Challenges with ASME "Code Qualifying" Graphite Irradiation Effects in Test Reactors

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What does the ASME B&PV Code Say (1/3)?

- Irradiated materials properties (HHA-2220 and HHA-III-3300):
  - **Dimensional change**
  - **Strength**
  - **Elastic modulus**
  - **Creep coefficient**
  - **Coefficient of thermal expansion (temperature dependent)**
  - **Thermal conductivity (temperature dependent)**
- Damage dose and irradiation temperature shall cover qualification envelope
  - **Maximum temperature increments of 200°C** shall be used (HHA-III-3300)
- Temperature-dependent properties will be measured with a **maximum temperature increments of 200°C** shall be used (HHA-III-3300)
  - Measurements at temperatures below the envelope temperature are permissible due to the necessity to retain rather than anneal radiation damage (HHA-III-3300)

ASME III-5, 2017, "ASME Boiler and Pressure Vessel Code An International Code", ASME International, New York, NY.

What does the ASME B&PV Code Say (2/3)?

- "Irradiated material properties data shall be obtained from **material that is representative of the material used for the generation of the irradiated material design data**" (HHA-III-4200)
  - "Designer is responsible for determination and justification of the representative data" (HHA-III-4200)
- "The historical data shall meet the requirements of this Appendix and be on the same graphite grade. **If credit is taken from previous qualification programs, then testing shall also demonstrate that the historic data is applicable to the current production material.**" (HHA-III-5000)

ASME III-5, 2017, "ASME Boiler and Pressure Vessel Code An International Code", ASME International, New York, NY.

What does the ASME B&PV Code Say (3/3)?

- Irradiation Fluence Limits (HHA-3142.1)
  - "(a) < 0.001 dpa the effects of neutron irradiation are negligible and may be ignored"
  - "(b) > 0.001 dpa the effect of neutron irradiation on thermal conductivity shall be taken into account."
  - "(c) > 0.25 dpa all effects of neutron irradiation (described in HHA-2200) shall be considered and a viscoelastic analysis applied."
- "HHA-3142.2 Stored (Wigner) Energy. **Should graphite be irradiated to significant fluence [> 0.25 dpa] at a temperature < 200°C, the effect of stored (Wigner) energy buildup shall be accounted for** when evaluating reactor thermal transients."

ASME III-5, 2017, "ASME Boiler and Pressure Vessel Code An International Code", ASME International, New York, NY.

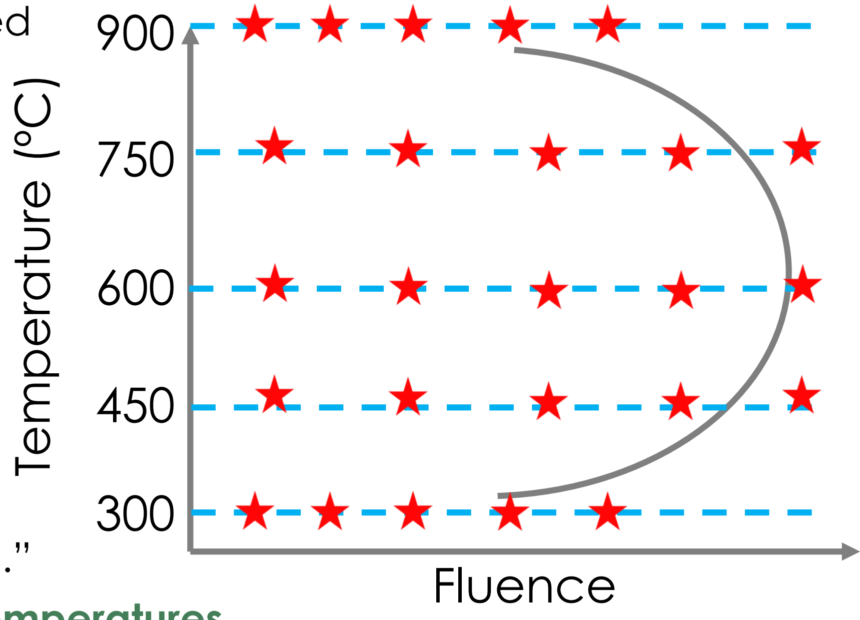
Graphite Cohesive Life Limit (HHA-3142.4)

- "A temperature-dependent cohesive life limit fluence is to be defined for the graphite grade used for Graphite Core Components. Material that exceeds this fluence limit is considered to provide no contribution to the structural performance (stiffness and strength) of the Graphite Core Component. **This fluence limit shall be set to the fluence at which the material experiences a +10% linear dimensional change in the with-grain direction.** For full assessment (HHA-3230), this material shall not be included in the volume of the Graphite Core Component assessed."

ASME III-5, 2017, "ASME Boiler and Pressure Vessel Code An International Code", ASME International, New York, NY.

ARTICLE HHA-II-4000 DETAILED REQUIREMENTS FOR DERIVATION OF THE MATERIAL DATA SHEET — IRRADIATED MATERIAL PROPERTIES

- "(a) The changes in these properties with neutron irradiation shall be determined at sufficient intervals over the design fluence and temperature range. **Temperature and fluence intervals shall be selected so as to provide adequate confidence in the accuracy of the interpolations**. Limited extrapolations are permitted, but such extrapolations shall be justified."
- "(b) Property changes (such as strength, thermal conductivity, the linear coefficient of thermal expansion, and the elastic modulus) are to be **reported in the form of the relative change with regard to value before irradiation as a function of the fluence over the relevant operating temperature range**. When reporting fractional change of these properties, for **near-isotropic graphite grades, distinction with respect to the preferred grain orientation is not required**."



- "(c) The creep coefficient (creep model parameters) **is to be given for those temperatures that occur in the irradiated components**, in accordance with HHA-3142.1(c)."
- "(d) The effect of **creep strain on coefficient of thermal expansion and elastic modulus shall be determined** and included in the Material Data Sheet."
- "(e) The irradiation-induced relative linear dimensional changes are to be given for the design service temperature range for the irradiated components. The relative linear dimensional changes are to be given separately for both grain orientations. For isotropic graphite, where the difference between dimensional change curves is insignificant, only one curve is necessary."

ASME III-5, 2017, "ASME Boiler and Pressure Vessel Code An International Code", ASME International, New York, NY.

MANDATORY APPENDIX HHA-III REQUIREMENTS FOR GENERATION OF DESIGN DATA FOR GRAPHITE GRADES

- "It is the policy of the ASME Boiler and Pressure Vessel Committee to adopt for inclusion **only such specifications adopted by the American Society for Testing and Materials (ASTM) and by other recognized national or international organizations**. Mandatory Appendix HHA-I contains two ASTM material specifications for nuclear grade graphites." (HHA-III-1000)
- "Material Specifications The following material specifications are accepted:" (HHA-I-1110)
  - "(a) ASTM D7219-08: Standard Specification for Isotropic and Near-isotropic Nuclear Graphites"
  - "(b) ASTM D7301-08: Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose"

ASME III-5, 2017, "ASME Boiler and Pressure Vessel Code An International Code", ASME International, New York, NY.

HHA-III-3000 Properties to be Determined

- "The Designer shall record the product cutting and sampling plan from which the specimens and various properties were taken, as well as all supporting test records. The **heterogeneity of the graphite and statistical nature of the data shall be considered**. Where property means and standard deviations are reported, the size of the sample population shall be reported as well."
  - "(a) The test standards specified in ASTM C781 shall be used."
  - "(b) Where a test standard does not exist or has to be customized for application, the test procedure used for the completion of the tests shall be filed with the material test data."

ASME III-5, 2017, "ASME Boiler and Pressure Vessel Code An International Code", ASME International, New York, NY.

ASTM Standards

- Bulk Density (ASTM C559-16) - mass of specimen >2000x balance sensitivity, Volume>500 mm^3
  - **Minimum dimension > 10 largest particle, and >2000x measurement device resolution**
- Flexural Strength
  - 4-point flexural strength (ASTM C651-15), **minimum dimension (t) > 5x particle size, length > 8t, width (w) t < w < 2t**
    - **Support span minimum length 40 mm** (and 3x longer than load span), **load span > 2t**
    - Specimen overhang support span by at least 1t at each end (**specimen length > 40 +2t**)
  - 3-point flexural strength (ASTM D7971-14) because of the small volume under stress so "four-point flexure is preferred and recommended for most characterization purposes"
    - **Minimum dimension (t) > 5x grain size, L/t > 6, and 1 < W/t < 2**
- Compressive strength (ASTM C695-15), **diameter (d) > 10x particle size**, length (l) **1.9d < l < 2.1d** (standard calls out recommended minimum of **9.5 mm x 19 mm**)
- Tensile strength (ASTM C749-15), **gauge diameter no smaller than 3-5x maximum particle size**, specifies specimen shapes and dimensions, at least **50% of specimens should fracture in gauge region**
  - Older version of ASTM C781-08 had annex about using glued-end rods (removed in the newest version of standard, still in ASTM D7775-16), diameter 6.5 mm, length 26 mm
- Elastic properties (ASTM C747-93, ASTM C769-15, C1198-09 ceramics)
  - Impulse/Resonance frequency – **"Obtain representative specimens", slender rod geometry (5 < L/T < 20)**
  - Sonic Velocity (ASTM C769-15) "Approximate" value, specimen size dependent on grain size and wavelength
    - **Wavelength longer than grain size (2.25 MHz frequency has λ=1.1 mm), diameter >5λ, length sufficiently long to include "more than a few grains"**
- Thermal conductivity (ASTM E1461-13), **thin disks (6-30 mm diameter), thickness to get thermal half-rise time 10-1000 ms** (in graphite 2.8-11 mm pre-irradiation and 0.5-5 mm post-irradiation)
- Coefficient of thermal expansion (ASTM E228-17), **length 25-60 mm, 5-10 mm diameter** (or equivalent)
  - **Cross section length > 5x grain size (no smaller than 4 mm), length > 25 mm** (50-150 preferred) (ASTM C781-18)
- Irradiation creep – **no ASTM standard**, safe to assume specimen dimensions would have similar requirements as compression or tensile/flexural specimens

Minimum Specimen Dimensions per ASTM Standards

Grade	Grain Size (mm)	Density	σ_{4F}	σ_{3F}	σ_C	σ_T	ThD	CTE	E_{IR}	E_{SV}
NBG-18	1.6	16^3 (4096 mm^3)	8x8x64	8x8x48	16x32	4.8	6x6x3	8x25	8x40	12x16[a]
PCEA	0.8	8^3 (512 mm^3)	4x4x32	4x4x24	8x16	0.8	6x6x3	4x25	4x20	5.5x15
IG-110	0.05	2^3 (8 mm^3)	0.25x0.25x2	0.25x0.25x2	0.5x1	0.15	6x6x3	0.25x25	0.25x1.25	5.5x15

[a] May require lower frequency since 2.25 MHz λ=1.1 mm (1.0 MHz λ=2.5 mm)

- Specimen sizes conforming to ASTM standards are typically **larger than those that can fit in capsules for irradiation testing**
- At ORNL, irradiations in the HFIR flux trap (~6-10 dpa per year) are usually limited to capsule irradiations, with internal space for specimens limited to **6 x 6 x 48 mm**
- Large instrumented test facilities possible at ORNL, INL, and Petten
  - Longer irradiation time, higher costs (instrumented), still have size limitations

How to use specimens that fit in irradiation capsules?

- ASTM D7775-16 covers the measurement of properties on specimens that are smaller than standard recommendations
  - “**responsibility of the user to demonstrate that the application of a standard outside any specified constraints is valid and reasonably provides properties of the bulk material from which the nonstandard specimen was extracted**”
- ORNL advises Size Effect Studies to quantify properties measured on specimens that conform at ASTM sizes and sub-sized specimens

ASTM D7775-16, 2016, "Standard Guide for Measurements on Small Graphite Specimens", ASTM International, West Conshohocken, PA, DOI: 10.1520/D7775-16, www.astm.org.

Major Challenges for Qualification Programs

- Need multiple temperature/fluence combination (10-30)
- Need replicate measurements of properties on multiple specimens (is triplicate sufficient?)
- Representative specimen sizes limits number of specimens that can be irradiated in a single capsule/zone
  - Multiple capsules/zones per temperature/fluence combination
- This results in 10's-100's of irradiation capsules in the ORNL HFIR to complete a full irradiation campaign
  - Can't complete irradiation of all specimens at one time

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