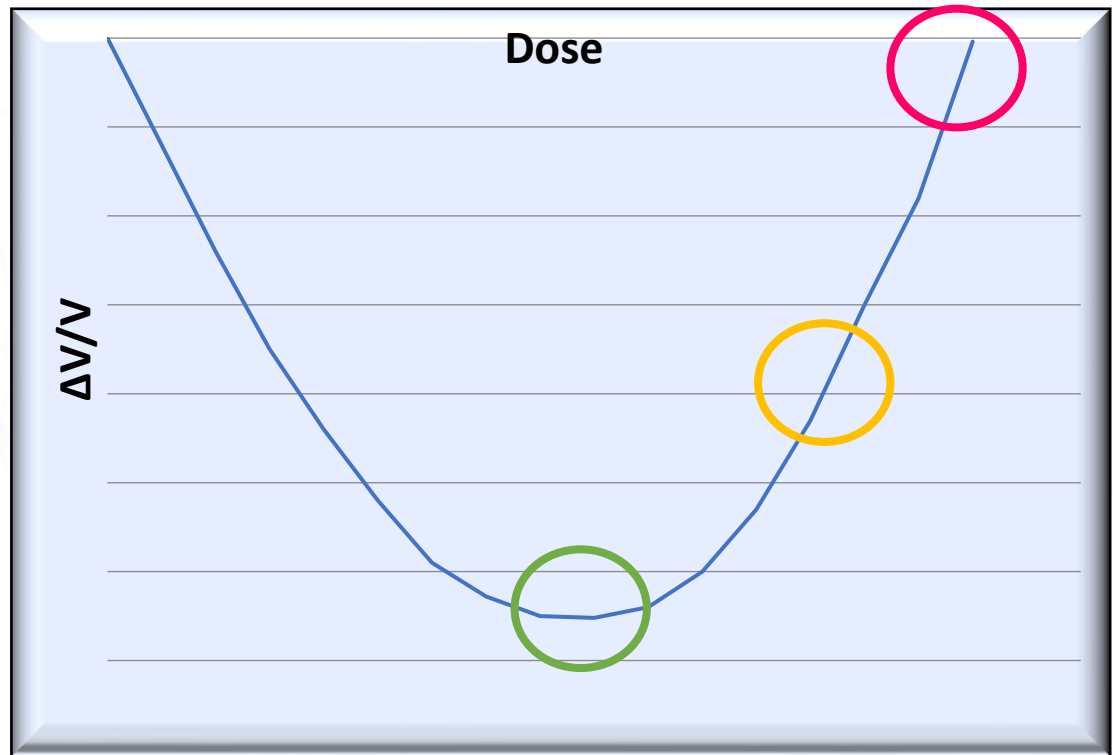
Operational Considerations – Operational Life

When do you replace the graphite?

**Most
Conservative
Dose Level**

**More Risk but
some Rx do
operate here**

Highest Risk



Operational Lifetime Considerations

Pebble Bed

- Highest component lifetime dose
 - What is expected lifetime dose?
 - Turnaround dose? After Turnaround?
- Continuous operation
 - Inspection of components is problematic
 - Component replacement is difficult
- Components in high-fluence regions should be designed for replacement
 - Will require shutdown and de-fueling of pebbles from core
- Large grain grades are possible
 - Higher Turnaround dose than fine grain
 - Lower oxidation rates than fine grain
- Irradiated test data validating models will be required
 - Currently only limited irradiation data for newer nuclear grades
 - Design life to be appropriately adjusted as data become available.
- Dust?

Prismatic

- Lower component lifetime dose
 - Still need expected lifetime dose
- Periodic shutdown
 - Much easier to inspect components
 - Components in high-fluence regions can be replaced or shuffled
- Finer grain grades required
 - Webbing between fuel/coolant channels requires smaller grain size
 - Slightly lower Turnaround dose
 - Higher oxidation rate
- Still requires irradiated test data to validate operational models
 - Currently only limited irradiation data for newer nuclear grades
 - Design life to be appropriately adjusted as data become available

Conclusions

- All graphite nuclear grades are proprietary
 - Graphite is porous – by design
 - Compressive applications only ($\sigma_c \gg \sigma_t$)
- Irradiation behavior is required for design
 - Dimensional change and creep is life limiting mechanism
 - Strength/internal stress is dose dependent
- Degradation/Oxidation of graphite
 - Graphite does not burn (but it does oxidize at high temperatures)
 - Oxidation limited to 10% mass loss. Then replace the component
- In-service Inspection
 - Easy for Prismatic designs. More difficult for Pebble designs
 - Visual and physical inspection of accessible areas during refueling or maintenance
 - In-situ Measurements (primarily interest to pebble reactors)
- ASME Code
 - Probabilistic design calculations
 - Some amount of failure (i.e., a crack) is nearly certain over time
- Operational considerations – Pebble and prismatic
 - What is the lifetime dose of component?
 - Is this after Turnaround dose?
 - Can core be inspected? How are components to be replaced if required?
 - Oxidation rates of graphite (small versus larger grain grade)

Suggested Reading

- Manufacturing

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- ASME Code and Licensing

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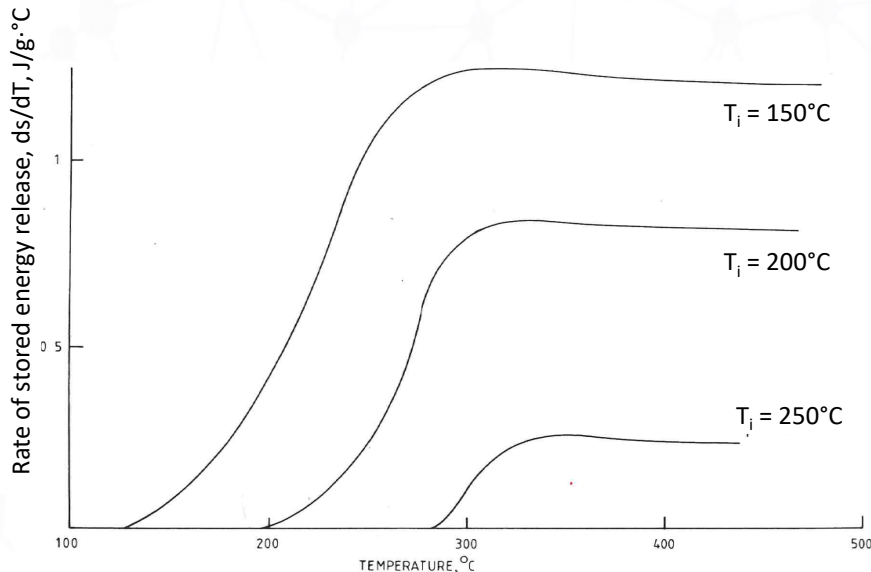
Source-dependence on graphite properties

- There is no generic “nuclear grade” graphite that can be made by all vendors
 - All nuclear graphite grades **are proprietary**. How they are made is secret to the individual vendor.
 - *Completely different than metals. There is no fabrication information available for any grade.*
 - Graphite users must select the grades that match their specific requirements.
 - And no, vendors won't give up their recipes. There is no customer base asking for it.
- As discussed in fabrication slide the unique graphite manufacturing processes dictate the graphite behavior – both unirradiated and irradiated.
 - Main fabrication parameters are:
 - *coke source: petroleum or coal-based coke source*
 - *grain size: coke particles (grains) range in size from 1800 μm to 15 μm*
 - *fabrication method: iso-static molded, vibration molded, or extruded fabrication*
 - *Grain-binder ratio: the amount of carbonaceous binder added to the grain particles*
 - Modifying these parameters can dramatically alter the unirradiated material properties and irradiation performance

Parameter	Unirradiated Behavior	Irradiated Behavior
Increased Density	Increased strength and modulus Higher fracture strength	A general decrease in Turnaround dose <ul style="list-style-type: none">• Shorter component lifetime
Isostatic fabrication	Higher isotropy (than extruded) Higher cost material	Better, more predictable, irradiation performance.
Smaller grain size	More uniform, finer microstructure <ul style="list-style-type: none">• Especially when isostatic molded Higher oxidation rate than larger grained	Super-fine grades <u>may</u> have lower Turnaround dose

Minimal effects to graphite from irradiation

- No significant changes occurs in:
 - Neutron moderation – Carbon atoms not removed
 - Specific heat capacity – Crystal structure remains intact
 - Oxidation rate - Minimal changes if any due to densification during irradiation.
 - Molten salt interaction – Graphite behavior (unirr. and irr.) similar to gas-cooled
 - Physical damage possible from salt intrusion into pores in graphite components
 - Emissivity:
 - Unaffected by irradiation but oxidation may leave impurity oxides on outer surface.



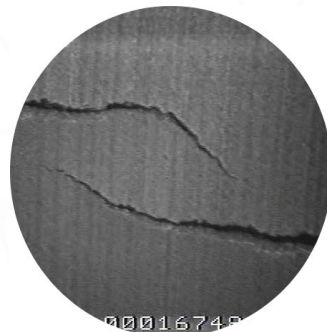
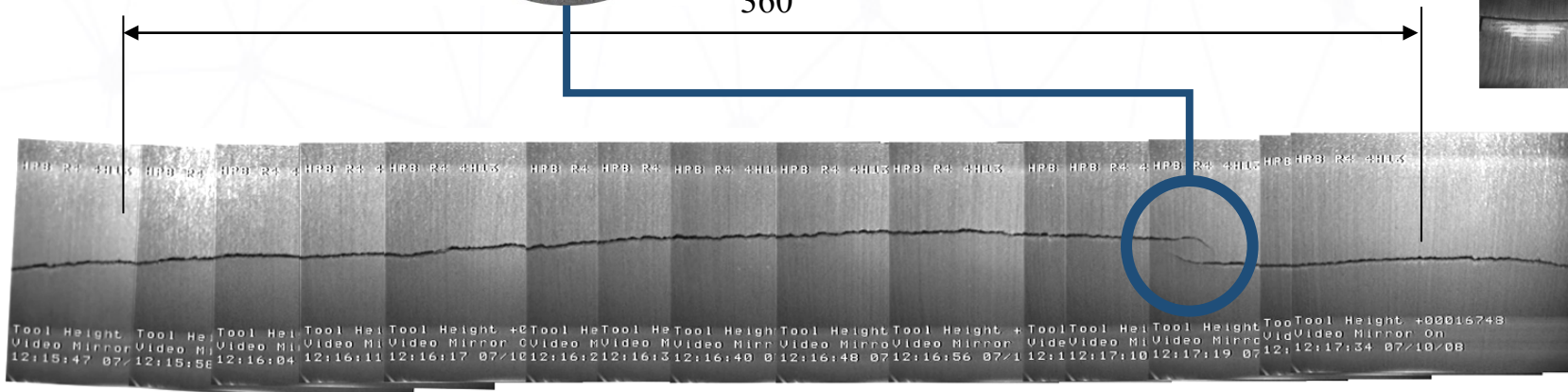
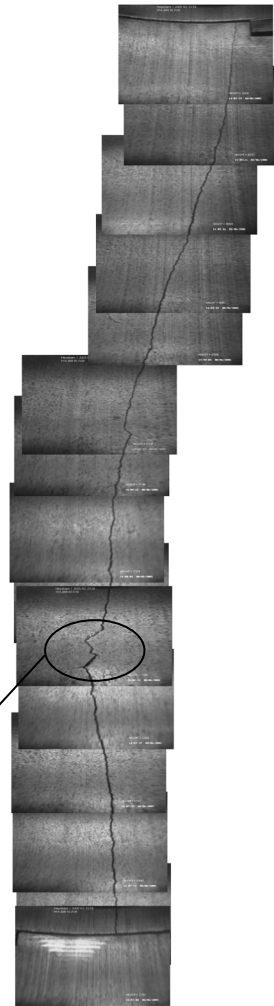
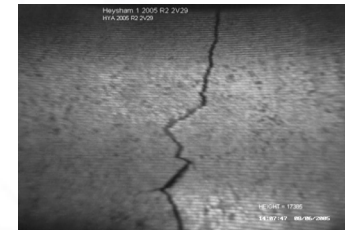
- Minimal Wigner energy release if components irradiated above 300°C.
 - Annealing of point defects in graphite is rapid above 250°C
 - Minimal accumulation of stored energy
- Need **high dose & low T_i**
 - Low dose/low T_i components have reduced risk

Graphite Component Failure



AGR Core components

- What do we mean by structural integrity
 - U.K.'s AGR bricks – ***Now past Turnaround dose***
 - Example of graphite component failure.
 - Both axial as well as radial cracking in components
- Lifetime is completely dependent upon graphite core now
 - Not fuel design/performance, metallic internals, or secondary systems

 360° 

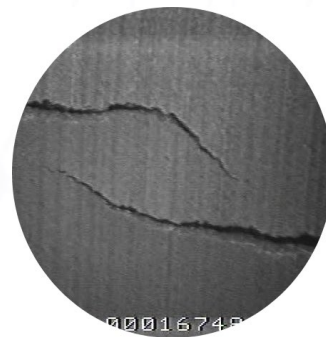
Graphite Component Failure



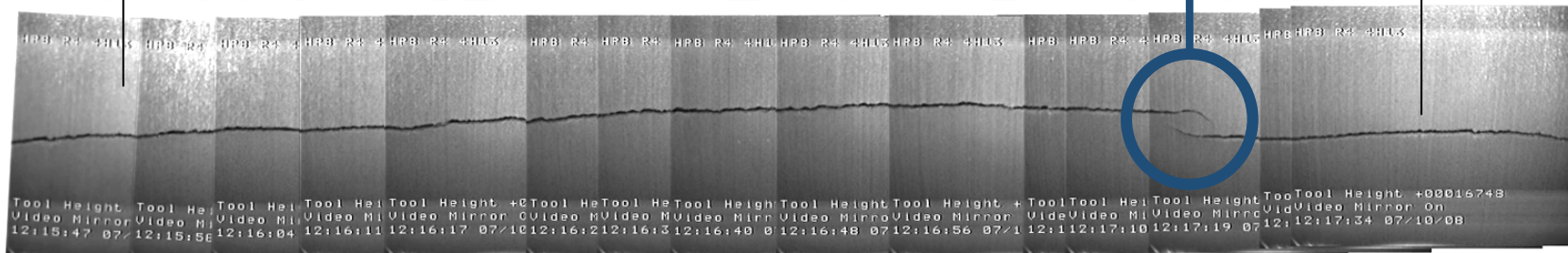
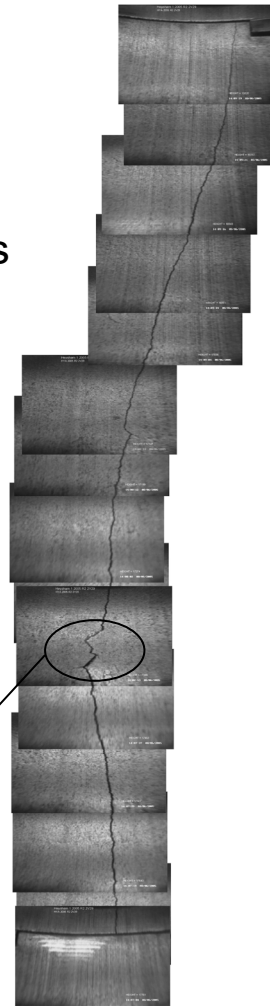
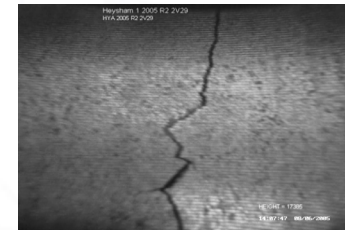
AGR Core components

- **CAUTION!**
 - U.K. AGR uses CO₂ for coolant
 - Radiolytic oxidation exacerbates all strength changes
- Inert gas cooled designs will be more robust
 - Component strength, internal stresses, and POF will be much different than CO₂ cooled AGRs

From: J. Reed, Summary of Recent Inspection Data at UK Advanced Gas Cooled Reactors with Implications for Assessment of Graphite Component Integrity, INGS-17, 4-8 September 2016, IAEA, Vienna, Austria

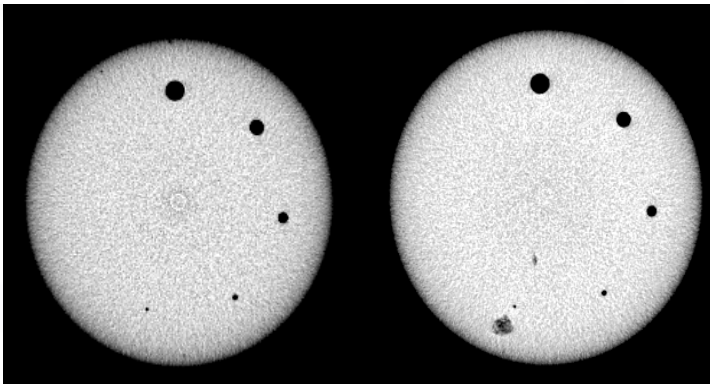


360°

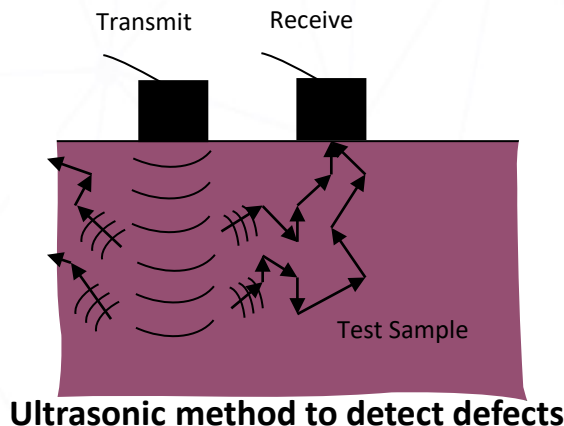


Component inspection (NDE techniques)

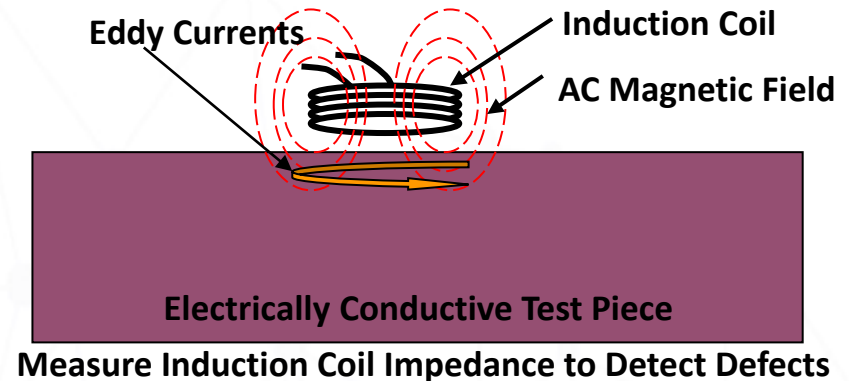
- Visual inspection, Eddy current, Ultrasonic, and X-ray inspection is possible
 - Thick graphite components are difficult to inspect
 - Flaw size resolution (i.e., cracks) are difficult to resolve in thick components
 - Visual, Eddy current, and small sample trepanning are current methods used
 - U.K.'s AGR inspection program
 - No good technique exists. Destructive analysis (trepanning) yields most information



Detection of flaws (drilled holes) from X-ray method



Ultrasonic method to detect defects



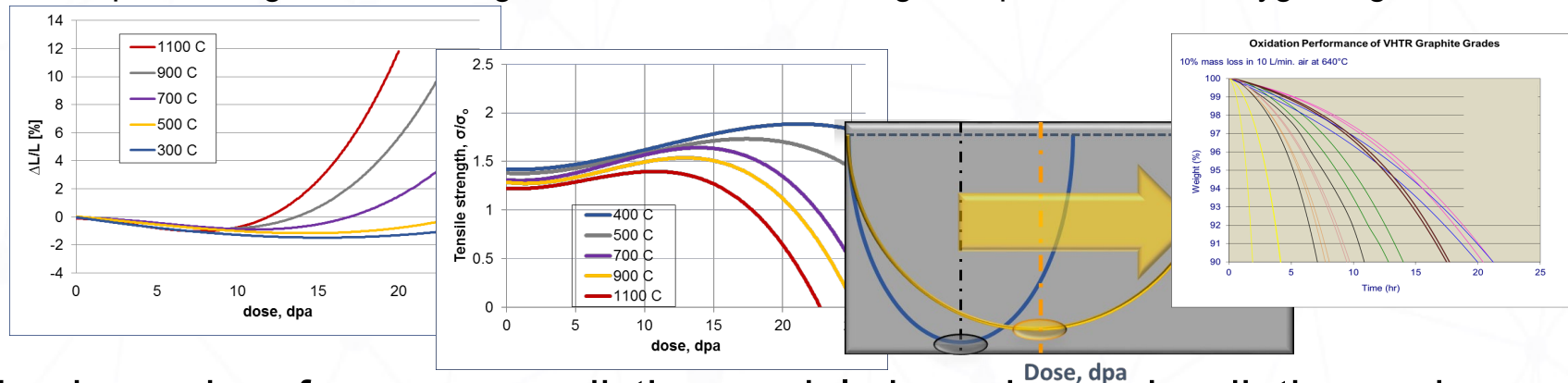
- ASTM D8093 Standard Guide for Nondestructive Evaluation of Nuclear Grade Graphite
 - Guideline on how to use various Non-Destructive Examination (NDE) techniques to graphite core components.

ASME code methodology for graphite - 1

- Two key points to keep in mind:
 1. All nuclear graphite is proprietary – Specific fabrication recipes are unknown
 - The properties for each grade are highly dependent on the recipe and **are optimized** (altered) to suit each users requirements.
 2. Graphite is brittle (quasi-brittle).
 - Metals are ductile giving them the ability to fail in a predictable manner.
 - Graphite fails much like ceramic – Probability of failure (POF) due to flaw size distributions
 - Weibull analysis historically used to predict the probability of failure and characteristic strength of brittle and flaw dependent materials.
- Consequently, there are no “standard” specifications such as metals have
 - ASTM D7219 specifies impurity levels only. Other properties are *desired* ranges.
 - It's like specifying “Stainless steel” for a component (not 304, 316, or 316L)
 - The selected grade is then fabricated to the specific requirements of component
 - **However**, not much variation over all the grades. Not like metals
 - $K_{Ic} \sim 0.5 - 1.5 \text{ Pa}\cdot\sqrt{\text{m}}$, $\sigma_t = 15\text{-}30 \text{ MPa}$, $4.5 - 5.5 \times 10^{-6}$, etc.
- Thus, graphite code is a “process” vs just picking a preapproved material
 - The reactor applicant must demonstrate the graphite grade selected will consistently meet the component requirements.
 - Requires property testing and analysis of the material properties **before** is durability as a nuclear component is analyzed.
 - Achieved through the “Material Data Sheets” required in Code.
 - Weibull parameters from strength tests used to predict the probability of failure of graphite.
 - Data used in both “simple” (deterministic) and “full” (probabilistic) determination

ASME code methodology for graphite -2

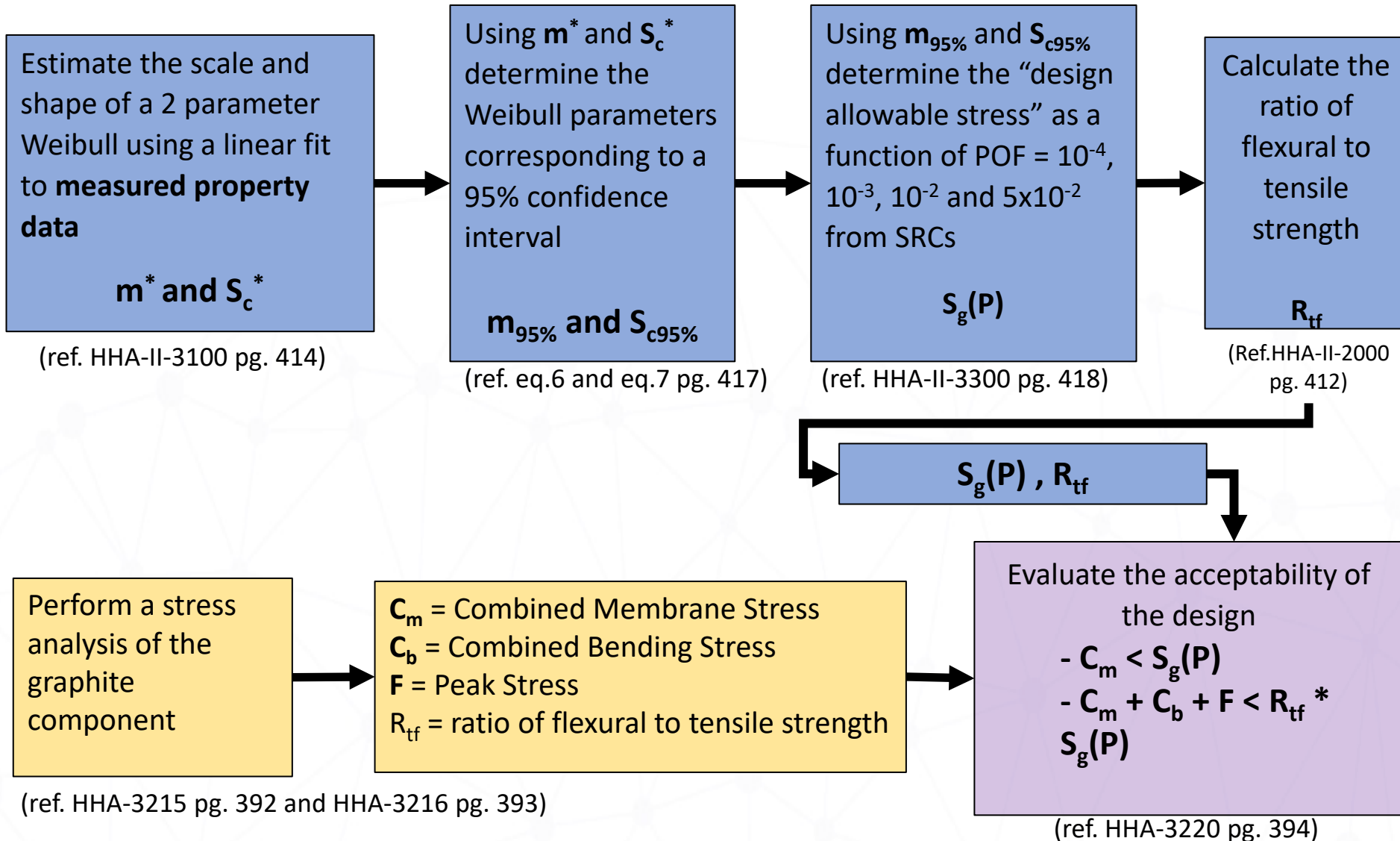
- Fundamental material properties change with irradiation/oxidation
 - Code must assess changes to design of component due to these changes
 - Irradiation: changes to density, strength, dimension, CTE, thermal conductivity
 - Oxidation: changes in density, strength, CTE, and thermal conductivity.
 - Code must also address these changes to in service and inspection
 - NDE and ISI are still outstanding issues that need to be addressed for graphite
- Material testing and analysis must be performed to determine changes
 - Property changes and irradiation creep to maximum expected dose levels.
 - Oxidation rates, property changes, and strength assessment to maximum expected oxidation levels
 - Expected degradation during off-normal events with high temperatures and oxygen ingress



- Behavior and performance prediction models based upon irradiation and oxidation experimental results.
 - Property degradation due to oxidation, irradiation, and dimensional stress buildup.
 - Fracture behavior and structural integrity = Primary

Summary of Simplified Graphite Assessment

Simple Assessment: 2 parameter Weibull (**Deterministic Analysis**)



Summary of Full Graphite Assessment

Full Assessment: 3 parameter Weibull (**Probabilistic Analysis**)

Define the “**Material Reliability Curve**” by fitting a 3 parameter Weibull model to the measurement data.

(ref. HHA-II-3200 pg. 417)

Estimate 3 parameter Weibull parameters using MLE's .

$(S_o, m_{095\%} \text{ and } S_{c095\%})$

(ref. HHA-3217 pg. 393)

Calculate the POF of the graphite core component using the “**Material Reliability Curve**” and stress distribution in the component.

$POF_{\text{component}}$

Determine the allowable POF from the **Structural Reliability Class (SRC)**, and **Service Level Design Loading**.

(ref. HHA-3230 thru HHA-3237 pg. 397)

$POF_{\text{allowable}} \leq 10^{-2} \text{ and } \geq 10^{-4}$

Evaluate the acceptability of the design

$POF_{\text{component}} < POF_{\text{allowable}}$

ASME Code modifications (Roadmap)

- Corrosion rate variability within a nuclear grade
 - Oxidation test specimens should require testing specimen be selected at different locations within a billet, over multiple billets, and over multiple batches.
 - *This will provide the oxidation rate variability across the entire specific grade*
 - Currently the oxidation mass loss for a component is limited to 10 wt%.
 - *After 10 wt% the component is recommended to be replaced.*
 - *Code needs to provide guidance on how the oxidation mass loss is applied.*
 - *Averaged over entire core? Only in central core region? Or only for select components?*
- High temperature mechanical testing isn't really necessary for graphite
 - As noted mechanical strength and modulus increase with increasing temperature.
 - Room temperature results are conservative for graphite
 - No elevated temperature testing standards exist to support this current requirement
 - *(i.e., no ASTM standards)*
 - How is elevated temperature testing of irradiated material to be conducted?
 - *Testing temperatures at (or above) T_{irr} will anneal out irradiation effects*
- Mechanical testing of irradiated material is unnecessary up to Turnaround
 - As noted mechanical strength and modulus increase with increasing dose – until Turnaround dose has been reached.
 - Room Temperature/unirradiated mechanical testing is conservative until Turnaround dose has been achieved.
 - If components will be used to dose levels above Turnaround (i.e., high dose levels) extensive testing will be required