Nuclear Graphite Components

Advanced Reactor Technologies

Idaho National Laboratory

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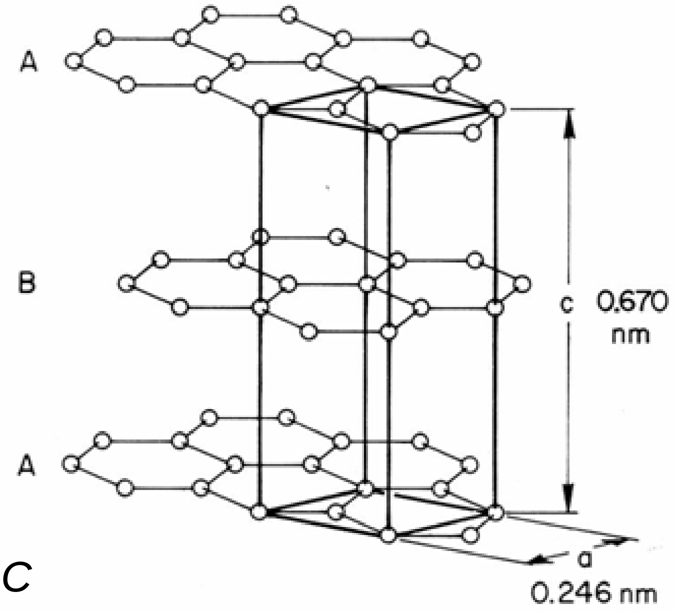
INL Idaho National Laboratory

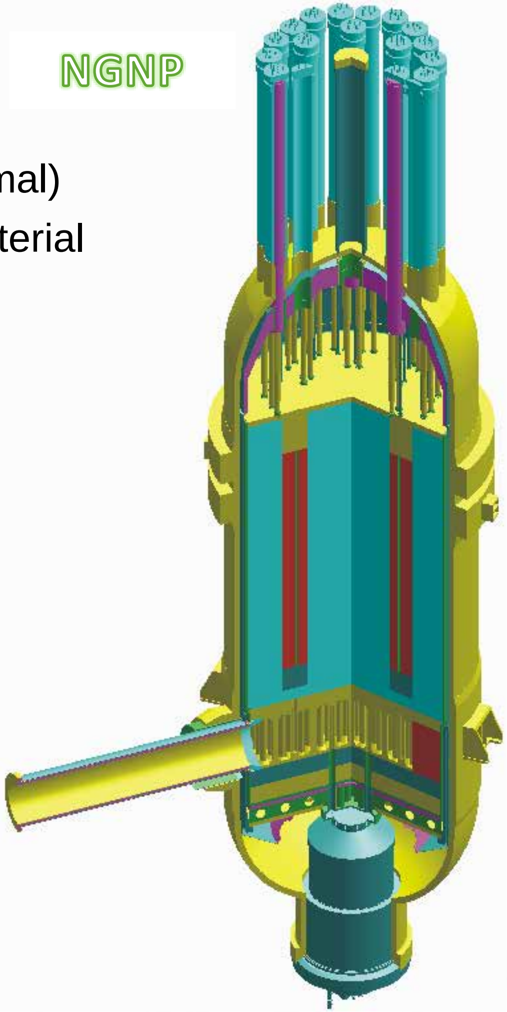
ART ADVANCED REACTOR TECHNOLOGIES

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



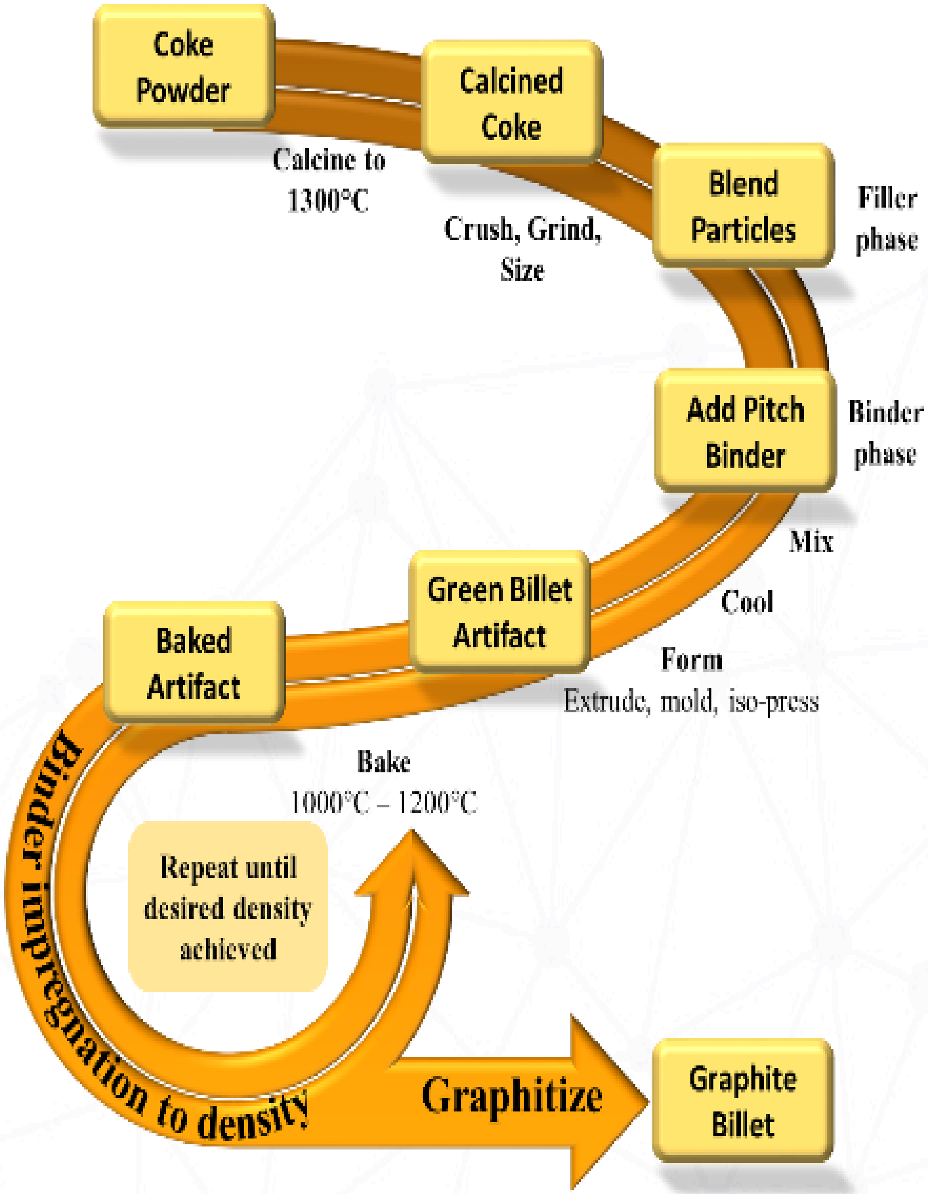
- Maintain core geometry and structural integrity
  - Maintain fuel configuration during all operations (normal and off-normal)
  - Maintain undisturbed access for the insertion of reactivity control material
  - Maintain proper core coolant configuration
    - *No blockage of coolant pathway*
    - *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*

- <u>How does it do this?</u>
  - Graphite does **NOT** melt or burn
  - Graphite **DOES** have high thermal conductivity and thermal stability
  - Relatively strong in compression, weak in tension.

Graphite Manufacture



Coke Powder

Calcine to 1300°C

Calcined Coke

Crush, Grind, Size

Blend Particles

Filler phase

Add Pitch Binder

Binder phase

Mix

Cool

Form

Extrude, mold, iso-press

Green Billet Artifact

Bake

1000°C – 1200°C

Baked Artifact

Binder impregnation to density

Repeat until desired density achieved

Graphitize

Graphite Billet

- 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

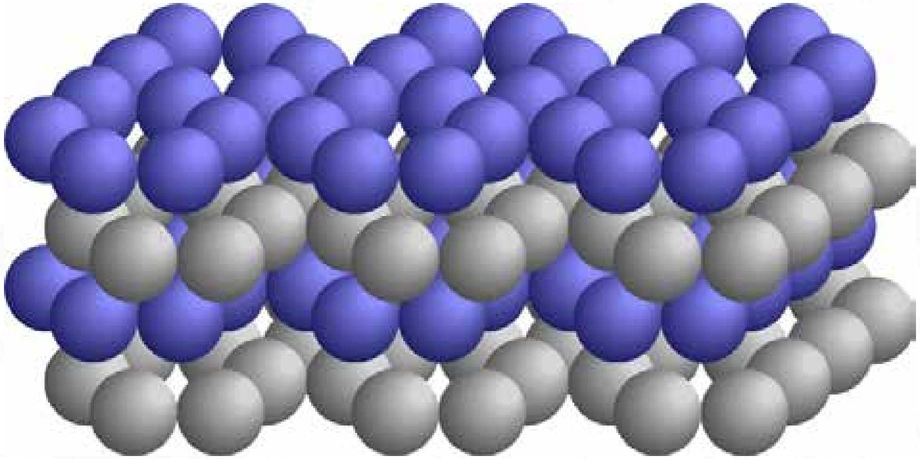
Property	Nominal Range	Performance Attributes
Density	1.7 - 1.9 g/cm^3	Neutron efficiency, Structural integrity, Thermal efficiency
Thermal Conductivity (at Room Temperature)	> 90 W/m/K	Heat transport
Purity (Total Ash Content)	< 300 ppm	Reduced component activity levels during replacement and/or disposal 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)

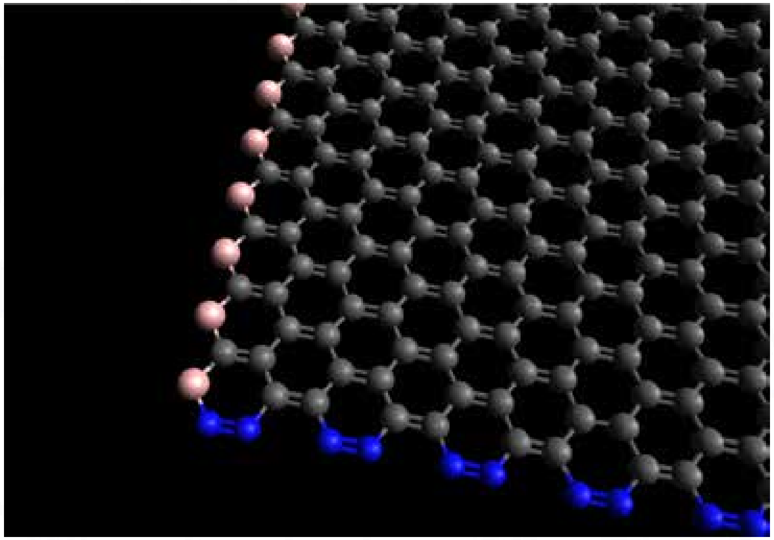
From ASTM D7219 : *Standard Specification for Isotropic and Near-isotropic Nuclear Graphites*

- **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

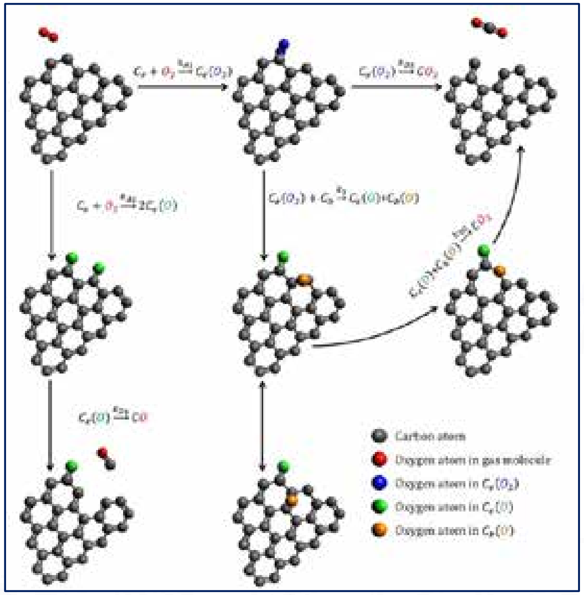
Acheson

White hot graphite from furnace

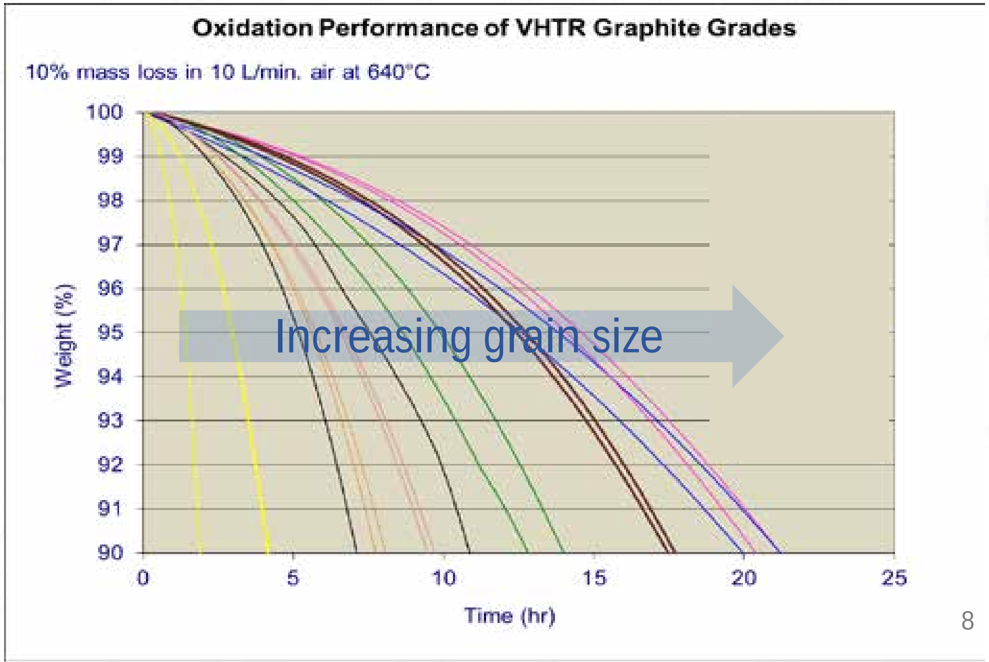
Corn (Maize) Dust

Graphite Oxidation and “Burning”

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

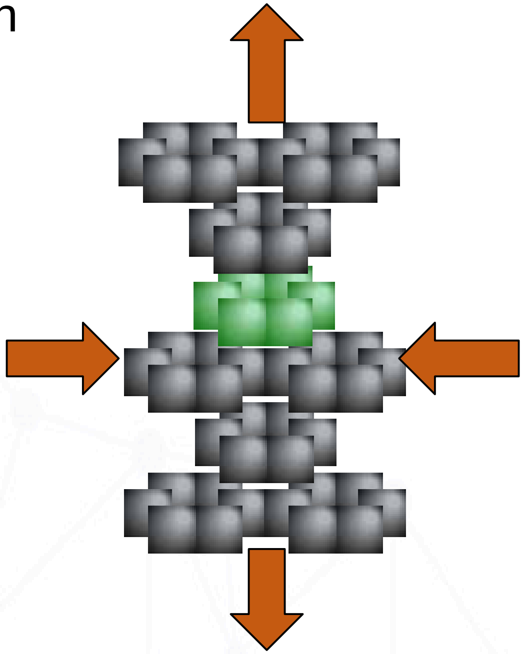


- Needs continuous oxygen and temperatures above 200°C – 300°C
  - Temperatures > 400°C needed for more rapid acute oxidation (accidents)
  - Temperatures < 400°C can still oxidize but at very slow rates (chronic oxidation)
- Oxidation still restricted to edges of crystallites with porosity dictating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



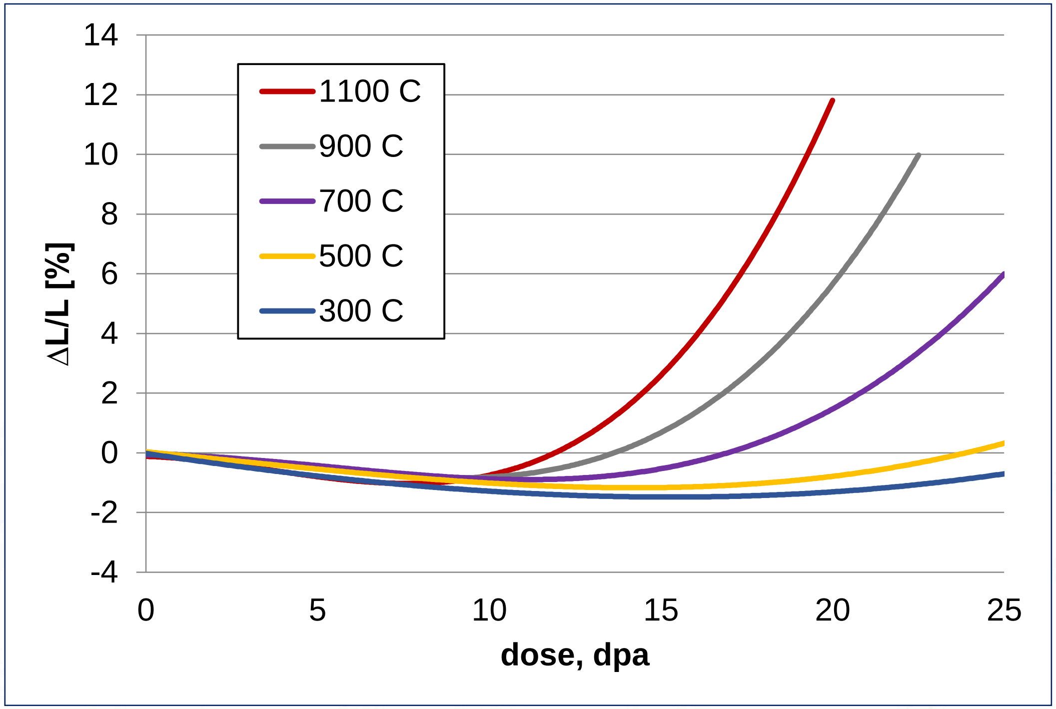
- Oxidation rates of different grades can be compared using ASTM D7542 standard, “Air Oxidation of Manufactured Carbon and Graphite in Kinetic Regime”
  - Grain size dependent
  - Oxidation of small grain grade >> than large grain size

Irradiation Effects on Graphite Properties



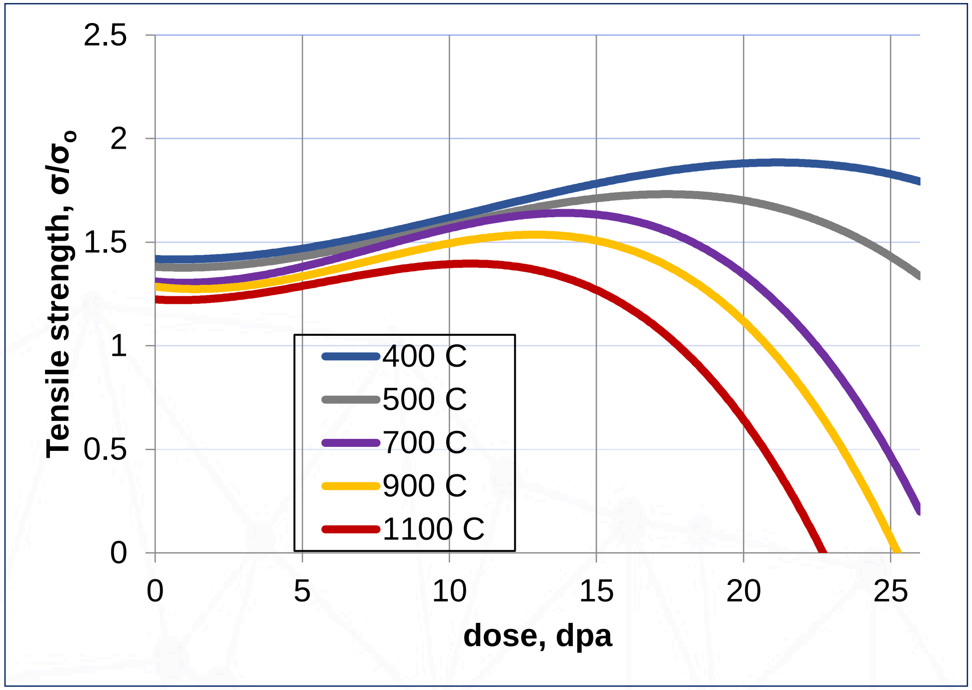
- Irradiation induced changes **must** be considered in design
- Significant changes occur during normal operation in:
  - Component dimensions
    - *Components actually shrink …*
    - *Until **Turnaround** when they begin to expand until failure*
  - Density
    - *Components become more dense …*
    - *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

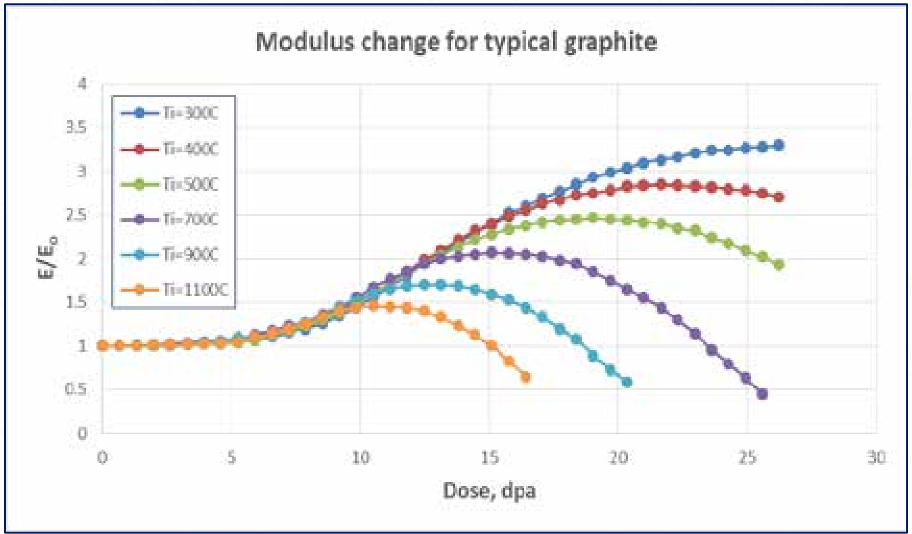


- Under neutron irradiation graphite components shrink (densify) – stop at **Turnaround** – then begin to expand (crack formation)
  - Change is dose dependent: Higher doses = larger change
  - Rate of change is highly temperature dependent
  - Rate and amount of change is grade specific
- Results in 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

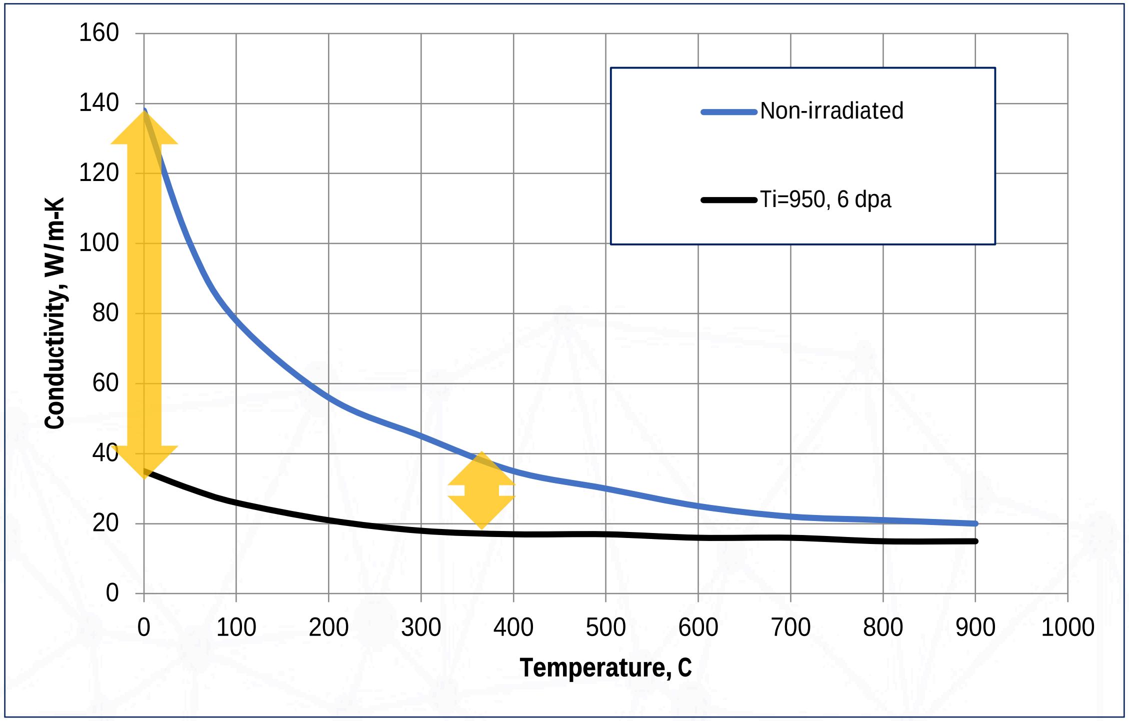


- Changes in strength and modulus somewhat parallel dimensional changes
- Strength/modulus initially increase
  - Maximum value is reached at approximately the **Turnaround** dose



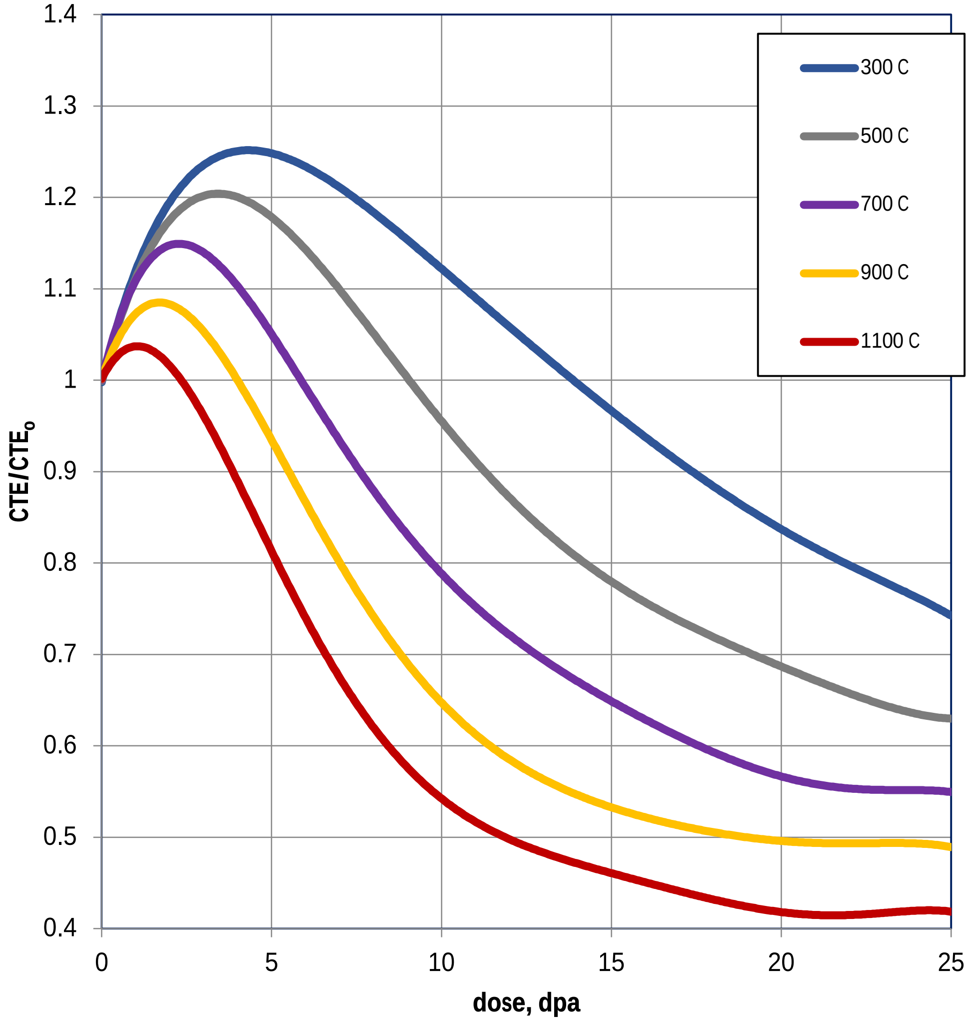
- **After Turnaround** pores start to form in microstructure
  - As porosity forms, strength and modulus fall at increasing rate
- As with dimensional changes, strong dependence on irradiation temperature

Irradiation-Induced Thermal Conductivity Changes



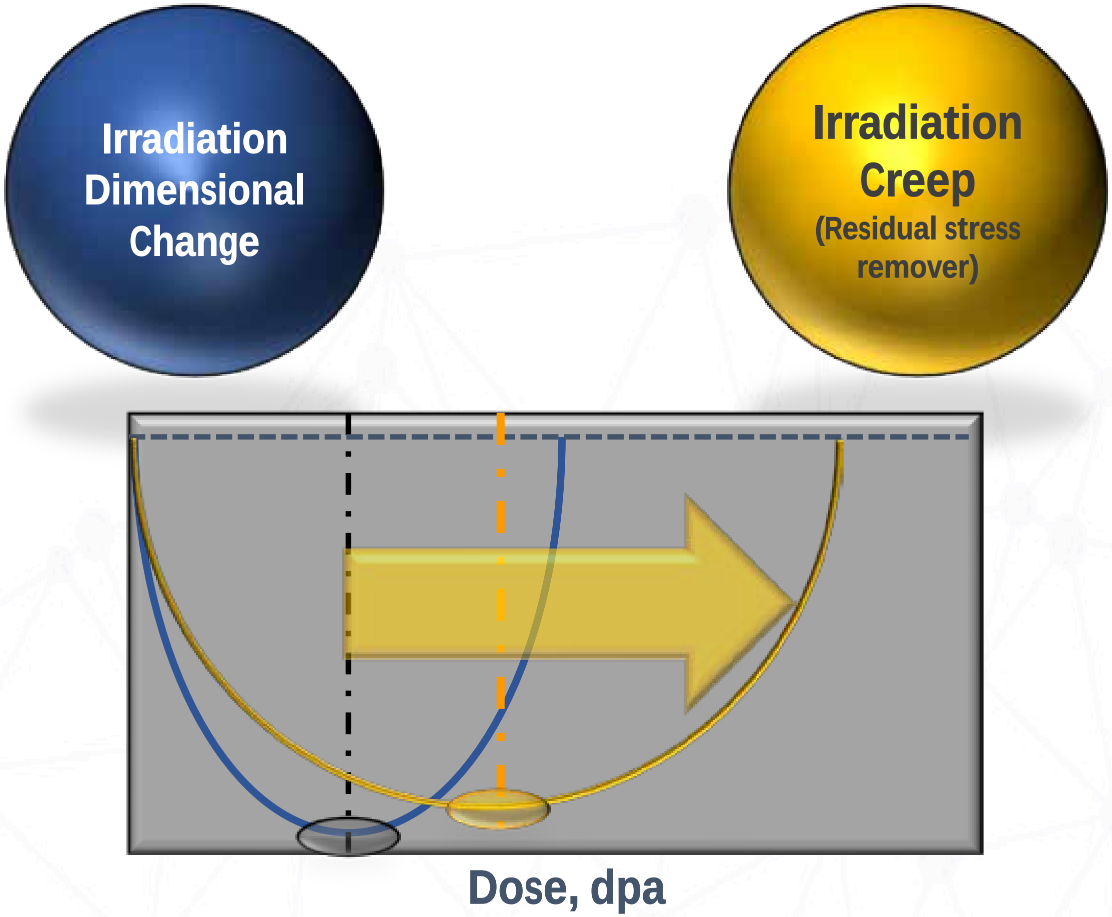
- Initial steep drop in conductivity followed by a saturation level
  - Point defects interrupt thermal diffusivity/conductance
  - Efficiency of recombination rate of point defects is dependent upon irradiation temperature = saturation
  - Further degradation of conductivity due to larger microstructure 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

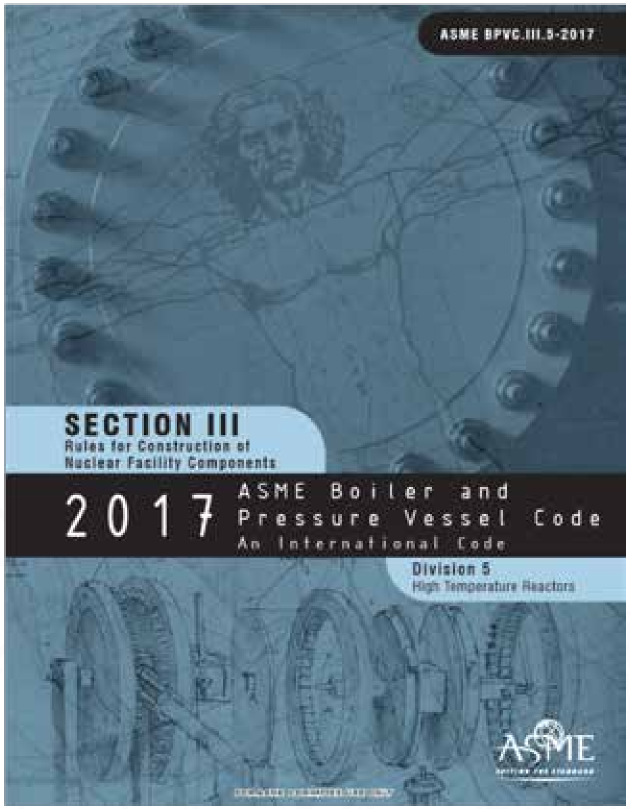
Irradiation Creep – Life Limiting Mechanism



- **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



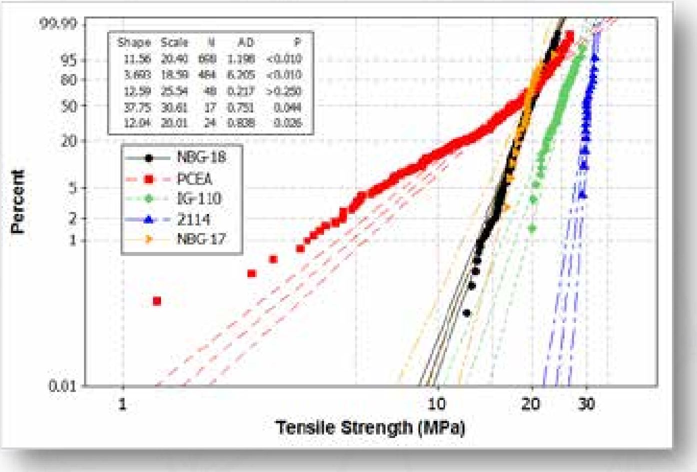
ASME Code for Graphite Core Components

Three methods are provided for assessing structural integrity

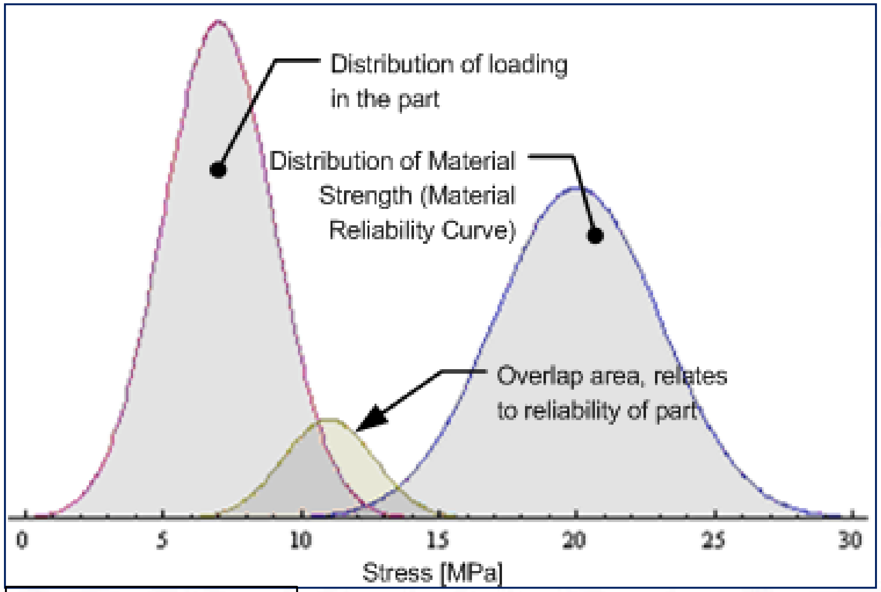
1. Deterministic
   - Simplified conservative method based on ultimate strength derived from Weibull statistics
2. Full Analysis Method
   - Detailed structural analysis taking into account loads, temperatures and irradiation history
   - Weibull statistics used to predict probability of failure
   - 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



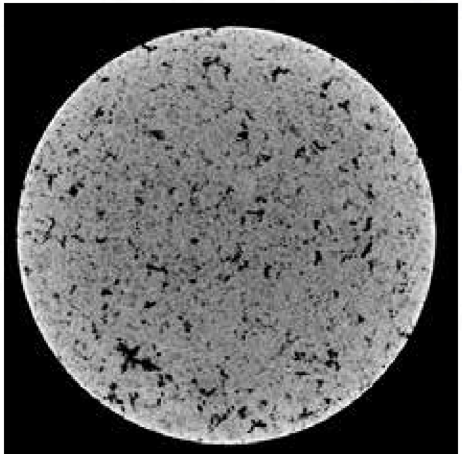
ASME Code for Graphite Core Components



From Dr. Mark Mitchell – PBMR Inc.

- 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
- **Probablistic** 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



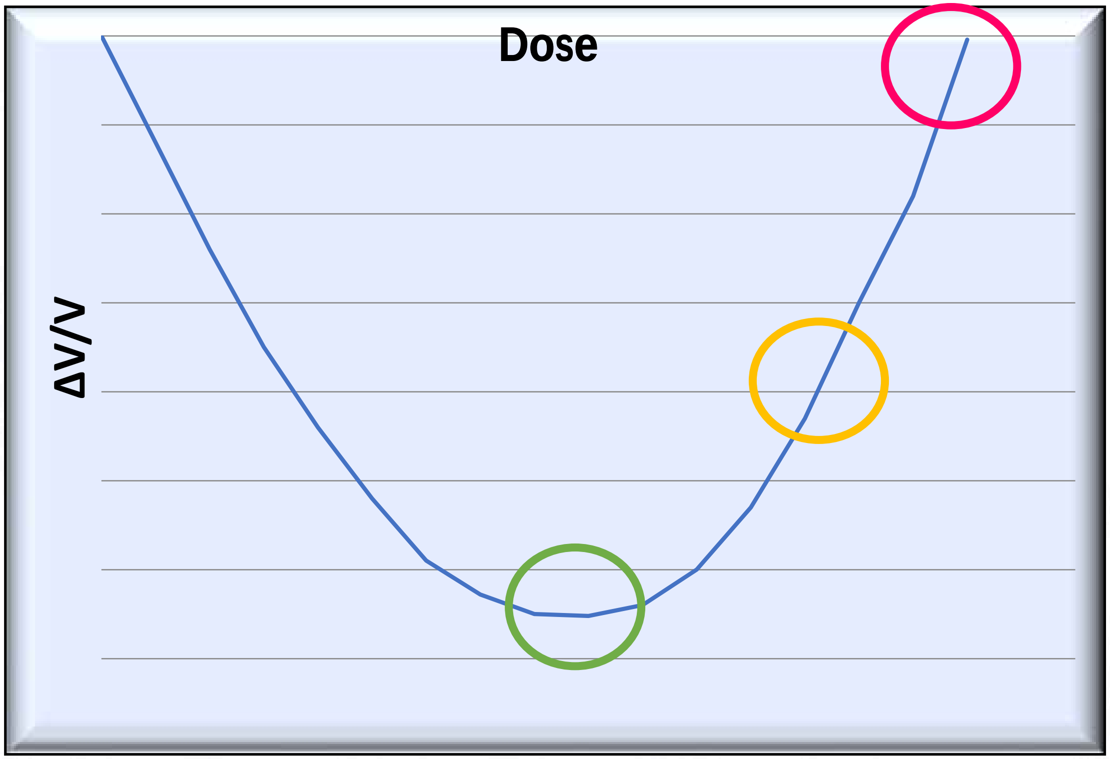
Operational Considerations – Operational Life

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 ($\sigma_c >> \sigma_t$)
- 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

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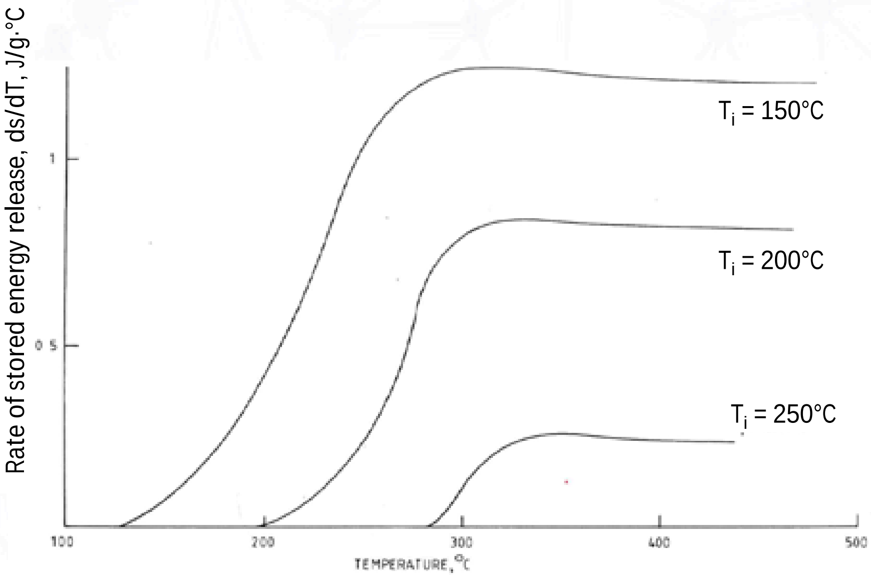
Source-dependence on graphite properties

- 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 wont 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 • Shorter component lifetime
Isostatic fabrication	Higher isotropy (than extruded) Higher cost material	Better, more predictable, irradiation performance.
Smaller grain size	More uniform, finer microstructure • 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 occur 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.

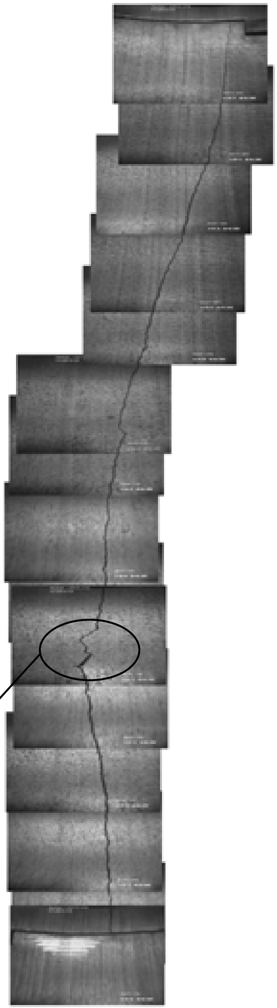


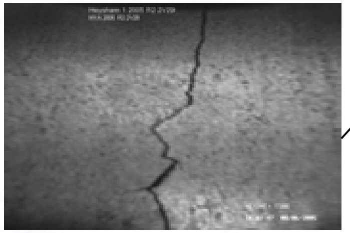
- Minimal Wigner energy release if components irradiated above 300°C.
  - Annealing of point defects in graphite is rapid above 250°C
  - Minimal accumulation of stored energy
- Need **high dose** & **low T_i**
  - Low dose/low T_i components have reduced risk

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





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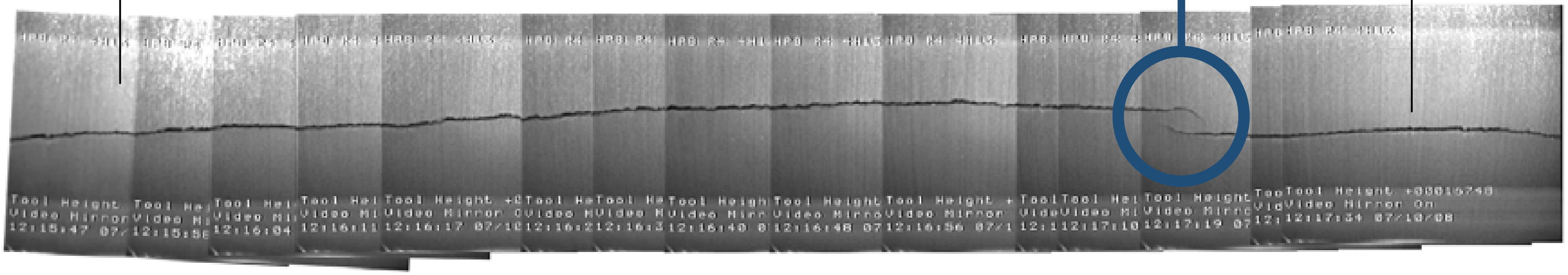
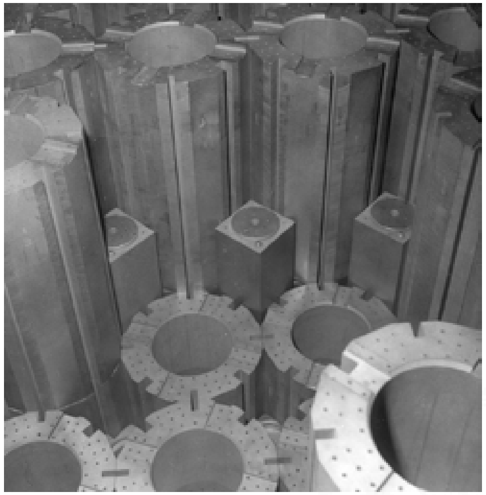


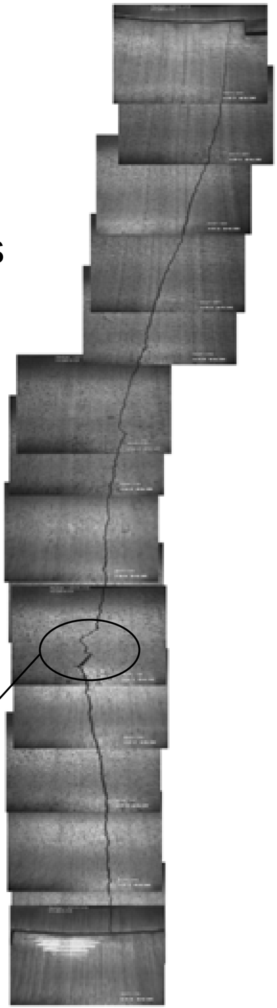
Graphite Component Failure

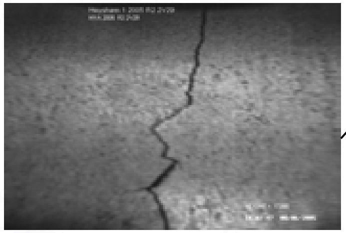


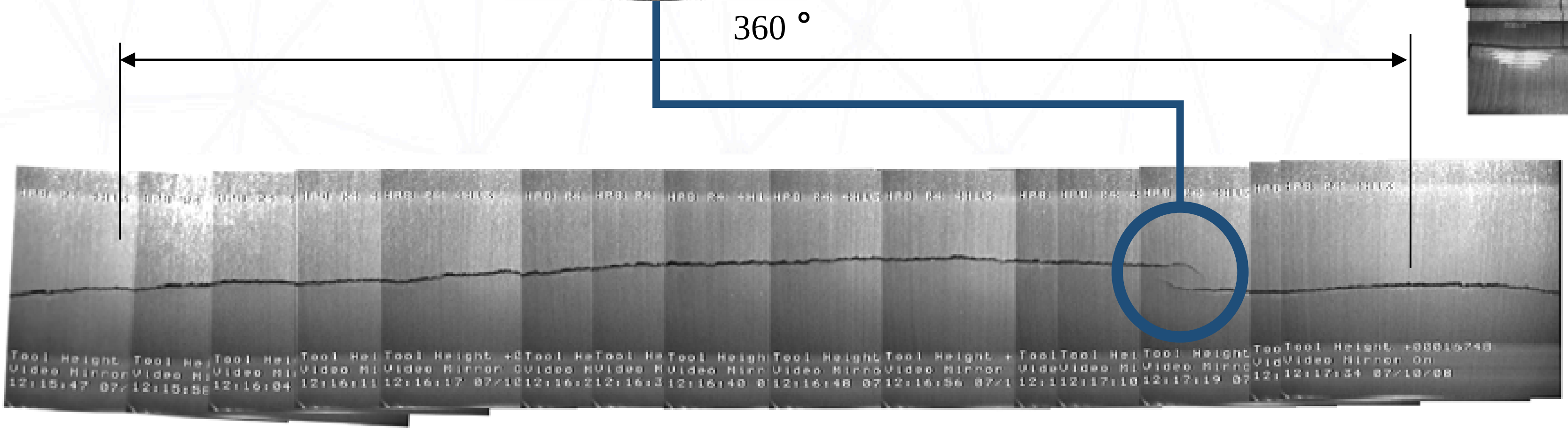
AGR Core components

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

- **CAUTION!**
  - U.K. AGR uses CO_2 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 CO_2 cooled AGRs

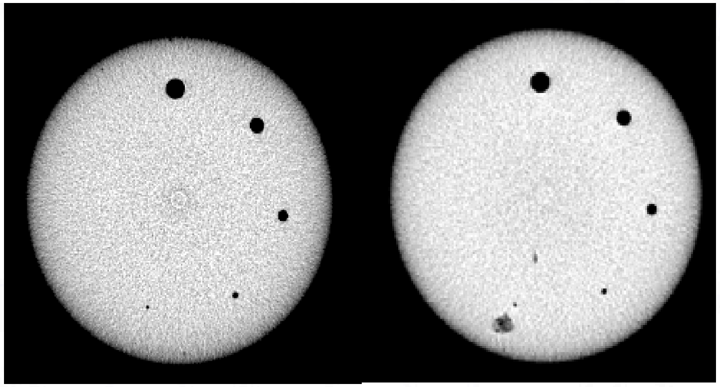




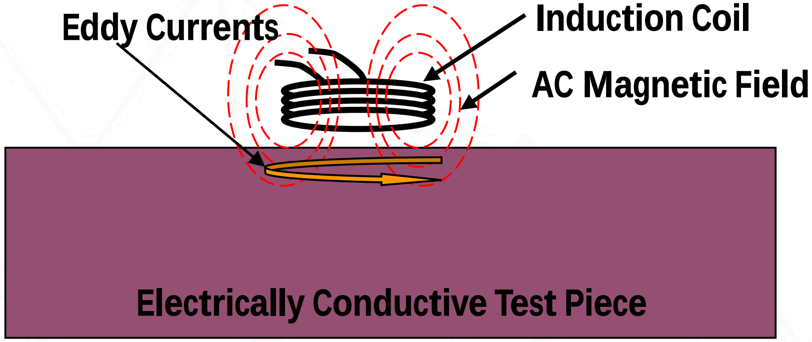


Component inspection (NDE techniques)

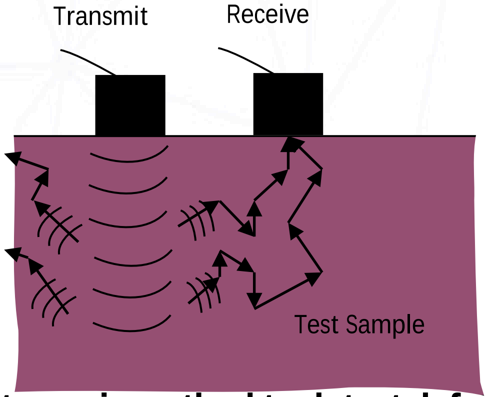
- Visual inspection, Eddy current, Ultrasonic, and X-ray inspection is possible
  - Thick graphite components are difficult to inspect
    - Flaw size resolution (i.e., cracks) are difficult to resolve in thick components
  - Visual, Eddy current, and small sample trepanning are current methods used
    - *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



Measure Induction Coil Impedance to Detect Defects



Ultrasonic method to detect defects

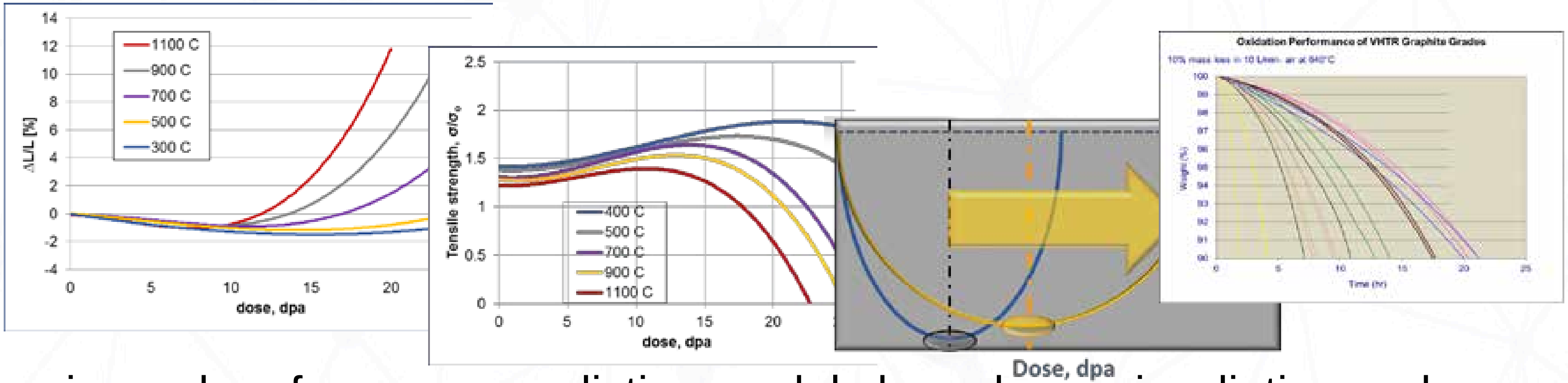
- ASTM D8093 Standard Guide for Nondestructive Evaluation of Nuclear Grade Graphite
  - Guideline on how to use various Non-Destructive Examination (NDE) techniques to graphite core components.

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} ~ 0.5 – 1.5 Pa·√m, σ_t = 15-30 MPa, 4.5 – 5.5 x 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 <u>consistently</u> 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

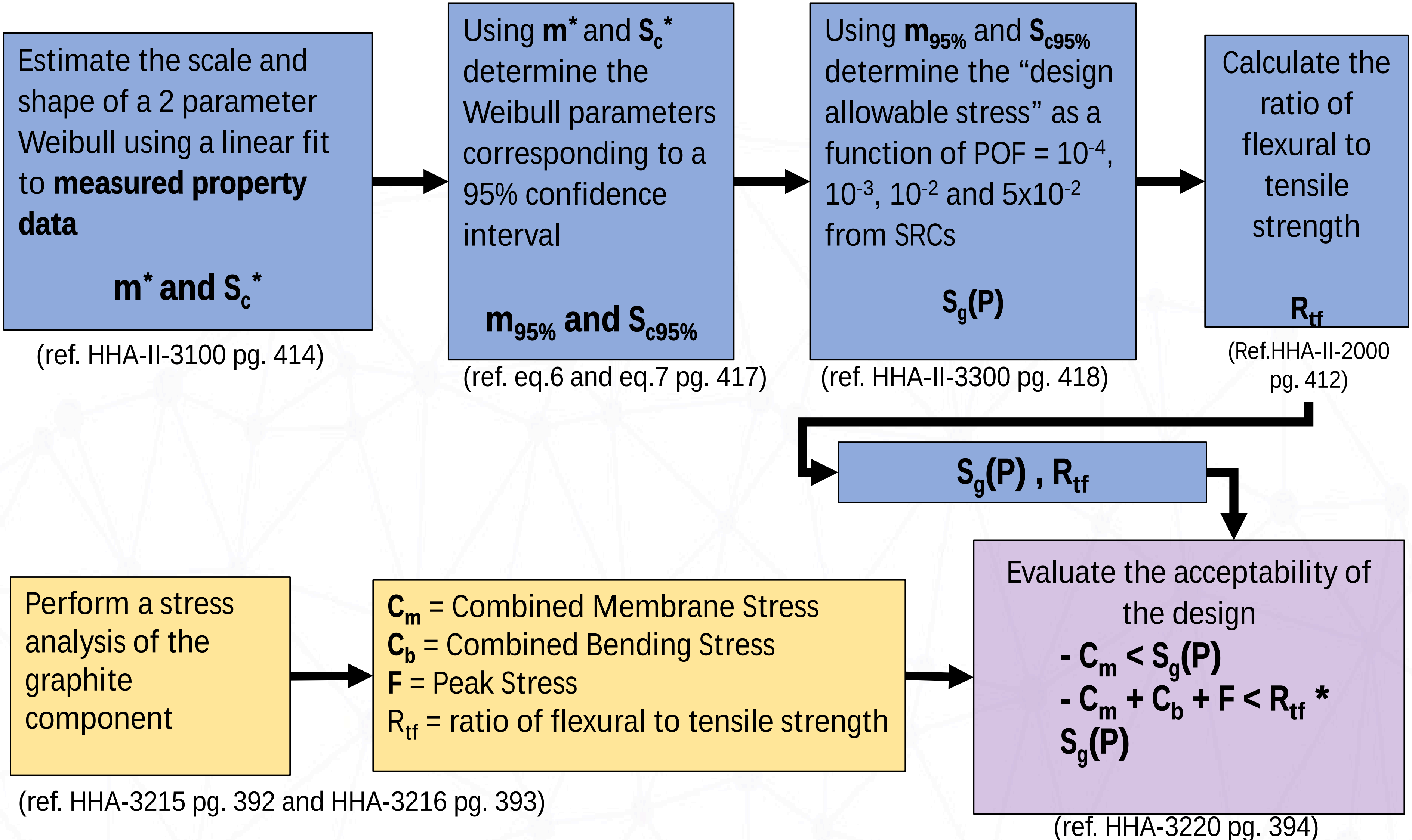
- Fundamental material properties change with irradiation/oxidation
  - Code must assess changes to design of component due to these changes
    - Irradiation: changes to density, strength, dimension, CTE, thermal conductivity
    - Oxidation: changes in density, strength, CTE, and thermal conductivity
  - 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)



Estimate the scale and shape of a 2 parameter Weibull using a linear fit to **measured property data**

m^* and S_c^*

(ref. HHA-II-3100 pg. 414)

Using **m^*** and **S_c^*** determine the Weibull parameters corresponding to a 95% confidence interval

$m_{95\%}$ and $S_{c95\%}$

(ref. eq.6 and eq.7 pg. 417)

Using **$m_{95\%}$** and **$S_{c95\%}$** determine the "design allowable stress" as a function of POF = 10^{-4}, 10^{-3}, 10^{-2} and 5×10^{-2} from SRCs

$S_g(P)$

(ref. HHA-II-3300 pg. 418)

Calculate the ratio of flexural to tensile strength

R_{tf}

(Ref.HHA-II-2000 pg. 412)

$S_g(P)$, R_{tf}

Perform a stress analysis of the graphite component

(ref. HHA-3215 pg. 392 and HHA-3216 pg. 393)

C_m = Combined Membrane Stress
C_b = Combined Bending Stress
F = Peak Stress
R_{tf} = ratio of flexural to tensile strength

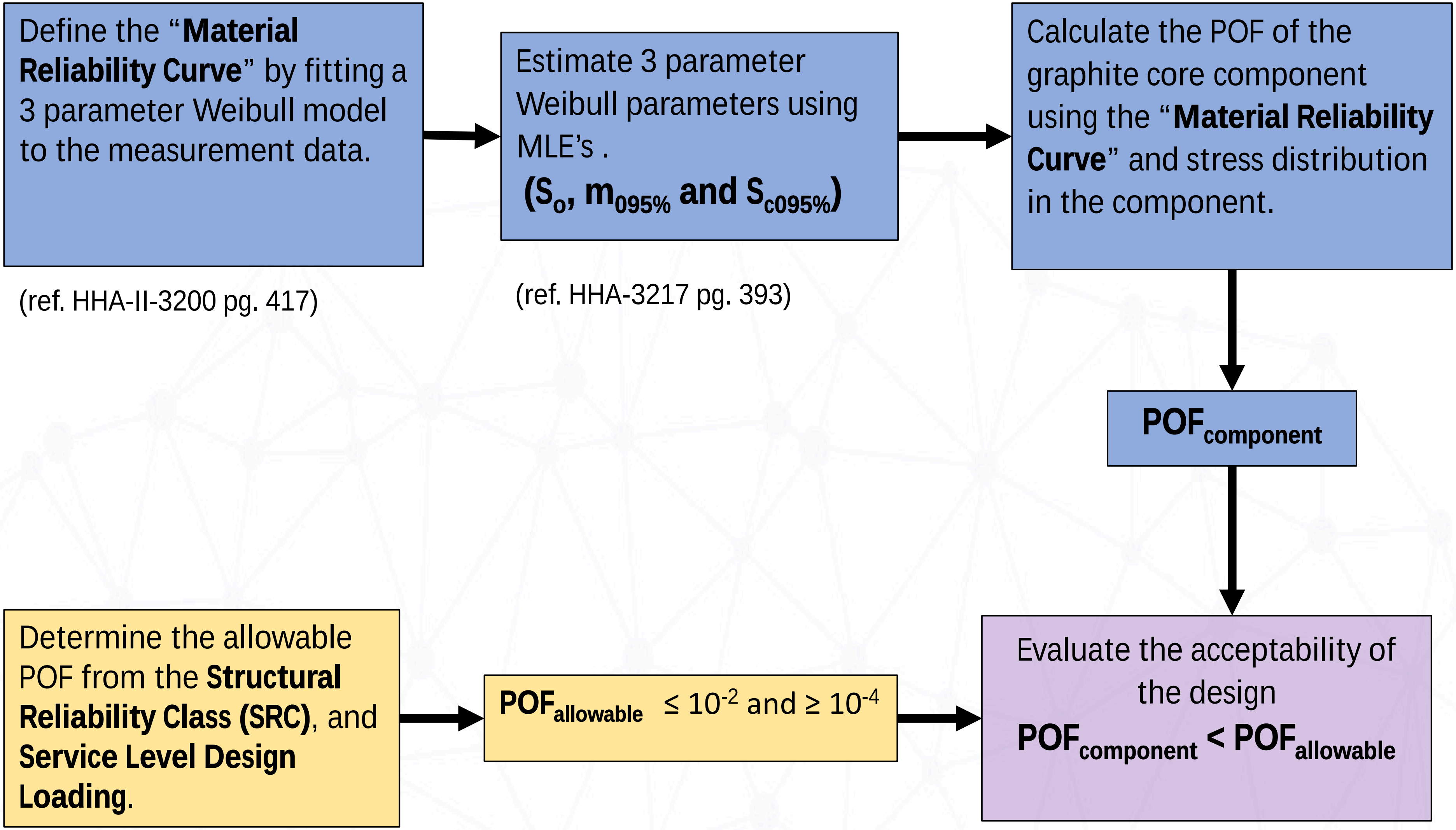
Evaluate the acceptability of the design

- $C_m < S_g(P)$
- $C_m + C_b + F < R_{tf} * S_g(P)$

(ref. HHA-3220 pg. 394)

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, $m_{095\%}$ 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.

$POF_{component}$

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

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

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

Evaluate the acceptability of the design

$POF_{component} < POF_{allowable}$

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