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Materials Science and Technology Division

# ASME: Ceramic Matrix Composites

Sec. III-5 Class SN – Nonmetallic Core Components

**DOE ART Gas-Cooled Reactor (GCR) Review Meeting**

Virtual Meeting

July 25 – 27, 2023





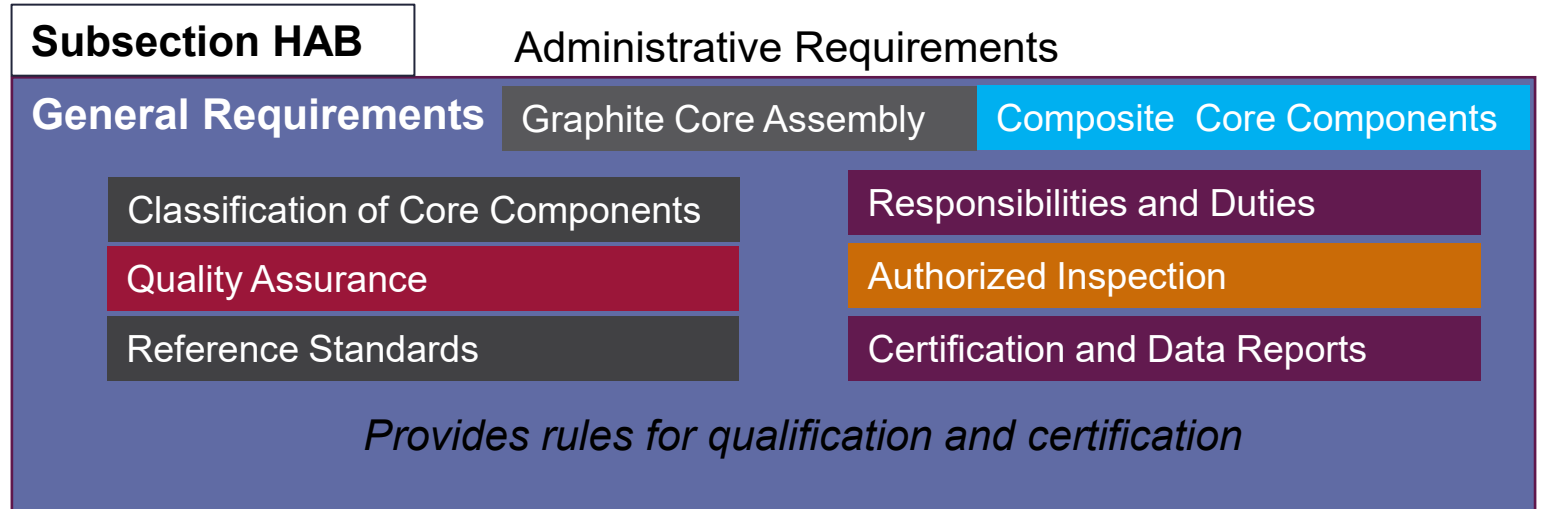
# Sec III-5 HHB (Composites) Development and Status

- **About CMCs in ASME BPV-III-5**
- Background on CMC and HHB rules
- Recent Code Changes - Sec III-5 2023 (HHB)
- Task Group Focus and Current Optimization Areas
- Future Projects
- Synergies

# About Non-metallic Core Components in Sec III

Graphite and composites rules integrated under Class SN Nonmetallic Core Components.

Code Classes allow a **choice of rules that provide a reasonable assurance of structural integrity and quality** commensurate with the relative importance assigned to the individual components of the advanced reactor plant.



## For Metals:

– HAA-1130 LIMITS OF THESE RULES :

“The rules of this Subpart and Subsection HH provide requirements for new construction and **include consideration of mechanical and thermal stresses due to cyclic operation**. They **do not** cover deterioration that may occur in service as a result of environmental effects such as radiation, corrosion, erosion or instability of materials.”

## For Graphite and Composite Materials:

– HAB-1130 LIMITS OF THESE RULES :

“The rules of this Subpart and Subsection HH provide requirements for new construction and **include consideration of mechanical and thermal stresses due to cyclic operation**. They **include consideration of deterioration that may occur in service as a result of environmental considerations**.”



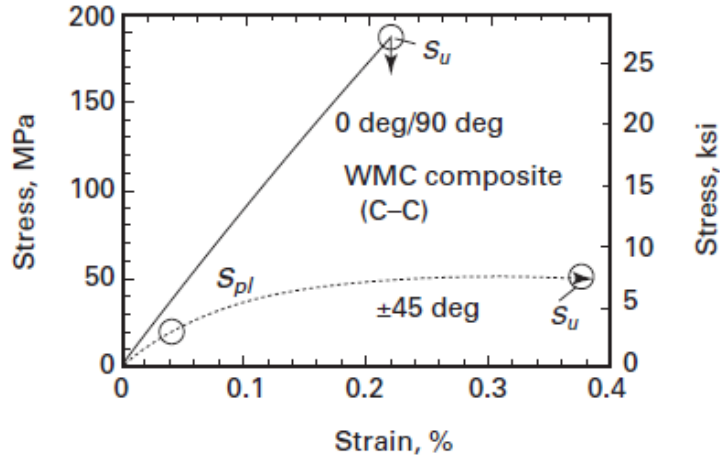
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# ASME - CMCs: Two permissible material systems

## C-C Composites

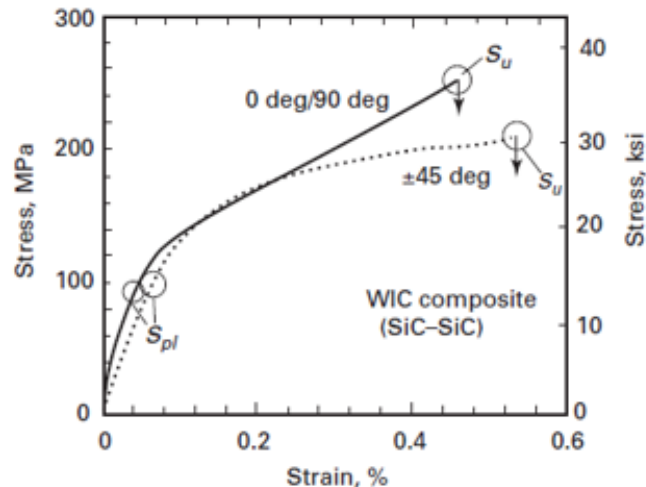
Weak matrix



- Pros**
- Eliminates metals from the core (specific components)
  - Good residual properties (greater strength and fracture resistance than graphite)
  - Good industrial experience
- Cons**
- Less tolerant to radiation damage ( $< \sim 8-10$ dpa,  $E=0.1$  MeV)

## SiC-SiC Composites

Weak interface



- Pros**
- Good oxidation resistance
  - Higher cracking stress than C-C
  - Great radiation damage resistance (up to  $\sim 40$ dpa,  $E=0.1$  MeV)
  - Longer Life: e.g. control rods need no change-out
- Cons**
- Higher cost than C-C
  - Less industrial experience

# Some key issues about composites

## ASME specification for C-C and SiC-SiC composites

- C-C and SiC-SiC **composites are complex** (fibers, matrix, porosity) with a wide range of constituents with **different properties** and many distinctly different densification techniques.
- **Reinforcement architectures can vary widely** with marked anisotropy giving anisotropic physical and mechanical properties.

- **Component properties can vary widely** based on constituents, architecture, and processing.
- **Component requirements can vary widely**, depending on design requirements and composite materials and architectures.

### DESIGN AND SPECIFICATION CONCERNS

Composites are a “new” material system that are tailored for a specific component. Composites have different design rules and different failure mechanisms than metals and monolithic ceramics.



# Some key concepts introduced in the HHB rules

The Design and Materials code for the use of CMCs (SiC-SiC and C-C) core components in HTR (like the graphite code)

The code is structured

- To allow for **multiple applications** and **continual development**
- To allow for future applications and the unique nature of the material, **the code is process based**
  - Guidance for permissibility of the materials, how to specify, how to qualify

- Design provides for two design approaches
  - **Design by Analysis (Simplified POF Assessment)**
  - **Design by Test**
- The **rules are probabilistic** as failure is derived from the variability in the material strength.
- It **includes** the evaluation of **environmental effects** such as irradiation, oxidation / chemical attack and STT (in the case of CMCs).

# HHB Rules: Requirements and Specification of Materials

## Guides to develop composite material specifications:

The purpose of the guides are to provide guidance on how to specify the constituents, the structure, the desired engineering properties, methods of testing, manufacturing process requirements, the quality assurance requirements, and traceability for composites for nuclear reactor applications.

- ASTM C-1783 (C-C)
- ASTM C-1793 (SiC-SiC)

## Material Specification

ASTM standards

## Populate the Material Data Sheet

Material parameters  
Reliability Curves

MDS requirements include characterization of

MDS form is exhaustive.  
Designer to specify the material properties.  
Subjected to HHB-2200.

## Design Specification

- Function & Boundaries
- Design and material requirements,
- Define environmental conditions (incl irradiation & corrosion)
- Design life

Design Report

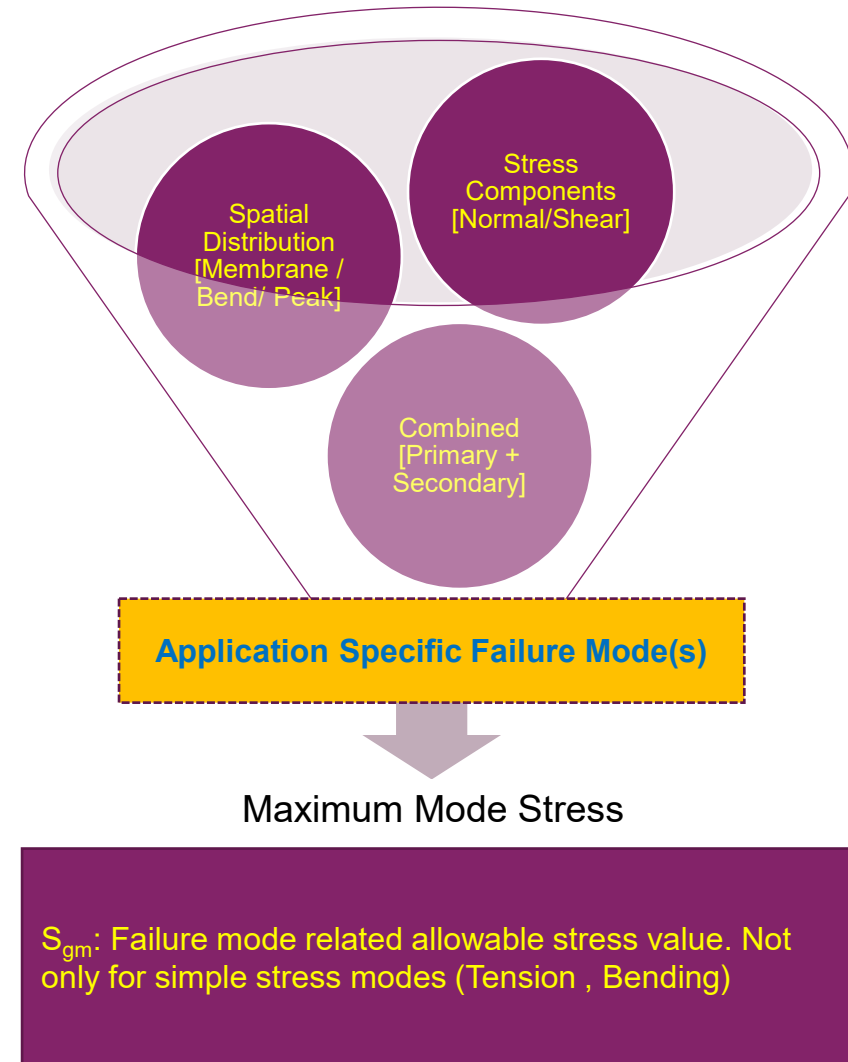
Design Drawings

Construction Specification



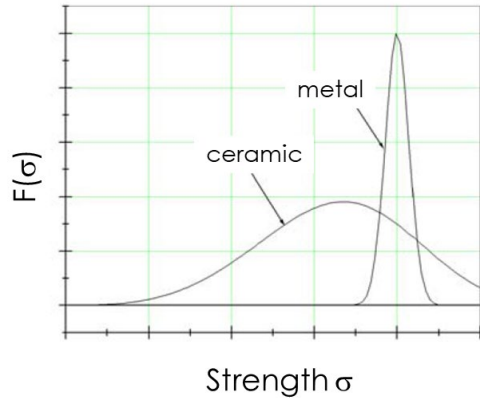
# Design by Analysis Approach

- Identify potential failure modes and loading criteria
  - Static
  - Time-dependant
  - Primary and secondary loads
- Define the component classification and acceptable POF
  - SRC-1: important to safety of the reactor core
  - SRC-3: not important to the safety of the reactor core
- Develop models for stress and strength
  - It is key to complete the stress analysis - the stress at failure need to be determined for the failure mode



# Design by Analysis Approach

- Statistically characterize the material performance



For metals and ductile materials, the scattering of strength values is within a small region around the mean value.

For ceramic materials the strength distribution differs in such a way that very small failure values can occur.

- Determine the design allowable stresses based on component POF

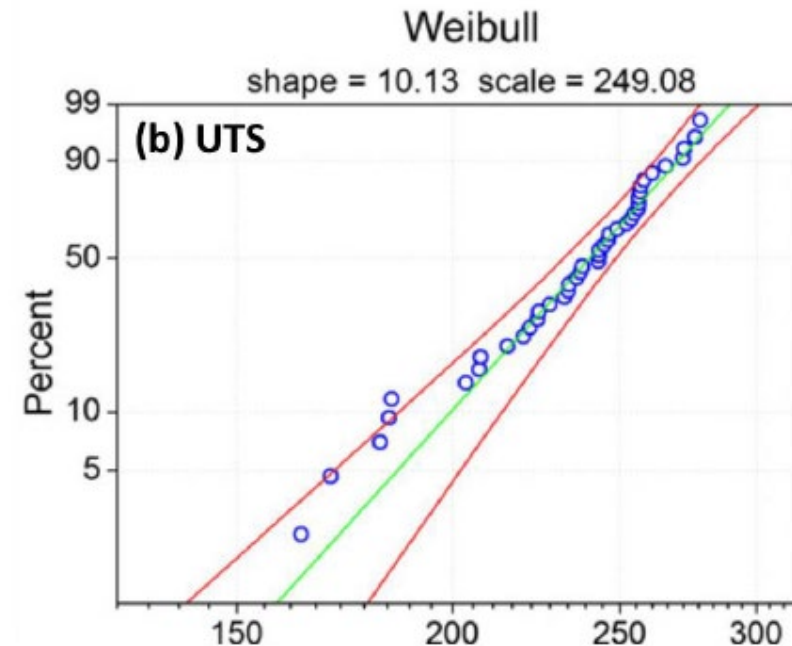
**Table HHB-3221-1**  
Allowable Probability of Failure

SRC Level	Design POF	Service Level Loadings			
		Level A POF	Level B POF	Level C POF	Level D POF
SRC-1	$10^{-4}$	$10^{-4}$	$10^{-4}$	$10^{-4}$	$10^{-3}$
SRC-3	$10^{-2}$	$10^{-2}$	$10^{-2}$	$5 \times 10^{-2}$	$5 \times 10^{-2}$

- Structural Reliability Assessment

The variability in material strength is characterised by the material reliability curve.

- Proposed. Use a Weibull distribution to characterise the material strength (Ho, Schmidt, Nemeth & Bratton)
- Conservatism introduced using 95% confidence limits.

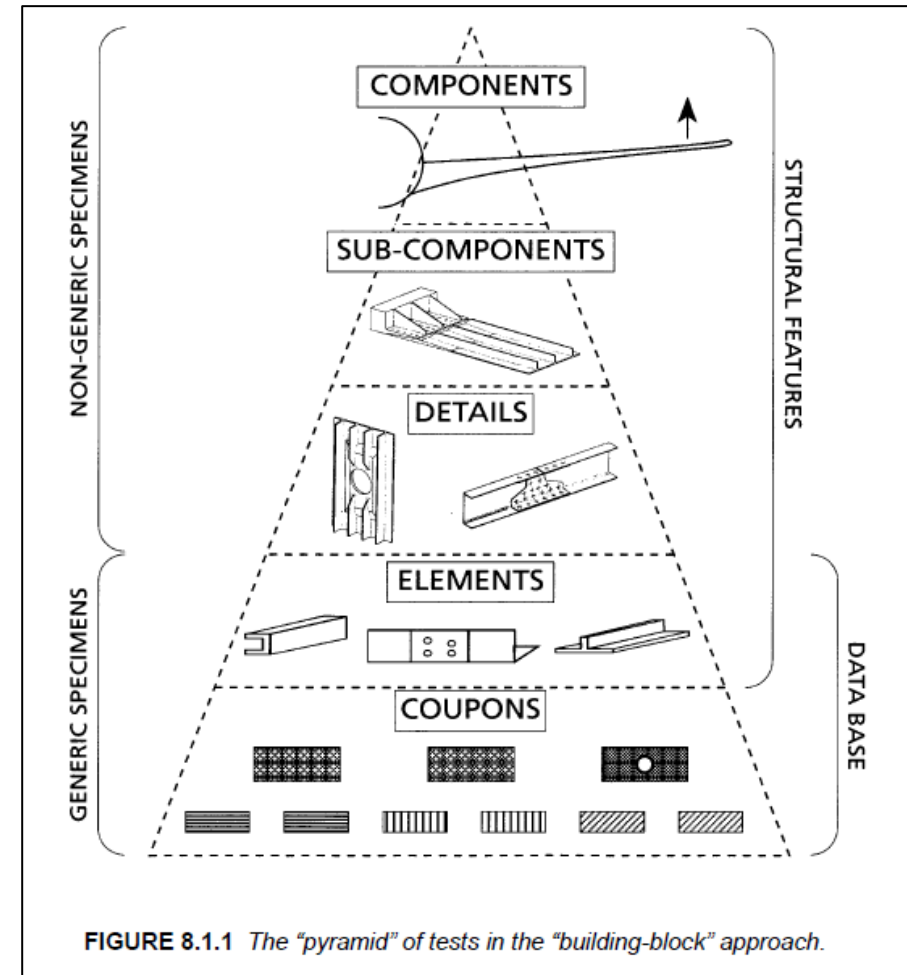


Tensile strength of CVI SiC-SiC composite tube with HNS fiber

Singh et al, Int. J. Appl. Ceram. Technol. (2018)

# HHB Rules: Design by Test

- Design by Test focuses on subcomponents / components
- Similar requirements to the derivation of  $S_{gm}$ 
  - Multiple components
  - Close similarity to actual components
  - May adjust for temperature and other environmental conditions
- **Experimental proof of strength and demonstration of POF**
  - Statistical analysis of the test results shall provide values within 95% certainty by lower bound.
- **Experimental Proof of Strength, Load Rating**
  - Method geared towards production component testing



# Current status code rules for CMCs (HHB): Pros and Cons

## Pros:

- Code rules established within the ASME design framework
- Allows the use of fiber reinforced CMCs for structural core components in HTRs.
- Provides a method to qualify new CMCs, acceptable for use of nuclear application (NQA-1)

## Cons:

- Not fully optimized (e.g. design by test, maximum failure mode, conservatism)
- Lacking technical basis and requires benchmarking
- Not endorsed by NRC (not part of initial review)



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# 2023 Code Change – Failure Mode Stress (Composites)

## Graphite

## Composites

### Technical Requirements

#### Graphite Core Components

HHA

- Graphite Core Assembly
- Maximum deformation energy theory (equivalent stress)
  - Design by Analysis
    - Simple assessment
    - Full assessment
  - Design by test

#### Composite Core Components

HHB

- CMC Components
- Failure Mode Stress Theory (equivalent stress do not apply)
  - Design by Analysis
    - Simple assessment
  - Design by test

Graphite uses the maximum deformation energy theory that combines stresses. This allows for an arbitrary stress state at a point to be converted to an equivalent stress which is then directly compared to the results of a uniaxial strength test. (HHA-3213)

- HHA Simple Assessment: calculate the peak equivalent stress
- HHA Full Assessment: calculate the combined equivalent stress

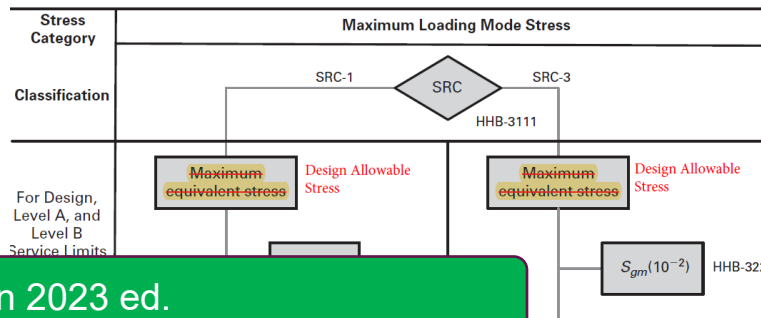
Composites design approach requires comparing the maximum stresses resulting from the loading of the component to the stress at failure of the material. It does not make use of the theory for combining stresses. The stress at failure needs to be determined for the mode at failure exercised by the applied stress. (HHB—3213)

- HHB Simple assessment: calculate the maximum loading mode stress

**The equivalent stress approach should not be used in HHB.**

The ratio of strengths is not applicable to composites

Figure HHB-3221-1  
Allowable Stresses Flowchart for SRC-1 and SRC-3 Composite Core Components



FORM MDS-3 (Cont'd)					
Design Strength and Material Reliability Curve Value					
Ratio of compressive to tensile strength, $R_{tc}$					
Ratio of flexure to tensile strength, $R_{ft}$					
For the selected failure mode: $S_{0.95\%}$ MPa $m_{0.95\%}$					
$S_0$	MPa	$S_{0.05\%}$	MPa	$m_{0.95\%}$	
$S_{gm}(10^{-4})$	MPa	$S_{gm}(10^{-3})$	MPa		

Revision published in 2023 ed.



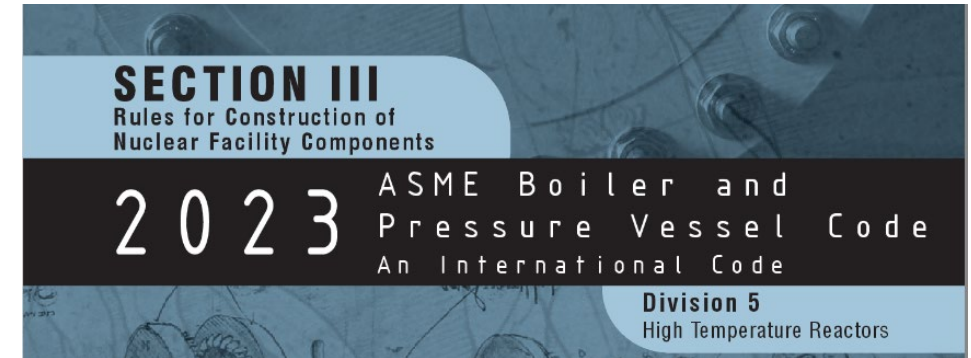
# HHB C-C CMC Non-mandatory Appendices

ASME BPVC.III.5-2023

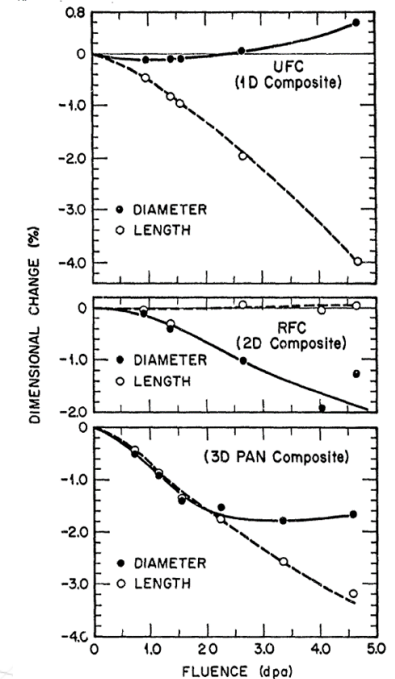
Published in 2023 ed.

## NONMANDATORY APPENDIX HHB-D CARBON-CARBON (C-C) COMPOSITE MATERIALS

## NONMANDATORY APPENDIX HHB-E CARBON-CARBON (C-C) COMPOSITE MATERIALS IRRADIATION AND ENVIRONMENTAL EFFECTS



Article	Paragraphs	Applicability
<b>New NMA Proposed</b>		
<p><b>(New) HHB-D C-C CMC Materials and Applications</b></p> <p>(general information on the composition, architecture, manufacture and properties of C-C composites.)</p>	<p>-1000 Introduction -2000 Manufacture -3000 Properties -4000 Potential Applications of composites in HTRs -5000 References</p>	<p>Address C-C specific aspects and list strength as well as physical material properties missing in HHB-B.</p> <p>It highlights potential applications of C-C composites in HTR, but some could be applicable for SiC-SiC.</p>
<p><b>(New) HHB-E C-C CMC Irradiation and Environmental Effects</b></p> <p>(Information on the effects of fast neutron irradiation on C-C composites)</p>	<p>-1000 Introduction -2000 Irradiation Induced Damage -3000 Irradiation Induced Dimensional Changes -4000 Irradiation Induced Changes in Physical Properties -5000 Irradiation Induced Changes in Mechanical Properties -6000 Effects of Chemical Attack/Oxidation on C/C Composites -7000 References</p>	<p>Specifically discuss the irradiation induced damage effects caused to C-C and highlights the typical irradiation induced changes to physical and mechanical properties.</p> <p>Specifically discuss corrosion / oxidation response on C-C composites.</p>



T.D. Burchell, *Physica Scripta* 64, 17-25, 1996.



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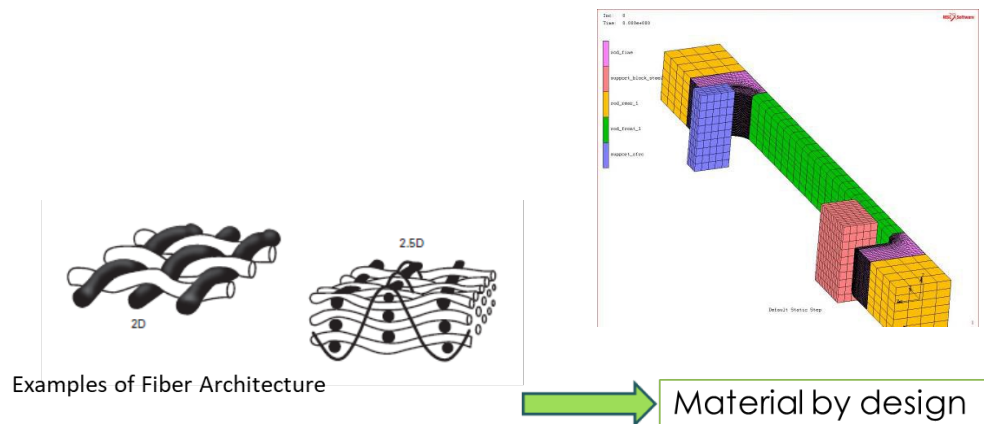
# WG-NDM: Task Group on Composites

The composites task group is a subset of the nonmetallic design and materials working group (NDM-WG) with specific focus to address mission relevant activities as it relates to Sec III.5 HAB and HHB on ceramic matrix composites.

Objective:

- Bring vendor community together on code related questions
- Identify type/current composite applications and code related issues that have been uncovered
- Identify areas that require review and/or further optimization
- Discuss strategies to demonstrate or benchmark the code methodology.

- Qualification Methodology:
  - Is it possible to optimize and/or accelerate the qualification process by reducing the material qualification effort?
  - How can technologies & analytical methods be used to reduce testing efforts?
  - What is truly mandatory or non-mandatory in the code?
- Composite design rule assessment
  - Does the “simplified assessment” design approach clearly explain how to address anisotropic differences in mechanical properties?
  - Is there sufficient detail for the design by test methodology?
  - When should which method be applied?
- Industry: code readiness
  - Identify optimization areas.





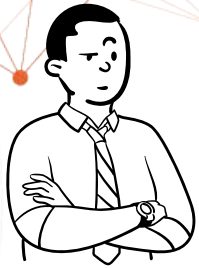
## Identified focus areas to optimize for 2025

- Design by Analysis – Structural Assessment Procedure
- Design by Analysis vs Design by Test
- Material Fabrication and Testing

Aim to revise for 2025 edition.



# Design by Analysis – Structural Assessment Procedure



**HHB-3214.2 Maximum Loading Mode Stress.** The maximum loading mode stress in a Composite Core Component is the highest loading mode stress computed from the total stress in the Composite Core Component in accordance with the provisions of HHB-3215.

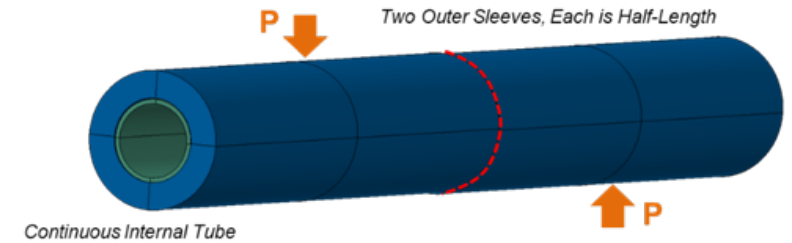
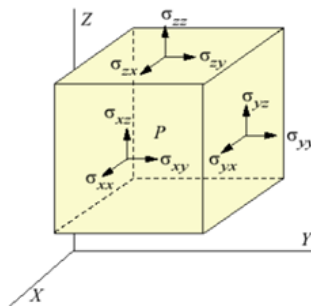
- *How is Maximum Loading Mode (MLM) stress determined and is it a singular global value?*
- *Is there an MLM stress for each direction and loading mode?*

**HHB-3213** Ba

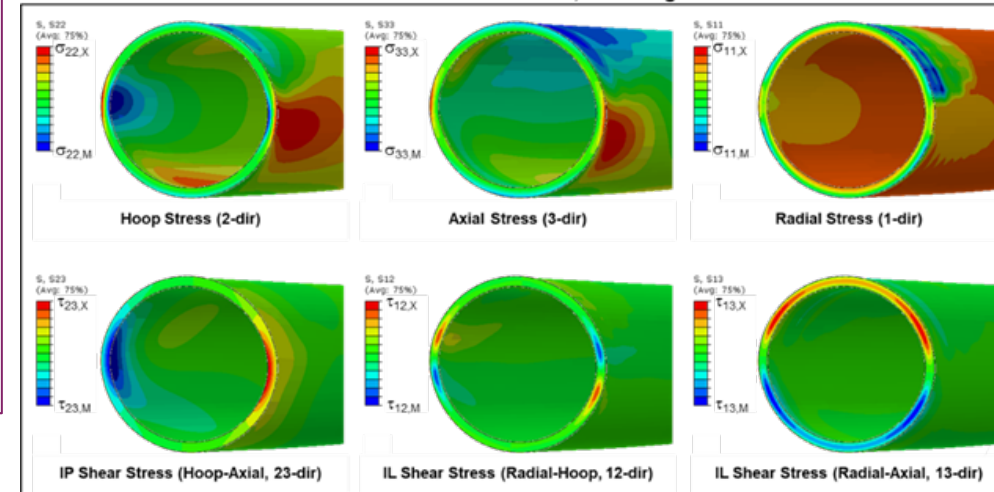
The design rule ory for combining comparing the m ing of the compo rial. It is key that the mode of failu

For example, if the stresses primarily result in shear stresses in the matrix, then the stress at failure for matrix shear stress shall be used for acceptance of the design.

Maximum Loading Mode Stress	$\sigma_{\max}$ tensile (xx,yy,zz), $\sigma_{\max}$ compression (xx,yy,zz), $\sigma_{\max}$ shear (x-y, x-z, y-z, y-z, z-x, z-y)
Design Allowable Stress (Reliability Factor)	$S_{\sigma}$ tensile (xx,yy,zz), $S_{\sigma}$ compression (xx,yy,zz), $S_{\sigma}$ shear (x-y, x-z, y-z, y-z, z-x, z-y)



Stress Contours for the Internal Tube, Mid-Length Section Cut



Courtesy MR&D Inc.

One should compare peak values for *all directional stresses*:

*Peak stress being the component stresses with corresponding POF-adjusted allowable stresses.*

*Not just one failure mode.*

# Design-by-Analysis and/or Design-by-Test

- Design by Analysis (HHB-3220) is unavoidable for larger components
  - Testing infrastructure to simultaneously apply all the necessary loads to properly qualify a component, does not exist
  - To create such test facility or setup often complex, expensive and introduce additional risk.
- Unfortunately, it requires extensive material characterization due to the added complexity of composite materials
  - Orthotropic vs. isotropic, more strengths to measure...
- A combined Analysis/Testing approach is desired to reduce the amount of material testing through supplemental subcomponent and component level testing.

**HHB-3100 GENERAL DESIGN**

Rules for the design of Composite Core Components (Core Components manufactured from C-C composites and SiC-SiC composites) and their integration into Core Assemblies are described in this Article.

Design of Composite Core Components is addressed in HHB-3200. Provisions are made for the following two approaches to design:

(a) design of Composite Core Components to meet the reliability targets based on stress limits derived from the material reliability curve (HHB-3220). This is referred to as a simplified assessment.

(b) design of Composite Core Components to meet the reliability targets based on experimental proof testing of Composite Core Component performance with margins derived from the material reliability curve (HHB-3240). This is referred to as design by test.

**HHB-3240 EXPERIMENTAL LIMITS — DESIGN BY TEST**

It is permissible to declare a design in compliance with the requirements of this Article based on component testing or design by test. Design by test either demonstrates that the POF of the Composite Core Component subjected to an enveloping load meets the requirements of this Article or establishes a Load Rating for the component consistent with the limits provided in this Article. The POF limits are summarized in Table HHB-3221-1.

Note that not all parts and loadings are suitable to design by test, as complex loadings and environmental effects may not be adequately reproducible in a test. In such a case, the method in HHB-3220 shall apply for the design of such a part (in the applicable loading mode).

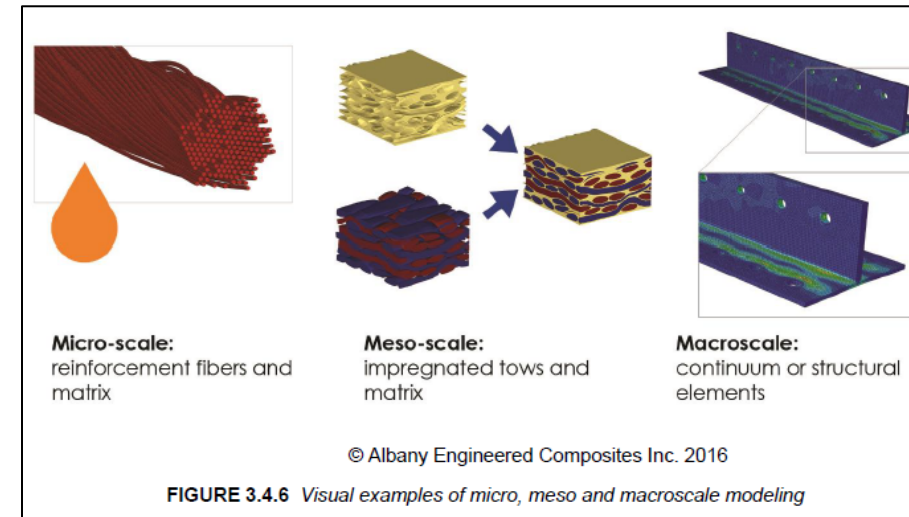
ASME BPV III-5 (2021)

Case study: Ceramic Stationary Gas Turbine

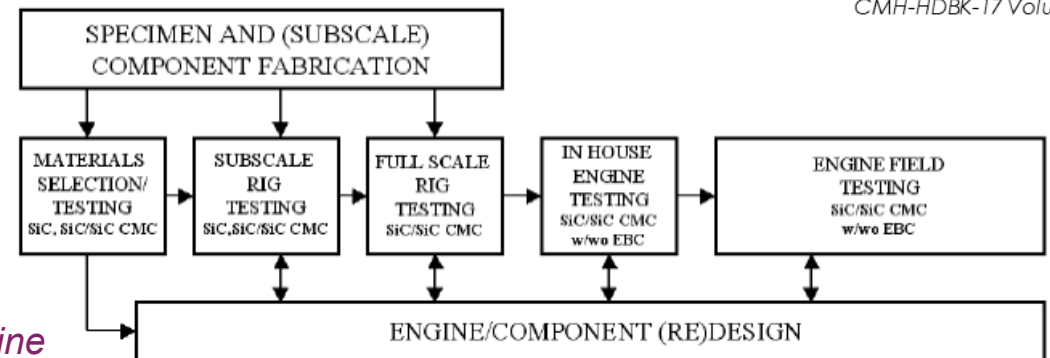
*Optimization area:*

*Combined Analysis/Testing approach is not explicitly outlined in the code.*

*Requires emphasis!*



CMH-HDBK-17 Volume 5





# Material Fabrication and Testing

- MDS data is utilized to determine POF and allowable stress considered in analysis
- **ASME requires generation of the MDS forms per lot**; co-processed composite material of the same composition and architecture
  - MDS form associated with production component
  - Generation of design strength, modulus and reliability curves specific to production lot
  - Includes environmental effects such as temperature, oxidation, irradiation etc.
    - Testing at maximum temp increments of 200°C
- This approach differs from that described in CMH-17; where qualification testing is performed to accumulate testing data (generation of a data base) utilized to generate design allowables, followed by acceptance testing of production components

*Example paragraphs:*

## **HHB-III-4100 AS-MANUFACTURED CERAMIC COMPOSITE MATERIAL**

As-manufactured material property data shall be obtained from tests of composite components or witness coupons from **each production lot of material** meeting all of the requirements of the Designer (Mandatory Appendix HHB-I). (Witness coupons are composite pieces of the same composition and architecture, co-processed in the same lot/batch and with the same nominal thick-

*Optimization area:*

*The objective is to qualify the production process, then to obtain the material properties for the data base generation*

*Requires change!*

## **MATERIAL**

Irradiation, chemical attack/oxidation, and STT material property data shall be obtained from material that is **representative of the material** used for the generation of the irradiation, chemical attack/oxidation, and STT material design data.

The Designer is responsible for the determination and justification of the representative data.



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# Benchmark Study

## Benchmark study

- Generate Micromechanics Models for CMC material properties in as-fabricated and irradiated condition from test data.
- Demonstrate allowable stress using the POF methodology
- Determine failure mode and perform structural assessment applying code rules

Provide technical support basis for ASME

Question on code conservatism / knockdown factors:

ASME:

SRC-1 – Design POF =  $10^{-4}$

SRC-3 – Design POF =  $10^{-2}$

A-basis allowables (CMH-17)

Sample collection – Design POF =  $1.0 \times 10^{-2}$

Aircraft structures (U.S. DOT)

Composites Design POF =  $1.0 \times 10^{-7}$  or  $1.0 \times 10^{-9}$

U.S. DOT guidance: “The agency certifying the structure should be responsible for setting this overall specification for the structure.”

Material: Carbon fiber-reinforced carbon (C/C)

Application: High-temperature applications

### Material data of SIGRABOND® Premium

Typical properties	Units	Premium
Density	g/cm <sup>3</sup>	1.6
Flexural strength	MPa	230
Flexural modulus	GPa	75
Tensile strength	MPa	400
Interlaminar shear strength	MPa	11
Ash content	ppm	1000
Ash content (purified grade)	ppm	< 10
Max. application temperature	°C [°F]	2000 [3600] in vacuum or inert gas

Values without tolerance represent typical average values. For any engineering/design purposes please always contact our technical sales team.

<https://www.sgicarbon.com/en/markets-solutions/material/sigrabond-carbon-fiber-reinforced-carbon/>

Example:

Interlaminar shear on C-C presents a challenge for designers.

For SRC-1: If  $m^* = 20$  then  $S_g(10^{-4}) = 6.6$  MPa

If  $m^* = 10$  then  $S_g(10^{-4}) = 3.85$  MPa

$m^*$  = Weibull modulus (95% lower bound confidence interval)

# Other Identified Challenges

- Are composite components always Material by Design?
  - With sufficient standardization in manufacturing specifications, testing specifications, and design / analysis / qualification approaches, composites could move to material in design

➡ A long-term process?

- Challenges
  - Reconciling the depth of the material specification with the proprietary nature of composite fabrication techniques
  - Current status of composite material testing standards
  - Availability of elevated temperature testing facilities
  - Timeline, cost, and dimensional limitations associated with obtaining irradiated material properties



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# CMC Synergistic Activities & Industry Alignment



## SUBSECTION HH CLASS SN NONMETALLIC CORE COMPONENTS – SUBPART B COMPOSITE MATERIALS

FORM MDS-3 MATERIAL DATA SHEET — CERAMIC COMPOSITE MATERIAL (SI UNITS)

**Identification, Classification, and Description**

Design/material specification ID \_\_\_\_\_ Purchase document \_\_\_\_\_

Producer/source name or ID \_\_\_\_\_

Composite classification (if specified per HHB-A) \_\_\_\_\_

Component ID \_\_\_\_\_ Production lot number \_\_\_\_\_

Date of manufacture \_\_\_\_\_

**Constituents and Fabrication Description**

Description and pedigree of fiber (carbon/graphite/silicon carbide) \_\_\_\_\_

Description and pedigree of fiber architecture \_\_\_\_\_

Description and pedigree of fiber coating/interface, if used \_\_\_\_\_

Description and pedigree of matrix \_\_\_\_\_

Description and pedigree of composite fabrication \_\_\_\_\_

Description and pedigree of component seal coating, if used \_\_\_\_\_

Meets chemical purity specification? Yes \_\_\_\_\_ No \_\_\_\_\_ High purity \_\_\_\_\_ Low purity \_\_\_\_\_

Constituent bulk volume fractions: Fiber \_\_\_\_\_ vol. % Matrix \_\_\_\_\_ vol. %

Bulk density by physical measurement \_\_\_\_\_ g/cm<sup>3</sup>

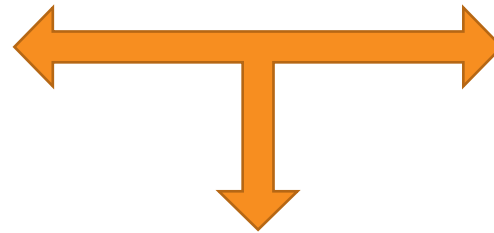
Bulk density by immersion \_\_\_\_\_ g/cm<sup>3</sup>

Apparent porosity by immersion \_\_\_\_\_ %

Orientation/Directional Factors of the Fiber/Fabric Architecture

Direction	Description	Fiber, vol. %
1		
2		
3		

(07/19)



Units	ASTM Test Methods	ASTM Test Methods
Tensile Properties (ultimate, fracture, PropL)	MPa & strain	C1275, C1359
Flexure Properties (ultimate, fracture, PropL)	MPa & strain	C1341
Compression Properties (ultimate, fracture, PropL)	MPa & strain	C1358
Shear Properties (ultimate, fracture, PropL)	MPa & strain	C1292, C1425
Trans thickness Tensile Properties (ultimate, fracture, PropL)	MPa & strain	C1468
Hoop Strength Properties (ultimate, fracture, PropL)	MPa & strain	NA
Elastic/Shear Modulus by Mechanical Loading	GPa	E111
Elastic/Shear Modulus by Sonic Resonance	GPa	C1198
Elastic/Shear Modulus by Impulse Excitation	GPa	C1259
Elastic Modulus by Sonic Velocity	GPa	C769
Poisson's Ratio	nd	E132
Modulus of Resilience (in Tension)	J/m <sup>3</sup>	C1275, C1359
Modulus of Toughness (in Tension)	J/m <sup>3</sup>	C1275, C1359
Open Hole Tensile Strength Properties	MPa & strain	D5766 <sup>A</sup>
Open Hole Compression Strength Properties	MPa & strain	D6484 <sup>A</sup>
Notch Tensile Strength Properties	MPa & strain	B
Notch Compression Strength Properties	MPa & strain	B
Pin Bearing Strength Properties	MPa & strain	D5961 <sup>A</sup>
Fracture Toughness / Strain Energy Release Rate	kJ/m <sup>2</sup>	D5528 <sup>A</sup> , D6671 <sup>A</sup> , E1922 <sup>A</sup>

<sup>A</sup> Modification of this polymer matrix composite test method may be required.  
<sup>B</sup> New test methods are required.  
nd = no dimensions.

## CMH-17 Ceramic Matrix Composites Volume 5 Handbook

### Handbook Sections – Part C. Testing:

- Chapter 8 – Thermo-Mechanical-Physical Test Methods – Overview
- Chapter 9 – Material Testing & Characterization for Submission of Data to CMH-17
- **Chapter 10 – Evaluation of Reinforcements**
- **Chapter 11 – Evaluation of Matrix Materials**
- Chapter 12 – Evaluation of Interface Material
- **Chapter 13 – Evaluation of Composites**

### Section 13 – Evaluation of Composites

- 13.2 – Fiber Volume Fraction (ASTM D3171 & Optical Methods)
- 13.3 – Coefficient of Thermal Expansion (CTE) (ASTM E228)
- 13.4 – Thermal Conductivity (Multiple ASTMs)
- 13.5 – Specific Heat (Multiple ASTMs)
- 13.7 – Compression (ASTM C1358)
- 13.8 – Flexure (ASTM C1341)
- 13.10 – Interlaminar Tension (ASTM C1468/D6415)
- 13.11 – Notched Testing (Multiple ASTMs)
- 13.12 – Interlaminar Fracture Toughness
- 13.14 – Creep Testing (ASTM C1337)
- 13.15 – Fatigue Testing (ASTM C1360)
- 13.17 – Wear Testing
- 13.18 – Bearing Testing
- 13.19 – Biaxial Testing

CMH-17 works towards partnership with C28.07 - Joint meetings



# Conclusion

- ASME Sec III-5 HHB = Class SN Nonmetallic Core Components
  - Rules includes environmental in-service conditions
  - HHB has not been reviewed by NRC. (Not endorsed)
- It is structured to allow for multiple applications and continual development as it is process based.
  - The code needs to be flexible (unique nature of the CMCs)
  - It currently provides two design approaches.
- There are new code additions and revisions in the ASME BPV 2023 edition.
- From strong community interaction, optimization areas have been identified.
  - Target - 2025 edition
- Technical basis (for ASME) is lacking and could benefit from a benchmark study to validate the approach
- Aiming to improve industry alignment (ASTM & CMH-17 community)



Questions?

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